UNIVERSITY OF CALIFORNIA,
IRVINE

Development of Nickel-Catalyzed Coupling Reactions: Intramolecular Alkyl-Heck and Reductive Cross-Electrophile Cyclizations and Hydroarylation of Alkynes

DISSERTATION

submitted in partial satisfaction of the requirements
for the degree of

DOCTOR OF PHILOSOPHY

in Chemistry

by

Mikhail Olegovich Konev

Dissertation Committee:
Professor Elizabeth R. Jarvo, Chair
Professor Christopher D. Vanderwal
Professor Jennifer A. Prescher

2017

DEDICATION

To

my family and friends.

"Poets say science takes away from the beauty of the stars – mere globs of gas atoms. Nothing is 'mere.' I too can see the stars on a desert night, and feel them. But do I see less or more?

-Richard Feynman

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8. "Alkylaluminum-Mediated Stereospecific Nickel-Catalyzed Coupling Reactions of Benzylic Electrophiles" **Konev, M. O**. Endean, T. B. D.; Jarvo, E. R. *manuscript in preparation.*

7. "Rhodium-Catalyzed Intramolecular C-H Arylation Using Inert Phenol Derivatives" **Konev, M. O**.; Higashino, M.; Yasui, K.; Jarvo, E. R.; Tobisu, M.; Chatani, N. *manuscript in preparation.*

6. "Nickel-Catalyzed Directed Hydroarylation of Alkynes" Hanna, L. E.; **Konev, M. O.**; Jarvo, E. R. *ready to submit*.

5. "Synthesis of Substituted *Z*-Styrenes by Hiyama-type Coupling of Oxasilacycloalkenes: Application to the Synthesis of 1-Benzocanes and the Carbon Skeleton of Glandulone B" Engles, C. A.; Bray, S. L.; Wold, E. D.; Porter, C. L.; **Konev, M. O.**; Vyvyan, J. R. *manuscript in preparation.*

4. "Nitroxyl Surprise: A Simple Amine Additive Revealed as Copper's Co-catalyst in the Aerobic Oxidation of Alcohols" **Konev, M. O**.; Jarvo E. R. *ACS Cent. Sci.* **2017**, *3*, 272

3. "Decarboxylative Alkyl-Alkyl Cross-Coupling Reactions" **Konev, M. O**.; Jarvo E. R. *Angew. Chem. Int. Ed.* **2016**, *55*, 11340.

2. "Nickel-Catalyzed Reductive Cross-Electrophile Coupling Reactions of Primary and Secondary Benzylic Esters with Aryl Halides" **Konev, M. O.**; Hanna, L. E.; Jarvo, E. R. *Angew. Chem. Int. Ed.* **2016**, *55*, 6730.
*Article highlighted in *Org. Process Res. Dev.* **2016**, *20,* 1109.

1. "Enantiospecific Intramolecular Heck Reactions of Secondary Benzylic Ethers" Harris, M. R.; **Konev, M. O.**; Jarvo, E. R. *J. Am. Chem. Soc*. **2014**, *136*, 7825. *Article highlighted in *Synfacts*, **2014**, *10*, 932.

Presentations and Conferences

7. Oral Presentation: "Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Primary and Secondary Benzylic Esters with Aryl Halides" *251st ACS National Meeting*, San Diego, CA. March 13th-17th, **2016**.

6. Oral Presentation: "Nickel-Catalyzed Intramolecular Enantiospecific Heck Reactions of Secondary Benzylic Ethers" *45th ACS Western Regional Meeting*, San Marcos, CA. November 6th-8th, **2015**.

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Awards

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References

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ABSTRACT OF THE DISSERTATION

Development of Nickel-Catalyzed Coupling Reactions: Intramolecular Alkyl-Heck and Reductive Cross-Electrophile Cyclizations and Hydroarylation of Alkynes

by

Mikhail Olegovich Konev

Doctor of Philosophy in Chemistry

University of California, Irvine, 2017

Professor Elizabeth R. Jarvo, Chair

Transition metal catalyzed reactions are ubiquitous in the realm of synthetic chemistry, allowing for the strategic construction of complex molecular frameworks of pharmaceuticals, natural products, and synthetic materials. Palladium-catalyzed cross-coupling reactions are part of the foundation of these transformations, insofar as they were recognized with the 2010 Nobel Prize in chemistry. Traditionally, these reactions have relied on aryl and vinyl electrophiles, whereas the alkyl counterparts have only recently begun to emerge in the literature. Nickel has been on the forefront of enantioconvergent alkyl cross-coupling reactions due to its propensity to undergo single electron chemistry. However, under special conditions, it has a unique ability to break strong carbon–oxygen bonds in a stereospecific manner, making research into its reactivity a valuable endeavor to the field of organometallic chemistry.

Chapter 1 describes the development of a stereospecific intramolecular alkyl-Heck cyclization of benzylic ethers. The reaction proceeds with inversion at the electrophilic carbon, for the synthesis of methylenecyclopentanes of both extended π-electron and simple

aromatic systems. The enantioenriched products can be effectively derivatized to cyclic α-aryl ketones in good yields with good transfer of chirality. Avenues to expand the utility of this reaction have been identified and further studies are ongoing.

Chapter 2 discusses the development of nickel-catalyzed cross-electrophile coupling reactions of benzylic esters and aryl halides. An intermolecular reaction proceeds in high yields for primary benzylic esters for the synthesis of pharmacologically relevant diarylmethanes. The corresponding intramolecular cyclization proceeds under mild conditions, demonstrating the first example of a stereospecific cross-electrophile coupling of secondary benzylic esters. A variety of heterocyclic and functionalized substrates are tolerated under the reaction conditions.

Chapter 3 examines the development a regio- and stereoselective nickel-catalyzed hydroarylation of alkynes with arylboronic acids. The reaction is facilitated by propargyl carbamates as directing groups. The reaction is tolerant of a range of functional groups and heterocycles. Mechanistic studies reveal that the acidic protons of the arylboronic acid coupling partner serve as the origin of hydrogen. Furthermore, the synthesis of tamoxifen can be completed in two steps from a simple hydroarylation product.

Chapter 1

Stereospecific Intramolecular Heck Cyclization of Secondary Benzylic Ethers

1.1 Introduction

Transition metal-catalyzed cross-coupling reactions have seen exhaustive use in chemical applications, particularly for the development of carbon-carbon bond forming reactions.[1] These transformations are an indispensable tool insofar as they were recognized in 2010 with the Nobel Prize in chemistry for their use organic synthesis. The Mizoroki–Heck reaction was one of the reactions highlighted in this award and as such, it is part of the foundation of modern organometallic chemistry and a key disconnection in the synthetic chemist's gamut.[2,3] Traditional Heck reactions employ aryl or vinyl (pseudo)halides as electrophiles. The development of alkyl-Heck reactions of alkyl (pseudo)halides is undergoing revitalization in part due to synergy with recent advances in alkyl cross-coupling reactions.[4,5] Exciting results employing primary alkyl halides have been reported, where catalyst control suppresses undesired side reactions and provides regioselectivity and asymmetric induction in the migratory insertion step.[6] Nickel catalysts have also recently been employed for the branched selective Heck reaction of primary benzylic chlorides with

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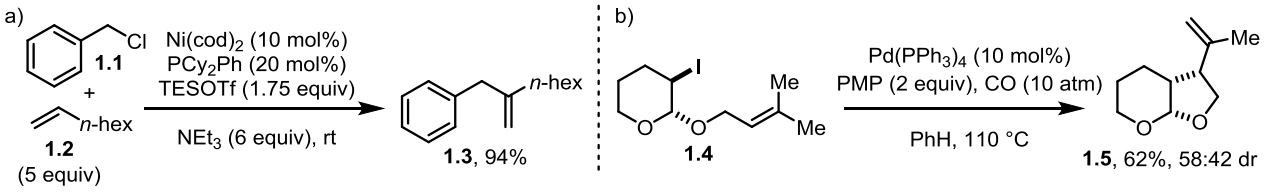
[1] Nicolaou, K. C.; Bulger, P. G.; Sarlah, D. *Angew. Chem. Int. Ed*. **2005**, *44*, 4442.
[2] A portion of this chapter was originally published in journal format: Harris, M. R.; Konev, M. O.; Jarvo, E. R. *J. Am. Chem. Soc*. **2014**, *136*, 7825.
[3] (a) Seechurn, C. C. C. J.; Kitching, M. O.; Colacot, T. J.; Snieckus, V. *Angew. Chem., Int. Ed.* **2012**, *51*, 5062. (b) Heck, R. F. *Org. React.* **1982**, *27*, 345. (c) The Mizoroki–Heck Reaction; Oestreich, M., Ed.; Wiley: Chichester, 2009. (d) Review of asymmetric Heck reactions in synthesis: Dounay, A. B.; Overman, L. E. *Chem. Rev*. **2003**, *103*, 2945. (d) Review of industrial applications of the Heck reaction: Torborg, C.; Beller, M. *Adv. Synth. Catal.* **2009**, *351*, 3027.
[4] Firmansjah, L.; Fu, G. C. *J. Am. Chem. Soc*. **2007**, *129*, 11340.
[5] (a) Swift, E. C.; Jarvo, E. R. *Tetrahedron* **2013**, *69*, 5799. (b) Jana, R.; Pathak, T. P.; Sigman, M. S. *Chem. Rev*. **2011**, *111*, 1417.
[6] Yang, Z.; Zhou, J. *J. Am. Chem. Soc*. **2012**, *134*, 11833.

terminal olefins (Scheme 1.1 a).[7] While this preliminary work demonstrates the feasibility of these reactions, no reaction occurred when substitution at the benzylic position was introduced. Intramolecular Heck-like reactions of secondary alkyl iodides that proceed through radical intermediates have also been reported and provide cyclization with high diastereoselectivity; however, these processes are mechanistically distinct compared to genuine alkyl-Heck reactions (Scheme 1.1 b).[8] Important challenges remain: these methods have been unsuccessful in employing secondary electrophiles to furnish enantioenriched coupled products and control of the absolute configuration at the site of oxidative addition had not been reported at the time.

Scheme 1.1. Nickel-catalyzed Heck reaction of a) primary benzylic chlorides and b) Heck-like cyclization of secondary alkyl iodides

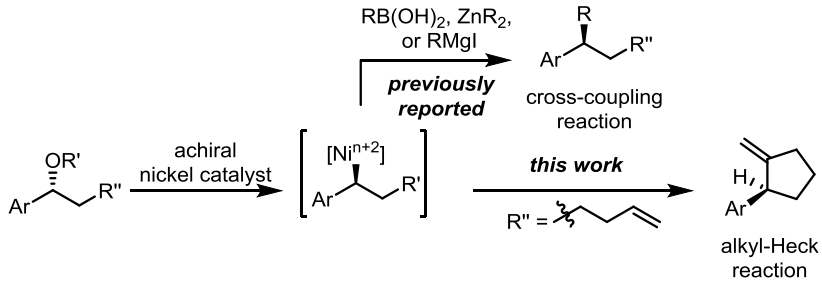


We hypothesized that enantioenriched secondary benzylic ethers functionalized with a pendant alkene should undergo nickel-catalyzed Heck cyclization and the reactions would be highly stereospecific. This work builds on our group's development of related stereospecific nickel-catalyzed cross-coupling reactions of benzylic C–O electrophiles (Scheme 1.2).[9] We propose that oxidative addition occurs with inversion, providing a single

[7] (a) Matsubara, R.; Gutierrez, A. C.; Jamison, T. F. *J. Am. Chem. Soc.* **2011**, *133*, 19020. (b) Standley, E. A.; Jamison, T. F. *J. Am. Chem. Soc*. **2013**, *135*, 1585.
[8] (a) Affo, W.; Ohmiya, H.; Fujioka, T.; Ikeda, Y.; Nakamura, T.; Yorimitsu, H.; Oshima, K.; Imamura, Y.; Mizuta, T.; Miyoshi, K. *J. Am. Chem. Soc*. **2006**, *128*, 8068. (b) Bloome, K. S.; McMahen, R. L.; Alexanian, E. J. *J. Am. Chem. Soc*. **2011**, *133*, 20146.
[9] Tollefson, E. J.; Hanna, L. E.; Jarvo, E. R. *Acc. Chem. Res*. **2015**, *48*, 2344.

enantiomer of the key secondary alkylnickel intermediate that can continue through the cross-coupling or Heck catalytic cycle.

Scheme 1.2. Stereospecific nickel-catalyzed reactions of benzylic C–O electrophiles

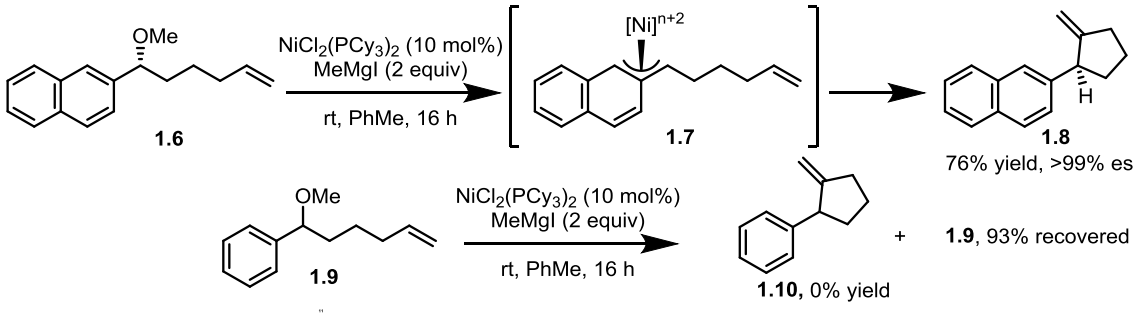


1.2 Development of Stereospecific Heck Cyclization of Simple Benzylic Ethers

Preliminary experiments, carried out by Michael Harris, showed that using an air-stable $NiCl_2(PCy_3)_2$ precatalyst with methylmagnesium iodide as a base resulted in naphthalene-containing Heck product **1.8** in 76% yield and >99% enantiospecificity.[10] The corresponding reaction with simple phenyl derivative **1.9** did not provide any desired product and only starting material was observed. Metal-catalyzed cross-coupling reactions of benzylic electrophiles have been largely limited to extended π-electron aromatic systems, such as benzylic ether **1.6** (Scheme 1.3).[11] This limitation of non-extended π-electron aromatics is attributed to the higher aromatic stabilization energy of these substrates. It has been proposed that the product of oxidative addition is stabilized by the formation of a π-benzylnickel intermediate **1.7**, which requires dearomatization of the arene. The energy required for naphthalene dearomatization is 25.2 kcal/mol, whereas for benzene, it is 30.6 kcal/mol.[12]

[10] Harris, M. R. PhD. Dissertation, University of California, Irvine, **2015**.
[11] Greene, M. A.; Yonova, I. M.; Williams, F. J.; Jarvo, E. R. *Org. Lett.* **2012**, *14*, 4293.
[12] Brauer, D. J.; Krueger, C. *Inorg. Chem.* **1977**, *16*, 884.

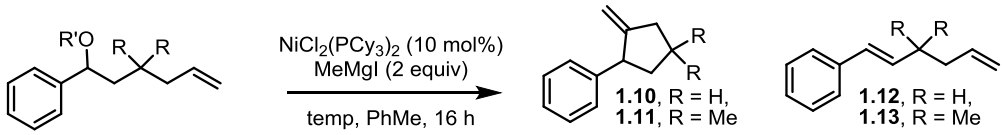
Scheme 1.3. Initial results of the intramolecular Heck reaction performed by Michael Harris



Based on the initial success of the Heck reaction with naphthalene containing substrates, we were hopeful that the reaction could be applied to non-extended π-electron substrates. Upon heating to 75 °C under the optimized conditions of the naphthyl system, use of simple benzylic ether **1.9** afforded desired product in 8% yield with trace β-hydride elimination side product and a majority of starting material recovered. To improve conversion, we implemented an methoxy ethyl ether moiety as a traceless directing group to accelerate oxidative addition by weakening the benzylic C–O bond through coordination of magnesium salts.[8] Furthermore, to disfavor β-hydride elimination and assist in cyclization by the Thorpe–Ingold effect,[13] we synthesized substrates bearing geminal methyl groups, which increased the yield of desired product to 67% with only 22% β-hydride elimination side product. Finally, addition of one equivalent of exogenous magnesium iodide suppressed β-hydride elimination while also increasing the yield of the desired product when performing the reaction under lower temperatures (Table 1.1).

[13] Beesley, R. M.; Ingold, C. K.; Thorpe, J. F. *J. Chem. Soc. Trans.* **1915**, *107*, 1080.

Table 1.1. Optimization of non-extended π-electron aromatic substrates



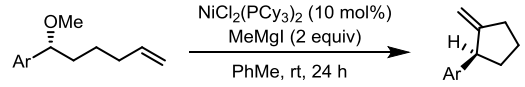
Entry	R	R'	temp (°C)	recovered SM (%)	yield **1.10** or **1.11** (%)	yield **1.12** or **1.13** (%)
1	H	Me	75	66	8	<5
2	H	$(CH_2)_2OMe$	75	10	29	54
3	Me	Me	80	18	59	19
4	Me	$(CH_2)_2OMe$	80	0	67	22
5[a]	Me	$(CH_2)_2OMe$	60	0	81	7

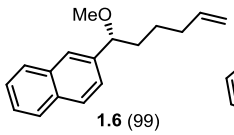
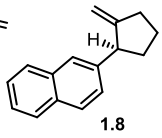
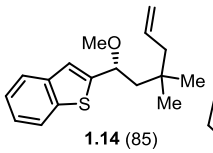
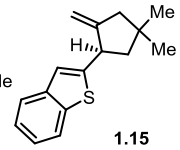
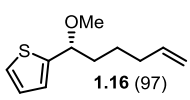
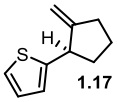
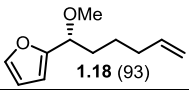
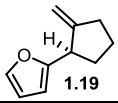
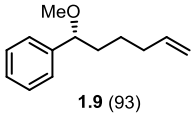
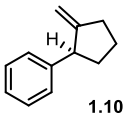
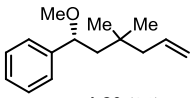
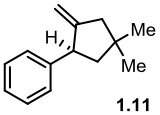
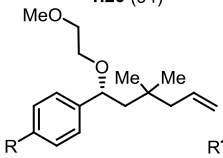
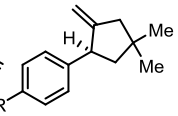
[a] Reaction performed with added MgI_2 (1 equiv).

1.3 Scope of Intramolecular Heck Cyclization

Next, we examined the cyclization of a range of enantioenriched benzylic ethers (Table 1.2). Benzylic methyl ethers of π-extended arenes proceed in excellent yield to afford highly enantioenriched methylenecyclopentanes (entries 1 and 2). Simple heteroarenes such as thiophene **1.16** and furan **1.18** also perform well under the reaction conditions (entries 3 and 4). Taking advantage of the Thorpe−Ingold effect by substitution of the alkyl chain with geminal dimethyl substituents improves the yield of the cyclization in general. Simple benzylic substrates such as **1.9** presented a challenge, where high enantiospecificity but modest yields were typically observed (entry 5). In this case, geminal disubstitution failed to improve yield (entry 6), but modification of the ether provided a solution (entries 7–9). As previously mentioned, our laboratory has developed the methoxyethyl ether as a traceless directing group that accelerates sluggish cross-coupling reactions. This strategy proved fruitful in the context of Heck reactions as well; methoxyethyl ethers **1.21**, **1.23**, and **1.25** afforded the desired methylenecyclopentanes at 60 °C. Yields could typically be further improved by approximately 10% with the addition of MgI_2 in all cases (1 equiv).

Table 1.2. Scope of intramolecular Heck cyclization



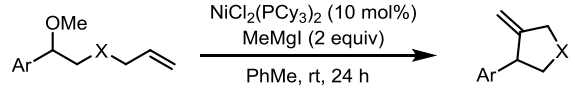
Entry	Substrate (ee (%))[a]	Product	yield (%)[b]	product ee (%)[a]	es (%)[c]
1	 **1.6** (99)	 **1.8**	74	99	>99
2	 **1.14** (85)	 **1.15**	74	84	99[f]
3	 **1.16** (97)	 **1.17**	73	97	>99
4[e]	 **1.18** (93)	 **1.19**	76	93	>99
5[g,h]	 **1.9** (93)	 **1.10**	31 (64)[i]	93	>99
6[g,h]	 **1.20** (94)	 **1.11**	35 (60)[i]	94	>99
7[g,h]	 **1.21** R = H (92)	 **1.22** R = H	67 (81)[i]	92	>99
8[g,h]	**1.23** R = *p*-F (90)	**1.24** R = *p*-F	57 (77)[i]	90	>99[f]
9[g,h]	**1.25** R = *p*-TMS (92)	**1.26** R = *p*-TMS	85	92	>99[f]

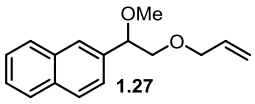
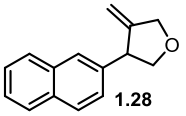
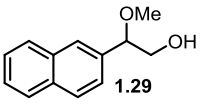
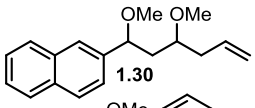
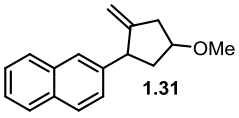
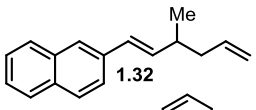
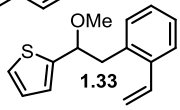
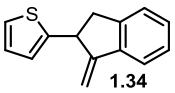
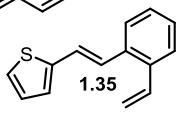
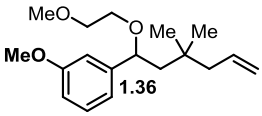
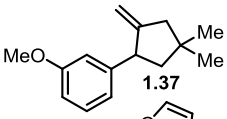
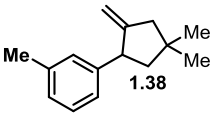
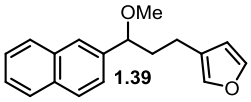
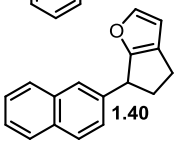
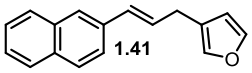
[a]Determined by SFC. [b]isolated yield.[c]$ee_{product}/ee_{substrate}$x100.[d]Reaction on 1.0 mmol scale.[e]Determined by GC.[f]ee determined from alcohol instead of ether.[g]Reaction at 60 °C.[h]Reaction with MgI_2 (1 equiv).[i]^{1}H NMR yield based on PhTMS as internal standard.

Representative examples of other substrates are presented to provide scope and limitations with respect to functional group compatibility and the formation of different ring sizes. Table 1.3 illustrates representative examples of substituent patterns that provide low yields or no desired Heck products. The Heck cyclization of substrates containing oxygenation in the tether failed to produce the desired tetrahydrofuran. This substrate class is susceptible to Tsuji–Trost type reactivity, and accordingly, we observed formation of the corresponding side product **1.29**. 1,3-Diol derivative **1.30** provided low yields of the desired product. Significant decomposition stems from the formation of an allylic ether side product that further reacts to a variety of products under the reaction conditions; upon β-hydride elimination, subsequent Tsuji–Trost methylation of the resulting allylic methoxy group occurs to provide **1.32** as the major side product. Subjecting thiophene **1.33** to cyclization

conditions afforded the desired substituted indane in low yield; however, elimination forming corresponding stilbene **1.35** was competitive with formation of desired product. The cyclization of simple benzylic substrate **1.36** went smoothly; however, the aryl methyl ether underwent undesired methylation. This reaction was recently explored by the Chatani group and they showed that the alkylation of anisole derivatives could even be performed using Grignard reagents with β-hydrogens.[14] Finally, any attempt to perform a heteroaryl Heck reaction with **1.39** did not provide desired product. The sole product of these reaction was styrene **1.41** from β-hydride elimination.

Table 1.3. Incompatible substrates for Heck cyclization



Entry	Substrate	Heck product	yield (%)	side product
1	 **1.27**	 **1.28**	<5	 **1.29**
2	 **1.30**	 **1.31**	23	 **1.32**
3	 **1.33**	 **1.34**	18	 **1.35**
4	 **1.36**	 **1.37**	<5[a]	 **1.38**
5	 **1.39**	 **1.40**	<5	 **1.41**

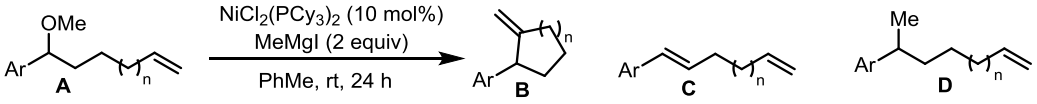
[a]Reaction performed with MgI_2 (1 equiv)

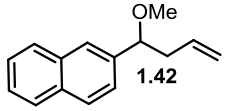
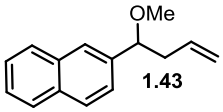
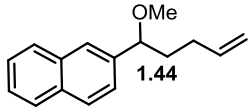
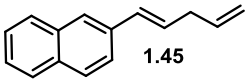
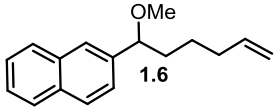
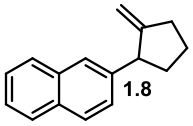
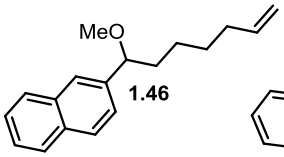
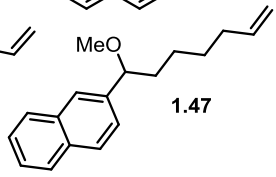
The length of the alkane tether was varied to determine if alternate rings sizes could be formed in the Heck cyclization. When the tether length is too small, we observe only

[14] Tobisu, M.; Takahira, T.; Morioka, T.; Chatani, *J. Am. Chem. Soc.* **2016**, *138*, 6711.

recovered starting material when the substrate is subjected to cyclization conditions (Table 1.4). Interestingly, no β-hydrogen elimination to form a conjugated styrenyl diene was observed. In contrast, when a similar benzylic ether lacking a terminal olefin is subjected to cyclization conditions, it is converted to the styrene in excellent yield. This observation is consistent with coordination of the olefin to the catalyst prior to oxidative addition. When the length of the alkane tether is such that 4-exo or 5-endo cyclization could occur, we observe high conversion to the elimination product (entry 2) and the Kumada cross-coupling product is also observed. When the tether is the appropriate length to form 6-membered rings in the Heck cyclization, only recovered starting material is observed.

Table 1.4. Attempted cyclization for varied ring sizes

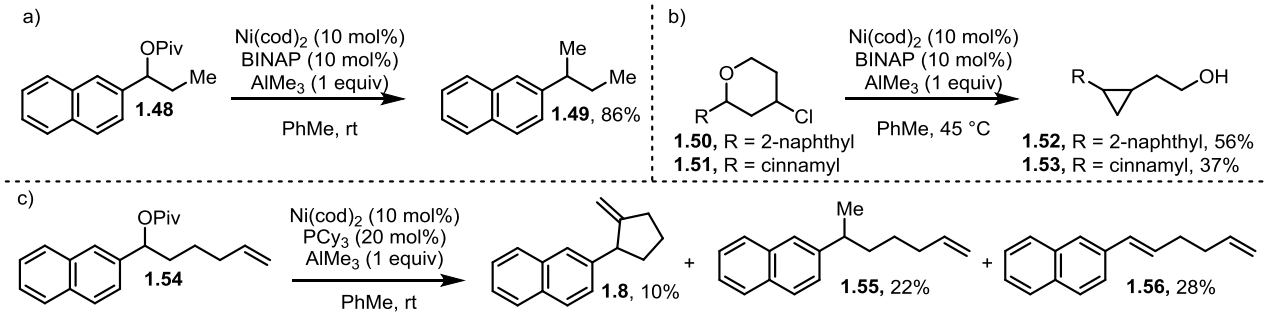


Entry	Substrate	Major Product	recovered **A** (%)	yield **B** (%)	yield **C** (%)	yield **D** (%)
1	 **1.42**	 **1.43**	99	< 5	< 5	< 5
2	 **1.44**	 **1.45**	< 5	< 5	61	36
3	 **1.6**	 **1.8**	< 5	81	15	< 5
4	 **1.46**	 **1.47**	99	< 5	< 5	< 5

Expanding the utility of the alkyl-Heck reaction can be achieved by utilizing a terminal reductant and base that is more compatible with functional groups than Grignard reagents.

Recent work in the Jarvo lab has shown that $AlMe_3$ can serve both as an alkylating reagent of benzylic electrophiles and a mediator for reductive ring contractions of 2-substituted-4-chlorotetrahydropyrans (Scheme 1.4 a, b). Applying this reagent to substrate **1.54** with PCy_3 yields the desired cyclized product in modest yields (Scheme 1.4 c). Use of alkylaluminum reagents is a promising strategy for the development of a mild Heck cyclization and will continue to be investigated in future studies.

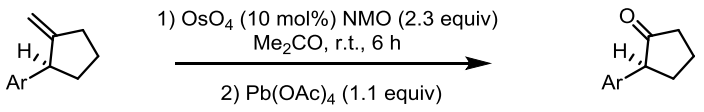
Scheme 1.4. Recent developments of aluminum-mediated coupling reactions



1.4 Derivatization of Methylenecyclopentanes to Cyclic α-Aryl Ketones

Methylenecyclopentanes are valuable synthetic intermediates as the exocyclic olefin provides a synthetic handle for further elaboration to more complex products. For example, methylenecyclopentanes **1.8**, **1.10,** and **1.15** are readily converted to the corresponding enantioenriched α-aryl cyclopentanones by a two-step procedure (Scheme 1.5). Dihydroxylation of the olefin with OsO_4 followed by mild oxidative cleavage of the resultant diol with $Pb(OAc)_4$ affords α-aryl cyclopentanones **1.57-1.58** in good yields and enantiopurities.

Scheme 1.5. Two step sequence for the synthesis of cyclic α-aryl ketones



1.8, Ar = 2-naphthyl, 99% ee
1.10, Ar = Ph, 93% ee
1.15, Ar = 2-thianaphthyl

1.57, Ar = 2-naphthyl, 52% yield, 98% ee
1.58, Ar = Ph, 40% yield, 76% ee
1.59, Ar = 2-thianaphthyl, 58% yield

1.5 Conclusion

In summary, selective formation of methylenecyclopentanes containing tertiary stereogenic centers has been achieved by stereospecific, nickel-catalyzed intramolecular Heck cyclization of secondary ethers. The use of simple aromatic substrates in the intramolecular Heck reaction has been addressed by tuning of the leaving group and exogenous Lewis acid. Efforts to expand the scope of the transformation and elucidate mechanistic details are underway.

1.6 Experimental Details

General Procedures

All reactions were carried out under an atmosphere of N_2, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (CH_2Cl_2), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 ºC for 12 h) to remove H_2O. All other solvents utilized were purchased "anhydrous" commercially, or purified as described. ^1H NMR spectra were recorded on Bruker DRX-400 (400 MHz ^1H, 100 MHz ^{13}C, 376.5 MHz ^{19}F), GN-500 (500 MHz ^1H, 125.7 MHz ^{13}C), or CRYO-500 (500 MHz ^1H, 125.7 MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of triplets (tt), quartet (q), quintet (quin), apparent doublet (ad), apparent triplet (at), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 ºC. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm^{-1}). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F_{254} precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with $KMnO_4$, ceric ammonium molybdate (CAM), or *p*-anisaldehyde (PAA) solutions. Flash

chromatography was performed using Silica Gel 60 (170-400 mesh) from Fisher Scientific. Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Automatic Polarimeter. SFC determinations of enantiopurity were performed on a Berger Analytical instrument using a Daicel™ Chiralpak® column (OD-H, OJ-H, or AD-H; 100 bar, 50 °C, 215 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

Bis(tricyclohexylphosphine)nickel(II) chloride was purchased from Strem, stored in a glovebox under an atmosphere of N_2, and used as received. All other reagents were purchased commercially and used as received.

Where noted, silver nitrate impregnated silica gel was used to separate cyclization products from alkene byproducts, which was prepared as follows.[1] To a 1 L round bottom flask was added $AgNO_3$ (15 g) followed by H_2O (5 mL) and CH_3CN (100 mL), with the exclusion of light. The resulting solution was agitated for 15-20 min. Silica gel (100 mL) was added and agitated for 15-20 min. The solvent was removed in vacuo, the silica gel was dried overnight at reduced pressure (~ 1 torr), and then stored in the dark.

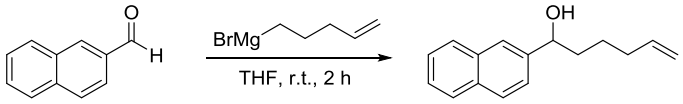
Preparation of Methylmagnesium Iodide

Under an argon atmosphere, dry Et_2O (10 mL) was added to magnesium turnings (1.1 g, 45 mmol) in a 3-neck flask equipped with a reflux condenser and Schlenk filtration apparatus. Iodomethane (1.9 mL, 31 mmol) was then added slowly (over 30 min), so as to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a Schlenk flask under an argon-atmosphere. The Schlenk flask was sealed and removed from the rest of the apparatus.

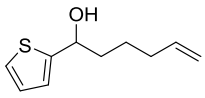
The resulting Grignard reagent titrated with LiCl and iodine and was typically between 2.5 and 3.0 M.[2] The Grignard reagent could be stored (sealed, under argon) for at least 4 weeks without detrimental effects. For satisfactory yields, the Grignard reagent must be prepared from the alkyl iodide in Et_2O at a concentration of at least 2.0 M.

Synthesis and characterization of substrates

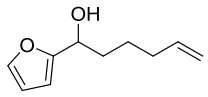
General Procedure A. *Grignard addition to aldehydes.*



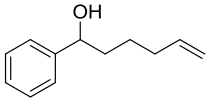
1.60. In a flame-dried round-bottom flask, to a solution of 2-naphthaldehyde (3.12 g, 20.0 mmol, 1.00 equiv) in THF (30 mL) was added at 0 °C pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 17 mL, 30 mmol, 1.5 equiv). After stirring at room temperature for 2 h, saturated ammonium chloride (25 mL) was added at 0 °C and the reaction was extracted with EtOAc (3 x 25 mL). The combined organic layers were washed with brine (1 x 40 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by column flash chromatography (10% EtOAc/hexanes) to afford the title compound as a white solid (4.30 g, 19.0 mmol, 95%). Analytical data is consistent with the values listed for (*R*)-**1.60** (vide infra).



1.61. Using representative procedure A outlined above, the following amounts of reagents were used: 2-thiophenecarboxaldehyde (0.56 mL, 6.0 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 5.0 mL, 9.0 mmol, 1.5 equiv). The product was purified by flash column chromatography (20% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.09 g, 6.00 mmol, quantitative). Analytical data is consistent with the values listed for (*R*)-**1.61** (vide infra).

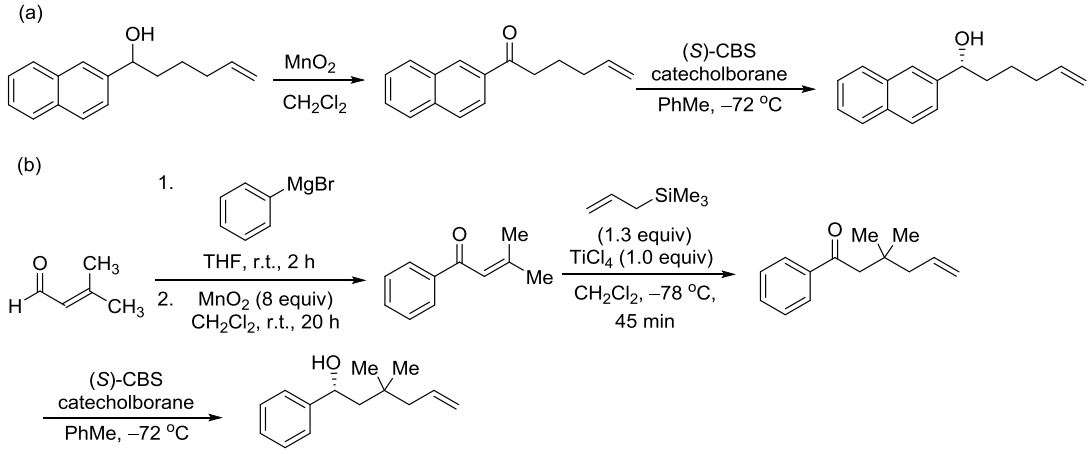


1.62. Using representative procedure A outlined above, the following amounts of reagents were used: furfural (0.83 mL, 10 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 8.3 mL, 15 mmol, 1.5 equiv). The product was purified by flash column chromatography (20% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.47 g, 8.85 mmol, 86%). Analytical data is consistent with the values listed for (*R*)-**1.62** (vide infra).

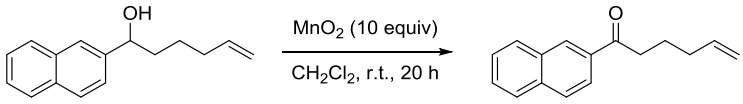


1.63. Using representative procedure A outlined above, the following amounts of reagents were used: benzaldehyde (0.71 mL, 7.0 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 5.8 mL, 11 mmol, 1.5 equiv). The product was purified by flash column chromatography (15% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.14 g, 6.44 mmol, 92%). Analytical data is consistent with the values listed for (*R*)-**1.63** (vide infra).

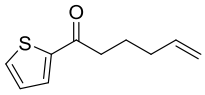
Scheme 1.6. Representative synthetic routes to enantioenriched alcohols



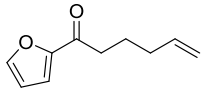
General Procedure B: *Oxidation of benzylic alcohols*



1.64. The product was prepared according to a modified procedure reported by Wipf.[3] To a solution of **1.60** (1.18 g, 5.20 mmol, 1.00 equiv) in wet CH_2Cl_2 (120 mL) was added in a single portion MnO_2 (4.52 g, 52.0 mmol, 10.0 equiv). The reaction was allowed to stir overnight at room temperature. The resulting slurry was filtered through celite, and the celite was washed with Et_2O. Solvent was removed in vacuo to afford the title compound as a yellow solid (1.08 g, 4.82 mmol, 93%). **TLC R_f** = 0.5 (10% EtOAc/hexanes, UV active); **m.p.** = 36–37 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.44 (s, 1H), 8.02 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.85 (t, *J* = 8.1 Hz 2H), 7.60–7.50 (m, 2H), 5.85 (ddt, *J* = 17.0, 10.2, 3.2 Hz, 1H), 5.07 (ad, *J* = 17.0 Hz, 1H), 5.02 (ad, *J* = 10.2 Hz, 1H), 3.08 (t, *J* = 7.7 Hz, 2H), 2.19 (q, *J* = 7.1 Hz, 2H), 1.90 (quin, *J* = 7.7 Hz 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.2, 138.2, 135.6, 134.4, 132.6, 129.7, 129.6, 128.5, 128.4, 127.8, 126.8, 124.0, 115.4, 37.8, 33.3, 23.5; **IR** (neat) 3058, 2829, 1678 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{16}H_{16}OH$ $(M + H)^+$ 225.1279, found 225.1278.

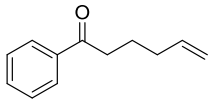


1.65. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol **1.61** (0.73 g, 4.0 mmol, 1.0 equiv), MnO_2 (3.50 g, 40.0 mmol, 10.0 equiv), CH_2Cl_2 (50 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (0.69 g, 3.8 mmol, 95%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 3.7, 1.0 Hz, 1H), 7.62 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.13 (dd, *J* = 4.9, 3.9 Hz, 1H), 5.82 (ddt, *J* = 17.1, 10.2, 3.3 Hz, 1H), 5.05 (dd, *J* = 17.1, 1.6 Hz, 1H), 5.00 (d, *J* = 10.2 Hz, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.16 (q, *J* = 7.2 Hz, 2H), 1.86 (quin, *J* = 7.3 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.3, 144.6, 138.0, 133.5, 131.8, 128.2, 115.5, 38.6, 33.3, 23.8; **IR** (neat) 3076, 2931, 1658 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{12}OSH$ $(M + H)^+$ 181.0687, found 181.0693.



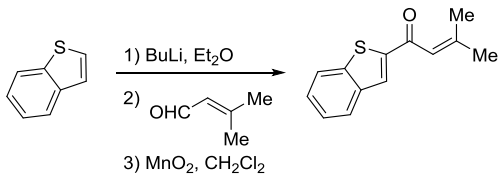
1.66. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol **1.62** (0.70 g, 4.2 mmol, 1.0 equiv), MnO_2 (3.7 g, 42 mmol, 10 equiv), CH_2Cl_2 (50 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (0.63 g, 3.9 mmol, 92%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.18 (d, *J* = 3.4, Hz, 1H), 6.53 (dd, *J* = 3.4, 1.6 Hz, 1H), 5.81 (ddt, *J* = 17.1, 10.1, 3.4 Hz, 1H), 5.04 (dd, *J* = 17.1, 1.6 Hz, 1H), 5.00 (d, *J* = 10.1 Hz, 1H), 2.83 (t, *J* = 7.6 Hz, 2H), 2.14 (q, *J* = 7.3 Hz, 2H), 1.83 (quin, *J* = 7.3 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.7, 152.9, 146.4, 138.0, 117.0, 115.5, 112.3,

37.7, 33.3, 23.4; **IR** (neat) 3076, 2933, 1646, 1569 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{12}O_2H$ $(M + H)^+$ 165.0916, found 165.0920.



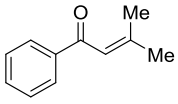
1.67. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol **1.63** (1.67 g, 9.50 mmol, 1.00 equiv), MnO_2 (8.26 g, 95.0 mmol, 10.0 equiv), CH_2Cl_2 (80 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (1.57 g, 9.00 mmol, 95%). Analytical data is consistent with literature values.[4] **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 5.83 (ddt, *J* = 17.0, 10.3, 6.7 Hz, 1H), 5.05 (d, *J* = 17.0 Hz, 1H), 5.00 (d, *J* = 10.3 Hz, 1H), 2.98 (t, *J* = 7.5 Hz, 2H), 2.16 (dd, *J* = 14.4, 7.2, Hz, 2H), 1.86 (tt, *J* = 14.4, 7.2 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.4, 138.2, 137.2, 133.1, 128.7, 128.2, 115.4, 37.8, 33.3, 23.4.

General Procedure C. *Preparation of α,β-unsaturated ketones.*

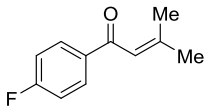


1.68. In a flame-dried round-bottom flask, to a solution of 3-methyl-2-butenal (3.80 mL, 39.4 mmol, 1.06 equiv) in THF (30 mL) was cooled to 0 °C. 2-lithiobenzothiophene[5] (1.5 M, 25 mL, 37 mmol, 1.0 equiv) was added. After stirring at room temperature for 2 h, saturated ammonium chloride (25 mL) was added at 0 °C and the reaction was extracted with EtOAc (3 x 25 mL). The combined organic layers were washed with brine (1 x 40 mL),

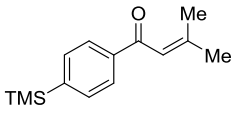
dried over $MgSO_4$, and concentrated in vacuo. The unpurified product was redissolved in 125 mL of CH_2Cl_2 in a 250 mL round bottom flask. Solid MnO_2 (12.4 g, 142 mmol, 3.84 equiv) was added in a single portion at room temperature. The reaction was allowed to stir open to air for 20 h before it was filtered through celite. The celite was washed with Et_2O and the solvents were removed in vacuo. The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a yellow solid (5.11 g, 23.6 mmol, 63%). **TLC R_f** = 0.6 (15% EtOAc/hexanes, UV active); **m.p.** = 56–58 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.88 (d, *J* = 8.2 Hz, 2H), 7.23 (m, 2H), 6.82 (quintet, *J* = 1.3 Hz, 1H), 2.30 (d, *J* = 1.1 Hz, 3H), 2.07 (d, *J* = 1.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 184.8, 158.5, 146.8, 142.6, 139.7, 127.9, 127.2, 126.0, 125.1, 123.1, 120.2, 28.4, 21.5; **IR** (neat) 3055, 2975, 1644, 1604, 1513, 1257, 1157 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{13}H_{12}OSNa$ $(M + Na)^+$ 239.0507, found 239.0513.



1.69. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.93 mL, 20.0 mmol, 1.00 equiv) in THF (15 mL), phenylmagnesium bromide (2.0 M, 15 mL, 30 mmol, 1.5 equiv), CH_2Cl_2 (125 mL), and manganese dioxide (13.91 g, 160.0 mmol, 8.000 equiv). The product was purified by flash column chromatography (5% EtOAc/ hexanes) to afford the title compound as a pale yellow solid (2.02 g, 12.5 mmol, 83%). Analytical data is consistent with literature values.[6] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 6.75 (s, 1H), 2.22 (s, 3H), 2.02 (s, 3H).



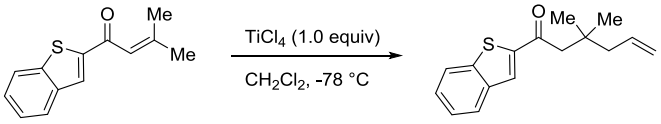
1.70. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.05 mL, 10.5 mmol, 1.00 equiv) in THF (15 mL), 4-fluorophenylmagnesium bromide (1.5 M, 7.0 mL, 10.5 mmol, 1.00 equiv), CH_2Cl_2 (50 mL), and manganese dioxide (4.47 g, 51.4 mmol, 4.90 equiv). The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a pale yellow oil (1.46 g, 8.19 mmol, 78%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.98–7.92 (m, 2H), 7.11 (t, *J* = 8.7 Hz, 2H), 6.71 (br s, 1H), 2.21 (s, 3H), 2.02 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 190.0, 165.3 (d, *J* = 253 Hz), 157.1, 135.6 (d, *J* = 3 Hz), 130.8 (d, *J* = 9 Hz), 120.9 (d, *J* = 1 Hz), 115.5 (d, *J* = 22 Hz), 28.1, 21.2; **IR** (neat) 2977, 1661, 1613, 1597, 1233, 1010, 822 cm^{-1}; [1]; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{11}H_{11}FOH$ $(M + H)^+$ 179.0872, found 179.0876.



1.71. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.10 mL, 11.4 mmol, 1.19 equiv) in THF (15 mL), 1-bromo-4-trimethylsilylphenylmagnesium bromide (1.4 M, 7.0 mL, 9.6 mmol, 1.0 equiv), CH_2Cl_2 (50 mL), and manganese dioxide (3.40 g, 39.1 mmol, 4.07 equiv). The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a pale yellow oil (1.33 g, 5.72 mmol, 60%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.9 Hz, 2H), 7.62 (d, *J* = 7.8 Hz, 2H), 6.76 (br s, 1H), 2.23 (s, 3H), 2.03 (s, 3H), 0.3 (s, 9H); **^{13}C NMR** (100 MHz,

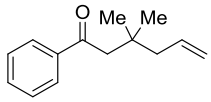
$CDCl_3$) δ 191.8, 156.8, 146.3, 139.6, 133.6, 127.4, 121.5, 28.2, 21.4, -1.1; **IR** (neat) 2955, 1661, 1613, 1246, 821 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{20}OSiH$ $(M + H)^+$ 233.1362, found 233.1360.

General Procedure D. *Hosomi–Sakurai reaction of benzylic enones*

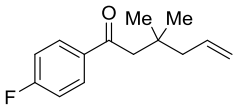


1.72. The product was prepared according to a modified procedure reported by Coates and co-workers.[7] To a flame-dried 50 mL round bottom flask was added **1.68** (4.52 g, 20.9 mmol, 1.00 equiv) dissolved in 20 mL CH_2Cl_2. Under an inert atmosphere was added neat titanium tetrachloride (2.60 mL, 23.7 mmol, 1.13 equiv) dropwise over 5 min at –78 °C. The reaction was allowed to stir at this temperature for 10 min before dropwise addition of allyltrimethylsilane (4.60 mL, 28.9 mmol, 1.38 equiv). The reaction was allowed to stir for an additional 5 min at –78 °C before it was placed in a room temperature water bath and stirred for 30 min. The reaction was cooled to 0 °C in an ice water bath and quenched with 2.25 M HCl. The reaction was diluted with Et_2O (80 mL) and washed with saturated $NaHCO_3$ (2 x 25 mL) and brine (1 x 30 mL). The organic layer was dried with $MgSO_4$, filtered, and concentrated in vacuo. The product was purified by flash column chromatography (25% EtOAc/hexanes) to afford the title compound as a yellow oil (1.73 g, 6.69 mmol, 32%). **TLC R_f** = 0.7 (15% EtOAc/hexanes, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.93–7.84 (m, 3H), 7.44 (m, 2H), 5.89 (quintet, *J* = 17.7, 10.4, 7.5 Hz, 1H), 5.13–5.04 (m, 2H), 2.88 (s, 2H), 2.21 (d, *J* = 7.5 Hz, 2H), 1.10 (s, 6H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 197.8, 146.0, 142.9, 139.5, 135.1, 129.2, 127.6, 126.2, 125.2, 123.2, 118.2, 49.0, 47.1, 34.7, 27.7

(2C); **IR** (neat) 3072, 2957, 1651, 1514, 1153 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{16}H_{18}OSNa$ $(M + Na)^+$ 281.0976, found 281.0974.

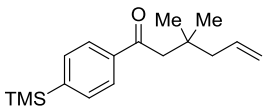


1.73. Using representative procedure D outlined above, the following amounts of reagents were used: **1.69** (1.98 g, 12.3 mmol, 1.00 equiv), titanium tetrachloride (1.4 mL, 12 mmol, 1.0 equiv), allyltrimethylsilane (2.54 mL, 16.0 mmol, 1.30 equiv). The product was purified by flash column chromatography (1–3% EtOAc/hexanes) to afford the title compound as a colorless oil (1.50 g, 7.40 mmol, 60%). **TLC R_f** = 0.5 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 5.85 (ddt, *J* = 17.2, 10.2, 4.2 Hz, 1H), 5.08–5.00 (m, 2H), 2.86 (s, 2H), 2.18 (d, *J* = 7.3 Hz, 2H), 1.05 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.4, 138.7, 135.2, 132.8, 128.6, 128.2, 117.8, 47.8, 46.9, 34.2, 27.7; **IR** (neat) 3077, 2957, 1689, 1673 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{18}OH$ $(M + H)^+$ 203.1436, found 203.1432.



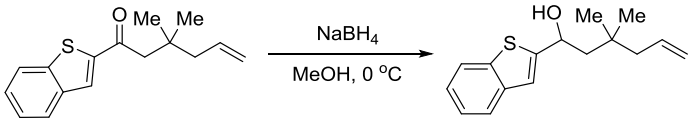
1.74. Using representative procedure D outlined above, the following amounts of reagents were used: **1.70** (1.35 g, 7.55 mmol, 1.00 equiv), titanium tetrachloride (0.91 mL, 8.8 mmol, 1.2 equiv), allyltrimethylsilane (1.60 mL, 10.1 mmol, 1.34 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (1.21 g, 5.49 mmol, 73%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99–7.92 (m, 2H), 7.15–7.07 (m, 2H), 5.85 (ddt, *J* = 17.7, 10.2, 7.5 Hz, 1H), 5.09–5.00 (m, 2H), 2.83 (s, 2H), 2.18 (d, *J* = 7.6 Hz, 2H), 1.05 (s,

6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 198.8, 165.7 (d, *J* = 254 Hz), 135.22 (d, *J* = 3 Hz), 135.18, 130.9 (d, *J* = 9 Hz), 118.0, 115.7 (d, *J* = 22 Hz), 47.8, 47.0, 34.3, 27.7; **IR** (neat) 2958, 1673, 1596, 1225, 1155 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{17}FOH$ $(M + H)^+$ 221.1342, found 221.1341.



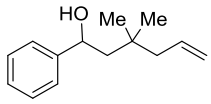
1.75. Using representative procedure D outlined above, the following amounts of reagents were used: **1.71** (1.23 g, 5.28 mmol, 1.00 equiv), titanium tetrachloride (0.65 mL, 5.9 mmol, 1.1 equiv), allyltrimethylsilane (1.10 mL, 6.92 mmol, 1.31 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (1.13 g, 4.12 mmol, 78%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (ad, *J* = 8.4 Hz, 2H), 7.61 (ad, *J* = 8.4 Hz, 2H), 5.86 (ddt, *J* = 18.2, 10.3, 7.4 Hz, 1H), 5.10–5.01 (m, 2H), 2.86 (s, 2H), 2.19 (d, *J* = 7.6 Hz, 2H), 1.06 (s, 6H), 0.30 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.7, 146.9, 138.9, 135.3, 133.7, 127.3, 117.9, 47.9, 47.0, 34.3, 27.8, -1.1; **IR** (neat) 2955, 1687, 1387, 1224, 835 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{26}OSiH$ $(M + H)^+$ 275.1831, found 275.1837.

General Procedure E. *Reduction of benzylic ketones with $NaBH_4$*

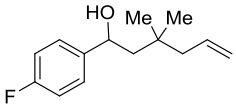


1.76. The product was prepared according to a modified procedure reported by Wang and Franzén.[8] A round bottom flask containing **1.72** (0.67 g, 2.6 mmol, 1.0 equiv) dissolved in MeOH (10 mL) was cooled to 0 °C in an ice water bath. Sodium borohydride (0.15 g, 4.0

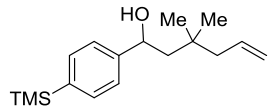
mmol, 1.5 equiv) was added in a single portion and the reaction was stirred for 30 min at 0 °C. The reaction was warmed to room temperature and stirred for an additional 1 h, after which time it was quenched with water. The reaction was extracted with Et_2O (3 x 25 mL) and washed with brine (1 x 40 mL). The combined organic layers were dried with $MgSO_4$, filtered and dried in vacuo. The residue was purified by flash column chromatography (5–10% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.58 g, 2.2 mmol, 85%). The analytical data is consistent with the values listed for (*R*)-**SI-21** (vide infra).



1.77. Using representative procedure E outlined above, the following amounts of reagents were used: **1.73** (0.73 g, 3.6 mmol, 1.0 equiv), MeOH (8 mL), sodium borohydride (0.33 g, 8.7 mmol, 2.4 equiv). The product was purified by flash column chromatography (8–15% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.64 g, 3.1 mmol, 86%). The analytical data is consistent with the values listed for (*R*)-**SI-23** (vide infra).

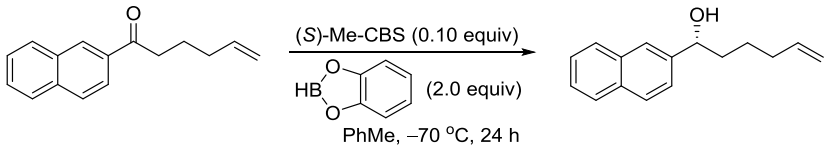


1.78. Using representative procedure E outlined above, the following amounts of reagents were used: **1.74** (0.50 g, 2.3 mmol, 1.0 equiv), MeOH (10 mL), sodium borohydride (0.30 g, 7.9 mmol, 3.4 equiv). The product was purified by flash column chromatography (3–10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.37 g, 1.7 mmol, 73%). The analytical data is consistent with the values listed for (*R*)-**SI-24** (vide infra).



1.79. Using representative procedure E outlined above, the following amounts of reagents were used: **1.75** (0.49 g, 1.8 mmol, 1.0 equiv), MeOH (10 mL), sodium borohydride (0.33 g, 8.7 mmol, 4.8 equiv). The product was purified by flash column chromatography (3–10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.35 g, 1.3 mmol, 86%). The analytical data is consistent with the values listed for (*R*)-**SI-25** (vide infra).

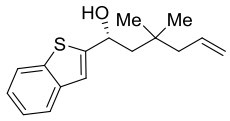
General Procedure F. *Enantioselective reduction of benzylic ketones*



(*R*)-1.60. The product was prepared according to a modified procedure reported by Okamura.[9] In a glovebox, (*S*)-Me-CBS (103 mg, 0.454 mmol, 0.100 equiv) was added to a flame-dried round bottom flask equipped with a stir bar. The flask was capped with a septum and removed from the box. Ketone **1.64** (1.02 g, 4.54 mmol, 1.00 equiv) was added to the flask as a solution in PhMe (20 mL). The reaction was then cooled to –70 °C and catecholborane (0.97 mL, 9.1 mmol, 2.0 equiv) was added dropwise via syringe. After stirring for 24 h at –70 °C, the reaction was warmed to ambient temperature and quenched with water. Sat. NH_4Cl was added to the reaction flask and the mixture was extracted with EtOAc (3 x 30 mL). The combined organics were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The product was purified by flash column chromatography (8–12% EtOAc/hexanes) to afford the title compound as a white solid (0.95 g, 4.3 mmol, 94%, 93% ee). The solid was recrystallized from hexanes to improve enantiomeric excess (99% ee).

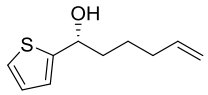
TLC R_f = 0.2 (10% EtOAc/hexanes, UV active); **m.p.** = 58–60 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88–7.80 (m, 3H), 7.78 (s, 1H), 7.52–7.43 (m, 3H), 5.78 (ddt, *J* = 17.3, 10.1, 3.3 Hz, 1H), 4.99 (dd, *J* = 17.1, 1.3 Hz, 1H), 4.94 (d, *J* = 10.3 Hz, 1H), 4.89–4.82 (m, 1H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.96–1.77 (m, 3H), 1.62–1.50 (m, 1H), 1.46–1.36 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.3, 138.7, 133.4, 133.1, 128.5, 128.1, 127.8, 126.3, 126.0, 124.7, 124.2, 114.9, 74.8, 38.5, 33.7, 25.2; **IR** (neat) 3279, 3056, 2932 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{16}H_{18}ONH_4$ $(M + NH_4)^+$ 244.1701, found 244.1701; **$[\alpha]^{26}_D$** +36 (*c* 3.6, $CHCl_3$); **SFC** analysis (OD-H, 8% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 7.2 minutes, t_R (minor) = 6.5 minutes.

E.J. Corey's model for stereoselectivity of CBS reductions was used to assign the absolute configuration of benzylic alcohols prepared from this method. [10]



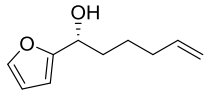
(*R*)-1.68. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.72** (0.500 g, 1.94 mmol, 1.00 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.13 equiv), catecholborane (0.40 mL, 3.8 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.280 g, 1.02 mmol, 56%, 85% ee). **TLC R_f** = 0.5 (15% EtOAc/hexanes, UV active); **m.p**. = 54–56 °C; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.84–7.80 (m, 1H), 7.74–7.70 (m, 1H), 7.33 (tdd, *J* = 14.6, 7.1, 1.4 Hz, 2H), 7.18 (s, 1H), 5.88 (, *J* = 16.8, 10.4, 7.4 Hz, 2H), 5.19 (dd, *J* = 7.7, 3.0 Hz, 1H), 5.11–5.03 (m, 2H), 2.13 (ddt, *J* = 7.4, 3.4, 1.3 Hz, 2H), 2.04 (s, 1H), 1.89 (d, *J* = 8.2, 1H), 1.85 (d, *J* = 3.8 Hz, 1H), 1.05 (s, 3H), 1.02 (s, 3H); **^{13}C**

NMR ($CDCl_3$, 100 MHz) δ 151.5, 139.7, 139.5, 135.6, 124.5, 124.4, 123.7, 122.7, 119.7, 117.6, 68.7, 50.8, 47.5, 33.6, 27.8, 27.7; **IR** (neat) 3403, 3071, 2956, 1458, 1436, 1366, 913 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{16}H_{20}OSNa$ $(M + Na)^+$ 283.1133, found 283.1122. **$[\alpha]^{30}_D$** +25 (*c* 1.1, $CHCl_3$); **SFC** analysis (OD-H, 15% IPA, 2.5 mL/min) indicated 85% ee: tR (major) = 6.3 minutes, t_R (minor) = 5.8 minutes.

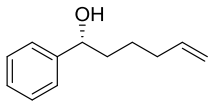


(*R*)-1.61. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.65** (0.45 g, 2.5 mmol, 1.0 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.10 equiv), catecholborane (0.53 mL, 5.0 mmol, 2.0 equiv), and PhMe (15 mL). After extraction with Et_2O, the combined organics were removed in vacuo, with much care taken to avoid heating. Heating the mixture under vacuum prior to column chromatography causes irreversible complexation of the desired product with boron reagents present from the reaction. The remaining solvent (mostly PhMe) was removed by running the mixture through a plug of silica. Flash column chromatography (10% EtOAc/hexanes) afforded the title compound as a clear oil (0.40 g, 2.2 mmol, 88%, 96% ee). **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.24 (dd, *J* = 4.3, 1.6 Hz, 1H), 6.99–6.94 (m, 2H), 5.79 (ddt, *J* = 17.3, 10.1, 3.3 Hz, 1H), 5.01 (add, *J* = 17.1, 1.5 Hz, 1H), 4.96 (d, *J* = 10.3, Hz, 1H), 4.92 (dd, *J* = 6.9, 3.9 Hz, 1H), 2.10 (q, *J* = 7.1 Hz, 2H), 2.05–2.00 (m, 1H), 1.94–1.78 (m, 2H), 1.60–1.51 (m, 1H), 1.48–1.38 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.9, 138.6, 126.7, 124.7, 123.9, 115.0, 70.3, 38.8, 33.6, 25.2; **IR** (neat) 3342, 3074, 2934, 2859 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{14}SOH$ $(M + H)^+$ 183.0844, found 183.0844; **$[\alpha]^{26}_D$** +21 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-

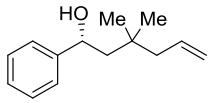
H, 3% IPA, 2.5 mL/min) indicated 96% ee: t_R (major) = 6.0 minutes, t_R (minor) = 5.1 minutes.



(*R*)-1.62. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.66** (0.52 g, 2.8 mmol, 1.0 equiv), (*S*)-Me-CBS (64 mg, 0.28 mmol, 0.10 equiv), catecholborane (0.60 mL, 5.6 mmol, 2.0 equiv), and PhMe (20 mL). After extraction with Et_2O, the combined organics were removed in vacuo, with much care taken to avoid heating. Heating the mixture under vacuum prior to column chromatography causes irreversible complexation of the desired product with boron reagents present from the reaction. The remaining solvent (mostly PhMe) was removed by running the mixture through a plug of silica. Flash column chromatography (10% Et_2O/hexanes) afforded the title compound as a clear oil (0.37 g, 2.2 mmol, 80%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (s, 1H), 6.33 (dd, *J* = 1.7, 1.1 Hz, 1H), 6.23 (d, *J* = 2.9 Hz, 1H), 5.80 (ddt, *J* = 17.1, 10.2, 3.3 Hz, 1H), 5.01 (add, *J* = 17.1, 1.5 Hz, 1H), 4.96 (d, *J* = 10.2, Hz, 1H), 4.68 (t, *J* = 7.0 Hz, 1H), 2.10 (q, *J* = 7.0 Hz, 2H), 1.94 (br s, 1H), 1.90–1.83 (m, 2H), 1.55 (sep, *J* = 7.2 Hz, 1H), 1.42 (sep, *J* = 7.3 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.9, 142.0, 138.6, 114.9, 110.3, 106.0, 67.8, 35.1, 33.6, 24.9; **IR** (neat) 3348, 3077, 2932, 2861 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{14}O_2NH_4$ $(M + NH_4)^+$ 184.1338, found 184.1329; **$[\alpha]^{24}_D$** +14 (*c* 1.0, $CHCl_3$).

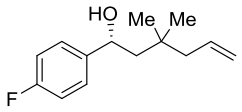


(*R*)-1.63. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.67** (0.435 g, 2.50 mmol, 1.00 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.10 equiv), catecholborane (0.53 mL, 5.0 mmol, 2.0 equiv), and PhMe (15 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear oil (0.396 g, 2.26 mmol, 90%). Analytical data is consistent with literature values.[11] **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38–7.31 (m, 4H), 7.30–7.25 (m, 1H), 5.78 (ddt, *J* = 17.0, 10.3, 6.6 Hz, 1H), 4.99 (d, *J* = 17.0 Hz, 1H), 4.94 (d, *J* = 10.3 Hz, 1H), 4.70–4.64 (m, 1H), 2.08 (dd, *J* = 14.2, 7.1 Hz, 2H), 1.86–1.76 (m, 2H), 1.76–1.68 (m, 1H), 1.57–1.48 (m, 1H), 1.43–1.33 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.9, 138.7, 128.6, 127.7, 126.0, 114.8, 74.7, 38.6, 33.7, 25.2; **$[\alpha]^{23}_D$** +38 (*c* 1.1, $CHCl_3$).

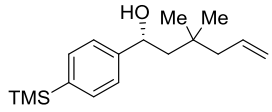


(*R*)-1.73. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.77** (0.42 g, 2.1 mmol, 1.0 equiv), (*S*)-Me-CBS (47 mg, 0.21 mmol, 0.10 equiv), catecholborane (0.44 mL, 4.1 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear oil (0.207 g, 1.01 mmol, 50%, 92% ee). Analytical data is consistent with literature values.[12] **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 4.3 Hz, 4H), 7.29–7.23 (m, 1H), 5.85 (ddt, *J* = 17.2, 10.1, 4.9 Hz, 1H), 5.08–4.99 (m, 2H), 4.85 (dt, *J* = 8.6, 3.2 Hz, 1H), 2.13–2.03 (m, 2H),

1.80–1.72 (m, 2H), 1.59 (dd, *J* = 14.7, 3.3 Hz, 1H), 1.00 (s, 3H), 0.97 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.6, 135.7, 128.7, 127.5, 125.8, 117.3, 72.3, 50.9, 47.4, 33.5, 27.82, 27.79; **IR** (neat) 3392, 2956, 1366 cm^{-1}; **$[\alpha]^{27}_D$** +55 (*c* 1.2, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.8 minutes, t_R (minor) = 2.1 minutes.

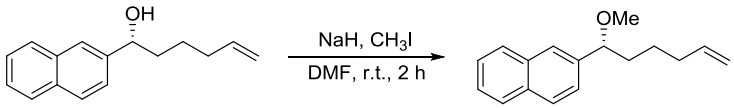


(*R*)-1.74. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.78** (0.632 g, 2.87 mmol, 1.00 equiv), (*S*)-Me-CBS (90 mg, 0.39 mmol, 0.17 equiv), catecholborane (0.62 mL, 5.8 mmol, 2.5 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.466 g, 2.10 mmol, 73%, 90% ee). **TLC R_f** = 0.3 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45–7.37 (m, 2H), 7.13 (at, *J* = 8.8 Hz, 2H), 5.97 (ddt, *J* = 17.6, 10.3, 7.4 Hz, 1H), 5.22–5.10 (m, 2H), 4.93 (dd, *J* = 8.5, 3.5 Hz, 1H), 2.19 (add, *J* = 7.4, 3.9 Hz, 1H), 2.15 (s, 1H), 1.85 (dd, *J* = 14.7, 8.6 Hz, 1H), 1.71 (dd, *J* = 14.7, 3.5 Hz, 1H), 1.11 (s, 3H), 1.08 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.2 (d, *J* = 245 Hz), 142.5 (d, *J* = 3 Hz), 135.7, 127.6 (d, *J* = 8 Hz), 117.4, 115.5 (d, *J* = 21 Hz), 71.6, 51.0, 47.5, 33.5, 27.91, 27.88; **IR** (neat) 3394, 2957, 1638, 1604, 1508, 1155 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{19}FOH$ $(M + H)^+$ 223.1498, found 223.1499; **$[\alpha]^{27}_D$** +45 (*c* 2.0, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 90% ee: t_R (major) = 9.5 minutes, t_R (minor) = 10.3 minutes.



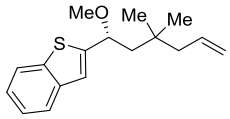
(*R*)-1.75. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **1.79** (0.612 g, 2.25 mmol, 1.00 equiv), (*S*)-Me-CBS (63 mg, 0.28 mmol, 0.12 equiv), catecholborane (0.50 mL, 4.5 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.283 g, 1.02 mmol, 46%, 92% ee). **TLC R_f** = 0.3 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 7.7 Hz, 2H), 5.84 (ddt, *J* = 17.6, 10.2, 7.5 Hz, 1H), 5.06–4.98 (m, 2H), 4.85–4.77 (m, 1H), 2.13–2.03 (m, 2H), 1.79–1.70 (m, 1H), 1.73 (ad, *J* = 8.8 Hz, 1H), 1.57 (dd, *J* = 14.8, 2.8 Hz, 1H), 1.00 (s, 3H), 0.97 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$ δ 147.2, 139.5, 135.7, 133.7, 125.1, 117.2, 72.2, 50.8, 47.4, 33.4, 27.8, 27.7, -1.0; **IR** (neat) 3388, 2955, 1638, 1600, 1386, 1248, 912 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{28}OSiH$ $(M + H)^+$ 277.1988, found 277.1990; **$[\alpha]^{27}_D$** +38 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.9 minutes, t_R (minor) = 2.3 minutes.

General Procedure G. *Methylation of benzylic alcohols.*



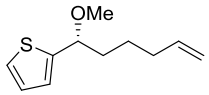
(*R*)-1.6. In a glovebox, NaH (42 mg, 1.8 mmol, 1.6 equiv) was added to a flame-dried round bottom flask equipped with a stir bar. The flask was removed from the glovebox, and anhydrous DMF (2 mL) was added. To this slurry was added a solution of alcohol (*R*)-**1.60**

(0.244 g, 1.10 mmol, 1.00 equiv) in DMF (4 mL) at 0 °C. The solution was warmed to room temperature over 30 min, then cooled to 0 °C and neat methyl iodide (0.14 mL, 2.2 mmol, 2.0 equiv) was added. The reaction was warmed to ambient temperature and stirred an additional 1.5 h. The reaction was quenched at 0 °C with 1M HCl and extracted with EtOAc (3 x 15 mL). The combined organic extracts were washed with water (1 x 50 mL) and brine (1 x 50 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by flash column chromatography (2–7% Et_2O/hexanes) to afford the title compound as a colorless oil (0.210 g, 0.890 mmol, 81%, 99% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88–7.80 (m, 3H), 7.70 (s, 1H), 7.51–7.41 (m, 3H), 5.76 (ddt, *J* = 17.1, 10.2, 3.2 Hz, 1H), 4.97 (ad, *J* = 17.1, Hz, 1H), 4.92 (d, *J* = 10.2 Hz, 1H), 4.25 (t, *J* = 6.9 Hz, 1H), 3.24 (s, 3H) 2.06 (q, *J* = 7.1 Hz, 2H), 1.95–1.86 (m, 1H), 1.77–1.68 (m, 1H), 1.58–1.48 (m, 1H) 1.41–1.31 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 139.9, 138.8, 133.3, 133.2, 128.5, 128.0, 127.9, 126.2, 126.0, 125.9, 124.6, 114.8, 84.2, 56.9, 37.6, 33.8, 25.3; **IR** (neat) 3057, 2859, 1601, 1098 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{17}H_{20}O$ $(M)^+$ 240.1514, found 240.1523; **$[\alpha]^{25}_D$** +82 (*c* 1.5, $CHCl_3$); **SFC** analysis (OD-H, 4% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 5.3 minutes, t_R (minor) = 5.0 minutes.

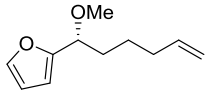


(*R*)-1.14. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.68** (0.190 g, 0.730 mmol, 1.00 equiv), NaH (36 mg, 1.5 mmol, 2.1 equiv), methyl iodide (0.10 mL, 1.1 mmol, 1.5 equiv) and THF (3 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.191 g, 0.696 mmol, 95%). **TLC R_f** = 0.9 (15%

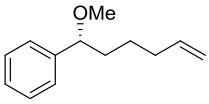
EtOAc/hexanes, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.86–7.82 (m, 1H), 7.76–7.72 (m, 1H), 7.34 (tdd, *J* = 14.8, 7.1, 1.4 Hz, 2H), 7.19 (s, 1H), 5.87 (m, 2H), 5.10–5.02 (m, 2H), 4.60 (dd, *J* = 8.5, 3.5 Hz, 1H), 3.30 (s, 3H), 2.09 (ddt, *J* = 7.5, 3.2, 1.1 Hz, 2H), 1.98, (dd, *J* = 14.7, 8.4 Hz, 1H), 1.70 (dd, *J* = 14.7, 3.4 Hz, 1H), 1.02 (s, 3H), 0.99 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 149.2, 139.9, 139.6, 135.7, 124.4, 124.3, 123.5, 122.8, 121.2, 117.4, 77.8, 56.6, 50.1, 47.6, 33.6, 27.8, 27.7; **IR** (neat) 3071, 2955, 1458, 1438, 1098, 912 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{17}H_{22}OSNa$ $(M + Na)^+$ 297.1289, found 297.1288; **$[\alpha]^{29}_D$** +60 (*c* 1.5, $CHCl_3$).



(*R*)-1.16. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.61** (0.219 g, 1.20 mmol, 1.00 equiv), NaH (46 mg, 1.9 mmol, 1.6 equiv), methyl iodide (0.15 mL, 2.4 mmol, 2.0 equiv) and DMF (6 mL). The product was purified by flash column chromatography (2–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.178 g, 0.910 mmol, 76%, 97% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 (d, *J* = 5.1 Hz, 1H), 6.98–6.94 (m, 2H), 5.79 (ddt, *J* = 17.1, 10.4, 3.3 Hz, 1H), 4.99 (adq, *J* = 17.1, 1.5 Hz, 1H), 4.94 (d, *J* = 10.2, Hz, 1H), 4.36 (t, *J* = 7.0 Hz, 1H), 3.25 (s, 3H), 2.06 (q, *J* = 7.2 Hz, 2H), 1.97–1.88 (m, 1H), 1.79–1.70 (m, 1H), 1.56–1.46 (m, 1H), 1.43–1.32 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.3, 138.7, 126.4, 125.4, 125.0, 114.8, 79.4, 56.6, 37.9, 33.6, 25.2; **IR** (neat) 3075, 2935, 1440 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{11}H_{16}OS$ $(M)^+$ 196.0922, found 196.0932; **$[\alpha]^{27}_D$** +70 (*c* 1.0, $CHCl_3$); **SFC** analysis (OD-H, 1% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.6 minutes.

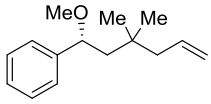


(*R*)-1.18. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.62** (0.20 g, 1.2 mmol, 1.0 equiv), NaH (46 mg, 1.9 mmol, 1.6 equiv), methyl iodide (0.15 mL, 2.4 mmol, 2.0 equiv) and DMF (6 mL). The product was purified by flash column chromatography (2–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.168 g, 0.936 mmol, 78%, 93% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 6.34 (dd, *J* = 2.8, 1.7 Hz, 1H), 6.26 (d, *J* = 3.1 Hz, 1H), 5.78 (ddt, *J* = 17.1, 10.3, 3.1 Hz, 1H), 4.99 (adq, *J* = 17.1, 1.5 Hz, 1H), 4.94 (d, *J* = 10.2, Hz, 1H), 4.16 (t, *J* = 7.0 Hz, 1H), 3.25, (s, 3H), 2.06 (q, *J* = 7.0 Hz, 2H), 1.95–1.86 (m, 1H), 1.85–1.76 (m, 1H), 1.54–1.43 (m, 1H), 1.40–1.28 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.5, 142.3, 138.7, 114.8, 110.0, 108.0, 76.6, 56.5, 33.6 (2C), 25.0; **IR** (neat) 2979, 2820, 1641, 1504 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{11}H_{16}O_2$ $(M)^+$ 180.1150, found 180.1145; **$[\alpha]^{26}_D$** +54 (*c* 1.3, $CHCl_3$); **GC** analysis: 93% ee (CYCLODEX B, inlet temp 220 °C, flow rate 5.3781 mL/min, initial temp 55 °C, hold 2 min, ramp 10 °C/min up to 180 °C, hold 3 min, ramp 40 °C/min up to 230 °C, hold 1 min, t_{R1} = 33.72 min, t_{R2} = 33.75.



(*R*)-1.9. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.63** (0.264 g, 1.50 mmol, 1.00 equiv), NaH (43 mg, 1.8 mmol, 1.2 equiv), methyl iodide (0.13 mL, 2.1 mmol, 1.4 equiv) and DMF (3 mL). The product was purified by flash column chromatography (2–5% Et_2O/hexanes) to afford the

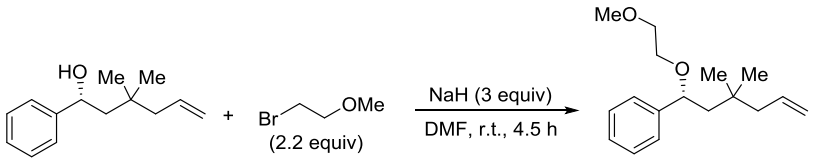
title compound as a colorless oil (0.257 g, 1.35 mmol, 90%, 93% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 7.1 Hz, 3H), 5.77 (ddt, *J* = 17.0, 10.3, 6.7 Hz, 1H), 4.98 (d, *J* = 17.0 Hz, 1H), 4.92 (d, *J* = 10.3 Hz, 1H), 4.09 (t, *J* = 6.9 Hz, 1H), 3.20 (s, 3H), 2.04 (dd, *J* = 14.2, 6.7 Hz, 2H), 1.86–1.77 (m, 1H), 1.68–1.58 (m, 1H), 1.55–1.45 (m, 1H), 1.39–1.29 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.5, 138.8, 128.5, 127.6, 126.8, 114.7, 84.1, 56.8, 37.8, 33.8, 25.3; **IR** (neat) 3056, 2925, 1601, 1154 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{13}H_{18}ONH_4$ $(M + NH_4)^+$ 208.1701, found 208.1706; **$[\alpha]^{23}_D$** +77 (*c* 0.7, $CHCl_3$); **SFC** analysis (OD-H, 5% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 1.9 minutes, t_R (minor) = 2.1 minutes.



(*R*)-1.20. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.73** (0.125 g, 0.610 mmol, 1.00 equiv), NaH (21 mg, 0.85 mmol, 1.4 equiv), methyl iodide (0.046 mL, 0.73 mmol, 1.2 equiv) and DMF (3 mL). The product was purified by flash column chromatography (2–5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.116 g, 0.530 mmol, 87%, 94% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.30–7.231 (m, 3H), 5.84 (m, 1H), 5.05–4.97 (m, 2H), 4.22 (dd, *J* = 8.7, 2.3 Hz, 1H), 3.15 (s, 3H), 2.09–1.99 (m, 2H), 1.78 (dd, *J* = 14.8, 8.7 Hz, 1H), 1.45 (dd, *J* = 14.8, 2.3 Hz, 1H), 0.96 (s, 3H), 0.94 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ144.0, 135.8, 128.5, 127.4, 126.6, 117.1, 81.7, 56.3, 50.2, 47.5, 33.5, 27.7; **IR** (neat) 3056, 2925, 1601, 1154 cm^{-1}; **HRMS** (TOF MS EI+) *m* / *z* calcd for $C_{15}H_{22}O$ $(M)^+$ 218.1671, found 218.1667; **$[\alpha]^{23}_D$** +88 (*c* 1.0, $CHCl_3$);

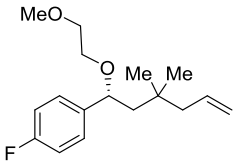
SFC analysis (OD-H, 1% IPA, 2.5 mL/min) indicated 94% ee: t_R (major) = 3.0 minutes, t_R (minor) = 3.2 minutes.

General Procedure H. *Alkylation of benzylic alcohol with 2-bromoethyl methyl ether*



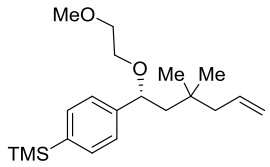
(*R*)-1.21. Alkylation of alcohols was performed according to a modified procedure reported by Lin.[13] Alcohol (*R*)-**1.73** (0.183 g, 0.900 mmol, 1.00 equiv) was dissolved in DMF (3.5 mL) and added to a slurry of NaH (65 mg, 2.7 mmol, 3.0 equiv) in DMF (1.3 mL) at 0 °C. The reaction mixture was stirred for 30 min at ambient temperature, and a solution of bromoethyl methyl ether (0.10 mL, 1.0 mmol, 1.1 equiv) in DMF (4.2 mL) was slowly added over 30 min at 0 °C. The reaction was stirred for an additional 1 h at ambient temperature, after which a second portion of bromoethyl methyl ether (0.10 mL, 1.1 mmol, 1.1 equiv) in DMF (4.2 mL) was slowly added over 30 min at 0 °C. After stirring for 2 h, saturated aqueous NH_4Cl (15 mL) and EtOAc (20 mL) were added at 0 °C. The layers were separated and the aqueous phase was extracted with EtOAc (3 x 15 mL). The combined organic layers were washed with brine (20 mL), dried over Na_2SO_4, and concentrated in vacuo. The crude mixture was purified by flash column chromatography (5–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.185 g, 0.704 mmol, 78%, 92% ee). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36–7.22 (m, 5H), 5.84 (ddt, *J* = 17.4, 9.8, 5.1 Hz, 1H), 5.04–4.97 (m, 2H), 4.39 (dd, *J* = 8.8, 2.6 Hz, 1H), 3.54–3.45 (m, 2H), 3.45–3.39 (m, 1H), 3.39–3.35 (m, 1H), 3.34 (s, 3H), 2.11–2.00 (m, 2H),

1.84 (dd, *J* = 14.6, 8.9 Hz, 1H), 1.44 (dd, *J* = 14.6, 2.2 Hz, 1H), 0.97 (s, 3H), 0.94 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.2, 136.0, 128.5, 127.4, 126.6, 117.0, 80.4, 72.2, 67.8, 59.0, 50.1, 47.4, 33.5, 27.8, 27.7; **IR** (neat) 2955, 2871, 1097 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{17}H_{26}O_2NH_4$ $(M + NH_4)^+$ 280.2277, found 280.2282; **$[\alpha]^{27}_D$** +66 (*c* 0.9, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.8 minutes, t_R (minor) = 2.1 minutes.



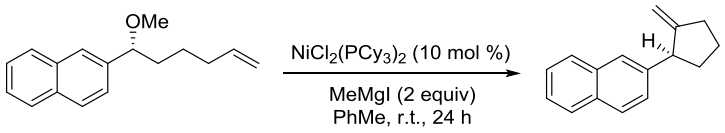
(*R*)-1.23. Using representative procedure H outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.74** (0.377 g, 1.70 mmol, 1.00 equiv), NaH (100 mg, 4.08 mmol, 2.40 equiv), bromoethyl methyl ether (0.50 mL, 5.3 mmol, 3.1 equiv) and DMF (8 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.252 g, 0.899 mmol, 53%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.6 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.28–7.22 (m, 2H), 7.01 (at, *J* = 8.8 Hz, 2H), 5.86 (ddt, *J* = 17.6, 10.4, 7.5 Hz, 1H), 5.04–4.97 (m, 2H), 4.37 (dd, *J* = 8.7, 2.8 Hz, 1H), 3.53–3.31 (m, 4H), 3.34 (s, 3H), 2.10–1.99 (m, 2H), 1.82 (dd, *J* = 14.7, 8.8 Hz, 1H) 1.42 (dd, *J* = 14.7, 3.0 Hz, 1H), 0.96 (s, 3H), 0.93 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.0 (d, *J* = 245 Hz), 139.8 (d, *J* = 3 Hz), 135.8, 128.0 (d, *J* = 8 Hz), 117.0, 115.3 (d, *J* = 21 Hz), 79.7, 72.1, 67.7, 58.9, 50.0, 47.3, 33.4, 27.7, 27.6; **IR** (neat) 2955, 2871, 1638, 1603, 1507, 1220, 1092, 912 cm-1; **HRMS** (TOF MS CI+)

m/z calcd for $C_{17}H_{25}FO_2NH_4$ $(M + NH_4)^+$ 298.2182, found 298.2185; $[\alpha]^{27}_D$ +54 (*c* 1.5, $CHCl_3$).

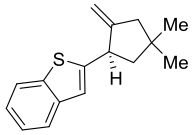


(*R*)-1.25. Using representative procedure H outlined above, the following amounts of reagents were used: alcohol (*R*)-**1.75** (0.197 g, 0.712 mmol, 1.00 equiv), NaH (52 mg, 2.2 mmol, 3.1 equiv), bromoethyl methyl ether (0.30 mL, 3.2 mmol, 4.5 equiv) and DMF (8 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.128 g, 0.382 mmol, 54%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.6 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 7.7 Hz, 2H), 5.86 (ddt, *J* = 17.9, 10.3, 7.5 Hz, 1H), 5.00 (m, 2H), 4.39 (dd, *J* = 9.2, 2.4 Hz, 1H), 3.53–3.35 (m, 4H), 3.35 (s, 3H), 3.36 (s, 3H), 2.11–2.01 (m, 2H), 1.83 (dd, *J* = 14.8, 9.2 Hz, 1H), 1.42 (dd, *J* = 14.8, 2.6 Hz, 1H), 0.98 (s, 3H), 0.95 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.7, 139.2, 135.9, 133.5, 125.8, 116.9, 80.3, 72.2, 67.8, 58.9, 50.1, 47.3, 33.5, 27.7, 27.6, -1.0; **IR** (neat) 2954, 1637, 1600, 1248, 1132, 911 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{20}H_{34}O_2SiNH_4$ $(M + NH_4)^+$ 352.2672, found 352.2670; $[\alpha]^{29}_D$ +48 (*c* 1.4, $CHCl_3$).

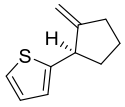
General procedure I*. Heck cyclization of benzylic ethers*



(*R*)-1.8. In a glovebox, a flame fried 7 mL vial equipped with a stir bar was charged with (*R*)-**1.6** (47 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The reaction was stirred 24 h before removing the vial from the glovebox, opening to atmosphere, quenching with isopropanol, and eluted through a silica gel plug (30% Et_2O/hexanes). The combined organics were concentrated in vacuo, internal standard was added (PhTMS, 17.2 μL, 0.100 mmol, 0.500 equiv), and ^{1}H NMR yield was collected. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (31 mg, 0.15 mmol, 74%, 99% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (t, *J* = 9.3 Hz, 3H), 7.66 (s, 1H), 7.44 (t, *J* = 6.8 Hz, 1H), 7.41 (t, *J* = 7.0 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 5.01 (s, 1H), 4.56 (s, 1H), 3.72 (at, *J* = 8.2 Hz, 1H), 2.63–2.49 (m, 2H), 2.26–2.18 (m, 1H), 1.96–1.81 (m, 2H), 1.78–1.66 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.2, 142.5, 133.6, 132.3, 128.1, 127.72, 127.67, 126.88, 126.87, 126.0, 125.3, 107.7, 51.6, 36.6, 33.7, 25.0; **IR** (neat) 3070, 2954, 2865, 1600 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{16}H_{16}H$ $(M + H)^+$ 209.1330, found 209.1326; **$[\alpha]^{24}_D$** –126 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 6.2 minutes, t_R (minor) = 5.8 minutes.

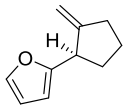


(*R*)-1.15. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**1.14** (55 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), and methylmagnesium iodide (0.16 mL, 2.5 M in Et2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (36 mg, 0.15 mmol, 74%, 84% ee). **TLC R_f** = 0.9 (100% pentane, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.75 (d, *J* = 7.9 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.27 (m, 2H), 7.10 (s, 1H), 5.06 (m, 1H), 4.87 (m, 1H), 4.17–4.09 (m, 1H), 2.40 (dq, *J* = 15.8, 2.5 Hz, 1H), 2.28 (dd, *J* = 15.8, 1.8 Hz, 1H), 2.08 (ddd, *J* = 12.5, 8.0, 1.9 Hz, 1H), 1.81 (dd, *J* = 12.4, 10.8 Hz, 1H), 1.15 (s, 3H), 1.06 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 154.9, 150.2, 140.2, 139.6, 124.2, 123.7, 123.0, 122.5, 120.7, 109.3, 51.1, 48.4, 45.5, 37.9, 29.2, 27.5; **IR** (neat) 3058, 2952, 2865, 1655, 1457, 885 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{16}H_{18}SH$ $(M + H)^+$ 243.1207, found 243.1206; **$[\alpha]^{27}_D$** –94 (*c* 1.7, $CHCl_3$); **SFC** analysis (OJ-H, 20% hexanes, 3.0 mL/min) indicated 84% ee: tR (major) = 6.3 minutes, t_R (minor) = 5.5 minutes.



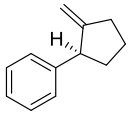
(*R*)-1.17. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**1.16** (37 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe

(1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). Care must be taken during workup as the product is relatively volatile. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (24 mg, 0.15 mmol, 73%, 97% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.15 (d, *J* = 5.0 Hz, 1H), 6.94 (t, *J* = 4.9 Hz, 1H), 6.86 (d, *J* = 2.9 Hz, 1H), 5.01 (s, 1H), 4.80 (s, 1H), 3.86 (at, *J* = 7.3 Hz, 1H), 2.55–2.45 (m, 2H), 2.28–2.19 (m, 1H), 1.91–1.78 (m, 2H), 1.74–1.62 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.5, 148.6, 126.6, 124.3, 123.4, 107.7, 46.1, 37.3, 32.7, 24.6; **IR** (neat) 3072, 2957, 1440 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{12}SH$ $(M + H)^+$ 165.0738, found 165.0739; **$[\alpha]^{27}_D$** –164 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-H, 2% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.6 minutes.



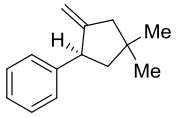
(*R*)-1.19. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**1.18** (36 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). Care must be taken while working up the product as it relatively volatile. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless and aromatic oil (22 mg, 0.15 mmol, 75%, 93% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (s, 1H), 6.29 (s, 1H), 6.05 (d, *J* = 2.6 Hz, 1H), 5.00 (s, 1H), 4.83 (s, 1H), 3.67 (t, *J* = 8.3 Hz, 1H), 2.44 (at, *J* = 6.6 Hz, 2H), 2.13–2.04 (m, 1H), 1.96–1.79 (m, 2H),

1.72–1.61 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 157.7, 153.3, 141.3, 110.1, 107.4, 105.0, 43.9, 32.92, 32.91, 24.9; **IR** (neat) 3075, 2959, 2870 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{10}H_{12}OH$ $(M + H)^+$ 149.0966, found 149.0971; **[α]$^{24}_D$** –133 (*c* 0.7, $CHCl_3$); **GC** analysis: 93% ee (CYCLODEX B, inlet temp 220 °C, flow rate 5.3781 mL/min, initial temp 55 °C, hold 2 min, ramp 10 °C/min up to 180 °C, hold 3 min, ramp 40 °C/min up to 230 °C, hold 1 min, t_{R1} = 15.29 min, t_{R2} = 15.48.

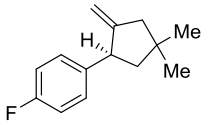


(*R*)-1.10. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**1.9** (38 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.15 mL, 2.7 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^{1}H NMR with PhTMS as internal standard was 64%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (12 mg, 0.073 mmol, 36%, 93% ee). **TLC R_f** = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.3 Hz, 2H), 7.24–7.16 (m, 3H), 4.97 (s, 1H), 4.53 (s, 1H), 3.55 (t, *J* = 7.9 Hz, 1H), 2.58–2.44 (m, 2H), 2.21 (m, 1H), 1.91–1.83 (m, 1H), 1.82–1.71 (m, 1H), 1.71–1.64 (m, 1H), 1.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.7, 145.1, 128.41, 128.39, 126.1, 107.4, 51.4, 36.6, 33.6, 29.9, 24.9; **IR** (neat) 3028, 2952, 1601 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{12}H_{14}H$ $(M + H)^+$ 159.1174, found

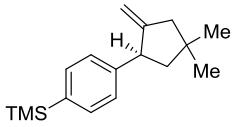
159.1168; **[α]$^{25}_{D}$** –93 (*c* 0.5, $CHCl_3$); **SFC** analysis (AD-H, 10% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 1.7 minutes, t_R (minor) = 1.9 minutes.



(*R*)-1.11. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**1.20** (52 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^{1}H NMR with PhTMS as internal standard was 81%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (25 mg, 0.13 mmol, 67%, 92% ee). **TLC R_f** = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.24–7.15 (m, 3H), 4.96 (s, 1H), 4.55 (s, 1H), 3.76 (tt, *J* = 10.6, 2.2 Hz, 1H), 2.38 (ad, *J* = 15.9 Hz, 1H), 2.29 (d, *J* = 15.9 Hz, 1H), 1.94 (dd, *J* = 12.5, 8.3 Hz, 1H), 1.76 (at, *J* = 12.0 Hz, 1H), 1.14 (s, 3H), 1.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.8, 145.6, 128.4, 128.3, 126.1, 108.3, 51.1, 49.9, 49.2, 37.7, 29.3, 27.6; **IR** (neat) 3027, 3063, 2952, 1602 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{18}H$ $(M + H)^+$ 187.1487, found 184.1479; **[α]$^{25}_{D}$** –126 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-H, 4% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.7 minutes, t_R (minor) = 1.6 minutes.



(*R*)-1.24. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**1.23** (56 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^1H NMR with PhTMS as internal standard was 77%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a colorless solid (23 mg, 0.11 mmol, 57%, 90% ee). **TLC R_f** = 0.6 (100% pentane, UV active, stain with $KMnO_4$); **m.p.** 32–35 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.25–7.19 (m, 2H), 7.03 (at, *J* = 8.7 Hz, 2H), 5.02 (s, 1H), 4.59 (s, 1H), 3.81 (tt, *J* = 8.6, 2.2 Hz, 1H), 2.46–2.31 (m, 2H), 1.99 (ddd, *J* = 12.2, 8.1, 1.2 Hz, 1H), 1.67 (at, *J* = 11.9 Hz, 1H), 1.20 (s, 3H), 1.11 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 161.3 (d, *J* = 243 Hz), 156.6, 141.0 (d, *J* = 3 Hz), 129.5 (d, *J* = 8 Hz), 115.0 (d, *J* = 21 Hz), 108.3, 51.1, 49.1, 48.9, 37.5, 29.2, 27.5, ; **IR** (neat) 2952, 1653, 1603, 1508, 1463, 1223, 815 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{17}F$ $(M)^+$ 204.1314, found 204.1316; **$[\alpha]^{27}_D$** –90 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 4% IPA, 2.0 mL/min) indicated 90% ee: t_R (major) = 2.1 minutes, t_R (minor) = 2.3 minutes.

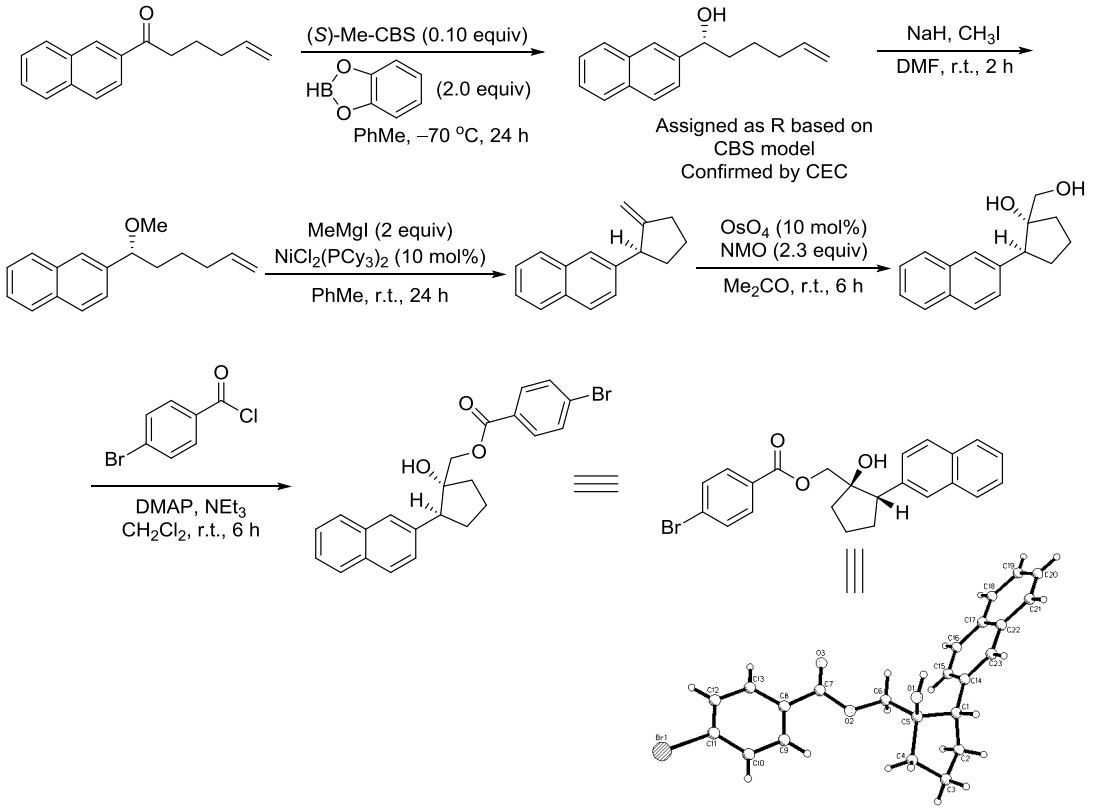


(*R*)-1.26. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**1.25** (67 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (44 mg, 0.17 mmol, 85%, 92% ee). **TLC R_f** = 0.3 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.97 (s, 1H), 4.57 (s, 1H), 3.78–3.72 (m, 1H), 2.38 (ad, *J* = 15.7 Hz, 1H), 2.28 (ad, *J* = 15.7 Hz, 1H), 1.93 (dd, *J* = 12.2, 8.3 Hz, 1H), 1.66 (t, *J* = 11.9 Hz, 1H), 1.13 (s, 3H), 1.05 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.5, 146.2, 137.5, 133.4, 127.6, 108.3, 50.9, 49.7, 49.2, 37.6, 29.2, 27.4, –1.0; **IR** (neat) 3067, 2952, 1653, 1600, 1247, 1109, 835 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{26}SiH$ $(M + H)^+$ 259.1882, found 259.1877; **$[\alpha]^{27}_D$** –109 (*c* 1.6, $CHCl_3$); **SFC** analysis (OJ-H, 3% hexanes, 2.0 mL/min) indicated 92% ee: t_R (major) = 2.6 minutes, t_R (minor) = 2.2 minutes

Stereochemical Proofs

The stereochemical course of the Heck cyclization is demonstrated for three examples. Those experiments are summarized below. In all examples, we confirm that the Heck cyclization proceeds with inversion. Full experimental details and characterization data for the derivatives synthesized to assign absolute configuration of products are provided below.

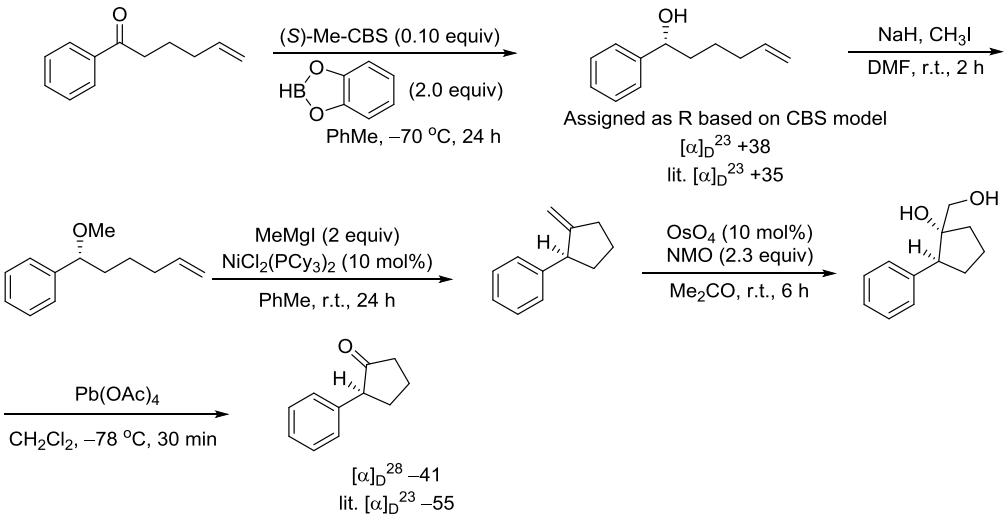
Scheme 1.7. Stereochemical course of the Heck cyclization.



Absolute configuration was assigned based on the accepted model for selectivity in CBS reductions[9] and confirmed by Competing Enantioselective Conversion (CEC) (vida infra). Conversion to methyl ether (*R*)-**1.6**, followed by stereospecific Heck cyclization produced (*R*)-**1.8**. Dihydroxylation of the olefin and conversion of the primary alcohol to

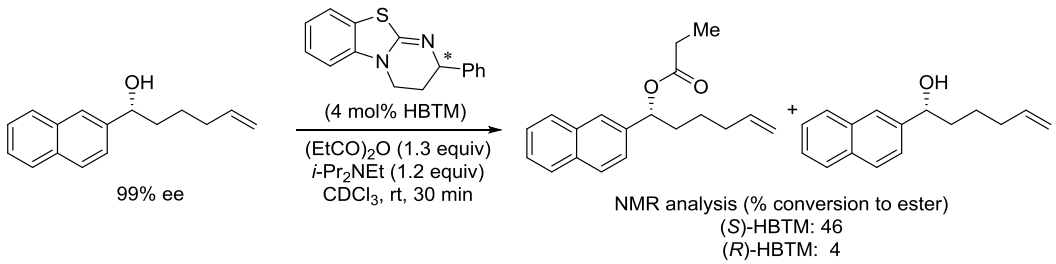
the 4-bromobenzoate afforded (*R*,*S*)-**1.77**, the absolute configuration of which was determined by X-ray crystallography. This product corresponds to net inversion in the Heck cyclization.

Scheme 1.8. Stereochemical course of the Heck cyclization

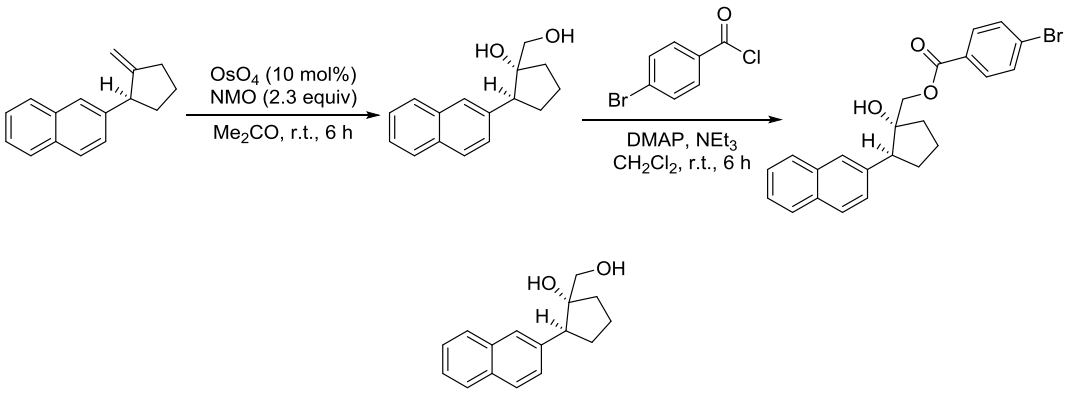


Enantioenriched alcohol (*R*)-**1.63** was prepared by enantioselective CBS reduction (vida infra). Absolute configuration was assigned based on the accepted model for selectivity in CBS reductions[10] and confirmed by comparison of the optical rotation to literature value.[11] Conversion to methyl ether (*R*)-**1.9**, followed by stereospecific Heck-cyclization produced (*R*)-**1.10**. Dihydroxylation of the olefin followed by oxidative cleavage of the diol (*R*,*S*)-**1.78** with $Pb(OAc)_4$ afforded α-aryl cyclopentanone (*S*)-**1.58**, the stereochemistry of which was determined by comparison of the optical rotation to the literature value.[14] This product corresponds to net inversion in the Heck cyclization.

Scheme 1.9. Confirmation of absolute configuration of alcohol (*R*)-**1.60**

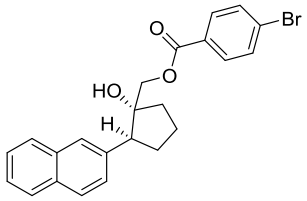


The competing enantioselective conversion experiment was performed according to the procedure outlined by Rychnovsky.[15] To a 7 mL vial under N_2 atmosphere was added alcohol (*R*)-**1.60** (11 mg, 0.050 mmol, 1.0 equiv), *i*-Pr_2NEt (11 µL, 0.060 mmol, 1.2 equiv), HBTM catalyst (0.20 mL, 0.010 M solution in $CDCl_3$, 0.0020 mmol, 0.040 equiv) and $CDCl_3$ (0.7 mL). The mixture was allowed to stir for 5 min before addition of propionic anhydride (8 µL, 0.07 mmol, 1.3 equiv). The reaction was stirred for 30 min and transferred to a NMR tube for ^1H NMR analysis. Percent conversion was measured for two reactions, one run with (*S*)-HBTM catalyst (46% conversion) and one with (*R*)-HBTM catalyst (4% conversion). Based on the pneumonic described by Rychnovsky, we assigned alcohol (*R*)-**1.60** as the *R* enantiomer. This assignment is consistent with E.J. Corey's stereochemical model for CBS reductions.



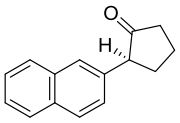
(*R*,*S*)-1.76. The title compound was prepared according to a modified procedure reported by Gaikwad.[16] A 20 mL scintillation vial charged with a stir bar and (*R*)-**1.8** (90 mg, 0.44 mmol, 1.0 equiv) and acetone (4 mL). To this solution was added *N*-methylmorpholine-*N*-oxide (119 mg, 1.01 mmol, 2.30 equiv) and OsO_4 (0.28 mL, 4% in H_2O, 0.044 mmol, 0.10 equiv). The reaction vessel was sealed and allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (5 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The diol was purified by flash column chromatography on silica gel (25% EtOAc/hexanes) to afford the title compound as a white, amorphous solid (88 mg, 0.36 mmol, 83%, >20:1 dr). **TLC R_f** = 0.3 (40% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83–7.71 (m, 3H), 7.64 (s, 1H), 7.48–7.40 (m, 2H), 7.38 (d, *J* = 8.6 Hz, 1H), 3.32 (t, *J* = 8.2 Hz, 1H), 3.29–3.23 (m, 1H), 3.23–3.15 (m, 1H), 2.86 (s, 1H), 2.33–2.23 (m, 1H), 2.11–1.77 (m, 5H), 1.74 (br s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.8, 133.5, 132.4, 128.1, 127.8, 127.7, 126.8, 126.29, 126.26, 125.7, 84.0, 66.9, 55.2, 36.2, 30.6, 22.1; **IR** (neat) 3371, 2954,

2874 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{16}H_{18}O_2Na$ $(M + Na)^+$ 265.1205, found 265.1204; **[α]$_{D}^{27}$** –29 (*c* 0.6, $CHCl_3$).



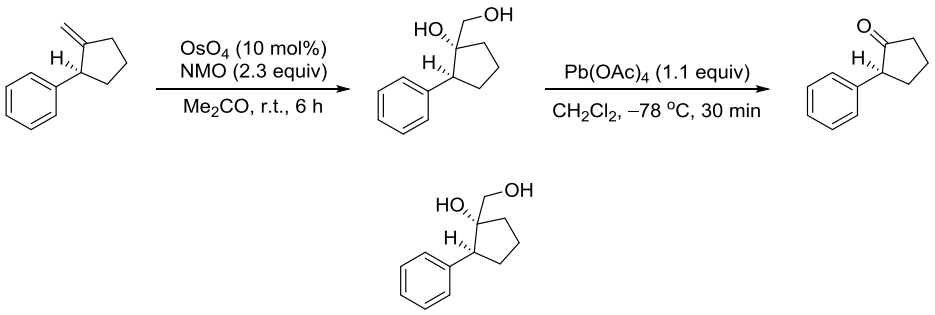
(*R*,*S*)-1.77. The title compound was prepared according to a modified procedure reported by Hassner.[17] Under an atmosphere of N_2, a 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**1.76** (40 mg, 0.17 mmol, 1.0 equiv) and dry CH_2Cl_2 (4 mL). To this solution was added 4-(dimethylamino)pyridine (2 mg, 0.02 mmol, 0.1 equiv), 4-bromobenzoylchloride (47 mg, 0.21 mmol, 1.3 equiv), and triethylamine (0.03 mL, 0.4 mmol, 2 equiv). The reaction was allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (4 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The product was purified by flash column chromatography on silica gel (15% EtOAc/hexanes) to afford the title compound as a white solid (62 mg, 0.15 mmol, 88%, 99% ee). A single crystal for X-ray crystallography was grown **TLC R_f** = 0.2 (20% EtOAc/hexanes, UV active, stain with $KMnO_4$); **m.p.** = 138 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.78–7.69 (m, 3H), 7.68 (s, 1H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.47–7.39 (m, 3H), 7.37 (d, *J* = 8.6 Hz, 2H), 4.11 (d, *J* = 11.7 Hz, 1H), 4.02 (d, *J* = 11.7 Hz, 1H), 3.45 (t, *J* = 8.5 Hz, 1H), 2.58 (br s, 1H), 2.39–2.30 (m, 1H), 2.22–2.12 (m, 1H), 2.09–1.92 (m, 4H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 165.9, 138.0, 133.5, 132.6, 131.6, 131.0, 128.5, 128.3, 128.1, 127.7, 127.6, 126.6, 126.5, 126.2, 125.7, 82.9, 69.3, 55.5, 36.7, 30.2, 21.8; **IR** (neat) 3487,

2956, 1717 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{23}H_{21}O_3BrNa$ $(M + Na)^+$ 447.0572, found 447.0581; $[\alpha]^{29}_D$ –68 (*c* 2.8, $CHCl_3$); **SFC** analysis (Whelk-O (R,R), 10% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 24.6 minutes, t_R (minor) = 23.5 minutes.



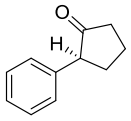
(*S*)-1.57. The title compound was prepared according to a modified procedure reported by Wicha.[18] A 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**1.76** (36 mg, 0.15 mmol, 1.0 equiv) and CH_2Cl_2 (4 mL). The reaction was cooled to –78 °C to avoid epimerization of the benzylic stereocenter. To this solution was added $Pb(OAc)_4$ (73 mg, 0.17 mmol, 1.1 equiv) in a single portion. The reaction was allowed to stir at –78 °C until TLC indicated the reaction had proceeded to conversion (30 min). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The ketone was purified by flash column chromatography on silica gel (10% EtOAc/hexanes) to afford the title compound as a white solid (20 mg, 0.095 mmol, 63%, 98% ee). Analytical data is consistent with literature values.[34] **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.76 (m, 3H), 7.64 (s, 1H), 7.49–7.41 (m, 2H), 7.31 (d, *J* = 8.6 Hz, 1H), 3.50 (at, *J* = 9.7 Hz, 1H), 2.61–2.54 (m, 1H), 2.52 (dd, *J* = 19.1, 8.6 Hz, 1H), 2.41–3.30 (m, 1H), 2.28–2.17 (m, 2H), 2.05–1.93 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 218.3, 136.0, 133.6, 132.6, 128.4, 127.82, 127.75, 126.9, 126.5, 126.2, 125.8, 55.6, 38.7, 31.9, 21.1; $[\alpha]^{24}_D$ –37 (*c* 0.9, $CHCl_3$); **SFC**

analysis (AS-H, 15% IPA, 2.5 mL/min) indicated 98% ee: t_R (major) = 3.9 minutes, t_R (minor) = 3.0 minutes.

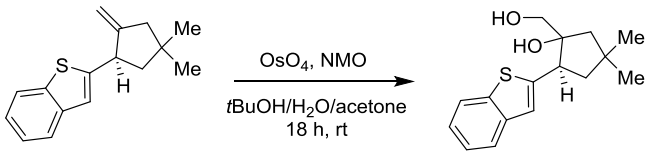


(*R*,*S*)-1.78. The title compound was prepared according to a modified procedure reported by Gaikwad.[36] A 20 mL scintillation vial charged with a stir bar and (*R*)-**1.10** (55 mg, 0.45 mmol, 1.0 equiv) and acetone (4 mL). To this solution was added *N*-methylmorpholine-*N*-oxide (122 mg, 1.03 mmol, 2.30 equiv) and OsO_4 (0.28 mL, 4% in H_2O, 0.045 mmol, 0.10 equiv). The reaction vessel was sealed and allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (5 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The diol was purified by flash column chromatography on silica gel (25% EtOAc/hexanes) to afford the title compound as clear oil (48 mg, 0.25 mmol, 56%, 7:1 dr). **TLC R_f** = 0.4 (40% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33–7.28 (m, 2H), 7.27–7.20 (m, 3H), 3.30 (d, *J* = 11.5 Hz, 1H), 3.24–3.13 (m, 2H), 2.75 (br s, 1H), 2.28–2.18 (m, 1H), 2.00–1.90 (m, 2H), 1.90–1.78 (m, 3H), 1.55 (br s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.3, 128.7, 128.0, 126.9, 83.8, 66.9, 55.2, 36.0, 30.5, 22.0; **IR** (neat) 3376, 2954,

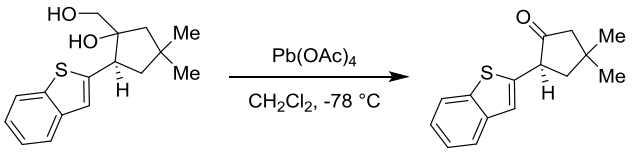
2874 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{12}H_{16}O_2Na$ $(M + Na)^+$ 215.1048, found 215.1044.



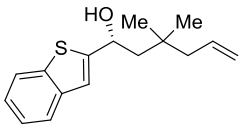
(*S*)-1.58. The title compound was prepared according to a modified procedure reported by Wicha.[38] A 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**1.78** (35 mg, 0.18 mmol, 1.0 equiv) and CH_2Cl_2 (4 mL). The reaction was cooled to –78 °C to avoid epimerization of the benzylic stereocenter. To this solution was added $Pb(OAc)_4$ (89 mg, 0.20 mmol, 1.1 equiv) in a single portion. The reaction was allowed to stir at –78 °C until TLC indicated the reaction had proceeded to conversion (30 min). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The ketone was purified by flash column chromatography on silica gel (10% EtOAc/hexanes) to afford the title compound as a white, amorphous solid (20 mg, 0.13 mmol, 72%, 76% ee). Analytical data is consistent with literature values.[34] **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.1 Hz, 2H), 7.28–7.22 (m, 1H), 7.19 (d, *J* = 7.1 Hz, 2H), 3.37–3.29 (m, 1H), 2.57–2.43 (m, 2H), 2.35–2.24 (m, 1H), 2.21–2.07 (m, 2H), 1.99–1.89 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 218.3, 138.6, 128.7, 128.3, 127.0, 55.5, 38.6, 31.9, 21.0; **$[\alpha]^{28}_D$** –41 (*c* 0.3, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 76% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.2 minutes.



(*R,S*)-1.79. A 5 mL round bottom flask equipped with a stir bar was charged with OsO_4 (100 mg, 2.5% wt% in *t*BuOH, 0.01 mmol), NMO (23 mg, 0.23 mmol), and water (1 mL). A solution of (*R*)-**1.15** (25 mg, 0.09 mmol) in acetone (1 mL) was added to the reaction mixture. After 18 hours, the reaction was quenched with sodium sulfite (100 mg) and diluted with EtOAc (10 mL). The aqueous layer was extracted with EtOAc (2 x 10 mL). The organic layers were dried with $MgSO_4$, filtered, and concentrated in vacuo. The crude mixture was purified by flash column chromatography (3:1 hexanes:EtOAc) to afford the title compound as a 1.5:1 mixture of diastereomers as a white solid (24 mg, 0.087 mmol, 84%). The diastereomeric ratio was determined based on integration of the benzylic methines in the ^{1}H NMR spectrum. The diastereomers were not assigned cis and trans configurations, but were carried forward as a mixture. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ 7.78 (dd, *J* = 7.8, 3.7 Hz, 1.5H), 7.70 (t, *J* = 8.3 Hz, 1.6H), 7.36–7.26 (m, 3.1H), 7.18 (s, 0.7H), 7.12 (s, 1.0H), 3.75 (dd, *J* = 13.3, 6.7 Hz, 1.0H), 3.62 (dd, *J* = 14.2, 11.5 Hz, 1.6H), 3.51 (dd, *J* = 12.8, 7.1 Hz, 1.3H), 3.32 (broad d, *J* = 10.8 Hz, 1.0H), 2.96 (s, 0.9H), 2.26 (t, *J* = 12.6 Hz, 0.8H), 2.05–1.72 (m, 7.4H), 1.25 (s, 2.8H), 1.25 (s, 2.8H), 1.15 (s, 2.7H), 1.12 (s, 2.0H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 144.0, 143.2, 139.9, 139.8, 139.4, 139.1, 124.5, 124.4, 124.04, 124.03, 123.11, 123.10, 122.24, 122.22, 122.19, 121.2, 82.8, 82.7, 68.8, 67.0, 51.3, 51.1, 50.6, 47.8, 47.6, 46.3, 35.7, 35.3, 31.5, 31.2, 30.90, 30.88, 29.8; **IR** (neat) cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{16}H_{20}O_2SNa$ $(M + Na)^+$ 299.1082, found 299.1074.



(*R*)-1.59. A 10 mL vial equipped with a stir bar was charged with a solution of (*R,S*)-**1.79** (24 mg, 0.087 mmol) in CH_2Cl_2 (5 mL). The reaction mixture was cooled to –78 °C and $Pb(OAc)_4$ (48 mg, 0.10 mmol) was added in a single portion. After 10 minutes, the reaction was at completion by TLC and saturated NH_4Cl (5 mL) was added to quench the reaction. The mixture was let to warm to room temperature and diluted with CH_2Cl_2 (5 mL). The aqueous layer was extracted with CH_2Cl_2 (2 x 5 mL. The organic layer was dried with $MgSO_4$, filtered, and concentrated in vacuo. The crude mixture was purified by flash column chromatography (4:1 hexanes:EtOAc) to afford the title compound as a white solid (12 mg, 0.49 mmol, 56%). **TLC R_f** = 0.75 (3:1 hexanes:EtOAc); **^{1}H NMR** ($CDCl_3$, 500 MHz) δ 7.84 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.40–7.31 (m, 2H), 7.22 (s, 1H), 3.96 (dd, *J* = 11.5, 9.2 Hz, 1H), 2.55–2.47 (m, 1H), 2.36 (q, *J* = 17.8 Hz, 2H), 2.21 (t, *J* = 12.4 Hz, 1H), 1.35 (s, 3H), 1.25 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 215.2, 141.6, 139.7, 139.4, 124.3, 123.9, 123.2, 122.2, 121.4, 52.8, 49.8, 45.7, 34.3, 29.6, 27.6; **$[\alpha]^{29}_D$** –47 (*c* 0.9, $CHCl_3$).



X-ray Data Collection, Structure Solution and Refinement for alcohol **(*R*)-1.68**

CCDC 1003301 contains the supplementary crystallographic data for this structure. These data can be obtained via www.ccdc.ac.uk/conts/retrieving.html. The crystal was obtained by slow evaporation from ethyl acetate over pentane.

A colorless crystal of approximate dimensions 0.062 x 0.120 x 0.329 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (120 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The systematic absences were consistent with the trigonal space groups $P3_1$ and $P3_2$. It was later determined that space group $P3_1$ was correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atom H(1) was located from a difference-Fourier map and refined (x,y,z and U_{iso}) with d(O-H) = 0.85Å. The remaining hydrogen atoms were included using a riding model. Carbon atoms C(12), C(13) and C(14) were disordered and included using multiple components with partial site-occupancy-factors (0.62:0.38).

At convergence, wR2 = 0.0960 and Goof = 1.039 for 196 variables refined against 3419 data (0.74Å), R1 = 0.0383 for those 3258 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

1. APEX2 Version 2014.1-1, Bruker AXS, Inc.; Madison, WI 2014.
2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.
3. Sheldrick, G. M. SADABS, Version 2012/1, Bruker AXS, Inc.; Madison, WI 2012.
4. Sheldrick, G. M. SHELXTL, Version 2014/2, Bruker AXS, Inc.; Madison, WI 2014.
5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.
6. Flack, H. D., Parsons, S., Wagner, T. Acta. Cryst., B69, 249-259, 2013

Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

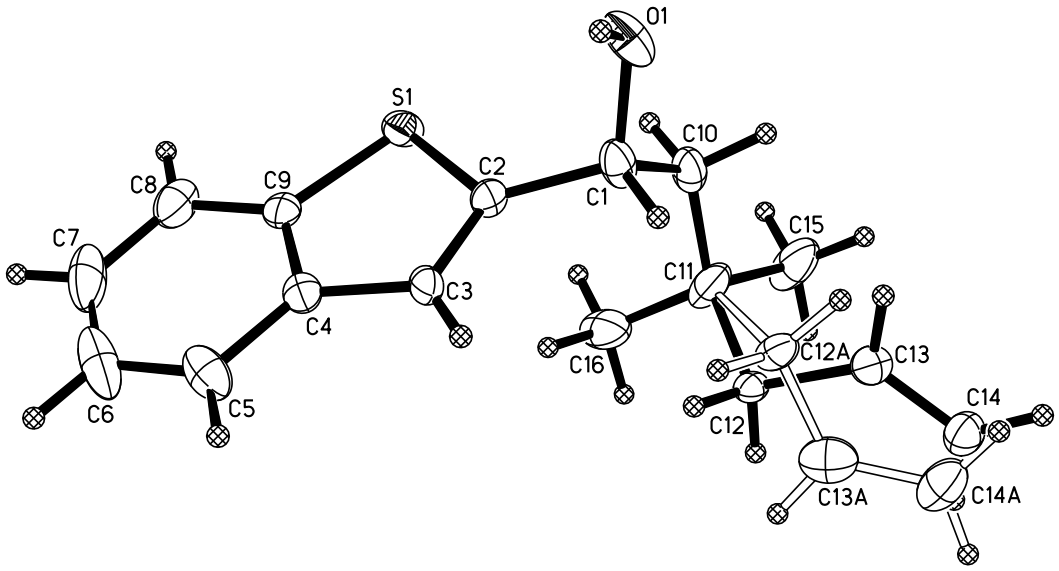


Table 1. Crystal data and structure refinement for erj15.

Identification code	erj15 (Mikhail Konev)	
Empirical formula	C_{16} H_{20} O S	
Formula weight	260.38	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Trigonal	
Space group	$P3_1$	
Unit cell dimensions	a = 13.4769(8) Å	□= 90°.
	b = 13.4769(8) Å	□= 90°.
	c = 6.8167(4) Å	□ = 120°.
Volume	1072.22(14) $Å^3$	
Z	3	
Density (calculated)	1.210 Mg/m^3	
Absorption coefficient	0.213 mm^{-1}	
F(000)	420	
Crystal color	colorless	
Crystal size	0.329 x 0.120 x 0.062 mm^3	

Theta range for data collection	1.745 to 28.670°
Index ranges	$-18 \le h \le 17$, $-17 \le k \le 17$, $-9 \le l \le 9$
Reflections collected	12748
Independent reflections	3419 [R(int) = 0.0230]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Numerical
Max. and min. transmission	1.0000 and 0.9139
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3419 / 2 / 196
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I) = 3258 data]	R1 = 0.0383, wR2 = 0.0935
R indices (all data, 0.74Å)	R1 = 0.0410, wR2 = 0.0960
Absolute structure parameter	0.02(2)
Largest diff. peak and hole	0.436 and -0.314 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for erj15. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	7101(1)	-1544(1)	2156(1)	24(1)
O(1)	9767(2)	457(2)	1758(3)	31(1)
C(1)	8870(2)	712(2)	1372(4)	26(1)
C(2)	7821(2)	-343(2)	652(4)	18(1)
C(3)	7341(2)	-502(2)	-1192(4)	21(1)
C(4)	6330(2)	-1637(2)	-1378(4)	20(1)
C(5)	5595(3)	-2117(3)	-2987(4)	31(1)
C(6)	4679(3)	-3219(3)	-2855(6)	43(1)
C(7)	4482(3)	-3858(3)	-1157(7)	44(1)
C(8)	5184(3)	-3414(3)	460(5)	33(1)
C(9)	6112(2)	-2292(2)	331(4)	21(1)
C(10)	8751(3)	1254(2)	3270(4)	27(1)
C(11)	7978(4)	1797(3)	3275(5)	39(1)
C(12)	8083(5)	2583(4)	1603(7)	17(1)
C(13)	9261(4)	3618(4)	1533(6)	22(1)
C(14)	9469(6)	4683(5)	1533(8)	26(1)
C(12A)	8673(9)	2719(7)	1334(11)	23(2)
C(13A)	8261(9)	3546(8)	1236(13)	36(2)
C(14A)	8926(12)	4666(9)	1432(15)	37(2)
C(15)	8229(4)	2481(3)	5197(5)	50(1)
C(16)	6711(3)	896(3)	3229(6)	41(1)

Table 3. Bond lengths [Å] and angles [°] for erj15.

S(1)-C(9)	1.731(3)
S(1)-C(2)	1.745(3)
O(1)-C(1)	1.438(4)
C(1)-C(2)	1.500(4)
C(1)-C(10)	1.533(4)
C(2)-C(3)	1.381(4)
C(3)-C(4)	1.459(4)
C(4)-C(5)	1.401(4)
C(4)-C(9)	1.401(4)
C(5)-C(6)	1.380(5)
C(6)-C(7)	1.387(6)
C(7)-C(8)	1.379(5)
C(8)-C(9)	1.403(4)
C(10)-C(11)	1.544(5)
C(11)-C(12)	1.513(5)
C(11)-C(16)	1.522(6)
C(11)-C(15)	1.539(4)
C(11)-C(12A)	1.734(9)
C(12)-C(13)	1.502(6)
C(13)-C(14)	1.318(7)
C(12A)-C(13A)	1.475(12)
C(13A)-C(14A)	1.322(15)
C(9)-S(1)-C(2)	91.55(13)
O(1)-C(1)-C(2)	109.8(2)
O(1)-C(1)-C(10)	105.1(2)
C(2)-C(1)-C(10)	117.0(2)
C(3)-C(2)-C(1)	126.3(2)
C(3)-C(2)-S(1)	113.0(2)
C(1)-C(2)-S(1)	120.71(19)
C(2)-C(3)-C(4)	111.2(2)
C(5)-C(4)-C(9)	118.9(3)
C(5)-C(4)-C(3)	128.7(3)
C(9)-C(4)-C(3)	112.3(2)

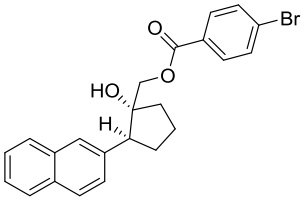
C(6)-C(5)-C(4)	119.4(3)
C(5)-C(6)-C(7)	120.9(3)
C(8)-C(7)-C(6)	121.4(3)
C(7)-C(8)-C(9)	117.8(3)
C(4)-C(9)-C(8)	121.6(3)
C(4)-C(9)-S(1)	111.9(2)
C(8)-C(9)-S(1)	126.5(2)
C(1)-C(10)-C(11)	118.9(2)
C(12)-C(11)-C(16)	102.6(3)
C(12)-C(11)-C(15)	107.9(3)
C(16)-C(11)-C(15)	108.5(3)
C(12)-C(11)-C(10)	118.7(3)
C(16)-C(11)-C(10)	112.0(2)
C(15)-C(11)-C(10)	106.7(3)
C(16)-C(11)-C(12A)	123.0(4)
C(15)-C(11)-C(12A)	110.0(4)
C(10)-C(11)-C(12A)	95.0(4)
C(13)-C(12)-C(11)	111.1(4)
C(14)-C(13)-C(12)	124.2(5)
C(13A)-C(12A)-C(11)	107.6(7)
C(14A)-C(13A)-C(12A)	124.2(10)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj15. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	18(1)	22(1)	27(1)	8(1)	-1(1)	7(1)
O(1)	20(1)	44(1)	19(1)	0(1)	-1(1)	8(1)
C(1)	25(1)	20(1)	19(1)	3(1)	-3(1)	2(1)
C(2)	19(1)	16(1)	16(1)	5(1)	2(1)	6(1)
C(3)	20(1)	19(1)	27(1)	-8(1)	-6(1)	11(1)
C(4)	18(1)	22(1)	24(1)	-5(1)	1(1)	12(1)
C(5)	24(1)	44(2)	24(1)	-12(1)	-2(1)	17(1)
C(6)	23(2)	49(2)	44(2)	-28(2)	-2(1)	9(1)
C(7)	21(2)	27(2)	72(3)	-19(2)	1(2)	3(1)
C(8)	20(1)	21(1)	56(2)	2(1)	6(1)	9(1)
C(9)	15(1)	19(1)	30(1)	-1(1)	0(1)	11(1)
C(10)	32(2)	20(1)	21(1)	0(1)	-9(1)	6(1)
C(11)	72(3)	21(1)	26(2)	-6(1)	-20(2)	26(2)
C(12)	14(2)	17(2)	22(2)	3(2)	2(2)	9(2)
C(13)	19(2)	23(2)	23(2)	4(2)	4(2)	11(2)
C(14)	27(3)	22(2)	26(2)	3(2)	1(2)	11(2)
C(12A)	32(5)	22(4)	20(4)	3(3)	0(3)	17(4)
C(13A)	42(5)	47(5)	30(4)	2(4)	1(4)	29(4)
C(14A)	53(7)	27(5)	34(5)	0(3)	2(5)	23(5)
C(15)	97(3)	30(2)	30(2)	-12(1)	-24(2)	36(2)
C(16)	59(2)	44(2)	41(2)	-18(2)	-20(2)	41(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj15.

	x	y	z	U(eq)
H(1)	9960(40)	290(40)	670(40)	67(16)
H(1A)	9137	1304	312	31
H(3A)	7630	55	-2208	25
H(5A)	5726	-1687	-4159	37
H(6A)	4176	-3544	-3942	52
H(7A)	3850	-4618	-1107	53
H(8A)	5044	-3854	1622	40
H(10A)	9529	1854	3672	33
H(10B)	8463	656	4299	33
H(12A)	7922	2160	347	20
H(12B)	7509	2829	1771	20
H(13A)	9897	3497	1485	26
H(14A)	8852	4833	1580	31
H(14B)	10237	5300	1486	31
H(12C)	9513	3126	1544	28
H(12D)	8501	2283	91	28
H(13B)	7467	3255	1020	44
H(14C)	9725	4985	1650	45
H(14D)	8605	5153	1354	45
H(15A)	8053	1963	6316	76
H(15B)	7753	2841	5263	76
H(15C)	9040	3074	5237	76
H(16A)	6535	363	4326	62
H(16B)	6532	473	1988	62
H(16C)	6251	1271	3345	62



X-ray Data Collection, Structure Solution and Refinement for **(*R,S*)-1.77**

CCDC 1003298 contains the supplementary crystallographic data for this structure. These data can be obtained via www.ccdc.ac.uk/conts/retrieving.html. The crystal was obtained by slow evaporation from ethyl acetate over pentane.

A colorless crystal of approximate dimensions 0.293 x 0.205 x 0.111 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (15 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model.

At convergence, wR2 = 0.0536 and Goof = 1.054 for 247 variables refined against 4644 data (0.73 Å), R1 = 0.0223 for those 4412 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

7. APEX2 Version 2014.1-1, Bruker AXS, Inc.; Madison, WI 2014.

8. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

9. Sheldrick, G. M. SADABS, Version 2012/1, Bruker AXS, Inc.; Madison, WI 2012.

10. Sheldrick, G. M. SHELXTL, Version 2014/2, Bruker AXS, Inc.; Madison, WI 2014.

11. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

12. Flack, H. D., Parsons, S., Wagner, T. Acta. Cryst., B69, 249-259, 2013.

Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

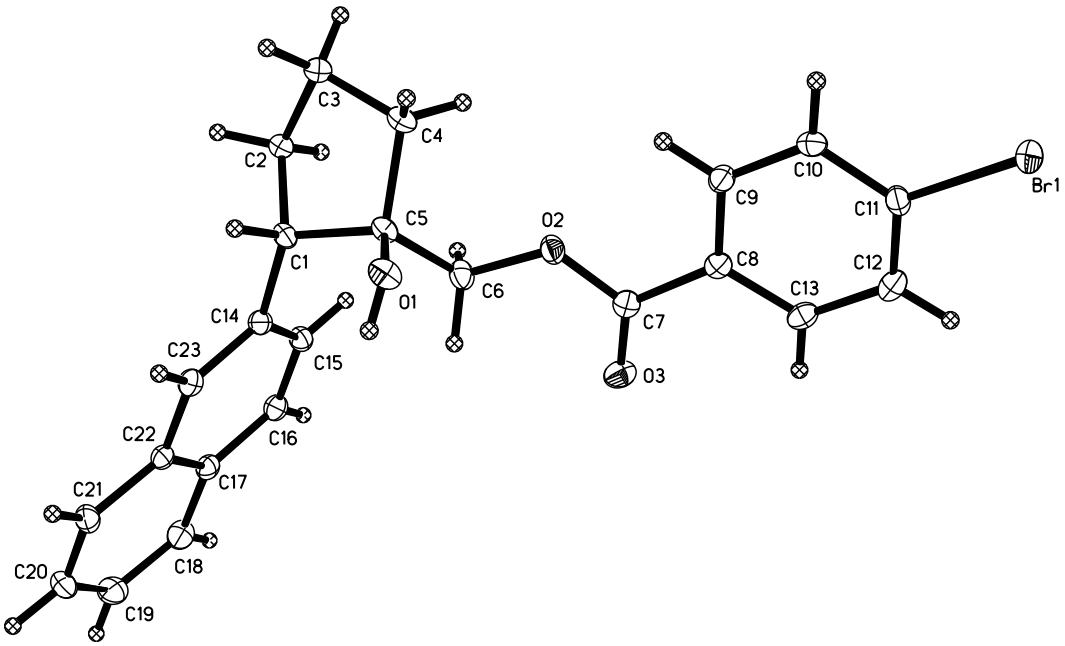


Table 1. Crystal data and structure refinement for erj16.

Identification code	erj16 (Michael Harris)	
Empirical formula	C_{23} H_{21} Br O_3	
Formula weight	425.31	
Temperature	88(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.9672(4) Å	□= 90°.
	b = 9.3042(4) Å	□= 90°.
	c = 24.7018(11) Å	□ = 90°.
Volume	1831.11(15) Å^3	
Z	4	
Density (calculated)	1.543 Mg/m^3	
Absorption coefficient	2.266 mm^{-1}	
F(000)	872	
Crystal color	colorless	
Crystal size	0.293 x 0.205 x 0.111 mm^3	

Theta range for data collection	1.649 to 29.026°
Index ranges	$-10 \le h \le 10$, $-12 \le k \le 12$, $-32 \le l \le 32$
Reflections collected	22622
Independent reflections	4644 [R(int) = 0.0251]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Numerical
Max. and min. transmission	0.8205 and 0.6133
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4644 / 0 / 247
Goodness-of-fit on F^2	1.054
Final R indices [I>2sigma(I) = 4412 data]	R1 = 0.0223, wR2 = 0.0528
R indices (all data, 0.73 Å)	R1 = 0.0245, wR2 = 0.0536
Absolute structure parameter	-0.007(2)
Largest diff. peak and hole	0.597 and -0.299 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for erj16. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	12231(1)	12759(1)	9891(1)	21(1)
O(1)	9393(2)	5010(2)	8236(1)	17(1)
O(2)	8218(2)	7849(2)	8350(1)	17(1)
O(3)	8638(2)	9449(2)	7682(1)	19(1)
C(1)	6594(3)	4163(2)	7945(1)	13(1)
C(2)	4933(3)	4160(2)	8260(1)	15(1)
C(3)	5549(3)	4243(2)	8848(1)	19(1)
C(4)	7006(3)	5317(2)	8826(1)	19(1)
C(5)	7648(3)	5352(2)	8234(1)	13(1)
C(6)	7414(3)	6842(2)	7985(1)	16(1)
C(7)	8726(3)	9114(2)	8156(1)	14(1)
C(8)	9453(3)	10031(2)	8591(1)	14(1)
C(9)	9538(3)	9552(2)	9125(1)	15(1)
C(10)	10340(3)	10367(2)	9517(1)	15(1)
C(11)	11033(3)	11682(2)	9366(1)	15(1)
C(12)	10900(2)	12208(3)	8842(1)	18(1)
C(13)	10105(3)	11377(2)	8455(1)	16(1)
C(14)	6411(3)	4202(2)	7334(1)	13(1)
C(15)	5344(3)	5226(2)	7076(1)	14(1)
C(16)	5088(3)	5208(2)	6529(1)	15(1)
C(17)	5859(3)	4150(2)	6198(1)	14(1)
C(18)	5577(3)	4084(3)	5629(1)	19(1)
C(19)	6305(3)	3026(3)	5325(1)	22(1)
C(20)	7352(3)	1985(2)	5571(1)	20(1)
C(21)	7666(3)	2027(2)	6119(1)	17(1)
C(22)	6920(2)	3113(2)	6443(1)	13(1)
C(23)	7176(3)	3179(2)	7016(1)	14(1)

Table 3. Bond lengths [Å] and angles [°] for erj16.

Br(1)-C(11)	1.895(2)
O(1)-C(5)	1.426(3)
O(2)-C(7)	1.334(3)
O(2)-C(6)	1.450(2)
O(3)-C(7)	1.212(3)
C(1)-C(14)	1.515(3)
C(1)-C(2)	1.535(3)
C(1)-C(5)	1.562(3)
C(2)-C(3)	1.535(3)
C(3)-C(4)	1.533(3)
C(4)-C(5)	1.551(3)
C(5)-C(6)	1.527(3)
C(7)-C(8)	1.490(3)
C(8)-C(9)	1.394(3)
C(8)-C(13)	1.397(3)
C(9)-C(10)	1.387(3)
C(10)-C(11)	1.393(3)
C(11)-C(12)	1.388(3)
C(12)-C(13)	1.384(3)
C(14)-C(23)	1.377(3)
C(14)-C(15)	1.427(3)
C(15)-C(16)	1.367(3)
C(16)-C(17)	1.420(3)
C(17)-C(22)	1.419(3)
C(17)-C(18)	1.425(3)
C(18)-C(19)	1.366(3)
C(19)-C(20)	1.415(3)
C(20)-C(21)	1.375(3)
C(21)-C(22)	1.420(3)
C(22)-C(23)	1.430(3)
C(7)-O(2)-C(6)	118.63(16)
C(14)-C(1)-C(2)	114.94(18)
C(14)-C(1)-C(5)	119.27(17)

C(2)-C(1)-C(5)	103.50(16)
C(1)-C(2)-C(3)	101.76(18)
C(4)-C(3)-C(2)	104.03(17)
C(3)-C(4)-C(5)	107.22(17)
O(1)-C(5)-C(6)	108.87(17)
O(1)-C(5)-C(4)	108.19(17)
C(6)-C(5)-C(4)	110.96(17)
O(1)-C(5)-C(1)	111.64(16)
C(6)-C(5)-C(1)	113.18(16)
C(4)-C(5)-C(1)	103.83(16)
O(2)-C(6)-C(5)	106.40(16)
O(3)-C(7)-O(2)	123.9(2)
O(3)-C(7)-C(8)	124.8(2)
O(2)-C(7)-C(8)	111.27(18)
C(9)-C(8)-C(13)	119.7(2)
C(9)-C(8)-C(7)	121.2(2)
C(13)-C(8)-C(7)	119.0(2)
C(10)-C(9)-C(8)	120.6(2)
C(9)-C(10)-C(11)	118.4(2)
C(12)-C(11)-C(10)	121.9(2)
C(12)-C(11)-Br(1)	119.28(17)
C(10)-C(11)-Br(1)	118.77(17)
C(13)-C(12)-C(11)	118.9(2)
C(12)-C(13)-C(8)	120.3(2)
C(23)-C(14)-C(15)	117.97(19)
C(23)-C(14)-C(1)	120.63(19)
C(15)-C(14)-C(1)	121.22(19)
C(16)-C(15)-C(14)	121.6(2)
C(15)-C(16)-C(17)	120.9(2)
C(22)-C(17)-C(16)	118.89(19)
C(22)-C(17)-C(18)	119.1(2)
C(16)-C(17)-C(18)	122.0(2)
C(19)-C(18)-C(17)	120.3(2)
C(18)-C(19)-C(20)	120.5(2)
C(21)-C(20)-C(19)	120.7(2)
C(20)-C(21)-C(22)	119.9(2)

C(17)-C(22)-C(21)	119.47(19)
C(17)-C(22)-C(23)	118.60(19)
C(21)-C(22)-C(23)	121.92(19)
C(14)-C(23)-C(22)	122.1(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj16. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	21(1)	21(1)	20(1)	-4(1)	1(1)	-8(1)
O(1)	14(1)	22(1)	15(1)	-3(1)	-2(1)	3(1)
O(2)	24(1)	13(1)	13(1)	-1(1)	-2(1)	-2(1)
O(3)	16(1)	25(1)	16(1)	4(1)	-2(1)	-4(1)
C(1)	15(1)	12(1)	12(1)	-2(1)	0(1)	2(1)
C(2)	18(1)	15(1)	12(1)	-1(1)	2(1)	-2(1)
C(3)	28(1)	16(1)	13(1)	0(1)	3(1)	-3(1)
C(4)	25(1)	21(1)	12(1)	-3(1)	1(1)	-3(1)
C(5)	13(1)	15(1)	11(1)	-2(1)	-1(1)	1(1)
C(6)	20(1)	15(1)	15(1)	-2(1)	-4(1)	-1(1)
C(7)	10(1)	15(1)	18(1)	0(1)	0(1)	2(1)
C(8)	11(1)	13(1)	16(1)	1(1)	2(1)	2(1)
C(9)	16(1)	13(1)	17(1)	0(1)	3(1)	-1(1)
C(10)	15(1)	16(1)	13(1)	1(1)	3(1)	1(1)
C(11)	12(1)	15(1)	18(1)	-5(1)	1(1)	-1(1)
C(12)	15(1)	14(1)	24(1)	2(1)	2(1)	-1(1)
C(13)	14(1)	16(1)	17(1)	4(1)	1(1)	1(1)
C(14)	13(1)	15(1)	12(1)	-1(1)	-1(1)	-2(1)
C(15)	14(1)	14(1)	16(1)	-2(1)	1(1)	0(1)
C(16)	14(1)	14(1)	17(1)	1(1)	-1(1)	-1(1)
C(17)	14(1)	14(1)	16(1)	0(1)	-1(1)	-3(1)
C(18)	21(1)	19(1)	17(1)	2(1)	-4(1)	-3(1)
C(19)	27(1)	26(1)	13(1)	-2(1)	-1(1)	-6(1)
C(20)	22(1)	19(1)	18(1)	-6(1)	4(1)	-2(1)
C(21)	15(1)	15(1)	20(1)	-4(1)	1(1)	-1(1)
C(22)	11(1)	13(1)	16(1)	-1(1)	1(1)	-3(1)
C(23)	12(1)	14(1)	16(1)	0(1)	-1(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj16.

	x	y	z	U(eq)
H(1A)	7156	3230	8031	16
H(2A)	4288	3268	8193	18
H(2B)	4231	5001	8165	18
H(3A)	4647	4591	9091	23
H(3B)	5937	3292	8976	23
H(4A)	7917	5012	9074	23
H(4B)	6619	6284	8938	23
H(6A)	7940	6885	7622	20
H(6B)	6205	7068	7948	20
H(9A)	9043	8659	9221	18
H(10A)	10414	10037	9881	18
H(12A)	11346	13124	8752	21
H(13A)	10003	11724	8095	19
H(15A)	4799	5935	7289	17
H(16A)	4385	5914	6368	18
H(18A)	4879	4779	5459	23
H(19A)	6107	2988	4946	26
H(20A)	7842	1250	5357	24
H(21A)	8382	1331	6279	20
H(23A)	7897	2495	7182	17
H(1)	9750(40)	4920(30)	7940(12)	21

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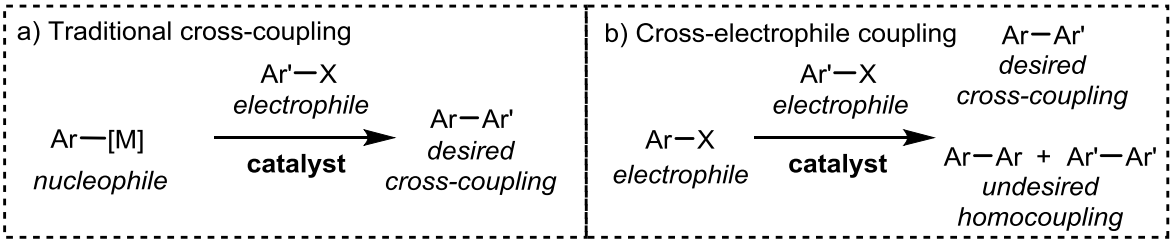
Chapter 2

Intra- and Intermolecular Nickel-Catalyzed Reductive Cross-Electrophile Coupling Reactions of Benzylic Esters with Aryl Halides

2.1 Introduction

Transition metal-catalyzed cross-coupling reactions have become an essential tool in the synthetic chemist's repertoire, allowing them to join two molecules together through the strategic placement of reactive functionalities. Such classic transformations are designed to be inherently selective by employing reaction partners that present dramatically different reactivity profiles. This is true in traditional cross-coupling reactions where selectivity is a direct result of one molecule being recruited to the metal catalyst through transmetallation by a nucleophilic partner and the other by oxidative addition of an electrophile.[1,2] In contrast, the selective coupling of two electrophiles has been a particularly interesting challenge, in part because such a reaction would avoid the need to preform a nucleophilic organometallic reagent.[3] However, both electrophiles can compete for oxidative addition with the catalyst, resulting in mixtures of cross- and homocoupling products.

Scheme 2.1. Selectivity in a) traditional and b) cross-electrophile coupling reactions



[1] A portion of this chapter was originally published in journal format: Konev, M. O.; Hanna, L. E.; Jarvo, E. R. *Angew. Chem. Int. Ed*. **2016**, *55*, 6730.
[2] Johansson Seechurn, C. C. C.; Kitching, M. O.; Colacot, T. J.; Snieckus, V. *Angew. Chem. Int. Ed.* **2012**, *51*, 5062.
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The foundation of nickel-mediated cross-electrophile coupling was first reported by Semmelhack for the Ullman-type coupling of two aryl electrophiles with a $Ni(cod)_2$ catalyst.[4] The first example of an in situ reductive coupling, mediated by zinc metal, was reported by Kende for the homocoupling of sp^2 electrophiles.[5] It was only recently that a resurgence began in this field with Weix developing the coupling of aryl and alkyl electrophiles that favored cross-selectivity over homocoupling.[6] In 2013, Weix investigated the mechanism of the cross-coupling of aryl iodides and alkyl iodides, giving further insight into the mechanistic aspects of sp^3 electrophile coupling reactions. [7] These findings proved crucial for the design of catalysts that control stereochemistry at the alkyl center. Weix proposed that the reaction begins with oxidative addition of a nickel(0) catalyst to an aryl halide bond, shown to occur selectively over the oxidative addition into alkyl halide bond. The arylnickel(II) intermediate then combines with an alkyl radical to form an arylalkylnickel(III) species which can undergo reductive elimination to form desired product. The resulting nickel(I) intermediate can further react with an alkyl iodide to generate an alkyl radical and NiI_2 which is then reduced by the stoichiometric reductant, regenerating the active nickel(0) catalyst.

Recently, several reports have shown that nickel complexes catalyze enantioselective cross-electrophile coupling reactions of a variety of sp^2 and sp^3 electrophiles, displaying control of stereochemical information using chiral ligands. Reisman and co-workers have exploited this reactivity by demonstrating several examples of enantioselective reductive

[4] Semmelhack, M. F.; Helquist, P. M.; Jones, L. D.; *J. Am. Chem. Soc*. **1971**, *93*, 5908.
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couplings including using benzylic halides with vinyl bromides, acyl chlorides, or aryl halides.[8] These reactions appear to follow a similar mechanism as proposed by Weix, where the enantio-determining reductive elimination of a common nickel(III) intermediate forms the desired product (Scheme 2.2 a).[9] Most recently, Reisman and co-workers have reported an enantioselective decarboxylative reductive coupling of vinyl bromides with benzylic NHP esters (Scheme 2.2 b).[10] Weix further expanded the scope of these reaction to include primary benzylic mesylates as coupling partners (Scheme 2.2 c).[11] The Doyle and Sigman groups recently reported an investigation an enantioselective reductive coupling of styrenyl aziridines (Scheme 2.2 d).[12] To date, there have been no reports of a complimentary enantiospecific reductive coupling reaction of aryl halides and alkyl electrophiles where stereochemical information is transferred from starting materials to products. Herein is described the discovery and development of a nickel-catalyzed stereospecific reductive coupling of aryl halides and benzylic esters.

[8] (a) Cherney, A. H.; Kadunce, N. T.; Reisman, S. E. *J. Am. Chem. Soc.* **2013**, *135*, 7442. (b) Cherney, A. H.; Reisman, S. E. *J. Am. Chem. Soc.* **2014**, *136*, 14365. (c) Poremba, K. E.; Kadunce, N. T.; Suzuki, N.; Cherney, A. H.; Reisman, S. E. *J. Am. Chem. Soc*. **2017**, *139*, 5684. (d) Kadunce, N. T.; Reisman, S. E. *J. Am. Chem. Soc.* **2015**, *137*, 10480.
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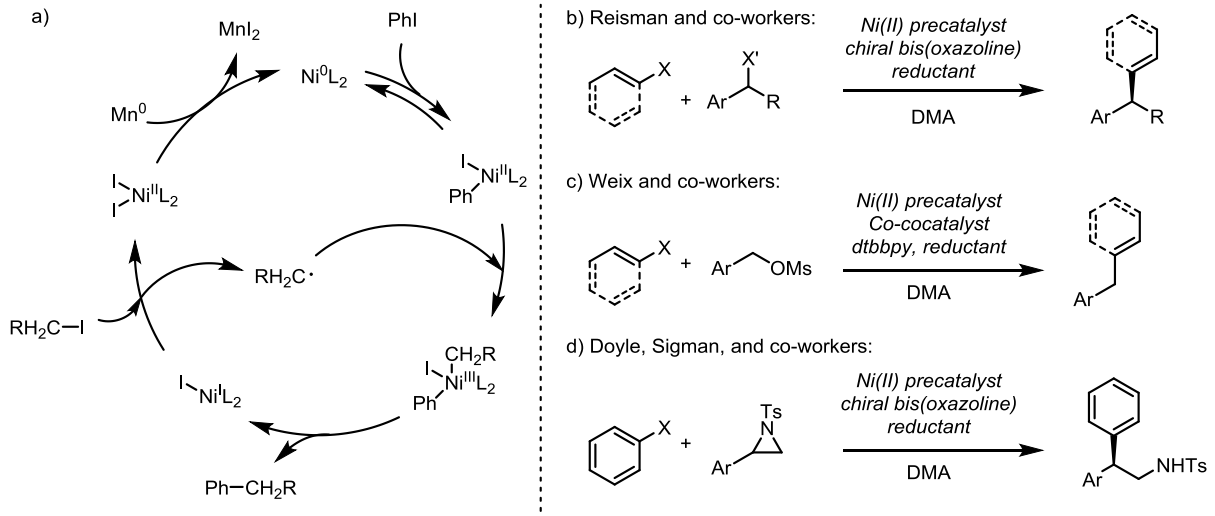
Scheme 2.2. a) Catalytic cycle for the cross-electrophile coupling of aryl iodides with alkyl iodides. Recent examples of cross-electrophile coupling reactions by b) Reisman, c) Weix, and d) Doyle and Sigman



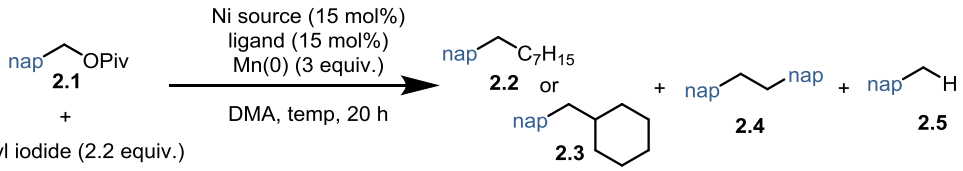
2.2 Development of an Intermolecular Cross-Electrophile Coupling Reaction of Benzylic Esters with Organic Halides

Following the mechanistic hypothesis proposed by Weix, our coupling partners were initially chosen on the premise that one substrate would undergo oxidative addition by a two-electron process, and the second by a radical chain mechanism. To satisfy these requirements, we envisioned using a benzylic ester as the initial two-electron oxidative addition partner, resembling substrates the Jarvo group has previously used for stereospecific cross-couplings,[13] with an alkyl iodide as the second electrophile. Initial conditions were inspired by Weix and co-workers, utilizing commercially available $NiCl_2$ precatalysts, Mn^0 as the stoichiometric reductant, and DMA as the solvent. Using a variety of ligands, the primary alkyl iodide was found mostly ineffective in the desired transformation, providing low yields of the coupled product (Table 2.1, entry 2). When the reaction

[13] Tollefson, E. J.; Hanna, L. E.; Jarvo, E. R. *Acc. Chem. Res.* **2015**, *48*, 2344.

conditions allowed for the consumption of the benzylic ester, either reduction at the benzylic center or homocoupling was observed. Using a secondary alkyl iodide provided up to 13% yield of product using dppf as a ligand (entry 4), again favoring either reduction or dimerization of the benzylic electrophile. The increased yield of desired product using secondary alkyl iodides compared to primary alkyl iodides is attributed to the generation of a more stable alkyl radical. Although alkyl iodides as coupling partners gave encouraging preliminary results, simultaneous investigation of other electrophilic coupling partners proved to be more fruitful.

Table 2.1. Investigation of reductive coupling of benzylic esters with alkyl iodides

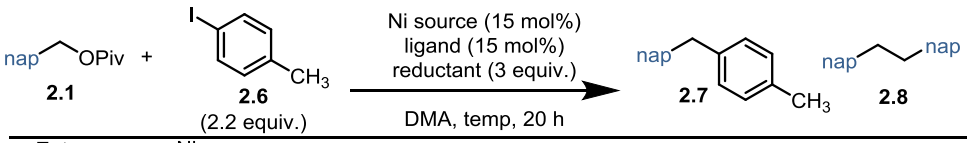


Entry	Ni source	ligand	alkyl iodide	temp (°C)	recovered **2.1** (%)	yield **2.2** or **2.3** (%)	yield **2.4** (%)	yield **2.5** (%)
1	$NiCl_2(PCy_3)_2$	PCy_3	I-C_7H_{15}	50	95	-	-	-
2	$NiCl_2$(dppf)	dppf	I-C_7H_{15}	50	-	7	59	36
3	$NiCl_2$(dppe)	dppe	I-C_7H_{15}	50	83	-	-	16
4	$NiCl_2$-glyme	dppf	I-Cy	50	-	13	62	29
5	$NiCl_2$-glyme	dppf	I-Cy	rt	-	7	67	11
6	$NiCl_2$-glyme	bipy	I-Cy	rt	33	-	5	45
7	$NiCl_2$-glyme	batho	I-Cy	rt	57	-	-	17

We hypothesized that replacing the alkyl iodide with an aryl iodide could lead to the formation of diarylmethanes, common moieties found in biologically active molecules. We chose to substitute the iodoalkane with iodotoluene **2.6** for coupling with benzylic pivalate **2.1**. Following an extensive investigation of ligands, a preformed $NiCl_2$(dppf) precatalyst was found to produce desired product in 66% yield, with dimerization of the benzylic electrophile as the major side product. Upon further optimization of the reaction conditions,

we found that using Zn^0 as the reductant and lowering reaction temperature increased the yield of the desired diarylmethane to 98% and suppressed dimerization completely (Table 2.2, entry 5).

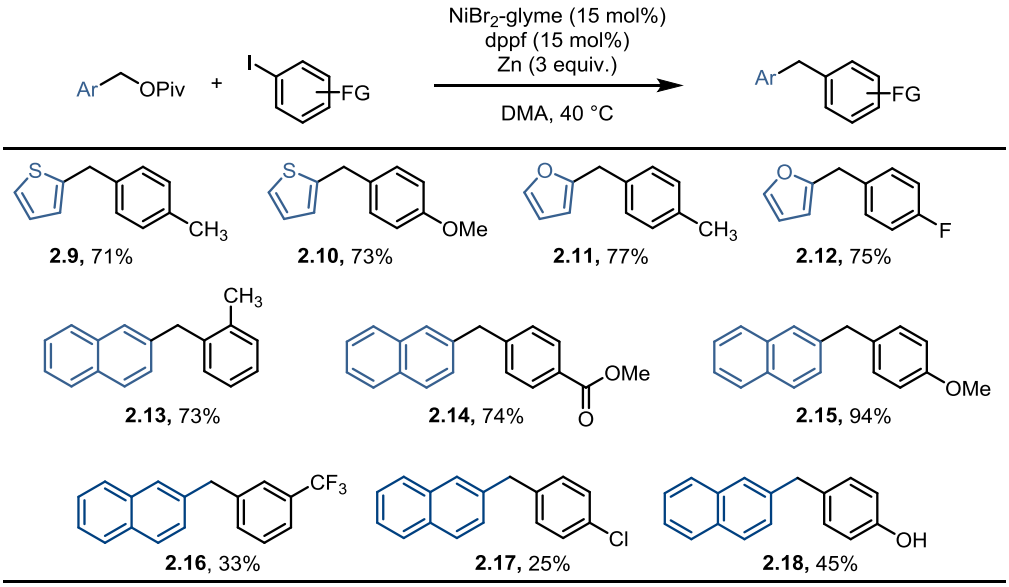
Table 2.2. Investigation of the reductive coupling of benzylic esters with iodotoluene



Entry	Ni source	ligand	reductant	temp (°C)	recovered **2.1** (%)	yield **2.7** (%)	yield **2.8** (%)
1	$NiCl_2(PCy_3)_2$	PCy_3	Mn	50	99	-	-
2	$NiCl_2$(dppe)	dppe	Mn	50	100	-	-
3	$NiCl_2$(dppf)	dppf	Mn	50	-	66	30
4	$NiCl_2$-glyme	dppf	Mn	rt	-	91	7
5	**$NiBr_2$-glyme**	**dppf**	**Zn**	**rt**	**-**	**98**	**-**

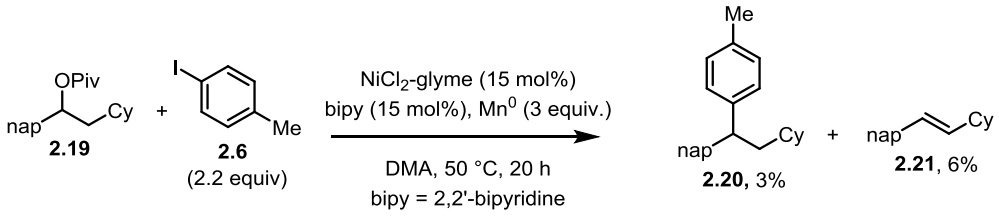
With suitable reaction conditions identified, we examined the scope of the intermolecular cross-electrophile coupling reaction (Table 2.3). With respect to the benzylic electrophile, coupling of pivalate esters of 2-naphthylmethanol proceeded at room temperature, whereas good yields of the analogous products of thiophene **2.9, 2.10** and furan **2.11, 2.12** required elevated temperatures for complete conversion. Simple benzylic esters, (e.g. benzyl pivalate), were unreactive under the optimized conditions even upon heating of the reaction mixture. In addition to iodotoluene, iodobenzene derivatives containing aryl ether, methyl benzoate, and fluoro functional groups were well tolerated. Additionally, the desired reaction proceeds with as little as 1.1 equivalents of the aryl iodide reagent. An excess is typically required in other methods, where adjusting stoichiometry improves selectivity for the cross-coupled product.

Table 2.3. Scope of intermolecular cross-electrophile coupling reaction



After examining the scope of the reaction with primary benzylic esters, we employed the optimized conditions with secondary benzylic esters and aryl iodides to explore the stereochemical fidelity of the reaction. Direct application of the previous reaction conditions yielded a small amount of β-hydride elimination side-product **2.21**, but no desired diarylalkane **2.20**. After an extensive investigation of ligands, only using 2,2'-bipyridine as a ligand was found to yield desired product, albeit in only a trace amounts with side-product **2.21** (Scheme 2.3). Given that preliminary efforts towards an intermolecular reaction were unsuccessful, we reasoned that an intramolecular reaction may more readily provide the desired reactivity through increased effective molarity of the electrophilic functionalities. We envisioned the intramolecular cross-electrophile coupling reaction between these functionalities would provide the straightforward synthesis of 1-arylindanes and tetralins, common motifs in natural products and pharmaceutical agents.

***Scheme 2.3*.** Preliminary result for intermolecular coupling of a secondary benzylic ester

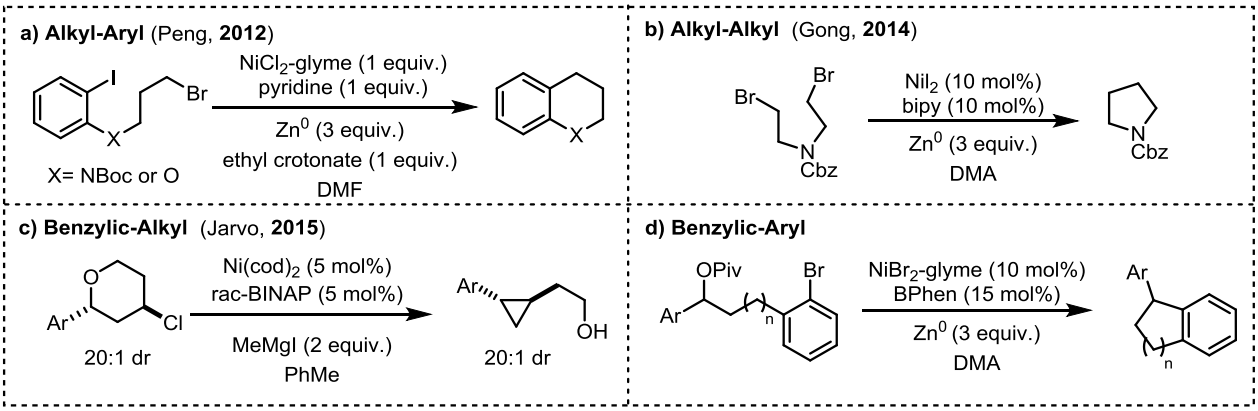


2.3 Development of an Intramolecular Cross-Electrophile Coupling Reaction of Benzylic Esters with Aryl Bromides.

While intermolecular reductive coupling reactions have undergone rapid development in recent years, few intramolecular variants have been reported. The Peng group disclosed a stoichiometric nickel-mediated reductive coupling reaction to access nitrogen- and oxygen-containing heterocycles (Scheme 2.4 a).[14] In 2014, Gong and co-workers reported a catalytic intramolecular cyclization of dihaloalkanes to access 5- and 6-membered rings (Scheme 2.4 b).[15] Recently, much interest has focused on the use of C–O electrophiles in reductive coupling reactions,[16] and our laboratory has reported a reductive ring-contraction of 4-chlorotetrahydropyrans to generate cyclopropanes (Scheme 2.4 c).[17] To further expand the scope of intramolecular cross-electrophile coupling reactions, we targeted cyclization reactions of benzylic esters with aryl halides to afford valuable indanes and tetralins (Scheme 2.4 d).

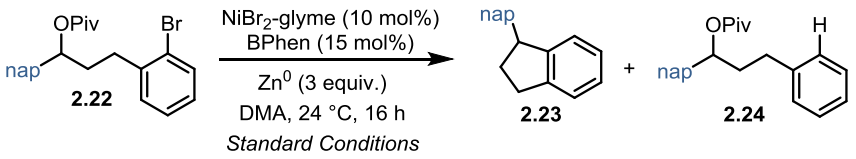
[14] Yan, C. S.; Peng, Y.; Xu, X. B.; Wang, Y. W. *Chem. Eur. J.* **2012**, *18*, 6039.
[15] Xue, W.; Xu, H.; Liang, Z.; Qian, Q.; Gong, H. *Org. Lett.* **2014**, *16*, 4984.
[16] Gu, J.; Wang, X.; Xue, W.; Gong, H. *Org. Chem. Front.* **2015**, *2*, 1411. (b) Moragas, T.; Correa, A.; Martin, R. *Chem. Eur. J*. **2014**, *20*, 8242.
[17] Tollefson, E. J.; Erickson, L. W.; Jarvo, E. R. *J. Am. Chem. Soc*. **2015**, *137*, 9760.

Scheme 2.4. Nickel-catalyzed intramolecular cross-electrophile coupling reactions



We designed the secondary benzylic pivalate **2.22** as a model substrate, based on our previous work in cross-coupling reactions of benzylic electrophiles. These substrates are easily prepared by lithiation of an arene and addition into the corresponding bromophenyl aldehyde. Alternatively, they can be prepared through the reduction and pivalylation of chalcones. Reaction of **2.22** in the presence of catalytic $NiBr_2$-glyme, BPhen, and Zn^0 provided the desired product **2.23** in excellent yield with negligible yields of hydrodehalogenation (Table 2.4, entry 1). Less sterically encumbered esters, such as acetate, provided lower conversion under the reaction conditions (entry 2). Alternative reducing agents such as Mn^0 also provided lower yields (entry 3). Utilizing the previously successful phosphine ligand dppf or other aromatic nitrogen-containing ligands, such as bipy or pybox, resulted in a dramatic decrease in product formation (entries 4–7). In the absence of a ligand or a nickel catalyst, the desired cyclization does not occur (entries 8 and 9). Additives known to promote reactivity in other reductive cross-electrophile coupling reactions were also examined; however, the addition of either pyridine or NaI favored hydrodehalogenation (entries 10 and 11).

Table 2.4. Optimization of reaction conditions for the intramolecular reductive coupling



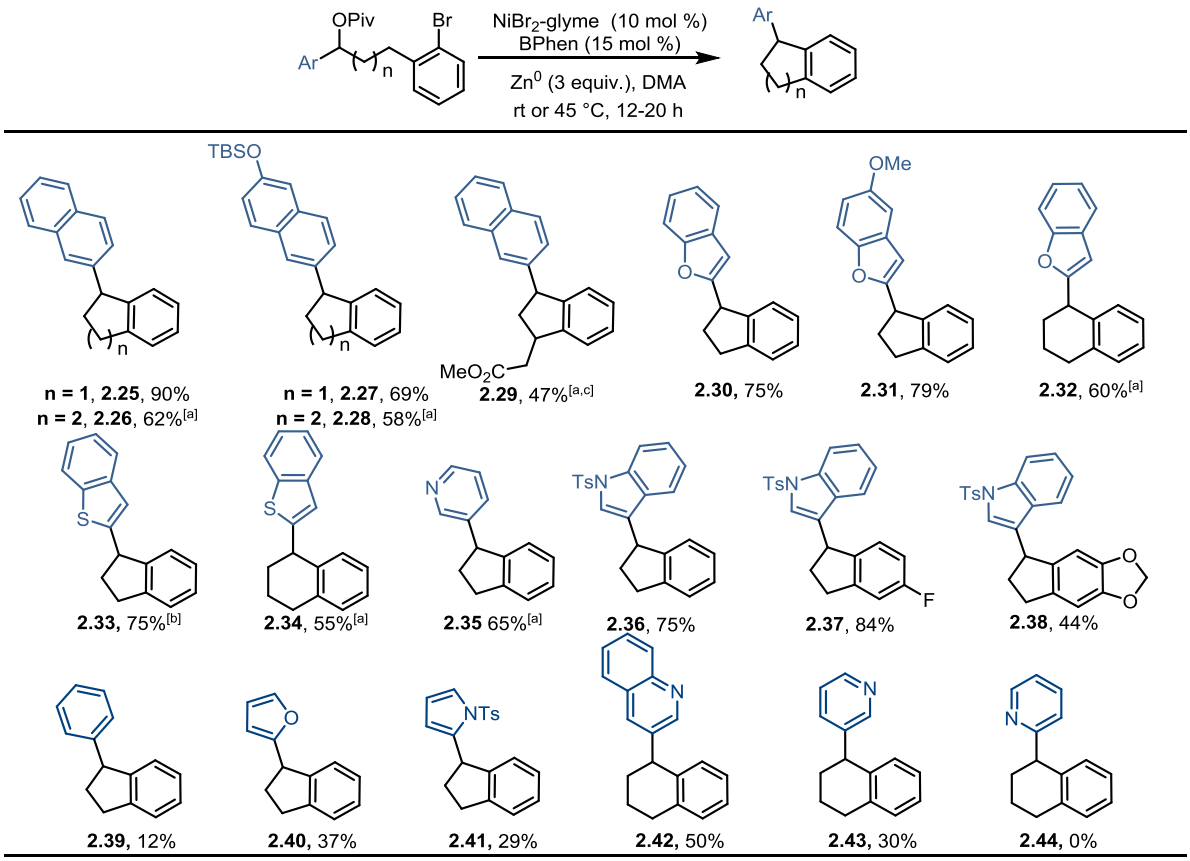
Entry	Variation From Standard Conditions	recovered **2.22** (%)	yield **2.23** (%)	yield **2.24** (%)
1	none	<2	90	<2
2	Ac instead of Piv	59	9	13
3	Mn instead of Zn	80	<2	<2
4	dppf instead of BPhen	26	25	49
5	bipy instead of BPhen	20	42	25
6	terpyridine instead of BPhen	86	<2	3
7	pybox instead of BPhen	98	<2	<2
8	no ligand	100	<2	<2
9	no $NiBr_2$-glyme	93	<2	<2
10	pyridine (40 mol%)	25	4	61
11	NaI (25 mol%)	<2	25	68
12	$NiCl_2$-glyme instead of $NiBr_2$-glyme	12	79	4
13	DMF instead of DMA	<2	44	35

Having established reaction conditions for the cyclization of the model substrate **2.22**, we set out to investigate the scope of the transformation (Table 2.5). Cyclization of a series of naphthylic esters provides the tetralins **2.26** and **2.28**, which correspond to the core of bicunningines A and B,[18] and the tetracyclic indanes **2.25**, **2.27**, and **2.29**. Benzofuran and benzothiophene moieties were well-tolerated, providing good yields for both indanes and tetralins (**2.30–2.34**). Additionally, substrates containing *N*-heterocycles were found to undergo the desired cyclization. The pyridine substituted indane **2.33** can be synthesized in 65% yield and *N*-tosylindoles **2.36–2.38** were obtained in good yields. Substrates containing methoxy, fluoro, silyl, and ester substituents were also well-tolerated under the reaction conditions. Substrates containing simple aromatic systems such as **2.39**, **2.40**, and **2.41**

[18] Hou, X. F.; Yao, S.; Mandi, A.; Kurtan, T.; Tang, C. P.; Ke, C. Q.; Li, X. Q.; Ye, Y. *Org. Lett.* **2012**, *14*, 460.

afforded low yields of product, primarily leading to decomposition of the starting material to undetermined by-products. 1-Pyridyl tetralins could also be obtained in modest yields; however, when 2-substituted pyridine **2.44** was subjected to reaction conditions only reduction of the benzylic pivalate was observed. Notably, these cyclization reactions proceed smoothly without the aid of a Thorpe–Ingold effect.[19]

Table 2.5. Scope of the intramolecular cross-electrophile coupling



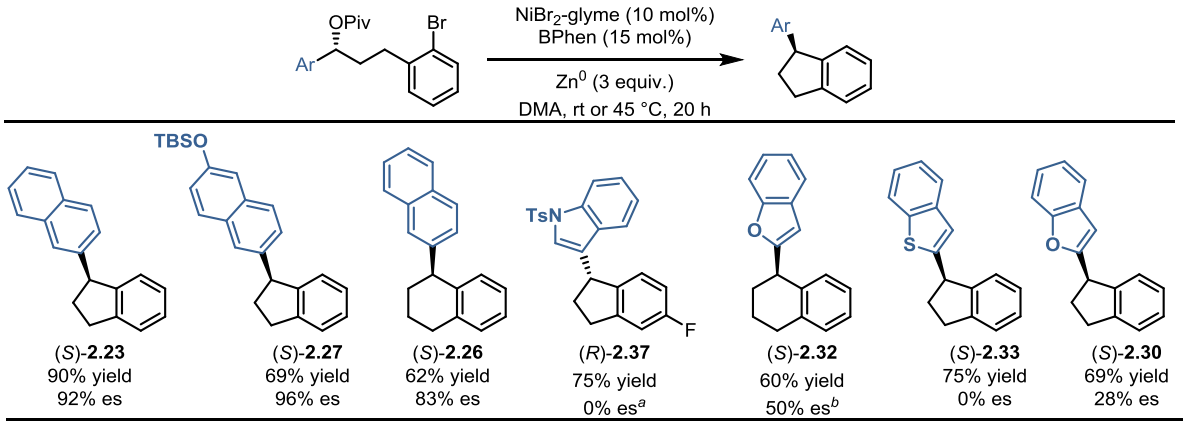
[a]Reaction run with 15 mol% $NiBr_2$-glyme and 45 °C. [b]Reaction run at 45 °C. [c]Both sm and product are 1:1 dr.

[19] Beesley, R. M.; Ingold, C. K.; Thorpe, J. F. *J. Chem. Soc. Trans.* **1915**, 107, 1080.

2.4 Scope of Enantiospecific Intramolecular Reductive Coupling

Finally, we sought to determine whether the intramolecular cyclization could proceed in an enantiospecific fashion. While several examples of stereoconvergent reductive coupling reactions have been reported (vide supra), to the best of our knowledge, there is only one example of an enantiospecific cross-electrophile coupling reaction.[16] Subjecting **(*R*)-2.22** to the optimized conditions afforded **(*S*)-2.23** in 90% yield in 88% enantiomeric excess with 92% enantiospecificity (Table 2.6). Indane **2.27** was also formed with high enantiospecificy, as was tetralin **2.26**. Notably, all three of these substrates contain the naphthylic ester moiety which we hypothesize is prone to rapid and stereospecific oxidative addition reactions. Substrates wherein the ester is activated by a heterocycle such as benzofuran, benzothiophene, or indole provided lower enantiospecificity. This change in stereoselectivity likely correlates to a change in the reaction mechanism. Investigation of the mechanistic details is ongoing.

Table 2.6. Nickel-catalyzed stereospecific reductive cyclization



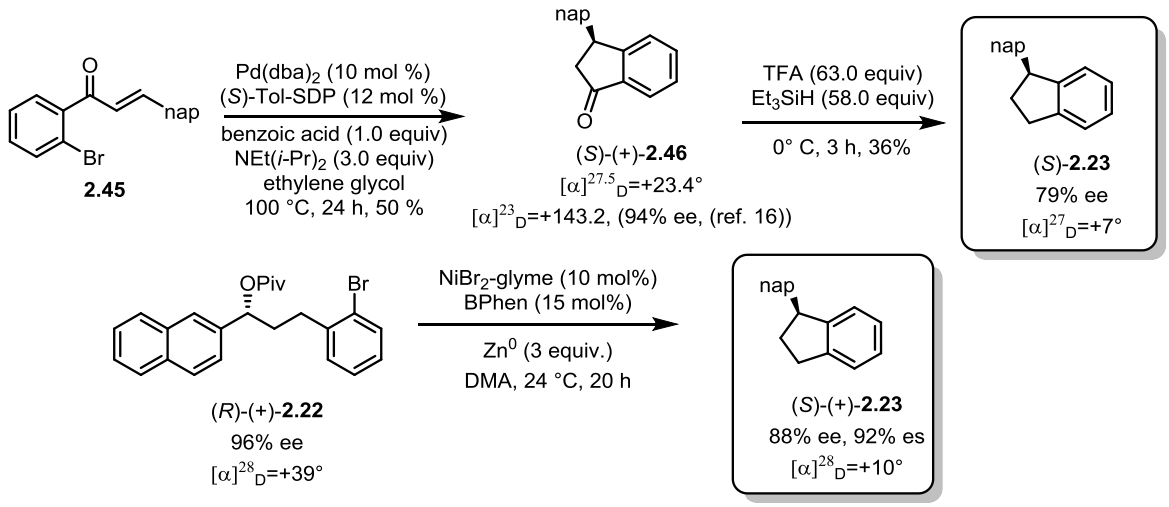
a) Starting material 23% ee b) $NiBr_2$-glyme (15 mol%), 45 °C

Based on comparison of **(*S*)-2.23** to literature values,[20] the reductive cross-

[20] (a) Yu, N-U.; Xu, M-H. *J. Org. Chem.* **2013**, *78*, 2736. (b) Yue, G.; Lei, K.; Hirao, H.; Zhou, J. *Angew. Chem. Int. Ed.* **2015**, *54*, 6531.

electrophile coupling reaction proceeds with inversion at the benzylic center. To verify whether the oxidative addition proceeded with retention or inversion, enantioenriched pivalate (*R*)-**2.22** was prepared by enantioselective CBS reduction and its absolute configuration was assigned based on the accepted model for selectivity in CBS reductions.[21] The absolute configuration of the enantioenriched indane **2.23** was assigned by derivatization of known enantioenriched indanone (*S*)-**2.46** to indane (*S*)-**2.23** (Scheme 2.5). Enantioenriched (*S*)-**2.46** was prepared by an asymmetric reductive Heck reaction as reported by Zhou. The stereochemistry was verified by comparison of the optical rotation to the literature value. Reduction to indane (*S*)-**2.23** and subsequent comparison of the optical rotation and SFC data matched that of (*S*)-**2.23** synthesized by the reductive cross-electrophile coupling reaction. This product corresponds to net inversion at the benzylic center in the reductive cross-electrophile coupling reaction.

Scheme 2.5. Stereochemical course of the reductive cross-electrophile coupling reaction



[21] Corey, E. J.; Helal, C. J. *Angew. Chem., Int. Ed*. **1998**, *37*, 1986.

2.5 Conclusion

In summary, we have developed an inter- and intramolecular reductive cross-coupling for the synthesis of diarylmethanes, indanes, and tetralins. The reactions are tolerant of a variety of heterocycles and functional groups. We have also demonstrated stereospecific cross-electrophile coupling reactions of benzylic esters for synthesis of enantioenriched 1-arylindanes and tetralins.

2.6 Experimental Details

General Procedures

All reactions were carried out under an atmosphere of N_2, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (CH_2Cl_2), and dimethylacetamide (DMA) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 ºC for 12 h) to remove H_2O. All other solvents utilized were purchased "anhydrous" commercially, or purified as described. ^1H NMR spectra were recorded on Bruker DRX-400 (400 MHz ^1H, 100 MHz ^{13}C, 376.5 MHz ^{19}F) or CRYO-500 (500 MHz ^1H, 125.7 MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of triplets (tt), quartet (q), quintet (quin), apparent doublet (ad), apparent triplet (at), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 ºC. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm^{-1}). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F_{254} precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with $KMnO_4$, ceric ammonium molybdate (CAM), or *p*-anisaldehyde (PAA) solutions. Flash chromatography was performed using Silica Gel 60 (170-400 mesh) from Fisher

Scientific. Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Automatic Polarimeter. SFC determinations of enantiopurity were performed on a Berger Analytical instrument using a Daicel™ Chiralpak® column (OD-H, OJ-H, or AD-H; 100 bar, 50 °C, 215 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

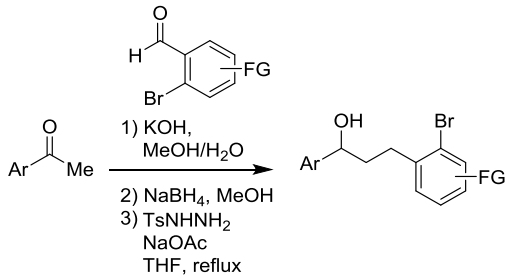
Nickel(II) bromide ethylene glycol dimethyl ether complex was purchased from Aldrich, stored in a glovebox under an atmosphere of N_2, and used as received.

Zinc powder (100 Mesh) was purchased from Alfa Aesar and used as received.

All other reagents were purchased commercially and used as received.

Synthesis and characterization of substrates

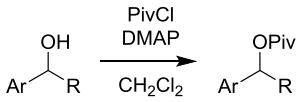
General Procedure A: *Aldol condensation, sodium borohydride reduction, and diimide reduction.*



The products were prepared according to a modified procedure reported by Franzen.[1] To a 250 mL round bottom flask equipped with a stir bar was added ketone (1.0 equiv), aldehyde (1.0 equiv), KOH (2.0 equiv), and MeOH (30 mL). The reaction was stirred overnight at room temperature. The resulting solid was filtered, washed with water, dried by vacuum filtration, and taken on to the next step without further purification. The unpurified chalcone was taken up in MeOH (50 mL) and $NaBH_4$ (1.2 equiv) was added in two portions over 10 minutes. Upon complete reaction of starting material, as judged by TLC, the mixture was quenched with careful addition of saturated NH_4Cl (15 mL). EtOAc (50 mL) was added, the layers were separated, and the aqueous layer was extracted with EtOAc (3 x 30 mL). The organic extract was washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure and the unpurified allylic alcohol was taken on to the next step without further purification. The unpurified residue was dissolved in THF (100 mL) and tosylhydrazide (4.0 equiv) and $NaOAc•3H_2O$ (4.0 equiv) were added. The reaction vessel was equipped with a reflux condenser and the mixture was heated at reflux for 24 hours. The mixture was cooled to room temperature and quenched with H_2O (30 mL), and the aqueous layer was extracted with EtOAc (3 x 40 mL). The organic extract was washed with brine, dried over $MgSO_4$, and

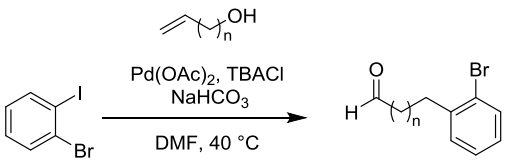
concentrated under reduced pressure. The product was then purified by flash column chromatography.

General Procedure B: *Pivalation of benzylic alcohols.*



The product was prepared according to a modified procedure reported by Martin.[2] To a solution of benzylic alcohol (1.0 equiv) in CH_2Cl_2 (20 mL) was added pivaloyl chloride (1.1 equiv) and dimethylaminopyridine (1.1 equiv). The reaction was allowed to stir overnight at room temperature. The reaction was quenched with water (10 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The organic extract was washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

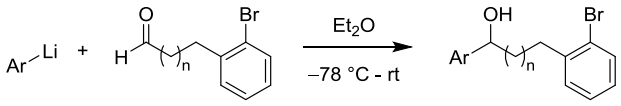
General Procedure C: *Heck reaction for the preparation of 2-bromophenylaldehydes.*



The product was prepared according to a modified procedure reported by Tietze.[3] A flame-dried 100 mL round bottom flask equipped with a stir bar and septum was charged with TBACl (1.0 equiv), $NaHCO_3$ (2.5 equiv), $Pd(OAc)_2$ (0.05 equiv), and dry DMF (30 mL). The mixture was stirred for 5 minutes followed by simultaneous addition of 1,2-iodobromobenzene (1.0 equiv), and alkenol (1.5 equiv). The reaction mixture was heated to 40 °C and stirred for 30 hours. The reaction was slowly quenched with saturated NH_4Cl (25 mL) and extracted with EtOAc (3 x 30 mL). The organic extract was washed with brine, dried

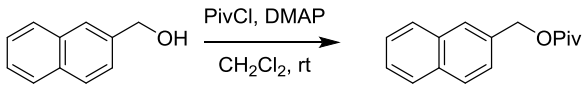
over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

General Procedure D*: Arene lithiation/lithium-halogen exchange, then addition into aldehyde.*



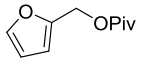
The product was prepared according to a modified procedure reported by O'Doherty.[4] A flame-dried 25 mL round bottom flask equipped with a stir bar and septum was charged with Et_2O (6 mL) and arene (1.3 equiv) and cooled to –78 °C. *n*-BuLi (2.5 M in hexanes, 1.4 equiv) was added dropwise over 10 minutes. The mixture was stirred cold for 30 minutes and then allowed to warm to room temperature and stir for an additional 20 minutes. A separate flask of requisite aldehyde (1.0 equiv) dissolved in Et_2O (10 mL) was cooled to –78 °C. The solution of aryl lithium was added dropwise over 30 minutes to the aldehyde. The reaction was warmed to room temperature overnight. The mixture was quenched with saturated NH_4Cl (15 mL). Layers separated and the aqueous layer was extracted with EtOAc (3 x 25 mL). The organic extract was washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

Synthesis and characterization of starting materials for Table 2.3

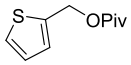


2.1. The product was prepared according to general procedure B using 2-naphthylmethanol (0.64 g, 5.0 mmol, 1.0 equiv), pivaloyl chloride (0.68 mL, 5.5 mmol, 1.1 equiv) and

dimethylaminopyridine (0.67 g, 5.5 mmol, 1.1 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (1.1 g, 4.5 mmol, 90%). Analytical data is consistent with literature values.[2] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85–7.77 (m, 4H), 7.51–7.41 (m, 3H), 5.26 (s, 2H), 1.24 (s, 9H).



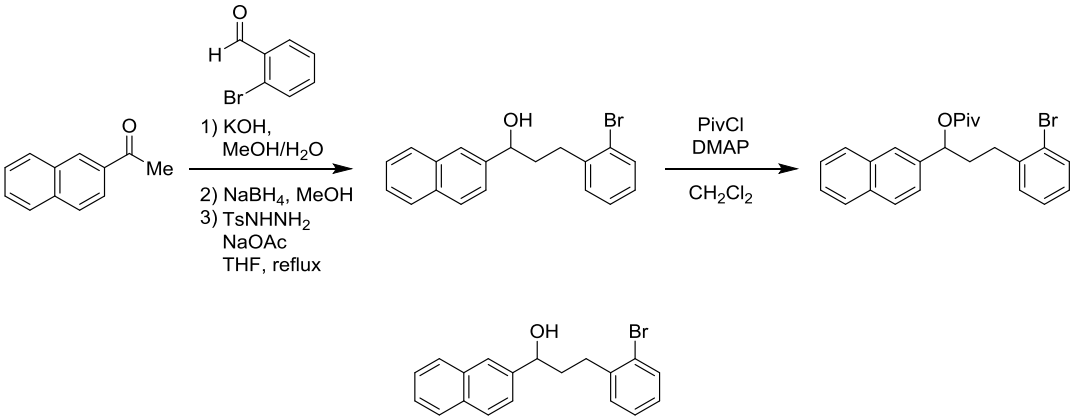
2.47. The product was prepared according to general procedure B using furfuryl alcohol (0.87 mL, 10 mmol), pivaloyl chloride (1.3 mL, 11 mmol), and dimethylaminopyridine (1.3 g, 11 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (1.6 g, 9.0 mmol, 82%). **TLC R_f** = 0.3 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (dd, *J* = 1.7, 0.7 Hz, 1H), 6.39–6.34 (d, *J* = 3.2 Hz, 1H), 6.35 (dd, *J* = 3.2, 1.7 Hz, 1H), 5.05 (s, 2H), 1.20 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 178.3, 150.1, 143.2, 110.7, 110.3, 58.3, 39.0, 27.3; **IR** (neat) 2973, 1728, 1279, 1153, 1136 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{10}H_{14}O_3Na$ $(M + Na)^+$ 205.0841, found 205.0836.



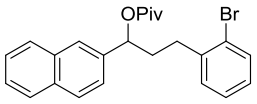
2.48. The product was prepared according to general procedure B using 2-thiophenemethanol (0.62 g, 5.5 mmol), pivaloyl chloride (0.74 mL, 6.1 mmol), and dimethylaminopyridine (0.75 g, 6.1 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (1.0 g, 5.4 mmol, 98%). **TLC R_f** = 0.3 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.05 (d, *J* = 3.3 Hz, 1H), 6.35 (dd, *J* = 5.0, 3.4 Hz, 1H), 5.25 (s, 2H), 1.20 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 178.3, 138.8, 127.6, 126.9, 126.6, 60.8, 39.0, 27.3; **IR** (neat)

2971, 1727, 1479, 1279, 1135 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{10}H_{14}O_2SNa$ $(M + Na)^+$ 221.0612, found 221.0609.

Synthesis and characterization for starting materials for Table 2.5

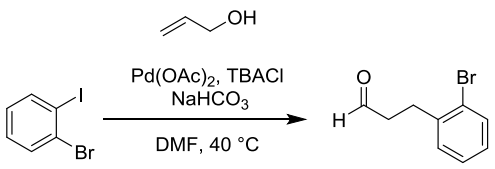


rac-**2.49.** The product was prepared according to general procedure A using 2-acetonaphthone (3.4 g, 20 mmol), 2-bromobenzaldehyde (2.3 mL, 20 mmol), and KOH (2.2 g, 40 mmol). Then $NaBH_4$ (1.0 g, 25 mmol), tosylhydrazide (15 g, 80 mmol) and $NaOAc•3H_2O$ (11 g, 80 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (6.2 g, 18 mmol, 91% over 3 steps). **TLC R_f** = 0.6 (10% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86–7.78 (m, 4H), 7.54–7.43 (m, 4H), 7.24–7.18 (m, 2H), 7.04 (ddd, *J* = 8.1, 6.2, 3.3 Hz, 1H), 4.89 (t, *J* = 6.4 Hz, 1H), 2.97–2.76 (m, 2H), 2.22–2.09 (m, 2H), 2.05 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.9, 141.3, 133.5, 133.3, 133.1, 130.7, 128.6, 128.2, 127.92, 127.90, 127.7, 126.4, 126.1, 124.9, 124.7, 124.3, 74.3, 39.0, 32.8; **IR** (neat) 3365, 3054, 2929, 1601, 1470, 1021 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{17}BrONa$ $(M + Na)^+$ 363.0360, found 363.0368.



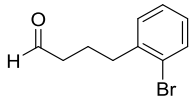
rac-**2.22.** The product was prepared according to general procedure B using *rac*-**2.49** (1.7 g, 5.0 mmol), pivaloyl chloride (0.68 mL, 5.5 mmol, 1.1 equiv) and dimethylaminopyridine (0.67 g, 5.5 mmol, 1.1 equiv). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (1.7 g, 4.1 mmol, 82%). **TLC** $\mathbf{R_f}$ = 0.7 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86–7.74 (m, 4H), δ 7.52–7.39 (m, 4H), δ 7.22–7.11 (m, 2H), 7.00 (t, *J* = 7.0 Hz, 1H), 5.95 (t, *J* = 6.5 Hz, 1H), 2.94–2.68 (m, 2H), 2.36–2.11 (m, 2H), 1.26 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.9, 140.9, 138.2, 133.5, 133.3, 133.2, 130.6, 128.7, 128.3, 128.1, 128.0, 127.8, 126.5, 126.3, 125.8, 124.6, 124.3, 75.5, 39.2, 36.9, 32.8, 27.5; **IR** (neat) 3055, 2969, 1725, 1471, 1279, 1147, 1025 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{24}H_{25}BrO_2Na$ $(M + Na)^+$ 447.0936, found 447.0926.

Synthesis of building blocks 2.50 and 2.51.

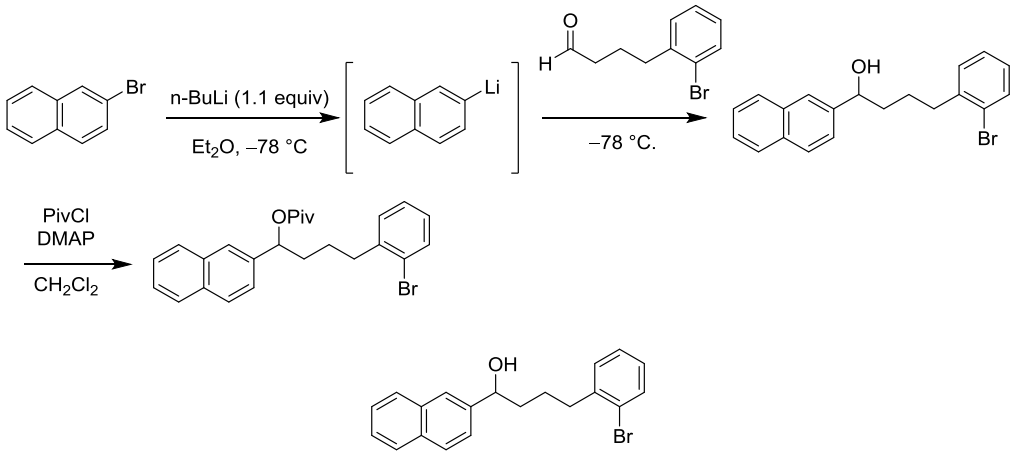


2.50. The product was prepared according to general procedure C using TBACl (5.6 g, 20.0 mmol), $NaHCO_3$ (4.2 g, 50 mmol), $Pd(OAc)_2$ (0.11 g, 0.5 mmol), iodobromobenzene (2.6 mL, 20 mmol, 1.0 equiv), and allyl alcohol (2.1 mL, 30 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (2.4 g, 10 mmol, 52%). Analytical data is consistent with literature values.[3] **^{1}H NMR** (400

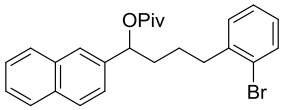
MHz, $CDCl_3$) δ 9.84 (s, 1H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.28–7.20 (m, 2H), 7.08 (q, *J* = 4.1 Hz, 1H), 3.07 (t, *J* = 7.4 Hz, 2H), 2.81 (t, *J* = 7.4 Hz, 2H).



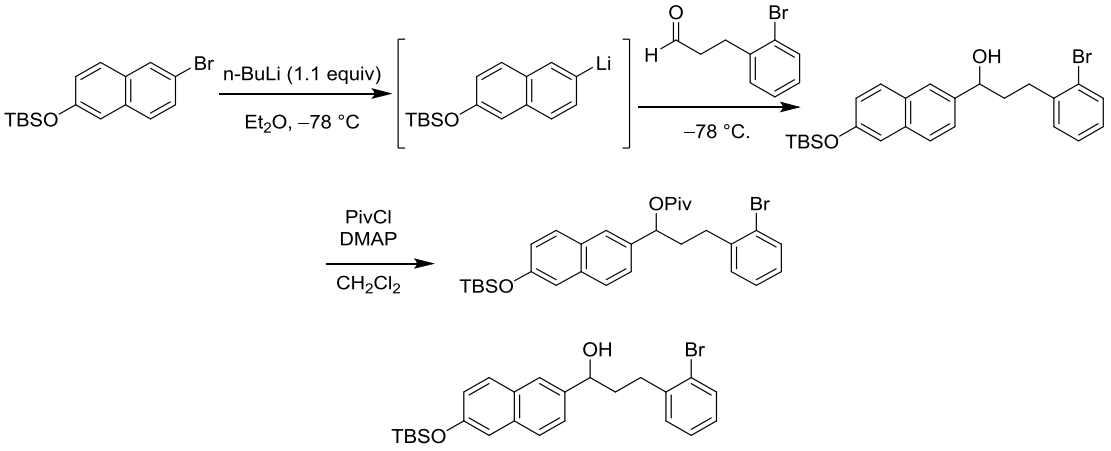
2.51. The product was prepared according to general procedure C using TBABr (6.5 g, 20.0 mmol), $NaHCO_3$ (4.2 g, 50 mmol), $Pd(OAc)_2$ (0.11 g, 0.5 mmol), iodobromobenzene (2.6 mL, 20 mmol, 1.0 equiv), and 3-buten-1-ol (2.6 mL, 30 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (2.4 g, 10 mmol, 52%). Analytical data is consistent with literature values.[5] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.53 (d, *J* = 7.9 Hz, 1H), 7.25–7.18 (m, 2H), 7.07 (dd, *J* = 7.9, 2.2 Hz, 1H), 2.79 (dt *J* = 7.5 Hz, 2H), 2.50 (td, *J* = 7.4, 1.5 Hz, 2H), 1.97 (d, *J* = 7.5 Hz, 2H).



rac-**2.52.** The product was prepared according to general procedure D using 2-bromonaphthalene (1.25 g, 6.00 mmol), *n*-BuLi (2.6 mL, 2.5 M in hexane, 6.6 mmol), and **2.51** (1.36 g, 6.00 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (1.89 g, 5.31 mmol, 85%).

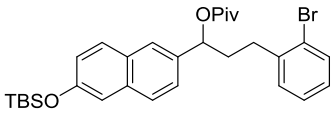


rac-**2.53.** The product was prepared according to general procedure B using *rac*-**2.52** (0.18 g, 0.50 mmol), pivaloyl chloride (0.080 mL, 0.60 mmol) and dimethylaminopyridine (0.80 g, 0.60 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.18 g, 0.40 mmol, 80%). **TLC** $\mathbf{R_f}$ = 0.4 (12% Et_2O/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.78 (m, 3H), 7.76 (s, 1H), 7.52–7.41 (m, 4H), 7.19 (td, *J* = 7.2, 1.2 Hz, 1H), 7.14 (dd, *J* = 7.6, 1.27 Hz, 1H), 7.03 (td, *J* = 7.6, 1.9 Hz, 1H), 5.91 (dd, *J* = 8.0, 5.6 Hz, 1H), 2.76 (t, *J* = 7.9 Hz, 2H), 2.07 (dddd, *J* = 13.4, 10.2, 8.0, 5.1 Hz, 1H), 1.93 (ddt, *J* = 13.8, 11.2, 5.6 Hz, 1H), 1.78–1.58 (m, 2H), 1.22 (s, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 177.8, 141.4, 138.5, 133.3, 133.2, 133.0, 130.5, 128.5, 128.2, 127.8, 127.8, 127.6, 126.3, 126.1, 125.6, 124.6, 124.3, 75.6, 39.0, 36.3, 36.0, 27.4, 26.0; **IR** (neat) 2971, 2866, 1726, 1500, 1279, 1148 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{25}H_{27}BrO_2$ $(M)^+$ 438.1194, found 438.1187.

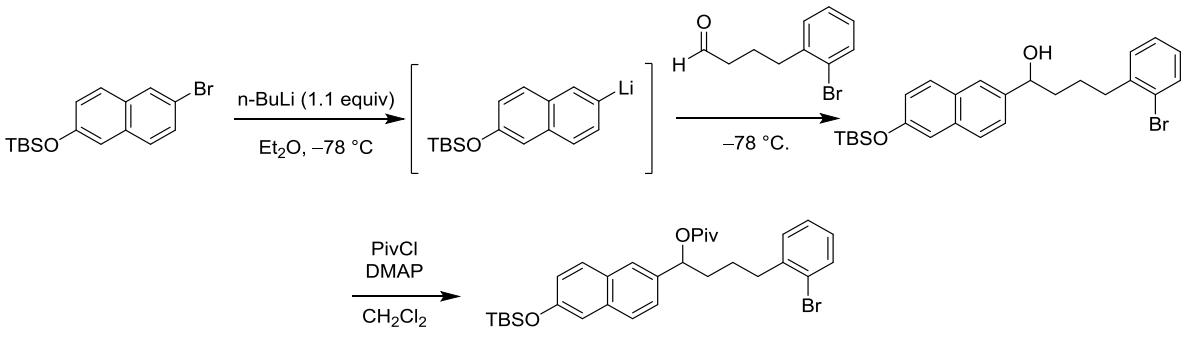


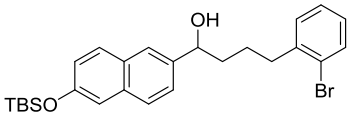
rac-**2.54.** The product was prepared according to general procedure D using 2-bromo-6-(*tert*-butyldimethylsiloxy)naphthalene (2.02 g, 6.00 mmol), *n*-BuLi (2.6 mL, 2.5 M in hexane, 6.6 mmol), and **2.50** (1.4 g, 6.0 mmol). The product was purified by flash column

chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (1.9 g, 4.0 mmol, 67%).

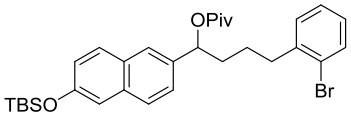


rac-**2.55.** The product was prepared according to general procedure B using *rac*-**2.54** (0.47 g, 1.0 mmol), pivaloyl chloride (0.13 mL, 1.1 mmol) and dimethylaminopyridine (0.13 g, 1.1 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.45 g, 0.81 mmol, 81%). **TLC R_f** = 0.5 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73–7.66 (m, 3H), 7.50 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.42 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.22–7.13 (m, 3H), 7.07 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.02 (ddd, *J* = 8.0, 6.9, 2.3 Hz, 1H), 5.91 (dd, *J* = 8.3, 5.3 Hz, 1H), 2.86 (ddd, *J* = 14.0, 11.2, 5.0 Hz, 2H), 2.73 (ddd, *J* = 13.5, 10.7, 5.7 Hz, 2H), 2.35–2.10 (m, 2H), 1.25 (s, 9H), 1.01 (s, 9H), 0.24 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 153.9, 140.9, 136.1, 134.5, 133.0, 130.5, 129.6, 129.1, 127.9, 127.7, 127.3, 125.5, 124.54, 124.50, 122.5, 114.9, 75.5, 39.1, 36.7, 32.7, 27.4, 25.9, 18.4, -4.2; **IR** (neat) 2956, 2930, 1728, 1605, 1505, 1261, 1152 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{30}H_{39}BrO_3Si$ $(M + Na)^+$ 554.1852, found 554.1850.

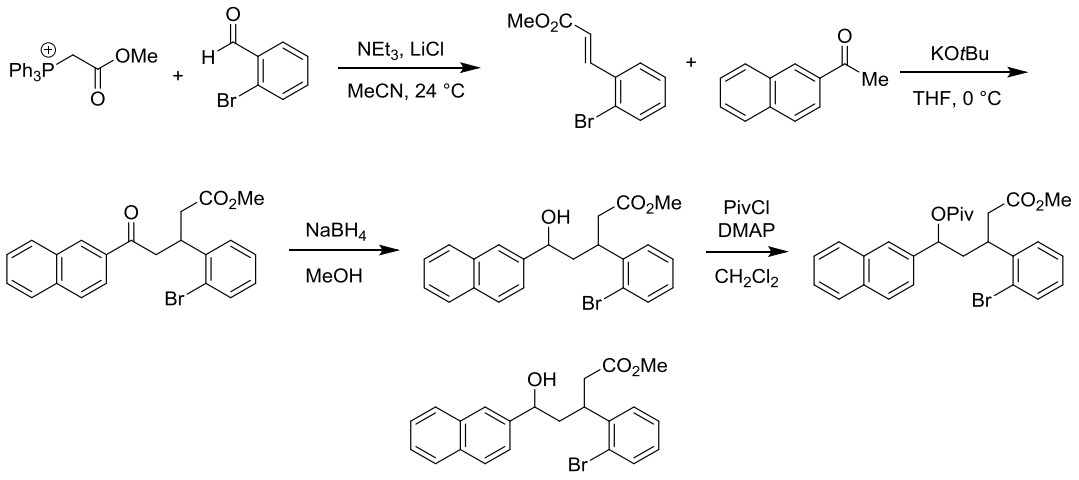




2.56. The product was prepared according to general procedure D using 2-bromo-6-(*tert*-butyldimethylsiloxy)naphthalene (1.01 g, 3.00 mmol), *n*-BuLi (1.3 mL, 2.5 M in hexane, 3.3 mmol), and **2.51** (0.68 g, 3.0 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (1.4 g, 2.9 mmol, 98%).



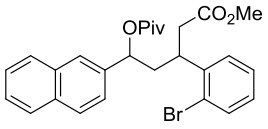
2.57. The product was prepared according to general procedure B using **2.56** (0.75 g, 1.5 mmol), pivaloyl chloride (0.22 mL, 1.8 mmol) and dimethylaminopyridine (0.22 g, 1.8 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.33 g, 0.77 mmol, 96%). **TLC R_f** = 0.5 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70–7.64 (m, 3H), 7.49 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.38 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.19–7.11 (m, 3H), 7.06 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.00 (ddd, *J* = 8.0, 7.0, 2.1 Hz, 1H), 5.88 (dd, *J* = 7.8, 5.6 Hz, 1H), 2.75 (t, *J* = 7.9 Hz, 2H), 2.11–1.85 (m, 2H), 1.80–1.56 (m, 2H), 1.21 (s, 9H), 1.01 (s, 9H), 0.23 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.9, 153.9, 141.5, 136.5, 134.5, 133.1, 130.6, 129.7, 129.2, 127.8, 127.7, 127.3, 125.5, 124.7, 122.6, 115.1, 75.8, 39.1, 36.4, 36.1, 27.5, 26.1, 26.0, 18.5, -4.04; **IR** (neat) 2954, 2929, 1726, 1605, 1479, 1260, 1150 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{31}H_{41}BrO_3SiNa$ $(M + Na)^+$ 591.1906, found 591.1910.



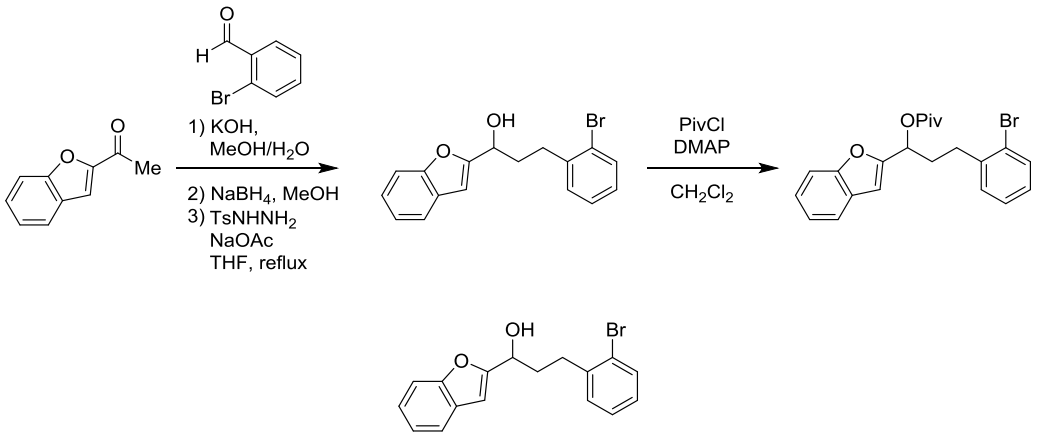
2.58. The product was prepared according to a modified procedure reported by Liu.[6] A 100 mL round bottom flask charged with LiCl (0.50 g, 10 mmol, 1.0 equiv) and a Teflon stir bar was flame-dried under vacuum. Once cooled to room temperature, the flask was removed from vacuum and charged with MeCN (50 mL), 2-bromobenzaldehyde (1.2 mL, 10 mmol, 1.0 equiv), trimethyl phosphonoacetate (2.00 mL, 12.5 mmol, 1.25 equiv), and triethylamine (1.4 mL, 10 mmol, 1.0 equiv), then sealed and stirred at 24 °C for 12 hr. The reaction mixture was passed through a cake of silica and the solvent was removed under reduced pressure affording a clear, colorless oil that was taken directly to the next step in the synthetic sequence.

A 100 mL round bottom flask containing methyl (*E*)-3-(2-bromophenyl)-acrylate was charged with 2-acetonaphthone (1.7 g, 10 mmol, 1.0 equiv), dissolved in THF (40 mL) and cooled to -5 °C in a brine-ice bath. Following this, potassium *tert*-butoxide (1.2 g, 10 mmol, 1.0 equiv) was added and the reaction was stirred at -5 °C to 0 °C for 3 hrs. The reaction was then quenched with saturated NH_4Cl (20 mL) and extracted with Et_2O (45 mL). The ether layer was then washed with brine and dried over Na_2SO_4, filtered and concentrated under vacuum. The unpurified product was purified by flash column chromatography (gradient:

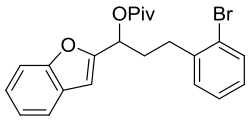
hexanes to 15% Et_2O/hexanes). The fractions containing product were concentrated in a 100 mL round bottom flask, dissolved in MeOH (45 mL), cooled to 0 °C and $NaBH_4$ (0.45 g, 12 mmol 1.2 equiv) was added. The reaction was allowed to warm to room temperature over the course of 30 min and stirred for an additional 2 hr. The unpurified mixture was then concentrated under reduced pressure and the unpurified oil was passed through a cake of silica with ethyl ether to afford the title compound as a pale yellow oil (1.4 g, 3.3 mmol, 33% over 3 steps).



2.59. The product was prepared according to general procedure B using **2.58** (1.3 g, 3.3 mmol), pivaloyl chloride (0.45 mL, 3.7 mmol) and dimethylaminopyridine (0.46 g, 3.8 mmol). The product was purified by flash column chromatography (10% Et_2O/hexanes) to afford the title compound as a colorless oil (1.6 g, 3.2 mmol, 99%, 1:1 dr). **TLC R_f** = 0.35 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84–7.76 (m, 3H), 7.63 (s, 1H), 7.55 (d, *J* = 8.3 Hz, 1H), 7.49–7.43 (m, 2H), 7.40 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.35–7.28 (m, 2H), 7.11–7.06 (m, 1H), 5.79 (t, *J* = 7.3 Hz, 1H), 3.68 (quint, *J* = 7.2 Hz, 1H), 3.56 (s, 3H), 2.68 (dd, *J* = 15.4, 7.2 Hz, 1H), 2.61 (dd, *J* = 15.5, 7.2 Hz, 1H), 2.49 (dt, *J* = 15.4, 8.2 Hz, 1H), 2.26–2.17 (m, 1H), 1.20 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 177.8, 172.0, 141.9, 137.5, 133.4, 133.27, 133.24, 128.6, 128.4, 128.2, 128.0, 127.8, 126.3, 126.2, 126.1, 124.8, 124.1, 74.0, 51.8, 40.8, 39.7, 38.9, 37.2, 27.2; **IR** (neat) 3056, 2972, 1728 (b), 1600, 1471, 1278, 1148, 1021 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{27}H_{29}BrO_4Na$ $(M + Na)^+$ 519.1147, found 519.1132.

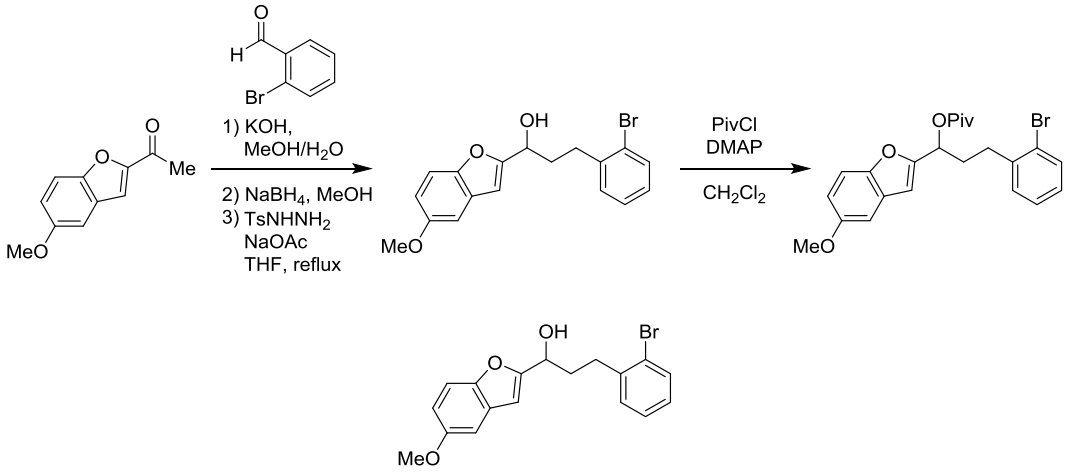


2.60. The product was prepared according to general procedure A using 2-benzofuran methyl ketone (2.9 g, 18 mmol), 2-bromobenzaldehyde (2.1 mL, 18 mmol), and KOH (2.0 g, 36 mmol). Then $NaBH_4$ (0.76 g, 20 mmol), tosylhydrazide (7.5 g, 40 mmol) and $NaOAc•3H_2O$ (5.4 g, 40 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (2.1 g, 6.3 mmol, 35% over 3 steps).

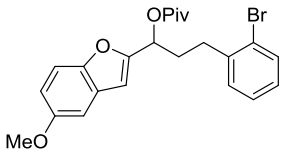


2.61. The product was prepared according to general procedure B using **2.60** (0.66 g, 2.0 mmol), pivaloyl chloride (0.27 mL, 2.2 mmol) and dimethylaminopyridine (0.27 g, 2.2 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.59 g, 1.4 mmol, 70%). **TLC R_f** = 0.5 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.3 Hz, 1H), 7.47 (d, *J* = 8.3 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 1H), 7.27–7.12 (m, 4H), 7.02–6.94 (m, 1H), 6.67 (s, 1H), 6.04 (t, *J* = 6.8 Hz, 1H), 2.90–2.72 (m, 2H), 2.41–2.27 (m, 2H), 1.25 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 155.6, 155.2, 140.6, 133.2, 130.7, 129.8, 128.3, 127.9, 124.74, 124.68, 123.2, 121.5, 111.6,

104.9, 68.8, 39.3, 33.3, 32.4, 27.5; **IR** (neat) 2970, 1729, 1453, 1278, 1142 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{23}BrO_3Na$ $(M + Na)^+$ 437.0728, found 437.0725.

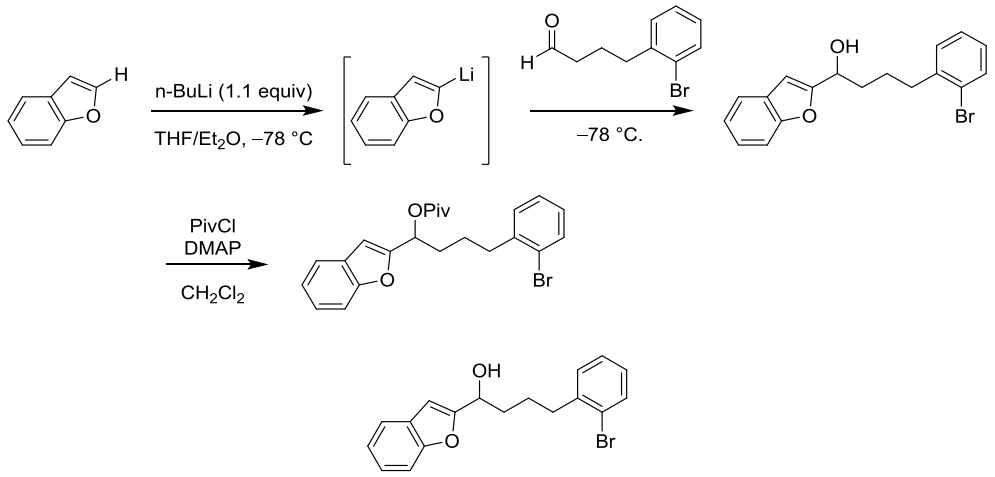


2.62. The product was prepared according to general procedure A using 2-acyl-5-methoxybenzofuran (2.0 g, 10 mmol), 2-bromobenzaldehyde (1.2 mL, 10 mmol), and KOH (1.2 g, 21 mmol). Then $NaBH_4$ (0.95 g, 25 mmol), tosylhydrazide (5.4 g, 29 mmol) and $NaOAc•3H_2O$ (3.9 g, 29 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (2.0 g, 5.5 mmol, 55% over 3 steps).

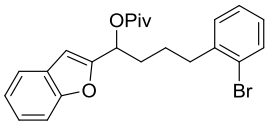


2.63. The product was prepared according to general procedure B using **2.62** (0.50 g, 1.6 mmol), pivaloyl chloride (0.19 mL, 1.6 mmol) and dimethylaminopyridine (0.19 g, 1.6 mmol). The product was purified by flash column chromatography (12% EtOAc/hexanes) to afford the title compound as a colorless oil (0.52 g, 1.2 mmol, 75%). **TLC R_f** = 0.4 (12% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.1 Hz, 1H), 7.33 (d, *J* = 8.9 Hz, 1H), 7.20–7.16 (m, 2H), 7.01 (ddd, *J* = 7.9, 6.0, 3.2 Hz, 1H), 6.99 (d, *J* = 2.6 Hz, 1H), 6.87 (dd, *J* = 8.9,

2.6 Hz, 1H), 6.62 (t, *J* = 0.6 Hz, 1H), 6.00 (t, *J* = 6.6 Hz, 1H), 3.78 (s, 3H), 2.90–2.72 (m, 2H), 2.41–2.26 (m, 2H), 1.25 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 156.34, 156.32, 150.1, 140.6, 133.2, 130.7, 128.8, 128.2, 127.9, 124.6, 113.5, 112.0, 105.0, 103.9, 68.8, 56.1, 39.2, 33.2, 32.3, 27.4; **IR** (neat) 2968, 1729, 1617, 1476, 1204, 1192 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{25}BrO_4Na$ $(M + Na)^+$ 467.0834, found 467.0832.

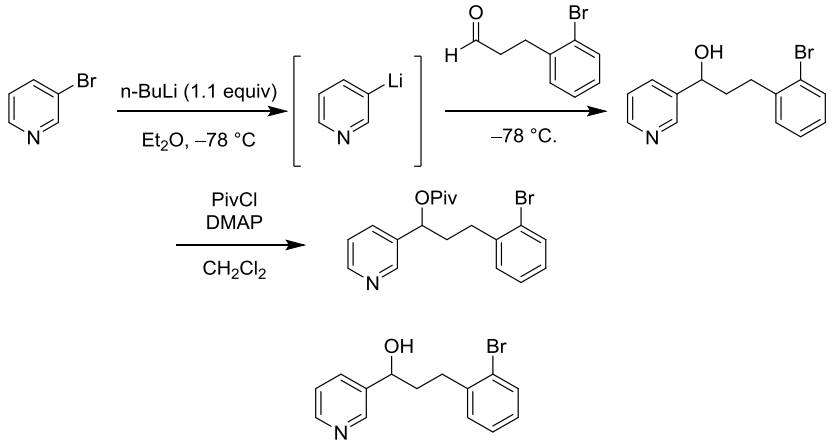


2.64. The product was prepared according to general procedure D using benzofuran (0.66 mL, 6.0 mmol), *n*-BuLi (2.6 mL, 2.5 M in hexane, 6.6 mmol), and **2.51** (1.0 g, 4.5 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (1.4 g, 3.8 mmol, 84%).

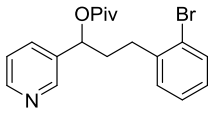


2.65. The product was prepared according to general procedure B using **2.64** (0.28 g, 0.80 mmol), pivaloyl chloride (0.11 mL, 0.90 mmol) and dimethylaminopyridine (0.11 g, 0.90 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.33 g, 0.77 mmol, 96%). **TLC R_f** = 0.5 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.3 Hz, 1H), 7.48 (d, *J* = 8.1 Hz, 1H),

7.44 (d, *J* = 8.2 Hz, 1H), 7.24 (t, *J* = 8.0 Hz, 1H), 7.21–7.11 (m, 3H), 7.00 (td, *J* = 7.8, 1.8 Hz, 1H), 6.63 (s, 1H), 6.01 (t, *J* = 6.8 Hz, 1H), 2.77 (t, *J* = 7.8 Hz, 2H), 2.18–2.03 (m, 2H), 1.80–1.60 (m, 2H), 1.21 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 155.9, 155.1, 141.4, 133.1, 130.6, 128.2, 128.0, 127.7, 124.7, 124.6, 123.1, 121.4, 111.6, 104.6, 69.0, 39.2, 36.0, 32.7, 27.4, 25.7; **IR** (neat) 3057, 2969, 2869, 1728, 1471, 1454, 1278, 1254, 1143 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{23}H_{25}BrO_3Na$ $(M + Na)^+$ 451.0885, found 451.0891.

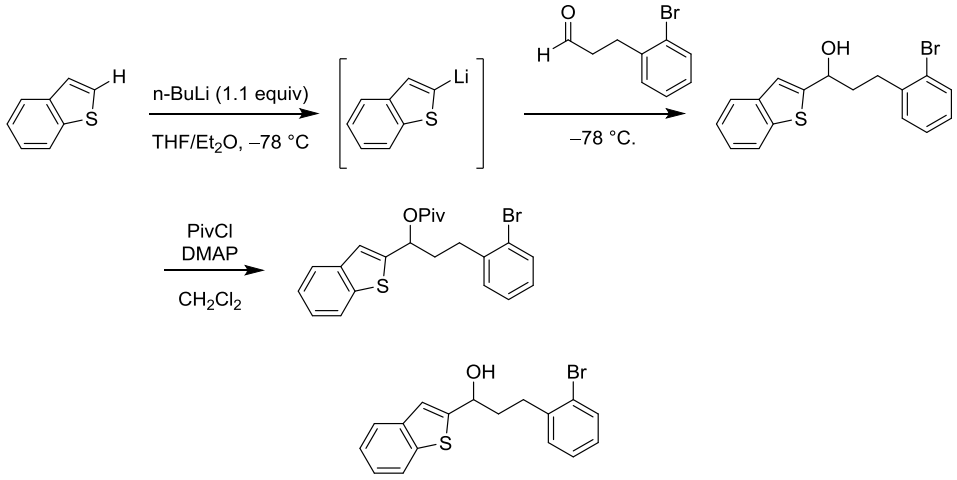


2.66. The product was prepared according to general procedure D using 3-bromopyridine (0.39 g, 4.0 mmol), *n*-BuLi (1.7 mL, 2.5 M in hexane, 4.4 mmol), and **2.50** (0.85 g, 4.0 mmol) and –78 °C. The product was purified by flash column chromatography (1% NEt_3 in EtOAc) to afford the title compound as a yellow oil (0.72 g, 2.5 mmol, 62%).

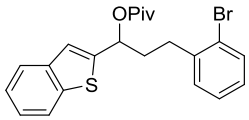


2.67. The product was prepared according to general procedure B using **2.66** (0.58 g, 1.5 mmol), pivaloyl chloride (0.22 mL, 1.8 mmol) and dimethylaminopyridine (0.22 g, 1.8 mmol). The product was purified by flash column chromatography (50% EtOAc/hexanes) to afford the title compound as a colorless oil (0.40 g, 1.1 mmol, 70%). **TLC R_f** = 0.2 (12% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.4 Hz, 1H), 8.54 (dd, *J* = 4.9, 1.2 Hz,

1H), 7.65 (dt, *J* = 8.2, 1.8 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.27 (dd, *J* = 7.3, 4.8 Hz, 1H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.16 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.05 (td, *J* = 7.8, 1.5 Hz, 1H), 5.80 (dd, *J* = 8.2, 5.1 Hz, 1H), 2.89–2.68 (m, 2H), 2.30–2.05 (m, 2H), 1.25 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.7, 149.5, 148.3, 140.4, 136.4, 134.1, 133.2, 130.5, 128.2, 127.9, 124.5, 123.6, 73.2, 39.1, 36.6, 32.5, 27.4; **IR** (neat) 3055, 2970, 1728, 1471, 1279, 1146 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{22}BrNO_2Na$ $(M + Na)^+$ 398.0732, found 398.0746.

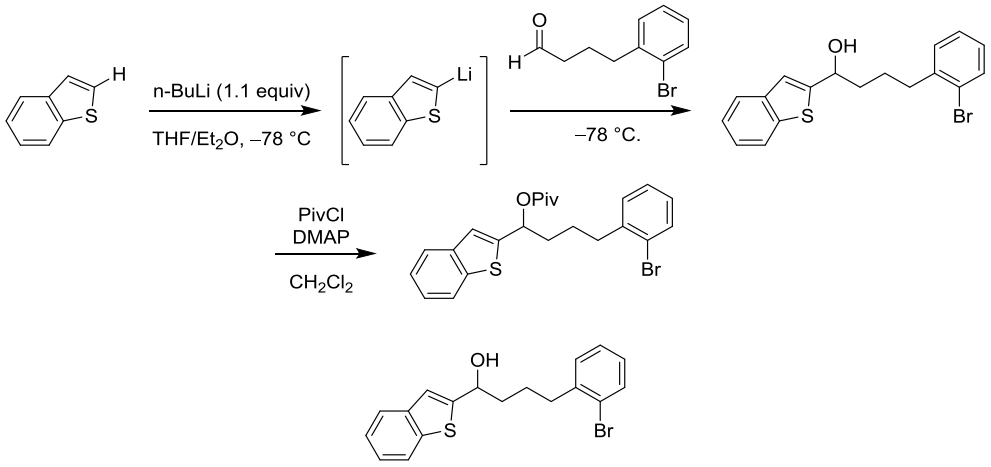


2.68. The product was prepared according to general procedure D using benzothiophene (0.54 g, 4.0 mmol), *n*-BuLi (1.7 mL, 2.5 M in hexane, 4.4 mmol), and **2.50** (0.85 g, 4.0 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (1.1 g, 3.2 mmol, 79%).

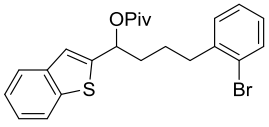


2.69. The product was prepared according to general procedure B using **2.68** (0.21 g, 0.60 mmol), pivaloyl chloride (0.08 mL, 0.7 mmol) and dimethylaminopyridine (0.080 g, 0.70 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.21 g, 0.48 mmol, 79%). **TLC R_f** = 0.5 (5%

EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.7 Hz, 1H), 7.77 (d, *J* = 7.4 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.40–7.30 (m, 3H), 7.28–7.20 (m, 2H), 7.01 (dd, *J* = 8.1, 2.2 Hz, 1H), 6.21 (dd, *J* = 7.6, 5.7 Hz, 1H), 2.99–2.79 (m, 2H), 2.46–2.27 (m, 2H), 1.32 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.7, 144.4, 140.5, 139.7, 139.5, 133.2, 130.6, 129.8, 128.2, 127.9, 124.7, 124.6, 124.0, 122.7, 122.3, 71.4, 39.2, 36.6, 32.6, 27.4; **IR** (neat) 3058, 2970, 1728, 1477, 1457, 1277, 1140, 1028 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{23}BrO_2SNa$ $(M + Na)^+$ 453.0500, found 453.0512.

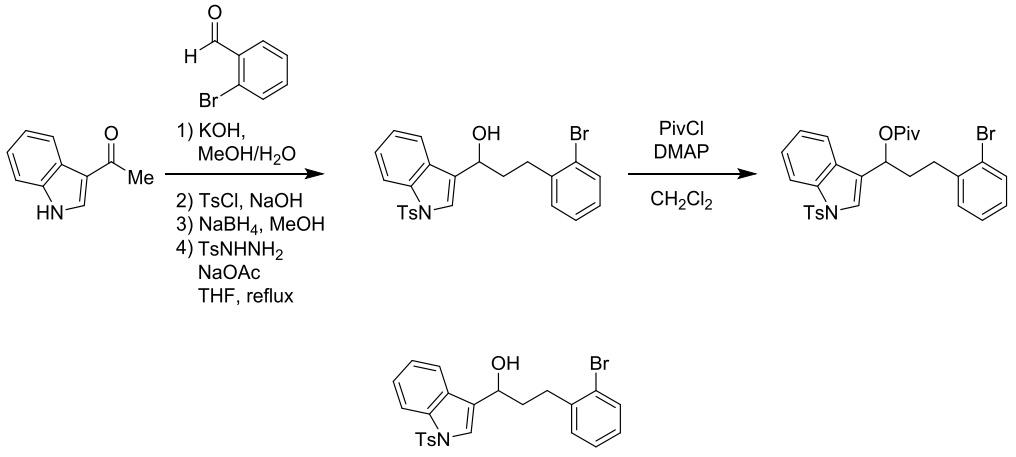


2.70. The product was prepared according to general procedure D using benzothiophene (0.34 g, 2.5 mmol), *n*-BuLi (1.1 mL, 2.5 M in hexane, 2.2 mmol), and **2.51** (0.45 g, 2.0 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (0.72 g, 2.0 mmol, 99%).

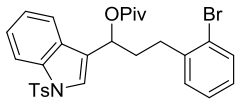


2.71. The product was prepared according to general procedure B using **2.70** (0.72 g, 2.0 mmol), pivaloyl chloride (0.27 mL, 2.3 mmol) and dimethylaminopyridine (0.27 g, 2.3 mmol). The product was purified by flash column chromatography (12% Et_2O/hexanes) to afford

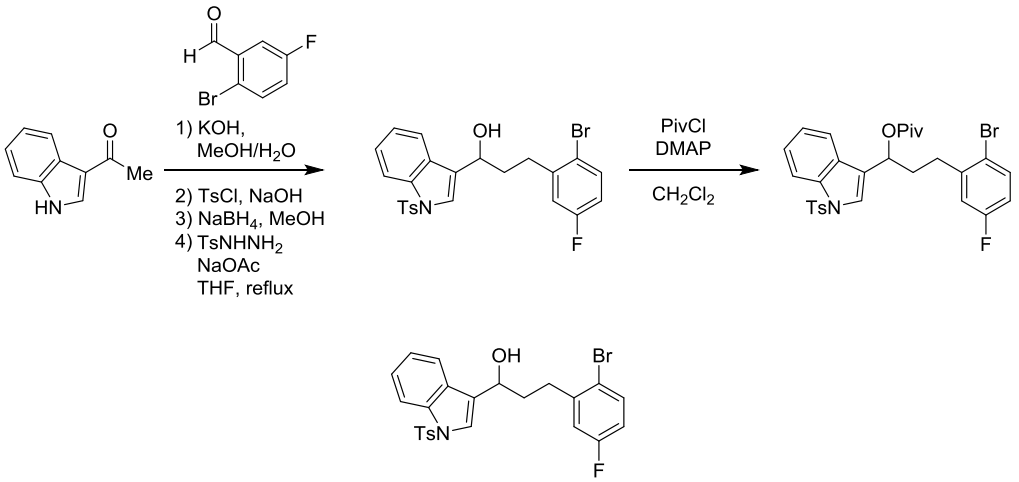
the title compound as a colorless oil (0.46 g, 1.0 mmol, 50%). **TLC R_f** = 0.6 (12% Et_2O/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81–7.77 (m, 1H), 7.73–7.69 (m, 1H), 7.51 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.35–7.27 (m, 2H), 7.23–7.15 (m, 3H), 7.04 (ddd, *J* = 7.9, 7.0, 2.1 Hz, 1H), 6.12 (dd, *J* = 7.6, 6.0 Hz, 1H), 2.78 (t, *J* = 7.8 Hz, 2H), 2.17–1.96 (m, 2H), 1.82–1.61 (m, 2H), 1.22 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 177.6, 144.5, 141.1, 139.4, 139.3, 132.9, 130.4, 127.7, 127.5, 124.5, 124.40, 124.35, 123.7, 122.4, 121.8, 71.4, 38.9, 35.9, 35.8, 27.2, 25.7; **IR** (neat) 3054, 2972, 1725, 1438, 1264, 1146 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{23}H_{25}BrO_2SNa$ $(M + Na)^+$ 467.0656, found 467.0652.



2.72. The product was prepared according to general procedure A using 3-acetylindole (1.6 g, 10 mmol), 2-bromobenzaldehyde (1.9 g, 10 mmol), and KOH (1.2 g, 22 mmol). The indolyl chalcone was then tosylated according to a modified procedure reported by Carreira. A using tosyl chloride (2.2 g, 12 mmol), TBABr (0.34 g, 0.12 mmol), and NaOH (30% w/v) and benzene.[7] The chalcone was then reduced by $NaBH_4$ (0.38 g, 10 mmol), and tosylhydrazide (7.5 g, 40 mmol) and $NaOAc•3H_2O$ (5.5 g, 40 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (3.6 g, 7.4 mmol, 74% over 4 steps).

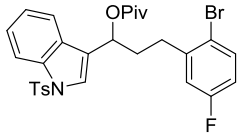


2.73. The product was prepared according to general procedure B using **2.72** (0.78 g, 1.6 mmol), pivaloyl chloride (0.25 mL, 2.0 mmol) and dimethylaminopyridine (0.25 g, 2.0 mmol). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title product as a colorless oil (0.86 g, 1.5 mmol, 94%). **TLC R_f** = 0.6 (15% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.62 (d, *J* = 6.5 Hz, 2H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.29 (t, *J* = 7.9 Hz, 1H), 7.20 (t, *J* = 7.7 Hz, 1H), 7.17–7.06 (m, 4H), 6.96 (td, *J* = 7.7, 1.7 Hz, 1H), 6.14 (t, *J* = 6.6 Hz, 1H), 2.84–2.64 (m, 2H), 2.39–2.19 (m, 2H), 2.17 (s, 3H), 1.21 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 145.3, 140.6, 135.7, 135.3, 133.1, 130.6, 130.2, 129.1, 128.2, 127.9, 127.0, 125.3, 124.6, 124.3, 123.7, 122.3, 120.6, 114.2, 68.9, 39.2, 35.1, 32.7, 27.5, 21.7; **IR** (neat) 3053, 2972, 1725, 1596, 1446, 1370, 1174, 1152, 1122 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{29}H_{30}BrNO_4SNa$ $(M + Na)^+$ 590.0977, found 590.0964.

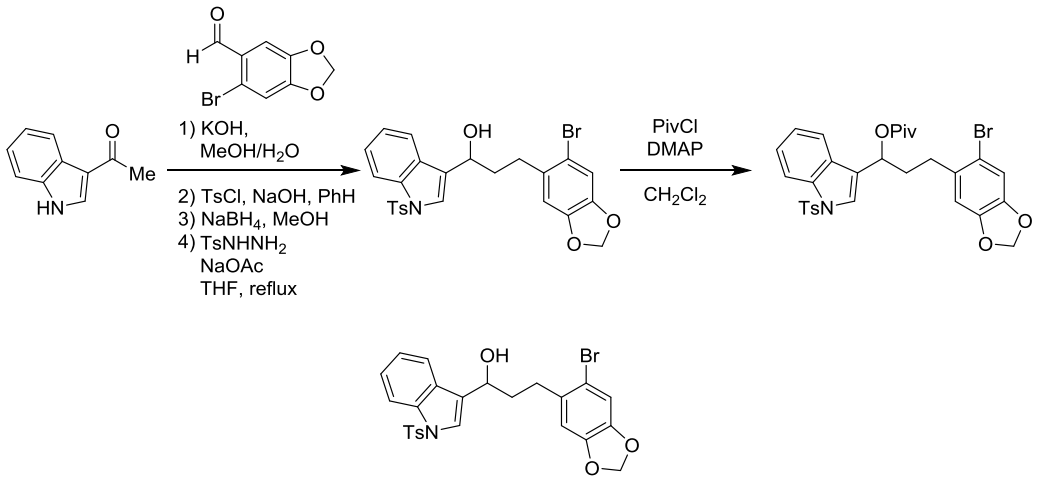


2.74. The product was prepared according to general procedure A using 3-acetylindole (1.6 g, 10 mmol), 2-bromo-5-fluorobenzaldehyde (2.0 g, 10 mmol), and KOH (1.4 g, 23 mmol).

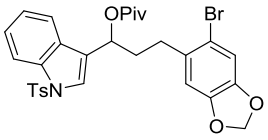
The indolyl chalcone was then tosylated according to a modified procedure reported by Carreira using tosyl chloride (2.2 g, 12 mmol), TBABr (0.34 g, 0.12 mmol), and NaOH (30% w/v) and benzene. The chalcone was then reduced by $NaBH_4$ (0.40 g, 10 mmol), and tosylhydrazide (7.5 g, 40 mmol) and $NaOAc•3H_2O$ (5.5 g, 40 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (3.6 g, 7.4 mmol, 64% over 4 steps).



2.75. The product was prepared according to general procedure B using **2.74** (0.75 g, 1.5 mmol), pivaloyl chloride (0.25 mL, 2.0 mmol) and dimethylaminopyridine (0.24 g, 2.0 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title product as a yellow oil (0.66 g, 1.1 mmol, 73%). **TLC R_f** = 0.6 (15% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 1H), 7.74 (d, *J* = 8.3 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.69 (s, 1H), 7.42 (dd, *J* = 8.7, 6.3 Hz, 1H), 7.32 (t, *J* = 7.7 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.85 (dd, *J* = 9.3, 3.0 Hz, 1H), 6.77 (td, *J* = 8.3, 3.0 Hz, 1H), 6.11 (t, *J* = 6.6 Hz, 1H), 2.80–2.61 (m, 2H), 2.36–2.17 (m, 5H), 1.21 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 162.1 (d, *J* = 247 Hz), 145.3, 142.7 (d, *J* = 7 Hz), 135.7, 135.3, 134.2 (d, *J* = 8 Hz), 130.1, 129.0, 127.0, 125.2, 124.2, 123.6, 122.0, 120.5, 118.6 (d, *J* = 3 Hz), 117.3 (d, *J* = 22 Hz), 115.3 (d, *J* = 22 Hz), 114.1, 68.8, 39.2, 34.7, 32.7, 27.4, 21.8; **IR** (neat) 3068, 2972, 1726, 1598, 1370, 1174, 1121 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{29}H_{29}BrFNO_4SNa$ $(M + Na)^+$ 608.0883, found 608.0860.



2.76. The product was prepared according to general procedure A using 3-acetylindole (1.6 g, 10 mmol), 2-bromo-5-fluorobenzaldehyde (2.3 g, 10 mmol), and KOH (1.4 g, 23 mmol). The indolyl chalcone was then tosylated according to a modified procedure reported by Carreira using tosyl chloride (2.2 g, 12 mmol), TBABr (0.34 g, 0.12 mmol), and NaOH (30% w/v) and benzene. The chalcone was then reduced by $NaBH_4$ (0.40 g, 10 mmol), and tosylhydrazide (7.5 g, 40 mmol) and $NaOAc•3H_2O$ (5.5 g, 40 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a yellow oil (3.6 g, 7.4 mmol, 11% over 4 steps).

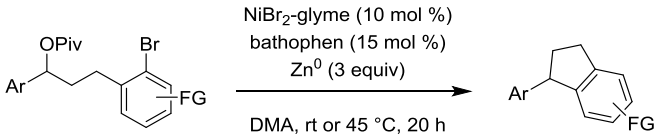


2.77. The product was prepared according to general procedure B using **2.76** (0.60 g, 1.1 mmol), pivaloyl chloride (0.16 mL, 1.3 mmol) and dimethylaminopyridine (0.16 g, 1.3 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title product as a yellow oil (0.25 g, 0.41 mmol, 36%). **TLC R_f** = 0.5 (15% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.2 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.58 (s, 1H), 7.30 (td, *J* = 8.4, 1.3 Hz, 1H), 7.22 (td, *J* = 8.1, 1.2 Hz, 1H), 7.17 (d, *J* = 8.0 Hz,

2H), 6.93 (s, 1H), 6.59 (s, 1H), 6.08 (t, *J* = 6.6 Hz, 1H), 5.89 (s, 2H), 2.73–2.54 (m, 2H), 2.32–2.11 (m, 5H), 1.20 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.8, 147.7, 147.1, 145.3, 135.7, 135.3, 133.5, 130.1, 129.0, 127.0, 125.2, 124.2, 123.6, 122.2, 120.6, 114.4, 114.1, 113.0, 110.1, 101.9, 68.8, 39.2, 35.2, 32.6, 27.4, 21.8; **IR** (neat) 2971, 1724, 1476, 1369, 1174, 1121 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{30}H_{30}BrNO_6SNa$ $(M + Na)^+$ 634.0875, found 634.0860.

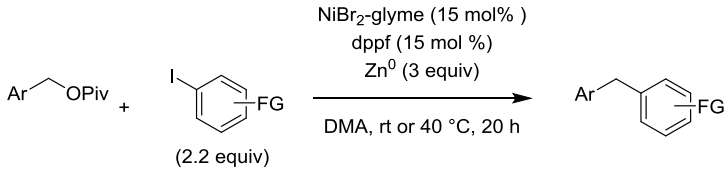
General procedures for reductive cross-electrophile coupling reactions

General Procedure E*: Intramolecular reductive coupling reactions*



In a glovebox, a flame-dried 7 mL vial equipped with a stir bar was charged with $NiBr_2$•glyme (10 or 15 mol %), bathophenanthroline (15 mol %), Zn^0 (3 equiv), DMA (0.60 mL), and substrate (1.00 equiv). The reaction was stirred for 20 h before removing the vial from the glovebox. The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography.

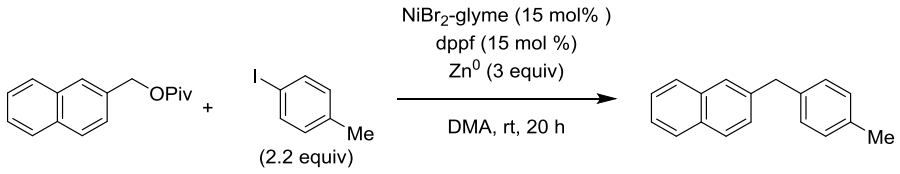
General Procedure F*: Intermolecular reductive coupling reactions*



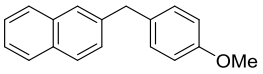
In a glovebox, a flame-dried 7 mL vial equipped with a stir bar was charged with $NiBr_2$•glyme (15 mol %), dppf (15 mol %), Zn^0 (3 equiv), aryl iodide (2.2 equiv), DMA (0.60 mL), and substrate (1.00 equiv). The reaction was stirred for 20 h before removing the vial from the

glovebox. The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography.

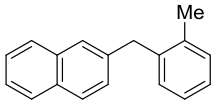
Characterization for products in Table 2.3



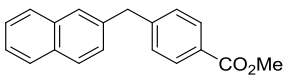
2.7. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4-iodotoluene (95.9 mg, 0.44 mmol), DMA (0.60 mL), and **2.1** (48.4, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (45.5 mg, 0.196 mmol, 98%). Analytical data is consistent with literature values.[8] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81–7.73 (m, 3H), 7.63 (s, 1H), 7.47–7.39 (m, 2H), 7.33–7.29 (m, 1H), 7.15–7.08 (m, 4H), 4.10 (s, 2H), 2.32 (s, 3H).



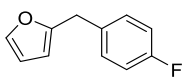
2.15. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4-iodoanisole (103.0 mg, 0.44 mmol), DMA (0.60 mL), and **2.1** (48.4 mg, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (46.6 mg, 0.188 mmol, 94%). Analytical data is consistent with literature values.[9] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80–7.72 (m, 3H), 7.60 (s, 1H), 7.46–7.38 (m, 2H), 7.30 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.16–7.11 (m, 2H), 6.85–6.81 (m, 2H), 4.08 (s, 2H), 3.77 (s, 3H).



2.13. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 2-iodotoluene (95.9 mg, 0.44 mmol, 2.2 equiv), DMA (0.60 mL), and **2.1** (48.4 mg, 0.200 mmol, 1.00 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (33.9 mg, 0.146 mmol, 73%). Analytical data is consistent with literature values.[9] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80–7.69 (m, 3H), 7.51 (s, 1H), 7.45–7.34 (m, 2H), 7.28 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.19–7.11 (m, 4H), 4.14 (s, 2H), 2.26 (s, 3H).

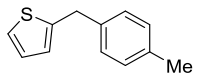


2.14. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), methyl 4-iodobenzoate (115.3 mg, 0.44 mmol), DMA (0.60 mL), and **2.1** (48.4 mg, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (40.9 mg, 0.148 mmol, 74%). Analytical data is consistent with literature values.[8] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.81-7.72 (m, 3H), 7.61 (s, 1H), 7.45–7.40 (m, 2H), 7.30–7.25 (m, 3H), 4.17 (s, 2H), 3.94 (s, 3H).

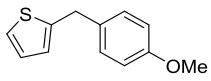


2.12. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4–iodofluorobenzene (98.0 mg, 0.44 mmol), DMA (0.60 mL), and **2.47** (36.4 mg, 0.200 mmol).

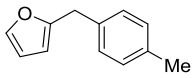
The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (26.4 mg, 0.150 mmol, 75%). Analytical data is consistent with literature values.[10] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 (dd, *J* = 1.8, 1.1 Hz, 1H), 7.20–7.15 (m, 2H), 7.01 (m, 2H), 6.28 (dd, *J* = 3.2, 1.9 Hz, 1H), 5.98 (dq, *J* = 3.2, 0.8 Hz, 1H), 3.93 (s, 2H).



2.9. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4-iodotoluene (95.9 mg, 0.44 mmol), DMA (0.60 mL), and **2.48** (39.6 mg, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (27.9 mg, 0.148 mmol, 74%). Analytical data is consistent with literature values.[10] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18–7.11 (m, 5H), 6.93 (dd, *J* = 5.1, 3.3 Hz, 1H), 6.82–6.79 (m, 1H), 4.13 (s, 2H), 2.34 (s, 3H).



2.10. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4-iodoanisole (103.0 mg, 0.44 mmol), DMA (0.60 mL), and **2.48** (39.6 mg, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (29.8 mg, 0.146 mmol, 73%). Analytical data is consistent with literature values.[11] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18–7.13 (m, 2H), 7.12 (dd, *J* = 5.2, 1.3 Hz, 1H), 6.97–6.93 (m, 3H), 6.90 (dd, *J* = 5.2, 3.5, 1H), 4.08 (s, 2H), 3.78 (s, 3H).

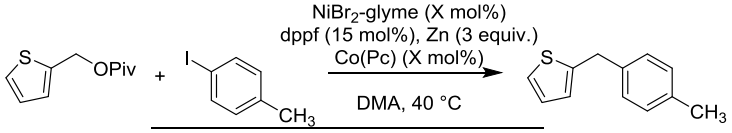


2.11. The product was prepared according to general procedure F using $NiBr_2$•glyme (9.2 mg, 0.030 mmol), dppf (16.6 mg, 0.030 mmol), Zn^0 (39.6 mg, 0.600 mmol), 4-iodotoluene (95.9 mg, 0.44 mmol,), DMA (0.60 mL), and **2.47** (36.4 mg, 0.200 mmol). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (26.5 mg, 0.153 mmol, 77%). Analytical data is consistent with literature values.[10] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (dd, *J* = 1.9, 0.9 Hz, 1H), 7.16–7.10 (m, 4H), 6.29 (dd, *J* = 3.1, 1.9 Hz, 1H), 6.00 (dq, *J* = 3.1, 0.9 Hz, 1H), 3.94 (s, 2H), 2.34 (s, 3H).

Control Experiments using different catalysts for intermolecular reductive coupling

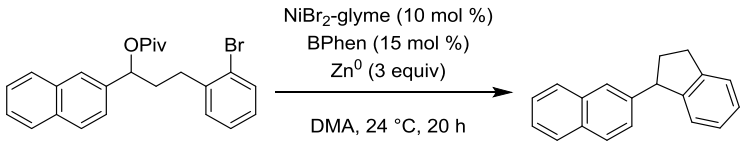
Cobalt co-catalysts have been employed in related reductive coupling reactions of benzylic alcohols. Including a cobalt co-catalyst slightly improves the yield of our nickel-catalyzed reductive coupling as well, although likely by changing the mechanism. In the presence of cobalt and the absence of nickel no reaction occurs.

Table 2.7. Effect of a Co(Pc) co-catalyst on an intermolecular cross-electrophile coupling reaction.

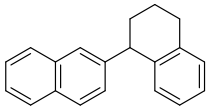


Ni loading (mol%)	Co(Pc) loading (mol%)	yield (%)
15	0	74
15	1.5	99
0	1.5	0

Characterization for products in Table 2.5

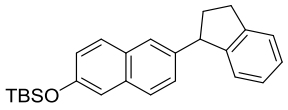


rac-**2.23**. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and *rac*-**2.22** (85.1 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (44.0 mg, 0.180 mmol, 90%). **TLC R_f** = 0.3 (100% hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80–7.71 (m, 3H), 7.63 (s, 1H), 7.44–7.36 (m, 2H), 7.30 (d, *J* = 7.5 Hz, 1H), 7.26 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.11 (t, *J* = 8.1 Hz, 1H), 6.95 (d, *J* = 7.6 Hz, 1H), 4.48 (t, *J* = 8.3 Hz, 1H), 3.12–2.92 (m, 2H), 2.65–2.55 (m, 1H), 2.19–2.07 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.1, 144.7, 143.1, 133.9, 132.7, 128.6, 127.98, 127.96, 126.99, 126.95, 126.82, 126.78, 126.3, 125.7, 125.4, 124.8, 52.1, 36.8, 32.3; **IR** (neat) 3050, 3018, 2937, 1599, 1506, 1477, 1457 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{19}H_{16}$ $(M)^+$ 224.1252, found 224.1257.

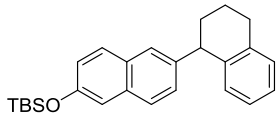


rac-**2.26**. The product was prepared according to general procedure E using $NiBr_2$•glyme (3.1 mg, 0.010 mmol, 10 mol %), bathophenanthroline (5.0 mg, 0.015 mmol, 15 mol %), Zn^0 (18.8 mg, 0.300 mmol, 3 equiv), DMA (0.40 mL), and **2.53** (44.0 mg, 0.100 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (10%

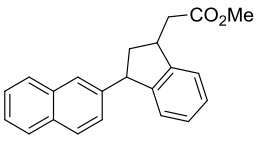
EtOAc/hexanes) to afford the title compound as a colorless oil (16 mg, 0.062 mmol, 62%). **TLC R_f** = 0.3 (hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84–7.73 (m, 3H), 7.53 (s, 1H), 7.49–7.41 (m, 2H), 7.29–7.23 (m, 1H), 7.21–7.11 (m, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 4.28 (t, *J* = 7.0 Hz, 1H), 3.02–2.84 (m, 2H), 2.27–2.18 (m, 1H), 2.03–1.90 (m, 2H), 1.86–1.75 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 145.1, 139.4, 137.8, 133.6, 132.3, 130.5, 129.2, 128.1, 127.79, 127.77, 127.5, 127.4, 126.2, 126.1, 125.9, 125.5, 46.0, 33.3, 30.0, 21.4; **IR** (neat) 3053, 2925, 2854, 1599, 1448 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{20}H_{18}$ $(M)^+$ 258.1408, found 258.1415.



rac-**2.27**. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and *rac*-**2.55** (111 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (51.7 mg, 0.138 mmol, 69%). **TLC R_f** = 0.3 (hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.6, 6.8 Hz, 2H), 7.57 (s, 1H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.23–7.10 (m, 4H), 7.05 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.97 (d, *J* = 7.5 Hz, 1H), 4.47 (t, *J* = 8.4 Hz, 1H), 3.13–2.93 (m, 2H), 2.66–2.57 (m, 1H), 2.13 (dq, *J* = 12.7, 8.8 Hz, 1H), 1.02 (s, 9H), 0.23 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.3, 147.1, 144.6, 140.8, 133.6, 129.5, 129.2, 127.2, 127.1 126.8, 126.6, 126.4, 125.2, 124.6, 122.4, 115.0, 51.8, 36.7, 32.1, 25.9, 18.5, –4.1; **IR** (neat) 2954, 2928, 1602, 1478, 1258 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{25}H_{30}OSi$ $(M)^+$ 374.2066, found 374.2054.

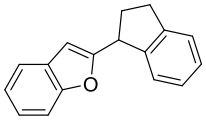


2.28. The product was prepared according to general procedure E using $NiBr_2$•glyme (3.1 mg, 0.010 mmol, 5.0 mol %), bathophenanthroline (5.0 mg, 0.015 mmol, 7.5 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.57** (113.9 mg, 0.200 mmol, 1.00 equiv). After 8 hours, the reaction vial was taken out of the oil bath. In the glovebox, an additional amount of $NiBr_2$•glyme (3.1 mg, 0.010 mmol, 5 mol %) and bathophenanthroline (5.0 mg, 0.015 mmol, 7.5 mol %) was added to the reaction mixture. The reaction vial was put back into the oil bath for the remainder of the reaction time. The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% pentane) to afford the title compound as a colorless oil (29.8 mg, 0.120 mmol, 58%). **TLC R_f** = 0.2 (pentane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (dd, *J* = 8.6, 6.9 Hz, 2H), 7.45 (s, 1H), 7.18 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.16–7.10 (m, 3H), 7.04 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.04–6.99 (m, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 4.23 (t, *J* = 6.7 Hz, 1H), 3.02–2.82 (m, 2H), 2.26–2.15 (m, 1H), 2.00–1.88 (m, 2H), 1.84–1.73 (m, 1H), 1.01 (s, 9H), 0.23 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.2, 142.8, 139.5, 137.7, 133.3, 130.4, 129.3, 129.04, 129.02, 127.6, 127.2, 126.8, 126.0, 125.7, 122.1, 114.8, 45.7, 33.2, 29.9, 25.8, 21.2, 18.3, -4.3; **IR** (neat) 3053, 2928, 1633, 1602, 1478, 1259 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{26}H_{32}OSi$ $(M)^+$ 388.2222, found 388.2227.



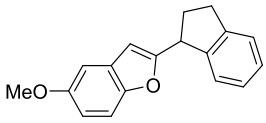
2.29. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0

(39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.59** (99.5 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the titlecompound as a colorless oil (29.7 mg, 0.094 mmol, 47%, 1:1 dr). **TLC R_f** = 0.6 (10% EtOAc in hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82–7.73 (m, 3H), 7.60–7.57 (m, 1H), 7.47–7.40 (m, 2H), 7.30 (d, *J* = 7.5 Hz, 1H), 7.27–7.22 (m, 2H), 7.18 (tdd, *J* = 7.5, 1.5, 0.5 Hz, 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 4.62 (t, *J* = 7.7 Hz, 1H), 3.87–3.78 (m, 1H), 3.72 (s, 3H), 2.76 (dd, *J* = 15.5, 6.1 Hz, 1H), 2.57 (dd, *J* = 15.4, 8.9 Hz, 1H), 2.51–2.36 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.3, 146.4, 146.3, 142.6, 133.7, 132.6, 128.5, 127.86, 127.85, 127.6, 127.4, 126.7, 126.5, 126.2, 125.7, 124.1, 123.1, 51.9, 49.9, 42.5, 40.6, 40.2; **IR** (neat) 3051, 2949, 1734, 1599, 1435, 1250, 1164 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{20}O_2Na$ $(M + Na)^+$ 339.1361, found 339.1367.

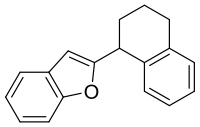


2.30. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.61** (83.1 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (neat Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (35.1 mg, 0.150 mmol, 75%). **TLC R_f** = 0.7 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54–7.50 (m, 1H), 7.48–7.44 (m, 1H), 7.36–7.29 (m, 2H), 7.27–7.19 (m, 4H), 6.43 (s, 1H), 4.61 (t, *J* = 7.7 Hz, 1H), 3.19–2.98 (m, 2H), 2.66–2.37 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.1, 155.2, 144.2, 143.5, 129.0, 127.5,

126.7, 125.1, 124.9, 123.6, 122.7, 120.7, 111.2, 102.4, 45.1, 32.4, 31.8; **IR** (neat) 3066, 3022, 2942, 1597, 1583, 1453, 1252 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{17}H_{14}ONH_4$ $(M + NH_4)^+$ 252.1388, found 252.1386.

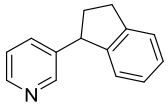


2.31. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.63** (89.0 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (41.2 mg, 0.156 mmol, 79%). **TLC R_f** = 0.3 (6% Et_2O/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31–7.26 (m, 2H), 7.25–7.14 (m, 3H), 6.95 (d, *J* = 2.6 Hz, 1H), 6.81 (dd, *J* = 8.9, 2.6 Hz, 1H), 6.34–6.32 (m, 1H), 4.53 (t, *J* = 7.7 Hz, 1H), 3.82 (s, 3H), 3.14–2.93 (m, 2H), 2.61–2.51 (m, 1H) 2.41–2.30 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.0, 156.0, 150.2, 144.2, 143.5, 129.5, 127.5, 126.7, 125.0, 124.9, 112.0, 111.5, 103.5, 102.6, 56.2, 45.1, 32.3, 31.8; **IR** (neat) 3068, 2929, 1615, 1599, 1474, 1202, 1030 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{18}H_{16}O_2NH_4$ $(M + NH_4)^+$ 282.1494, found 282.1494.

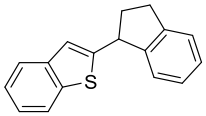


2.32. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.65** (85.9 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under

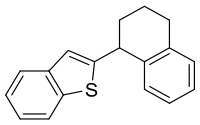
reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (29.8 mg, 0.120 mmol, 60%). **TLC R_f** = 0.8 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 (t, *J* = 8.3 Hz, 2H), 7.23 (td, *J* = 7.7, 1.4 Hz, 1H), 7.20–7.09 (m, 5H), 6.19 (s, 1H), 4.33 (t, *J* = 5.7 Hz, 1H), 2.91–2.78 (m, 2H), 2.35–2.08 (m, 2H), 1.95–1.75 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.6, 155.5, 137.6, 136.0, 130.1, 129.6, 128.9, 126.9, 125.9, 123.5, 122.7, 120.6, 111.2, 104.3, 39.9, 29.6, 28.7, 20.5; **IR** (neat) 3064, 2926, 2858, 1453, 1253 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{18}H_{16}OH$ $(M + H)^+$ 249.1279, found 249.1276.



2.35. The product was prepared according to general procedure E using $NiBr_2$•glyme (9.2 mg, 0.030 mmol, 15 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.67** (75.3 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (25.4 mg, 0.130 mmol, 65%). **TLC R_f** = 0.1 (12% EtOAc: hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 1.8 Hz, 1H), 8.50 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.45 (dt, *J* = 7.9, 2.0 Hz, 1H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.25–7.20 (m, 2H), 7.16 (t, *J* = 7.4 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 1H), 4.37 (t, *J* = 8.2 Hz, 1H), 3.13–2.95 (m, 2H), 2.63 (dtd, *J* = 12.8, 7.9, 7.9, 3.9 Hz, 1H), 2.08 (dq, *J* = 12.7, 8.7 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.0, 148.0, 145.7, 144.3, 140.8, 135.3, 127.0, 126.6, 124.7, 124.6, 123.6, 49.0, 36.5, 31.9; **IR** (neat) 3020, 2925, 1574, 1478, 1423 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{13}NH$ $(M + H)^+$ 196.1126, found 196.1119.

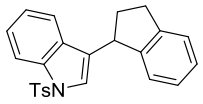


2.33. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.69** (86.3 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% pentane) to afford the title compound as a colorless oil (37.6 mg, 0.150 mmol, 75%). **TLC R_f** = 0.5 (pentane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.32–7.14 (m, 6H), 7.06 (s, 1H), 4.68 (t, *J* = 7.8 Hz, 1H), 3.14–2.90 (m, 2H), 2.66 (dtd, *J* = 12.6, 7.9, 4.6 Hz, 1H), 2.25 (dq, *J* = 12.7, 7.8 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.2, 145.6, 143.9, 140.2, 139.7, 127.4, 126.8, 125.2, 124.8, 124.4, 123.9, 123.2, 122.5, 120.9, 47.4, 36.6, 31.9; **IR** (neat) 3060, 2922, 1567, 1475, 1456, 1133 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{17}H_{14}OH$ $(M + H)^+$ 251.0894, found 251.0894.

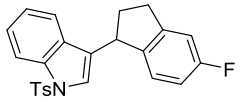


2.34. The product was prepared according to general procedure E using $NiBr_2$•glyme (9.2 mg, 0.030 mmol, 15 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.71** (89.0 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (29.1 mg, 0.110 mmol, 55%). **TLC R_f** = 0.5 (hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H),

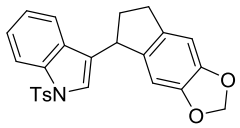
7.32–7.27 (m, 1H), 7.25–7.08 (m, 5H), 6.87 (s, 1H), 4.46 (t, *J* = 5.9 Hz, 1H), 2.96–2.79 (m, 2H), 2.29–2.07 (m, 2H), 2.00–1.75 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.0, 139.8, 139.6, 137.9, 137.1, 130.2, 129.3, 126.6, 125.8, 124.1, 123.6, 123.0, 122.3, 122.1, 41.2, 32.8, 29.4, 20.4; **IR** (neat) 3060, 2915, 1456, 1435, 1308, 1129 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{18}H_{16}S$ $(M)^+$ 264.0973, found 264.0966.



2.36. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.73** (114 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (59.4 mg, 0.153 mmol, 75%). **TLC R_f** = 0.4 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.34–7.26 (m, 3H), 7.25–7.09 (m, 6H), 7.01 (d, *J* = 7.6 Hz, 1H), 4.53 (t, *J* = 8.1 Hz, 1H), 3.08–2.92 (m, 2H), 2.55 (dtd, *J* = 12.4, 7.8, 4.6 Hz, 1H), 2.32 (s, 3H), 2.12 (dq, *J* = 12.6, 8.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.99, 144.98, 144.2, 136.0, 135.5, 130.6, 130.0, 127.2, 127.0, 126.7, 126.4, 124.90, 124.86, 124.8, 123.3, 123.2, 120.4, 114.1, 42.3, 34.2, 31.9, 21.8; **IR** (neat) 3065, 2926, 2850, 1596, 1446, 1368, 1171 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{24}H_{21}NO_2SNa$ $(M + Na)^+$ 410.1191, found 410.1193.



2.37 The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.75** (117.3 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (68.1 mg, 0.168 mmol, 84%). **TLC R_f** = 0.4 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.6 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.32–7.27 (m, 2H), 7.23–7.20 (m, 3H), 7.16 (td, *J* = 7.7, 0.9 Hz, 1H), 7.01–6.97 (m, 1H), 6.92 (dd, *J* = 8.3, 5.4 Hz, 1H), 6.81 (td, *J* = 9.0, 2.3 Hz, 1H), 4.49 (t, *J* = 8.1 Hz, 1H), 3.05–2.91 (m, 2H), 2.57 (dtd, *J* = 12.6, 8.0, 4.7 Hz, 1H), 2.35 (s, 3H), 2.17 (dq, *J* = 12.7, 8.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.3 (d, *J* = 243 Hz), 146.2 (d, *J* = 8 Hz), 144.8, 140.2 (d, *J* = 2 Hz), 135.8, 135.3, 130.2, 129.9, 126.8, 125.5 (d, *J* = 9 Hz), 126.0, 124.7, 123.1, 123.0, 120.1 113.9, 113.4 (d, *J* = 22 Hz), 111.7 (d, *J* = 22 Hz), 41.4, 34.4, 31.7, 21.6; **IR** (neat) 2923, 1595, 1482, 1368, 1172, 1121 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{24}H_{20}FNO_2SNa$ $(M + Na)^+$ 428.1096, found 428.1081.

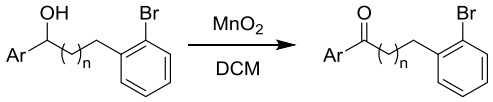


2.38. The product was prepared according to general procedure E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and **2.77** (122.5 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated

under reduced pressure. The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a colorless oil (38.0 mg, 0.088 mmol, 44%). **TLC R_f** = 0.5 (15% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.35–7.26 (m, 2H), 7.24–7.12 (m, 4H), 6.76 (s, 1H), 6.43 (s, 1H), 5.92 (d, *J* = 8.3 Hz, 2H), 4.43 (t, *J* = 7.7 Hz, 1H), 2.97–2.81 (m, 2H), 2.60–2.48 (m, 1H), 2.33 (s, 3H), 2.13 (dq, *J* = 12.7, 7.9 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.3, 146.9, 145.0, 137.7, 137.0, 136.1, 135.5, 130.5, 130.0, 127.0, 126.6, 124.9, 123.3, 123.2, 120.4, 114.1, 105.33, 105.31, 101.2, 42.2, 34.6, 31.7, 21.8; **IR** (neat) 2926, 1596, 1474, 1367, 1172, 1120, 1021 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{25}H_{21}NO_4SNa$ $(M + Na)^+$ 454.1089, found 454.1085.

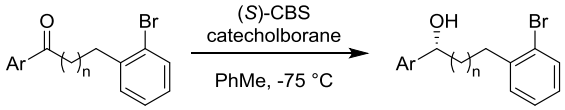
Synthesis and characterization of enantioenriched esters for Table 2.6 and stereospecific intramolecular reductive cross-electrophile coupling reaction

General Procedure G: *Manganese dioxide oxidation of benzylic alcohols*

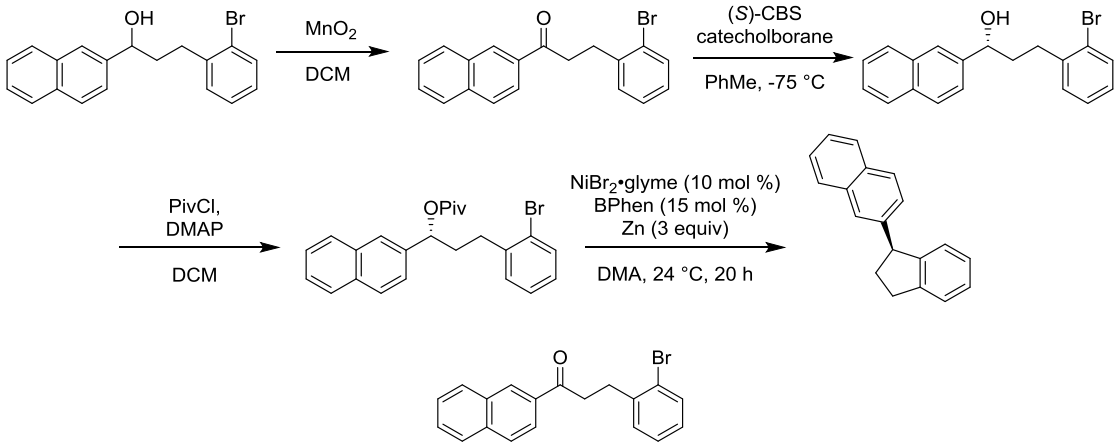


The product was prepared according to a modified procedure reported by Wipf.[12] To a solution of *rac*-benzylic alcohol (1.0 equiv) in CH_2Cl_2 (30 mL) was added in a single portion MnO_2 (8 equiv). The reaction was allowed to stir overnight at room temperature. The resulting slurry was filtered through celite, and the celite was washed with CH_2Cl_2. Solvent was removed under reduced pressure to afford the pure title compound.

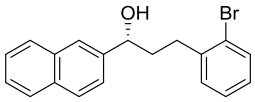
General Procedure H*: CBS reduction of benzylic ketones*



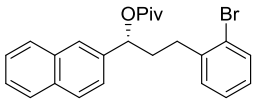
The product was prepared according to a modified procedure reported by Okamura.[13] In a glovebox, (*S*)-Me-CBS (0.100 equiv) was added to a 50 mL flame-dried round bottom flask equipped with a stir bar. The flask was capped with a septum and removed from the box. Benzylic ketone (1.0 equiv) was added to the flask as a solution in PhMe (20 mL). The reaction was then cooled to –78 °C and catecholborane (2.0 equiv) was added dropwise. After stirring for 24 h at –78 °C, the reaction was warmed to ambient temperature and quenched with water. Saturated $NaHCO_3$ (15 mL) was added to the reaction flask and the mixture was extracted with EtOAc (3 x 30 mL). The combined organics were washed with brine, dried over MgSO4, and concentrated under reduced pressure. The product was purified by flash column chromatography to afford the title compound.



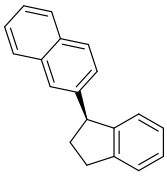
2.78. The product was prepared according to general procedure G using *rac*-**2.49** (1.32 g, 3.86 mmol, 1.0 equiv), MnO_2 (2.08 g, 24.0 mmol, 6.22 equiv). The product was purified by flash column chromatography to afford the pure title compound as a yellow oil (0.962 g, 2.84 mmol, 74%). **TLC R_f** = 0.6 (10% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.04 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.86 (t, *J* = 8.6 Hz, 2H), 7.61–7.50 (m, 3H), 7.34 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.27–7.21 (m, 1H), 7.08 (td, *J* = 7.8, 1.8 Hz, 1H), 3.44 (t, *J* = 8.3 Hz, 2H), 3.24 (t, *J* = 8.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 199.1, 140.9, 135.8, 134.3, 133.2, 132.8, 131.1, 130.0, 129.8, 128.7 (2C), 128.3, 128.0, 127.9, 127.0, 124.6, 124.1, 39.0, 31.2; **IR** (neat) 3057, 2928, 1677, 1626, 1469, 1182, 1122 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{15}BrONa$ $(M + Na)^+$ 361.0204, found 361.0218.



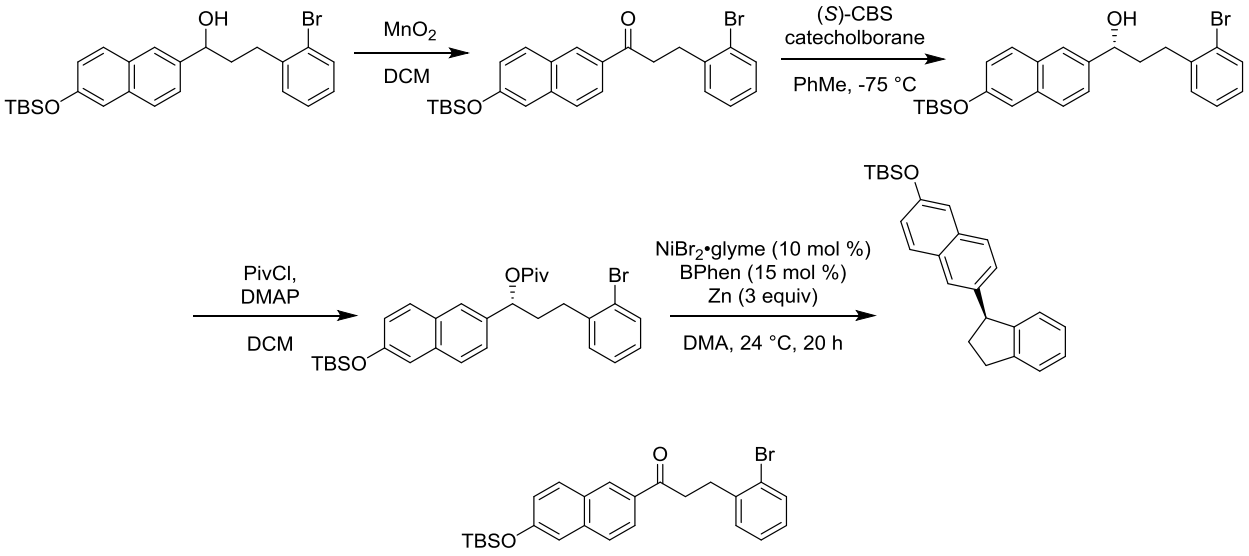
(*R*)**-2.49**. The product was prepared according to a general procedure H using (*S*)-Me-CBS (98 mg, 0.28 mmol, 0.100 equiv), **2.78** (0.96 g, 2.8 mmol, 1.0 equiv), and catecholborane (0.59 mL, 5.5 mmol, 2.0 equiv). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.91 g, 2.6 mmol, 94%, 96% ee). Analytical data is consistent with the values listed for **2.49** (vide supra).



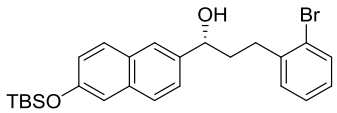
(*R*)-**2.22.** The product was prepared according to general method B using (*R*)-**2.49** (0.60 g, 1.7 mmol, 1.0 equiv), pivaloyl chloride (0.24 mL, 1.9 mmol, 1.1 equiv) and dimethylaminopyridine (0.24 g, 1.9 mmol, 1.1 equiv). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.49 g, 1.1 mmol, 66%). Analytical data is consistent with the values listed for *rac*-**2.22** (vide supra). $[\alpha]^{28}_{D}$ +39 (c 1.7, $CHCl_3$); **SFC** analysis (OD-H, 6% IPA, 2.5 mL/min) indicated 96% ee: t_R (major) = 13.6 minutes, t_R (minor) = 14.9 minutes.



(*S*)-**2.25**. The product was prepared according to general method E using $NiBr_2$•glyme (6.2 mg, 0.020 mmol, 10 mol %), bathophenanthroline (10.0 mg, 0.030 mmol, 15 mol %), Zn^0 (39.6 mg, 0.600 mmol, 3 equiv), DMA (0.60 mL), and (*R*)-**2.22** (85.1 mg, 0.200 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (44.0 mg, 0.180 mmol, 90%, 88% ee). Analytical data is consistent with the values listed for *rac*-**2.25** (vide supra); $[\alpha]^{28}_{D}$ +10 (c 0.9, $CHCl_3$); **SFC** analysis (OD-H, 6.0% IPA, 2.5 mL/min) indicated 88% ee: t_R (major) = 11.3 minutes, t_R (minor) = 10.5 minutes.

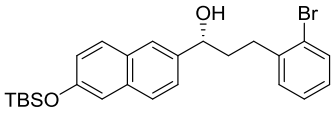


2.79. The product was prepared according to general procedure G using *rac*-**2.54** (0.94 g, 2.0 mmol, 1.0 equiv), MnO_2 (1.8 g, 20.0 mmol, 10 equiv). The product was purified by flash column chromatography to afford the pure title compound as a yellow oil (0.56 g, 1.2 mmol, 60%). **TLC R_f** = 0.4 (5% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.98 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.78 (d, *J* = 9.0 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.51 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.30 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.22–7.17 (m, 2H), 7.10 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.02 (td, *J* = 7.6, 1.6 Hz, 1H), 3.37 (t, *J* = 7.9 Hz, 2H), 3.20 (t, *J* = 7.8 Hz, 2H), 1.00 (s, 9H), 0.25 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 198.5, 156.0, 140.8, 137.3, 133.0, 132.4, 131.4, 130.9, 129.8, 128.2, 128.1, 127.8, 127.2, 124.5, 124.4, 123.1, 115.0, 38.6, 31.1, 25.8, 18.4, -4.1; **IR** (neat) 3056, 2954, 1680, 1598, 1467, 1256 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{25}H_{29}BrO_2Si$ $(M)^+$ 468.1120, found 468.1132.

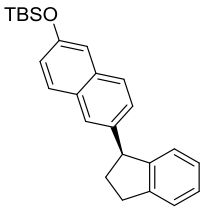


(*R*)**-2.54**. The product was prepared according to a general procedure H using (*S*)-Me-CBS (66 mg, 0.24 mmol, 0.200 equiv), **2.79** (0.56 g, 1.2 mmol, 1.0 equiv), and catecholborane

(0.26 mL, 2.4 mmol, 2.0 equiv). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.42 g, 0.90 mmol, 75%). Analytical data is consistent with the values listed for **2.54** (vide supra).

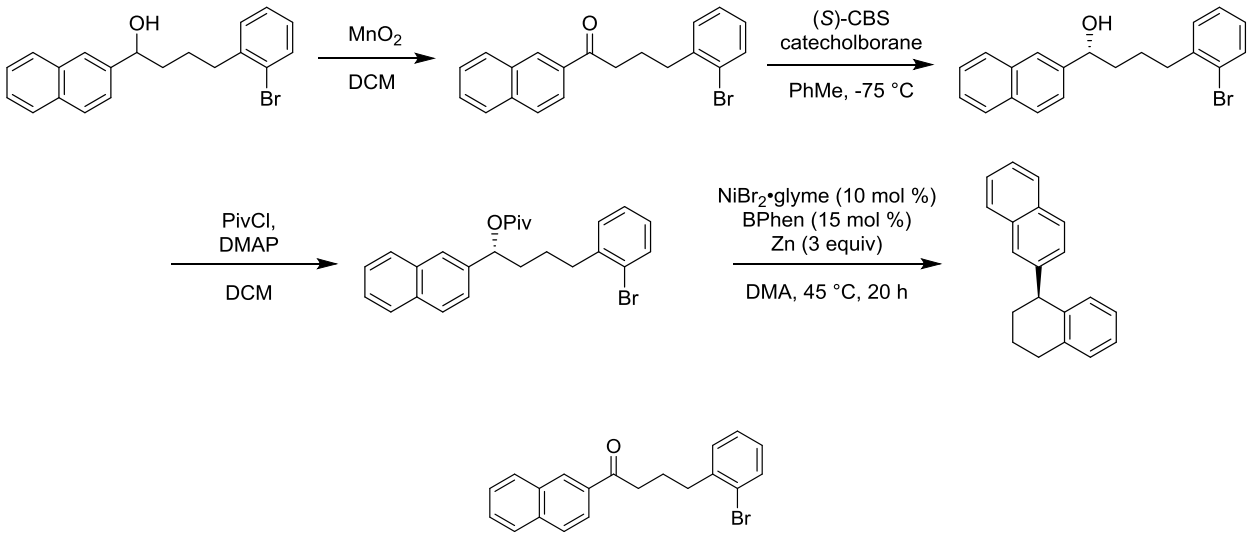


(*R*)**-2.55.** The product was prepared according to general procedure B using (*R*)-**2.54** (0.40 g, 0.85 mmol, 1.0 equiv), pivaloyl chloride (0.12 mL, 0.98 mmol, 1.1 equiv) and dimethylaminopyridine (0.14 g, 1.0 mmol, 1.1 equiv). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.23 g, 0.41 mmol, 52%). Analytical data is consistent with the values listed for *rac*-**2.55** (vide supra). $[\alpha]^{27.5}_{D}$ +67 (c 3.7, $CHCl_3$); **SFC** analysis (OD-H, 8% IPA, 2.5 mL/min) indicated 96% ee: t_R (major) = 8.1 minutes, t_R (minor) = 8.9 minutes.

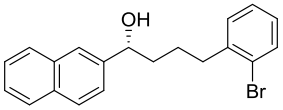


(*S*)**-2.27**. The product was prepared according to general procedure E using $NiBr_2$•glyme (3.1 mg, 0.010 mmol, 10 mol %), bathophenanthroline (5.0 mg, 0.015 mmol, 15 mol %), Zn^0 (18.6 mg, 0.300 mmol, 3 equiv), DMA (0.40 mL), and (*R*)**-2.25** (55.5 mg, 0.100 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (26 mg, 0.069 mmol, 69%, 92% ee). Analytical data is consistent with the values listed for *rac*-**2.27** (vide supra); $[\alpha]^{26.6}_{D}$ +91 (c

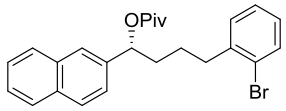
0.55, $CHCl_3$); **SFC** analysis (AD-H, 7.0% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 7.1 minutes, t_R (minor) = 8.6 minutes.



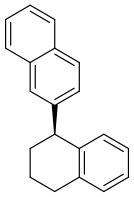
2.80. The product was prepared according to general procedure G using *rac*-**2.52** (1.79 g, 5.03 mmol, 1.0 equiv), MnO_2 (4.4 g, 51 mmol, 10 equiv). The product was purified by flash column chromatography to afford the pure title compound as a yellow oil (1.22 g, 3.69 mmol, 73%). **TLC R_f** = 0.5 (12% Et_2O/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 8.03 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.97–7.93 (m, 1H), 7.91–7.85 (m, 1H), 7.58 (dddd, *J* = 18.5, 7.7, 6.6, 1.3 Hz, 3H), 7.29–7.22 (m, 3H), 7.07 (ddd, *J* = 8.0, 7.0, 2.1 Hz, 1H), 3.17 (t, *J* = 7.3 Hz, 2H), 2.90 (t, *J* = 7.7 Hz, 2H), 2.20–2.11 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.0, 141.2, 135.7, 134.4, 133.0, 132.7, 130.7, 129.86, 129.74, 128.62, 128.58, 128.0, 127.9, 127.7, 126.9, 124.8, 124.1, 38.0, 35.6, 24.7; **IR** (neat) 2939, 2893, 1679, 1620, 1438, 1165 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{20}H_{17}BrOH$ $(M + H)^+$ 353.0541, found 353.0442.



(*R*)-**2.52**. The product was prepared according to a general procedure H using (*S*)-Me-CBS (67 mg, 0.29 mmol, 0.100 equiv), **2.80** (1.0 g, 2.9 mmol, 1.0 equiv), and catecholborane (0.62 mL, 5.8 mmol, 2.0 equiv). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.83 g, 2.3 mmol, 80%). Analytical data is consistent with the values listed for **2.52** (vide supra).



(*R*)-**2.53.** The product was prepared according to general procedure B using (*R*)-**2.52** (0.728 g, 2.05 mmol, 1.0 equiv), pivaloyl chloride (0.30 mL, 2.4 mmol, 1.2 equiv) and dimethylaminopyridine (0.33 g, 2.7 mmol, 1.3 equiv). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.23 g, 0.41 mmol, 52%). Analytical data is consistent with the values listed for *rac*-**2.53** (vide supra). $[\alpha]^{27.6}_{D}$ +84 (c 2.1, $CHCl_3$); **SFC** analysis (OD-H, 6% IPA, 2.5 mL/min) indicated 94% ee: t_R (major) = 18.2 minutes, t_R (minor) = 21.6 minutes.



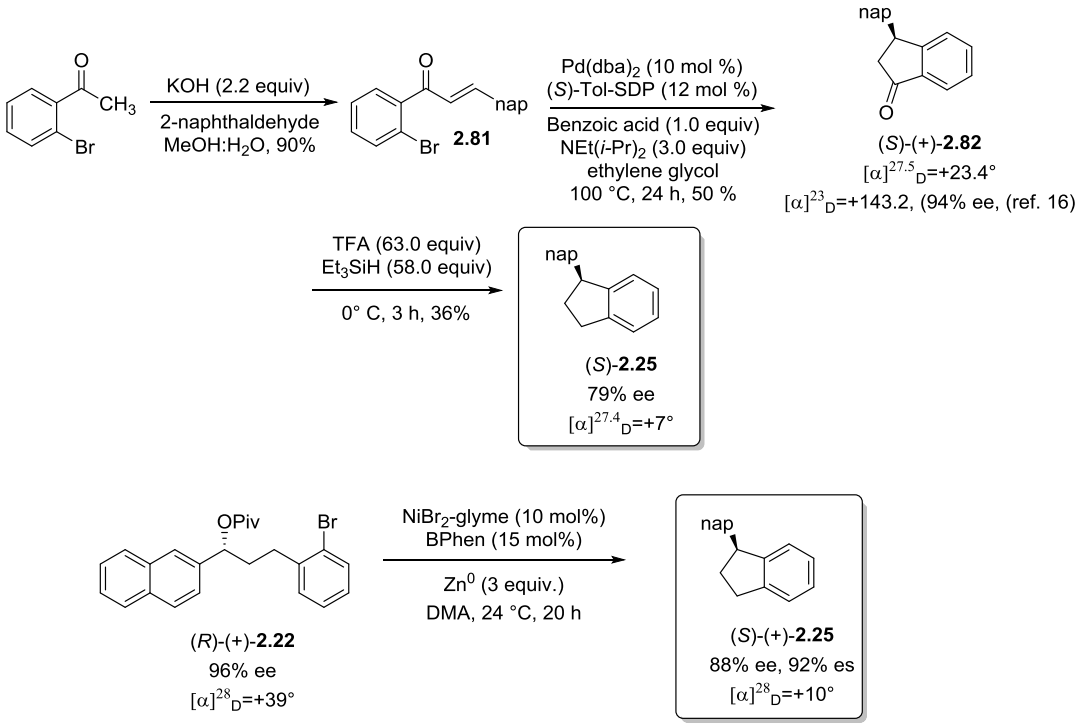
(*S*)-**2.56**. The product was prepared according to general procedure E using $NiBr_2$•glyme (3.1 mg, 0.010 mmol, 10 mol %), bathophenanthroline (5.0 mg, 0.015 mmol, 15 mol %), Zn^0 (18.6 mg, 0.300 mmol, 3 equiv), DMA (0.40 mL), and (*R*)-**2.53** (44.0 mg, 0.100 mmol, 1.00 equiv). The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (16 mg, 0.062 mmol, 62%, 78% ee).

Analytical data is consistent with the values listed for *rac*-**2.26** (vide supra); $[\alpha]^{27.6}_{D}$ +43 (c 0.66, $CHCl_3$); **SFC** analysis (OD-H, 6.0% IPA, 2.5 mL/min) indicated 78% ee: t_R (major) = 12.5 minutes, t_R (minor) = 16.8 minutes.

Stereochemical proof

Enantioenriched alcohol (*R*)-**2.49** was prepared by enantioselective CBS reduction (vida supra). Absolute configuration of (*R*)-**2.49** and (*R*)-**2.22** were assigned based on the accepted model for selectivity in CBS reductions.[14]

The absolute configuration of the enantioenriched indane **2.25** was assigned by derivatization of known enantioenriched indanone (*S*)-**2.82** to indane (*S*)-**2.25.** Enantioenriched (*S*)-**2.82** was prepared by an asymmetric reductive Heck reaction as reported by Zhou. The stereochemistry was verified by comparison of the optical rotation to the literature value. Reduction to indane (*S*)-**2.25** and subsequent comparison of the optical rotation and SFC data matched that of (*S*)-**2.25** synthesized by the reductive cross-electrophile coupling reaction. This product corresponds to net inversion at the benzylic center in the reductive cross-electrophile coupling reaction.



2.81. The product was prepared according to a modified procedure reported by Zhou.[15] To a 100 mL round bottom flask equipped with a stir bar was added 2'-bromoacetophenone (0.67 mL, 5.0 mmol, 1.0 equiv), naphthaldehyde (0.78 g, 5.0 mmol, 1.0 equiv), KOH (0.62 g, 11 mmol, 2.2 equiv), and MeOH/H_2O (15:15 mL). The reaction was stirred overnight at room temperature. The resulting solid was filtered, washed with MeOH/H_2O, and dried by vacuum filtration to afford the title compound as a solid (1.3 g, 4.5 mmol, 90 % yield).

(*S*)-**2.82.** The product was prepared according to a modified procedure reported by Zhou.[15] In a glovebox, a flame-dried 7 mL vial equipped with a stir bar was charged with $Pd(dba)_2$ (14 mg, 0.025 mmol, 10 mol %), (*S*)- Tol-SDP (18 mg, 0.03 mmol, 12 mol %), benzoic acid (30 mg, 0.25 mmol, 1.0 equiv) and degassed ethylene glycol (1.25 mL). After stirring for 10 min, N-diisopropylethylamine (130 µL, 1.5 mmol, 3.0 equiv) and **2.81** (85 mg, 0.25 mmol, 1.0 equiv) were added and the mixture was taken out of the glove box. The reaction was stirred for 24 h in a pre-warmed oil bath at 100 °C then allowed to cool to room temperature. The reaction mixture was eluted through a silica plug (with Et_2O) and concentrated under reduced pressure. The product was purified by flash column chromatography (0%-10% Et_2O/hexanes) to afford the title compound as a colorless oil (27 mg, 0.13 mmol, 50% yield). Analytical data is consistent with literature values.[16] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.9 Hz, 1H), 7.84–7.77 (m, 3H), 7.67 (s, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.50–7.43 (m, 3H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.15 (dd, *J* = 8.4, 1.6 Hz, 1H), 4.77 (dd, *J* = 8.2, 3.7 Hz, 1H), 3.31 (dd, *J* = 19.3, 8.1 Hz, 1H), 2.79 (dd, *J* = 19.3, 3.9 Hz, 1H).

(*S*)-**2.25**. A 20 ml scintillation vial was charged with **2.82** (27 mg, 0.13 mmol, 1.0 equiv), dissolved in TFA (0.5 ml) and cooled to 0 °C in an ice bath. Et_3SiH (1.0 ml) was added dropwise over the course of 20 min and the reaction was allowed to stir for 3 h at 0 °C. The reaction was then allowed to warm to room temperature, was concentrated under reduced pressure, and the residue was taken up and purified by flash column chromatography (100% hexanes) to afford the title compound as a colorless oil (9.2 mg, 0.038 mmol, 36% yield, 79% ee). Analytical data is consistent with the values listed for (*S*)-**2.25** (vide supra). $[\alpha]^{27.4}_D$ +7 (c 0.46, $CHCl_3$); **SFC** analysis (OD-H, 6.0% IPA, 2.5 mL/min) indicated 79% ee: t_R (major) = 11.4 minutes, t_R (minor) = 10.6 minutes.

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Chapter 3

Directed Nickel-Catalyzed Hydroarylation of Alkynes with Boronic Acids

3.1 Introduction

Aryl-substituted alkenes are a common functional group present in pharmaceutical agents[1] and natural products[2], and are commonly used as synthons in synthesis[3] and polymer chemistry[4] (Figure 3.1). Hydroarylation reactions of alkynes offer a simple manifold for the facile synthesis of such substituted alkenes. The major challenges associated with the hydroarylation of alkynes has primarily been control of regioisomeric ratios and control of *E/Z* stereochemistry of the resulting olefin without relying on steric or electronic biases in the substrate.[5] The first example among transition metal-catalyzed reactions was reported by Hayashi and co-workers, where they demonstrated that rhodium(I) catalysts cleanly furnished hydroarylated products when using symmetrical alkynes and arylboronic acids; however, employing unsymmetrical starting materials that lacked strong electronic or steric bias resulted in mixtures of regioisomers (Scheme 3.1a).[6] The Lautens group judiciously addressed this problem with the use of pyridine or alcohol directing groups that could deliver hydroarylated products with excellent regio- and stereocontrol (Scheme 3.1b, c).[7]

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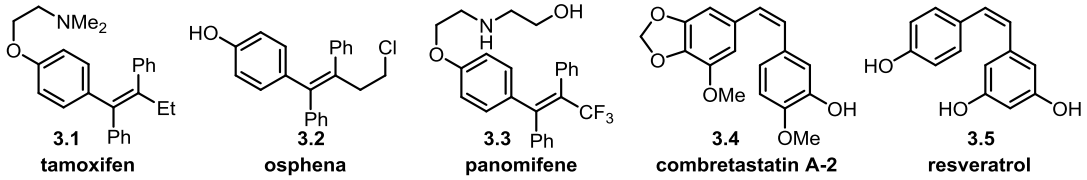
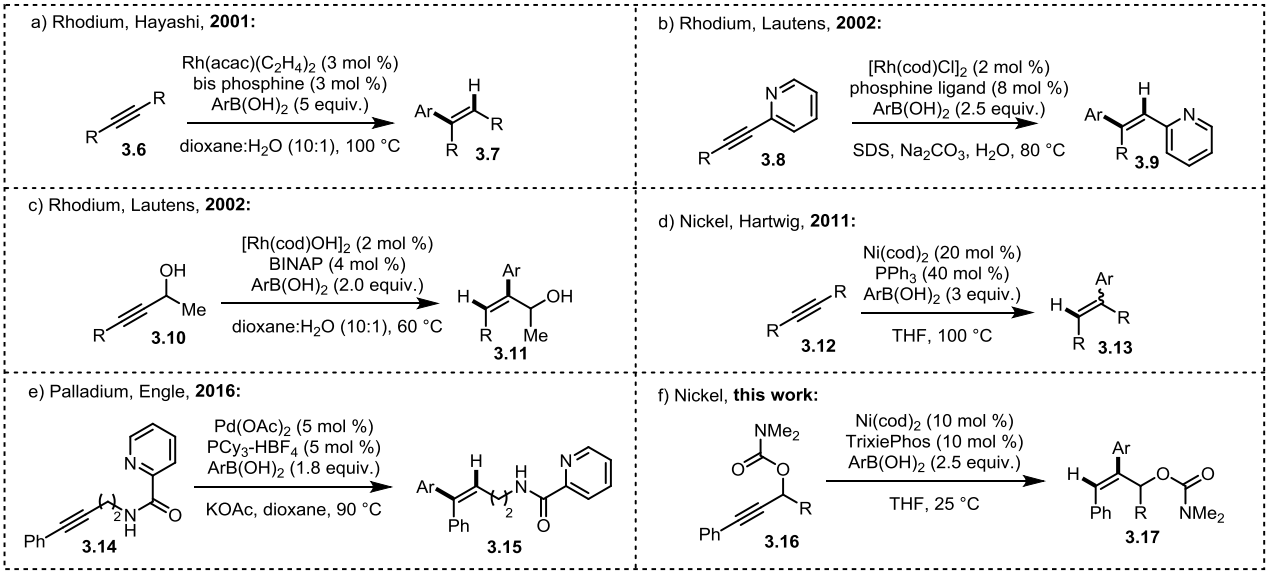
Efforts to employ group 10 metals such as palladium and nickel in hydroarylation reactions have been explored and found to proceed with low regio- and stereocontrol (Scheme 3.1d).[8] Similar to the first rhodium-catalyzed reactions, Oh and co-workers showed that even with electronically biased systems, the palladium-catalyzed hydroarylation proceeded with low regiocontrol.[9] However, Engle and co-workers have recently reported the use of pendant directing groups that address these obstacles with great success. (Scheme 3.1e).[10] To date, hydroarylation reactions that use nickel catalysts also suffer from both regio- and stereocontrol of the resulting olefin. Further development of these reactions using a similar directing group strategy would be a valuable addition to alkyne functionalization reactions.

Figure 3.1. Examples of stilbene-containing pharmaceuticals and natural products



[8]a) Hartwig, J. F. *Science* **2011**, 333, 1423. (b)
Shirakawa, E.; Takahashi, G.; Tsuchimoto, T.; Kawakami, Y. *Chem. Commun.* **2001**, 2688. (c)
Xu, X.; Chen, J.; Gao, W.; Wu, H.; Ding, J.; Su, W. *Tetrahedron* **2010**, 66, 2433.
[9] Oh, C. H.; Jung, H. H.; Kim, K. S.; Kim, N. *Angew. Chem., Int. Ed.* **2003**, 42, 805.
[10] Liu, Z.; Derosa, J.; Engle, K. M. *J. Am. Chem. Soc.*, **2016**, *138*, 13076.

Scheme 3.1. Summary of transition metal-catalyzed hydroarylations using boronic acids



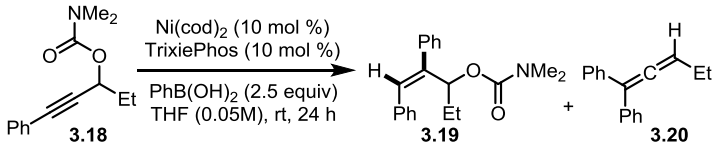
Moving forward, efforts in the Jarvo laboratory have focused on the use of base metals and propargylic directing groups to address this challenge. Previous work in our group has demonstrated that carbamates can serve as directing groups for nickel catalysts in Suzuki coupling reactions. Thus, we hypothesized that propargylic carbamates could also serve as directing groups for hydroarylation reactions. In this chapter, we report a regio- and stereoselective hydroarylation of alkynes under mild conditions, complimenting the recent developments made by Hartwig and Engle (Scheme 3.1f)

3.2 Development of Nickel-Catalyzed Directed Hydroarylation

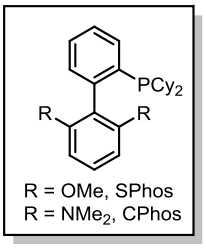
We began our investigation by examining a range of propargylic directing groups including pivalates, carbonates and carbamates. Luke Hanna in our laboratory showed that pivalates and carbonates provided undesired allene **3.20** with small amounts of desired product. A mixture of byproducts and only modest yields of hydroarylated product was observed when free secondary propargyl alcohols were used. Interestingly, nearly identical results were observed without the alcohol or directing groups present (Table 3.1, entries 4

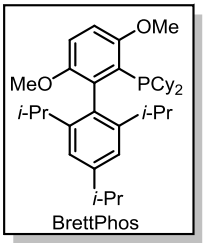
and 5). Employing bulky carbamates such as diisopropyl carbamate led to a severe decrease in product formation. However, moving to smaller alkyl groups such as pyrrolidine carbamate led to an increase in product formation, with a dimethyl carbamate giving the highest yield. A range of ligands were investigated during the optimization and the reaction was found to perform well with monodentate phosphine ligands. Further studies into ligand identity uncovered that catalysts with sterically encumbered Buchwald-type ligands such as TrixiePhos were uniquely capable of suppressing the formation of allene while still furnishing the desired product in good yields. The reaction performs much better in the absence of bases such as *t*-BuOK and K_3PO_4, bases that are commonly used for transmetallation of Suzuki reagents (Table 3.1, entry 13). Furthermore, the addition of one equivalent of LiCl to the reaction mixture led to a complete shutdown of reactivity and recovery of starting material (Table 3.1, entry 12). Additionally, during an investigation of solvents, it was found that the use of strongly coordinating solvents such as DMF also shut down reactivity (Table 3.1, entry 11).

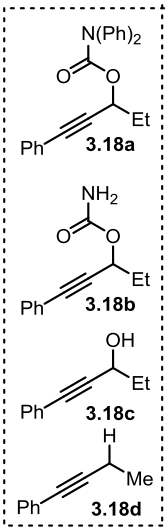
Table 3.1. Optimization of hydroarylation reaction conditions



entry	variation from standard conditions	recovered **3.18** (%)	**3.19** (%)	**3.20** (%)
1	none	<2	70	<2
2	**3.18a** instead of **3.18**	57	16	14
3	**3.18b** instead of **3.18**	81	8	<2
4	**3.18c** instead of **3.18**	73	20	<2
5	**3.18d** instead of **3.18**	57	19	<2
6	no nickel	100	0	0
7	no ligand	8	55	15
8	SPhos instead of TrixiePhos	16	63	2
9	BrettPhos instead of TrixiePhos	11	50	9
10	CPhos instead of TrixiePhos	12	50	12
11	DMF instead of THF	86	13	7
12	1 equivalent of LiCl	100	0	0
13	1 equivalent of K_3PO_4	10	40	6
14	PhBpin instead of $PhB(OH)_2$	78	8	4
15	$(PhBO)_3$ instead of $PhB(OH)_2$	65	19	6





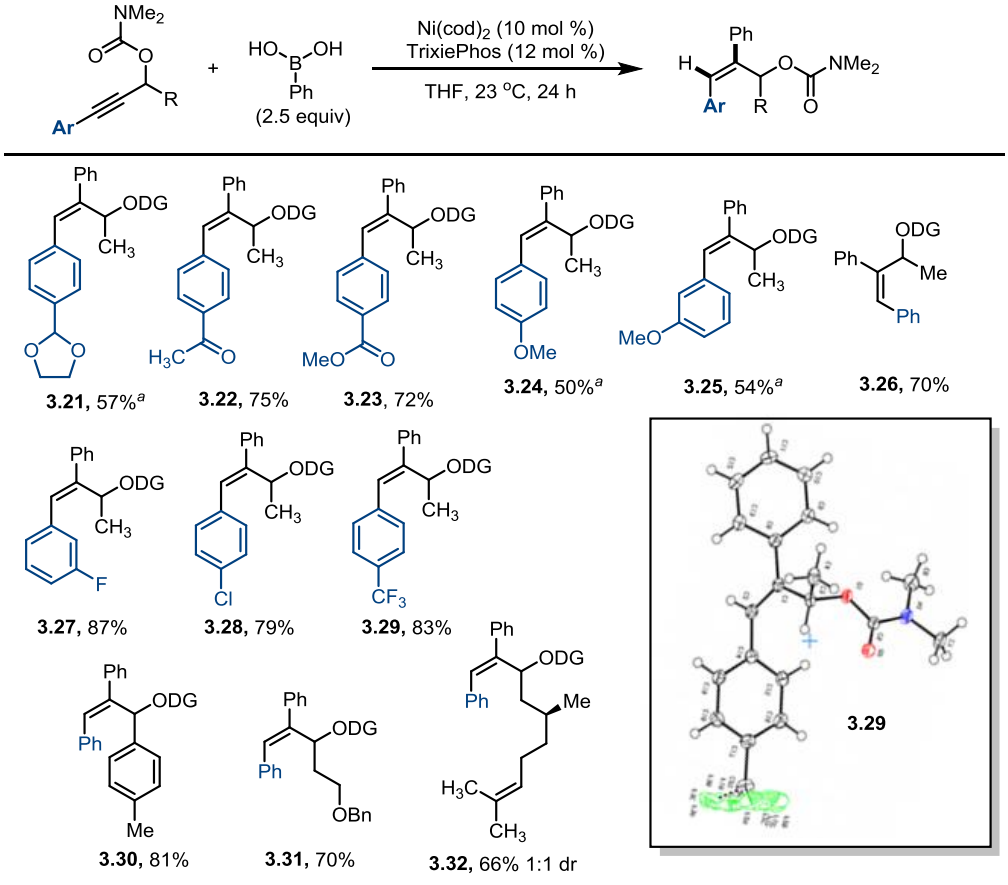


3.3 Scope of Substrates for Hydroarylation

Having identified suitable reaction conditions for model substrate **3.18**, we turned our attention to interrogating a range of internal alkynes (Table 3.2). Substrates bearing electron-deficient arenes provided the highest yields of hydroarylated product at room temperature while electron-rich arenes required heating to obtain high yields (Table 3.2, entry **3.22**, **3.23**, **3.29-3.29**). Ketone and ester substituents were well tolerated; however, subjecting the free aldehyde to the reaction conditions resulted in low yields of the desired product. Gratifyingly, a protected aldehyde could still be incorporated as an acetal, although heating was required. Aryl fluorides and chlorides were well tolerated and did not undergo the corresponding Suzuki cross-coupling, allowing for further orthogonal functionalization (Table 3.2, entry **3.27** and **3.28**). X-ray crystallographic analysis of a single crystal of trifluoromethyl-substituted **3.29** was obtained, supporting the assigned structure and *cis*-hydroarylation. Benzyl-protected alcohols on the alkyl moiety of the starting materials were

carried through the reaction without complications. Pendant alkenes and branched alkyl substituents did not hinder reactivity, forming citronellal-derived product **3.32** in good yield. Surprisingly, sp^2-hybridized side chains yielding both allylic and benzylic-activated carbamates did not undergo Tsuji–Trost type reaction and provided the desired products in high yields (entry **3.30**).

Table 3.2. Investigation of scope in alkynes for hydroarylation



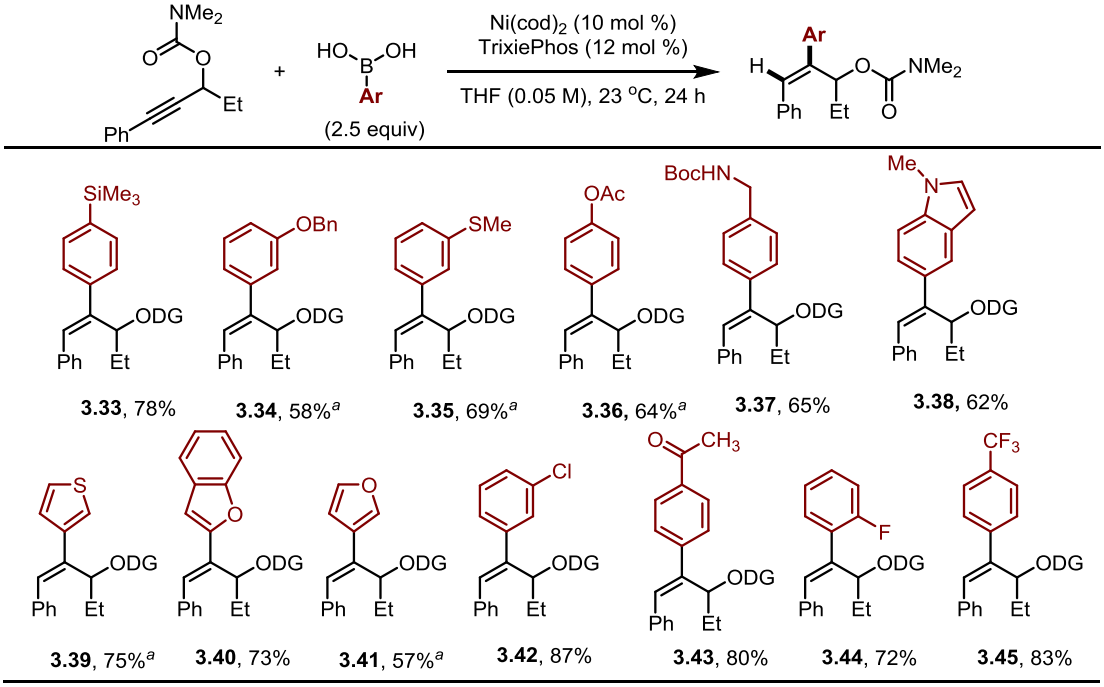
[a]Reaction run at 80 °C. DG = N,N'-dimethylcarbamoyl

3.4 Scope of Arylboronic Acid for Hydroarylation

Next, we turned our attention towards investigating the scope in arylboronic acid (Table 3.3). Again, a similar trend in sensitivity to electronics was observed where electron-deficient arylboronic acids provided the highest yields at room temperature while electron-

rich arylboronic acids required heating. A variety of functional groups were well-tolerated, allowing for the incorporation of phenyl TMS, acetoxy, ether, thioether, and trifluoromethyl moieties (Table 3.3, entries **3.33**-**3.36** & **3.45**). Ketones and Boc-protected amines were also well tolerated and provided hydroarylated product in good yields. Chloro- and fluorophenyl products, such as **3.42** and **3.44**, did not participate in either hydrodehalogenation or cross-coupling. Furthermore, boronic acids containing heterocycles such as furan and thiophene underwent hydroarylation smoothly at elevated temperatures while benzofuran and indole boronic acids could be coupled at room temperature (Table 3.3, entries **3.38** and **3.40**).

Table 3.3. Investigation of scope in arylboronic acid

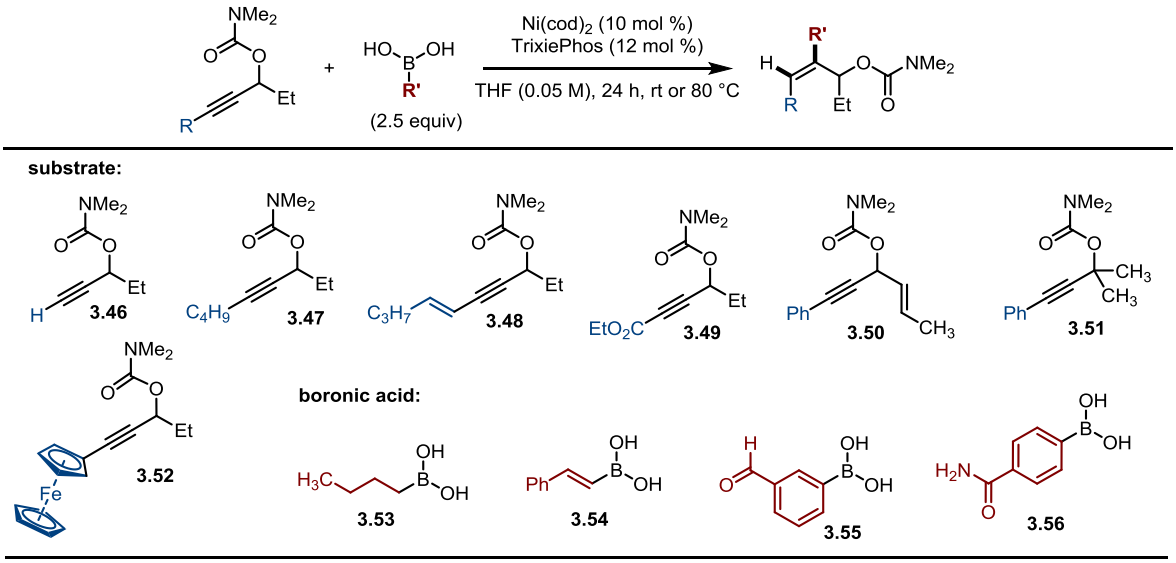


[a]Reaction run at 80 °C. DG = N,N'-dimethylcarbamoyl

Table 3.4 illustrates representative examples of substrates and boronic acids that provided low yields or no desired hydrofunctionalization products. The hydroarylation of substrates containing alkynyl substitution other than aryl groups failed to produce any desired product. Due to the lack of reactivity with terminal or alkyl substituted substrates,

we initially hypothesized that additional nickel-coordinating functional groups were required for activation of the catalyst. However, incorporation of either enyne **3.46** and ynoate **3.49** did not provide desired products. Interestingly, although the products of hydroarylation are allylic carbamates, employing **3.50** did not afford the desired carbamate and results in decomposition of the starting material. Furthermore, subjecting a tertiary carbamate to the reaction conditions results in trace yields of product and upon heating only decomposition of the starting material is observed. Similarly, any attempt at using alkyl or alkenylboronic acids failed to produce the desired hydroalkyl- or hydroalkenylated products. Future studies will be direct toward addressing the origin of the requirement of aryl substituted substrates and boronic acids.

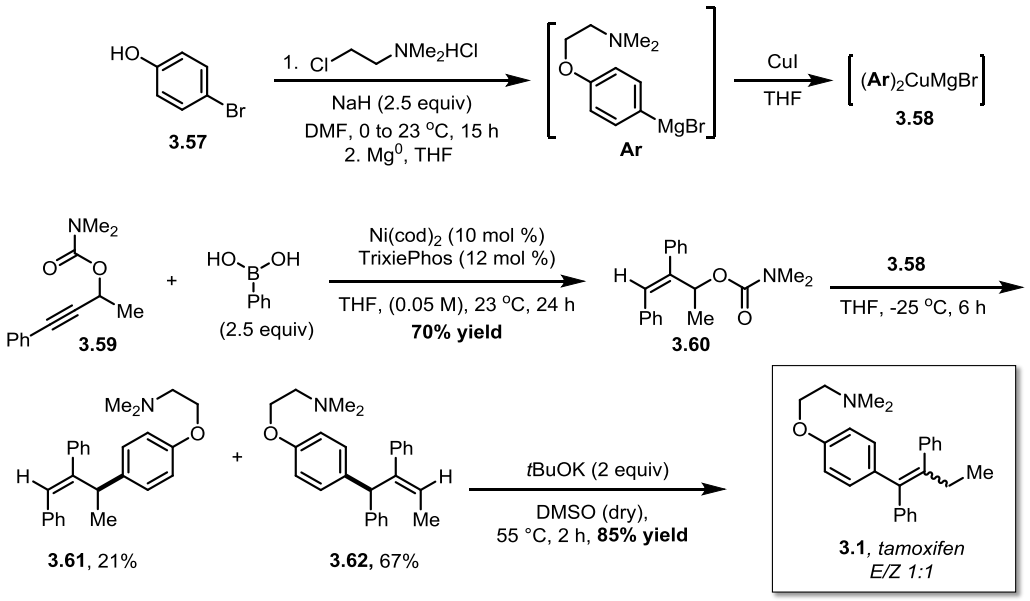
Table 3.4. Incompatible substrates and boronic acids for directed hydroarylation



3.5 Synthesis of Tamoxifen

Functionalized allylic alcohol derivatives are common synthetic building blocks en route to more complex molecular scaffolds. Allylic carbamates can be used in Tsuji–Trost-type cross-coupling reactions as well as copper-mediated substitutions, allowing for a range of back-to-back transition-metal-catalyzed reactions following hydroarylation. Scheme 3.2 demonstrates one of the many synthetic uses of these products. Allylic carbamate **3.60** can be converted to tamoxifen in two steps via a cuprate-mediated allylic substitution of the allylic carbamate followed by isomerization of the double bond to form tamoxifen (**3.1**). As previously stated, these stilbenes are known to be biologically active and **3.1** is a medication used to treat breast cancer and is on the WHO's list of essential medicines. Hence, this hydroarylation can serve as a useful platform to generate a library of tamoxifen-like structures for SAR studies.

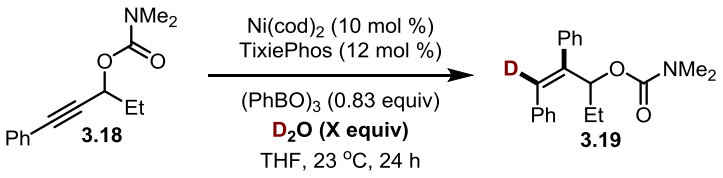
Scheme 3.2. Synthesis of tamoxifen via hydroarylation of a simple alkyne



3.6 Mechanistic Studies and Deuterium Labeling

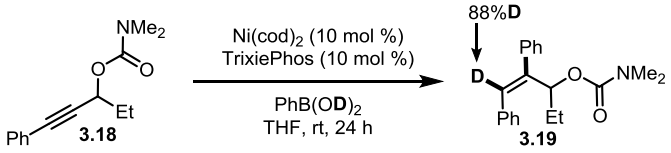
To gain insight into the mechanism of this reaction, we sought to determine the origin of the hydrogen atom in our hydroarylation reaction. Initial attempts to replace phenylboronic acid with phenyl boroxine and deuterium oxide resulted in low yields of product (17-30%) with 99% deuterium incorporation (Table 3.5). However, using $PhB(OD)_2$ led to a 68% yield of product **3.19** with 88% deuterium incorporation (Scheme 3.3). These results support the conclusion that the acidic protons in the arylboronic acid were the source of hydrogen in the hydroarylation. These results are consistent with a mechanism that involves a directed hydrometalation to afford a vinylnickel intermediate, which can then undergo transmetallation and reductive elimination to furnish the desired product. This mechanism is consistent with the previously proposed hydroarylation reactions reported that do not require base.[8c]

Table 3.5. Determination of source for deuterium incorporation



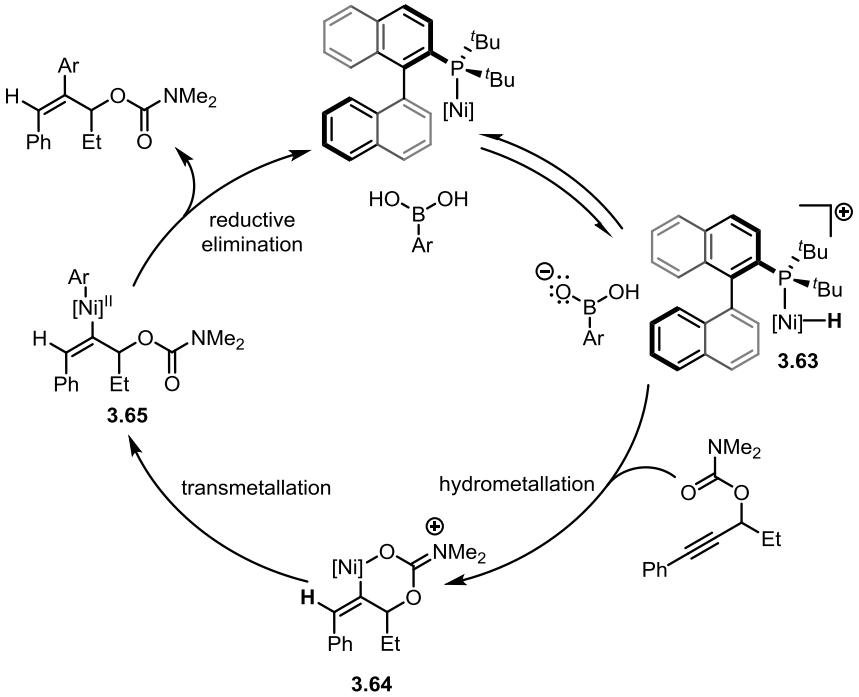
entry	D_2O (X equiv)	**3.18** (%)	**3.19** (%)	D incorporation (%)
1	1	65	32	>99
2	2	69	27	>99
3	3	70	25	>99
4	6	72	17	>99

Scheme 3.3. Deuterated phenylboronic acid reveals origin of hydrogen



Our current mechanistic hypothesis for the regio- and stereoselective hydroarylation is shown in Scheme 3.4. We propose that the nickel catalyst is initially protonated by the boronic acid (**3.63**) which then undergoes a directed *syn*-hydrometallation forming six-membered nickelocycle **3.64**. Transmetallation to the organonickel intermediate followed by reductive elimination provides the desired product and regenerates the nickel catalyst. We rule out an initial transmetallation and carbometallation based on the observed regioselectivity of the reaction. If carbometallation occurred first to generate the exocyclic six-membered metallocycle, the expected product would have the aryl group incorporated at the distal position, otherwise to obtain the desired regiochemistry, a seven-membered intermediate containing an (*E)*-olefin would result upon carbometallation.

Scheme 3.4. Tentative mechanistic hypothesis



3.7 Conclusion

In conclusion, we have developed a regio- and stereoselective nickel-catalyzed hydroarylation of alkynes using a propargyl carbamate as a directing group. The reaction is tolerant of a range of functional groups and heterocycles. Mechanistic studies into the origin of the hydrogen revealed that it is from the acid protons of the arylboronic acid. Furthermore, the synthesis of tamoxifen can be completed in two operationally simple steps in good overall yield.

3.8 Experimental Details

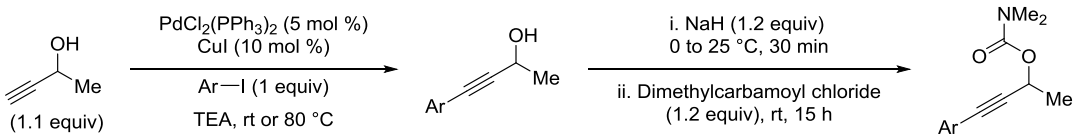
General Procedures

All reactions were carried out under an atmosphere of N_2, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (CH_2Cl_2), and dimethylacetamide (DMA) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h) to remove H_2O. All other solvents utilized were purchased "anhydrous" commercially, or purified as described. ^1H NMR spectra were recorded on Bruker DRX-400 (400 MHz ^1H, 100 MHz ^{13}C, 376.5 MHz ^{19}F) or CRYO-500 (500 MHz ^1H, 125.7 MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of triplets (tt), quartet (q), quintet (quin), apparent doublet (ad), apparent triplet (at), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 °C. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm^{-1}). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F_{254} precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with $KMnO_4$, ceric ammonium molybdate (CAM), or *p*-anisaldehyde (PAA) solutions. Flash chromatography was performed using Silica Gel 60 (170-400 mesh) from Fisher Scientific.

Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Automatic Polarimeter. High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center. Bis(cyclooctadiene)nickel [$Ni(cod)_2$] complex was purchased from Strem, stored in a glovebox under an atmosphere of N_2, and used as received. All other reagents were purchased commercially and used as received.

Synthesis and characterization of substrates

General Procedure A: *Sonogashira coupling and carbamate installation*



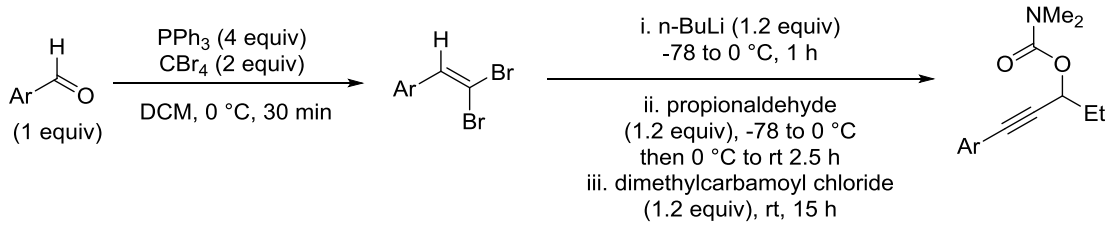
Sonogashira coupling:

The products were prepared according to a modified procedure reported by Lautens.[1] To a 100 mL round bottom flask equipped with a stir bar was added $PdCl_2(PPh_3)_2$ (5.0 mol %), CuI (10 mol %), 1-butyne-3-ol (1.1 equiv), aryl iodide (1.0 equiv), and triethylamine (25 mL). The reaction was stirred at room temperature for 4 hours. The reaction mixture was then filtered through silica to remove palladium and copper, and the silica was washed with diethyl ether. The solvents were removed under reduced pressure to provide the crude product as a dark yellow-brown oil. The crude mixtures were carried forward to carbamate installation without further purification.

Carbamate installation:

To a 100 mL round bottom flask was added NaH (1.1 equiv) and THF (30 mL). The suspension was cooled to 0 °C in an ice bath and a solution of the alcohol (1.0 M in THF) was added dropwise over 15 minutes. The ice bath was then removed and the mixture was allowed to warm to room temperature before dimethylcarbamoyl chloride (1.2 equiv) was added. The reaction was stirred at room temperature for 15 hours after which the mixture was quenched with saturated aqueous ammonium chloride, extracted with ethyl ether, dried over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

***General Procedure B**: Corey-Fuchs, 1, 2 addition, and carbamate installation*



Corey-Fuchs:

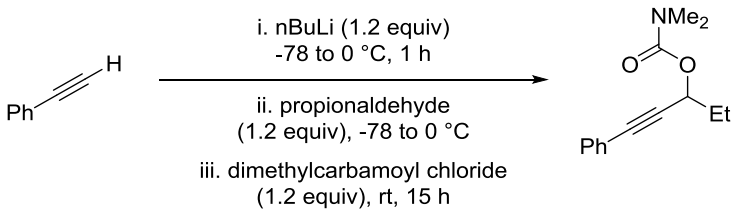
The product was prepared according to a modified procedure reported by Hoppe.[2] To a 250 mL round bottom flask was added PPh_3 (40 mmol, 4 equiv) and dichloromethane (35 mL) and was cooled to 0 °C, after which CBr_4 (20 mmol, 2 equiv) was added as a solution in dichloromethane (15 mL). The aryl aldehyde (10 mmol, 1 equiv) was then added as a solution in dichloromethane (10 mL) dropwise over the course of 10 minutes. The reaction was stirred for 30 min at 0 °C before the solvent was removed and the crude yellow oil was purified by column chromatography.

1, 2 addition and carbamate installation:

To a 250 mL round bottom flask was added *gem*-dibromo styrene and THF (50 mL). The solution was cooled to –78 °C in a dry ice and acetone bath after which *n*-BuLi (1.2 equiv, 2.5 M in hexane) was added dropwise over the course of 20 min. The bath was then removed and the reaction was allowed to warm to room temperature and stirred for 30 minutes before it was cooled back down to –78 °C, and a solution of propionaldehyde (1.0 M in THF) was added dropwise over the course of 10 minutes. The reaction was allowed to warm to room temperature and stirred for 4 hours before being quenched with dimethylcarbamoyl chloride (1.2 equiv). The resulting mixture was stirred at room temperature for 15 hours before being quenched with saturated aqueous ammonium chloride and extracted with ethyl

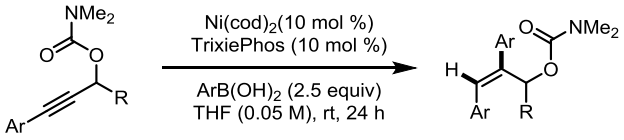
ether, dried over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

***General Procedure C**: 1, 2 addition and carbamate installation*



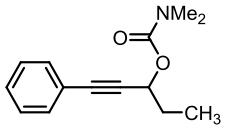
To a 250 mL round bottom flask was added phenylacetylene (1 equiv) and THF (50 mL). The solution was cooled to –78 °C in a dry ice and acetone bath after which *n*-BuLi (1.2 equiv, 2.5 M in hexane) was added dropwise over the course of 20 min. The bath was then removed and the reaction was allowed to warm to room temperature and stirred for 30 minutes before it was cooled back down to –78 °C. A solution of propionaldehyde (1.0 M in THF) was then added dropwise over the course of 10 minutes. The reaction was allowed to warm to room temperature and stirred for 4 hours before being quenched with dimethylcarbamoyl chloride (1.2 equiv). The resulting mixture was stirred at room temperature for 15 hours before being quenched with saturated aqueous ammonium chloride and extracted with ethyl ether, dried over $MgSO_4$, and concentrated under reduced pressure. The product was purified by flash column chromatography.

***General Procedure D**: Nickel-catalyzed hydroarylation*

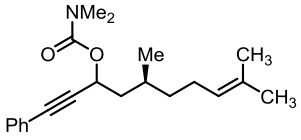


In a glove box, a flame dried 7 mL dram vial was charged with $Ni(cod)_2$ (5.5 mg, 0.020 mmol, 0.10 equiv), TrixiePhos (8.0 mg, 0.020 mmol, 0.10 equiv), arylboronic acid (0.50 mmol, 2.5 equiv), and the alkyne (0.20 mmol, 1.0 equiv) and dissolved in THF (4.0 mL). The reaction was then stirred for 24 hours at 24 ˚C or 85 ˚C depending on the substrate and arylboronic acid used. The reaction mixture was then filtered through a pad of silica and eluted with Et_2O to remove the catalyst and then concentrated under reduced pressure. The product was purified by flash column chromatography.

Characterization of starting materials

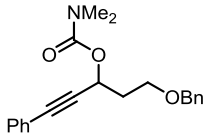


3.18. The product was prepared according to general procedure C using propionaldehyde (1.9 mL, 27 mmol), phenylacetylene (2.9 mL, 26 mmol), *n*-BuLi (11 mL, 28 mmol) and dimethylcarbamoyl chloride (2.5 mL, 28 mmol). The product was purified by flash column chromatography (20% Et_2O/pentane) to afford the title compound as a colorless oil (4.4 g, 19 mmol, 72% yield). **TLC R_f** = 0.4 (12% EtOAc/hexane); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.49–7.41 (m, 2H), 7.33–7.28 (m, 3H), 5.5 (t, *J* = 6.48 Hz, 1 H), 2.94 (s, 6H), 1.89 (quintet *J* = 7.5 Hz, 2H) 1.08 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.0, 132.2, 128.7, 128.5, 122.9, 87.6, 85.2, 66.9, 28.9, 9.8; **IR** (neat) 2970, 2935, 1702, 1489, 1128, 1392 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{17}NO_2Na$ $(M + Na)^+$ 254.1157, found 254.1151.

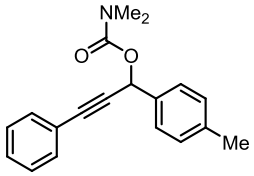


3.66. The product was prepared according to general procedure C using citronellal (2.0 mL, 11 mmol), phenylacetylene (1.1 mL, 10 mmol), *n*-BuLi (4.4 mL, 11 mmol) and dimethylcarbamoyl chloride (1.0 mL, 11 mmol). The product was purified by flash column chromatography (20% Et_2O/pentane) to afford the title compound as a colorless oil (3.3 g, 10 mmol, 92% yield). **TLC R_f** = 0.4 (20% Et_2O/pentane); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.46–7.40 (m, 2H), 7.31–7.27 (m, 3H), 5.65–5.58 (m, 1H), 5.14–5.07 (m, 1H), 2.94 (s, 6H), 2.08–1.84, (m, 3H), 1.83–1.65 (m, 5H), 1.60 (s, 3H), 1.47–1.37 (m, 1H), 1.29–1.18 (m, 1H), 1.00–0.96 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 156.0, 132.21, 132.19, 131.66, 131.65,

128.68, 128.66, 128.49, 128.48, 124.91, 124.90, 123.0, 88.3, 88.0, 85.3, 85.0, 64.9, 64.4, 42.8, 42.5, 37.4, 37.2, 29.7, 29.4, 26.0, 25.66, 25.62, 19.97, 19.89, 18.0; **IR** (neat) 2955, 2924, 1705, 1489, 1392, 1178 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{29}NO_2Na$ $(M + Na)^+$ 350.2096, found 350.2083.

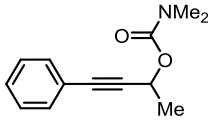


3.67. The product was prepared according to general procedure C using phenylacetylene (0.45 mL, 4.1 mmol), *n*-BuLi (1.9 mL, 4.6 mmol), 3-(benzyloxy)propanal (660 mg, 4.0 mmol), and dimethyl carbamoyl chloride (1.1 mL, 11 mmol). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a colorless oil (1.1 g, 3.4 mmol, 85% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.43–7.38 (m, 2H), 7.36–7.23 (m, 8H), 5.73 (t, *J* = 6.7 Hz, 1H), 4.53 (s, 2H), 3.74–3.62 (m 2H), 2.93 (s, 3H), 2.89 (s, 3H), 2.28–2.12 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.8, 138.6, 132.2, 128.8, 128.7, 128.5, 128.0, 127.8, 122.8, 87.5, 85.4, 73.3, 66.4, 63.4, 35.9; **IR** (neat) 2931, 2863, 1699, 1490, 1395, 1184 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{23}NO_3Na$ $(M + Na)^+$ 360.1576, found 360.1564.

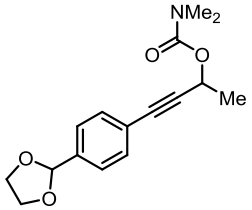


3.68. The product was prepared according to general procedure C using phenylacetylene (1.1 mL 10 mmol), *n*-BuLi (4.8 mL, 12 mmol), *p*-tolualdehyde (1.2 mL, 2.0 mmol), and dimethyl carbamoyl chloride (0.41 mL, 4.4 mmol). The product was purified by flash column chromatography (20% Et_2O/pentanes) to afford the title compound as a colorless oil (1.1 g,

3.4 mmol, 85% yield). **TLC R_f** = 0.3 (20% Et_2O/pentane); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.52–7.46 (m, 4H), 7.34–7.27 (m, 3H), 7.22–7.18 (m, 2H), 6.63 (s, 1H), 2.94 (s, 6H), 2.36 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.8, 138.9, 135.5, 132.2, 129.6, 128.9, 128.5, 127.9, 122.8, 87.0, 86.8, 67.2, 21.6; **IR** (neat) 2929, 1697, 1490, 1394, 1175 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{19}NO_2Na$ $(M + Na)^+$ 316.1313, found 316.1313.

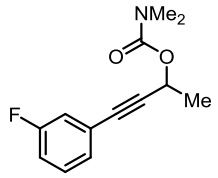


3.69. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (90 mg), CuI (45 mg), 1-butyne-3-ol (0.54 mL, 7.0 mmol), aryl iodide (0.78 mL, 7.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.71 mL, 7.7 mmol), NaH (200 mg, 8.4 mmol), THF (25 mL). The product was purified by flash column chromatography (20% Et_2O/pentanes) to afford the title compound as a colorless oil (1.3 g, 6.0 mmol, 86% yield). **TLC R_f** = 0.4 (12% EtOAc/hexane); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.46–7.42 (m ,2H), 7.32–7.25 (m, 3H), 5.65 (q, *J* = 6.6 Hz, 1H), 2.93 (s, 6H), 1.59 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.8, 132.1, 128.7, 128.5, 122.8, 88.6, 84.4, 61.9, 20.2; **IR** (neat) 2986, 2935, 1699, 1489, 1392, 1177 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{13}H_{15}NO_2Na$ $(M + Na)^+$ 240.1001, found 240.0989.

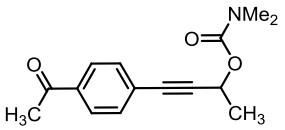


3.70 The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (350 mg, 0.50 mmol), CuI (190 mg, 1.0 mmol), 1-butyne-3-ol (0.83 mL, 11 mmol), aryl bromide

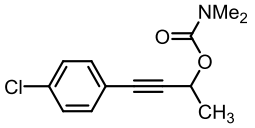
(2.3 g, 10 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (1.0 mL, 11 mmol), NaH (290 mg, 12 mmol), THF (25 mL). The product was purified by flash column chromatography (12% EtOAc/hexanes) to afford the title compound as a pale yellow oil (2.3 g, 8.0 mmol, 80% yield). **TLC R_f** = 0.2 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.47–7.39 (m, 4H), 5.79 (s,1H), 5.64 (q, *J* = 6.7 Hz, 1H), 4.14–3.99 (m , 4H), 2.94 (s, 6H), 1.58 (d, 3, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.9, 138.4, 132.2, 126.7, 123.7, 103.6, 89.2, 84.1, 65.6, 62.0, 22.3; **IR** (neat) 2985, 2886, 1699, 1391, 1176, 1079 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{16}H_{19}NO_4Na$ $(M + Na)^+$ 312.1212, found 312.1197.



3.71 The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (250 mg, 0.35 mmol), CuI (130 mg, 0.70 mmol), 1-butyne-3-ol (0.58 mL, 7.7 mmol), aryl iodide (0.82 mL, 7.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.71 mL, 7.7 mmol), NaH (200 mg, 8.2 mmol), THF (25 mL). The product was purified by flash column chromatography (12% EtOAc/hexanes) to afford the title compound as a pale yellow oil (1.9 g, 8.2 mmol, 82% yield)**. TLC R_f** = 0.4 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.29–7.20 (m, 2H), 7.16–7.12 (m, 1H), 7.05–6.98 (m, 1H), 5.63 (q, *J* = 6.8 Hz, 1H), 2.94 (s, 6H), 1.58 (d, *J* = 6.8 Hz , 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.5 (d, *J* = 246.0 Hz), 155.8, 130.1 (d, *J* = 8.8 Hz), 128.1 (d, *J* = 3.2 Hz), 124.7 (d, *J* = 9.7 Hz), 119.0 (d, *J* = 22.7 Hz), 116.1 (d, *J* = 21.4 Hz), 89.7, 83.2, 61.9, 22.2; **IR** (neat) 2936, 1701, 1579, 1486, 1392, 1170 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{13}H_{14}FNO_2Na$ $(M + Na)^+$ 258.0906, found 258.0905.

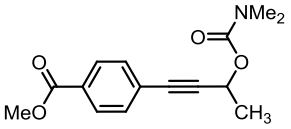


3.72. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (180 mg, 0.25 mmol), CuI (95 mg, 0.50 mmol), 1-butyne-3-ol (0.43 mL, 5.5 mmol), aryl iodide (1.2 g, 5.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.51 mL, 5.5 mmol), NaH (140 mg, 6.0 mmol), THF (25 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.86 g, 3.3 mmol, 66% yield). **TLC R_f** = 0.4 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 5.65 (q, *J* = 6.7 Hz, 1H), 2.95 (s, 6H), 2.60 (s, 3H), 1.60 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.7, 155.8, 136.7, 132.3, 128.4, 127.8, 92.0, 83.6, 61.8, 27.0, 22.1; **IR** (neat) 2987, 2936, 1701, 1682, 1392, 1260, 1176 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_{17}NO_3Na$ $(M + Na)^+$ 282.1106, found 282.1102.

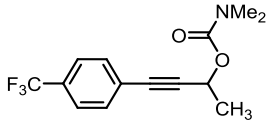


3.73. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (180 mg, 0.25 mmol), CuI (95 mg, 0.50 mmol), 1-butyne-3-ol (0.43 mL, 5.5 mmol), aryl iodide (1.2 g, 5.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.51 mL, 5.5 mmol), NaH (140 mg, 6.0 mmol), THF (25 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.89 g, 3.6 mmol, 71% yield). **TLC R_f** = 0.4 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 5.62 (q, *J* = 6.7 Hz, 1H), 2.94 (s, 6H), 1.58 (d, *J* = 6.6 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.8, 134.8, 133.4, 128.9, 121.4, 89.8, 83.3, 61.9,

22.2; **IR** (neat) 2987, 2934, 1701, 1488, 1391, 1177 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{13}H_{14}ClNO_2Na$ $(M + Na)^+$ 274.0611, found 274.0617.

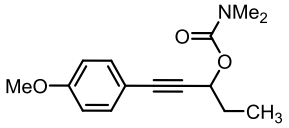


3.74. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (180 mg, 0.25 mmol), CuI (95 mg, 0.50 mmol), 1-butyne-3-ol (0.43 mL, 5.5 mmol), aryl iodide (1.3 g, 5.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.51 mL, 5.5 mmol), NaH (140 mg, 6.0 mmol), THF (25 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (1.0 g, 3.8 mmol, 75% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.6 Hz, 2H), 7.50 (d, *J* 8.6 = Hz, 2H), 5.64 (q, *J* = 6.7 Hz, 1H), 3.92 (s, 3H), 2.95 (s, 6H), 1.59 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 166.8, 155.8, 132.1, 130.0, 129.7, 127.6, 91.7, 83.7, 61.9, 52.6, 22.1; **IR** (neat) 2988, 2936, 1701, 1393, 1272, 1174, 1085 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_{17}NO_4Na$ $(M + Na)^+$ 298.1055, found 298.1047.

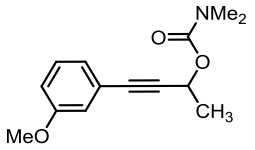


3.75. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (180 mg, 0.25 mmol), CuI (95 mg, 0.50 mmol), 1-butyne-3-ol (0.43 mL, 5.5 mmol), aryl iodide (0.73 mL, 5.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.51 mL, 5.5 mmol), NaH (140 mg, 6.0 mmol), THF (25 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (1.0 g, 3.7 mmol, 73% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ

7.58–7.52 (m, 4H), 5.64 (q, *J* 7.0 = Hz, 1H), 2.95 (s, 6H), 1.59 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.8, 132.4, 130.5 (q, *J* = 32.8 Hz), 126.7 (q, *J* = 1.8 Hz), 124.1 (q, *J* = 271.9 Hz), 125.5 (q, *J* = 3.7 Hz), 91.2, 83.1, 61.8, 22.0; **IR** (neat) 2938, 1703, 1394, 1319, 1165, 1064 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{14}F_3NO_2Na$ $(M + Na)^+$ 308.0874, found 308.0863.



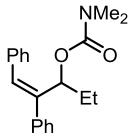
3.76. The product was prepared according to general procedure B using *p*-methoxy benzaldehyde (1.2 mL, 10 mmol), CBr_4 (6.6 g, 20 mmol), PPh_3 (11 g, 40 mmol), *n*-BuLi (8.4 mL, 21 mmol), propionaldehyde (0.72 mL, 10 mmol) and dimethyl carbamoyl chloride (1.0 mL, 11 mmol). The product was purified by flash column chromatography (12% EtOAc/hexanes) to afford the title compound as a pale yellow oil (1.2 g, 4.5 mmol, 45% yield). **TLC R_f** = 0.3 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.7, Hz, 2H), 5.50 (t, *J* = 6.5 Hz, 1H), 3.80 (s, 3H), 2.94 (s, 6H), 1.87 (quintet, *J* = 7.2 Hz, 2H), 1.07 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 160.0, 156.1, 133.7, 115.1, 114.1, 86.3, 85.1, 67.0, 55.6, 29.0, 9.8; **IR** (neat) 2962, 2930, 1698, 1509, 1248, 1176 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_{19}NO_3Na$ $(M + Na)^+$ 284.1263, found 284.1265.



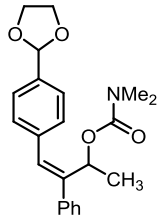
3.77. The product was prepared according to general procedure A using $PdCl_2(PPh_3)_2$ (180 mg, 0.25 mmol), CuI (95 mg, 0.50 mmol), 1-butyne-3-ol (0.43 mL, 5.5 mmol), aryl iodide (0.73 mL, 5.0 mmol), triethylamine (30 mL), dimethylcarbamoyl chloride (0.51 mL, 5.5 mmol), NaH (140 mg, 6.0 mmol), THF (25 mL). The product was purified by flash column

chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (1.0 g, 3.7 mmol, 73% yield). **TLC R_f** = 0.3 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.20 (t, *J* = 7.7 Hz, 1H), 7.06–7.02 (m, 1H), 6.99–6.95 (m, 1H), 6.89–6.84 (m, 1H), 5.64 (q, *J* = 6.9 Hz, 1H), 3.79 (s, 3H), 2.94 (s, 6H), 1.58 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.5, 155.9, 129.6, 124.7, 123.8, 116.9, 115.5, 88.5, 84.4, 62.0, 55.6, 22.3; **IR** (neat) 2936, 1700, 1574, 1392, 1173 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{13}H_{17}NO_3Na$ $(M + Na)^+$ 270.1106, found 270.1097.

Characterization of products

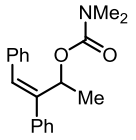


3.19. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), phenylboronic acid (60 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (43 mg, 70% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.46 (m, 2H), 7.43–7.23 (m, 8H), 6.71 (s, 1H), 5.84 (t, *J* = 6.85 Hz, 1H), 2.88 (s, 3H), 2.7 (s, 3H), 1.85 (m, 1H), 1.65 (m, 3H), 0.82 (t, *J* = 7.83 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 141.9, 141.4, 137.3, 132.6, 129.2, 128.9, 128.6, 128.2, 127.5, 127.3, 75.6, 27.5, 10.5; **IR** (neat) 2968, 2934, 1699, 1598, 1443 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{23}NO_2Na$ $(M + Na)^+$ 332.1627, found 332.1611.

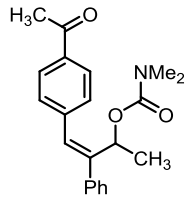


3.21. The product was prepared according to general procedure D using alkyne **3.70** (58 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (42 mg, 57% yield). **TLC R_f** = 0.3 (25% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.45 (m, 4H), 7.41–7.28 (m,

5H), 6.64 (s, 1H), 6.02 (q, *J* = 6.9 Hz, 1H), 5.83 (s, 1H), 4.16–4.01 (m, 4H), 2.86 (s, 3H), 2.70 (s, 3H), 1.39 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 143.4, 141.0, 138.1, 137.0, 131.0, 129.2, 128.9, 128.2, 127.6, 126.8, 103.9, 70.6, 65.7, 65.6, 30.1, 20.8; **IR** (neat) 2926, 1698, 1442, 1390, 1183, 1080 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{25}NO_4Na$ $(M + Na)^+$ 390.1681, found 390.1668.

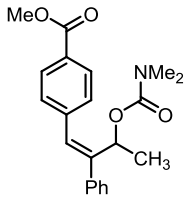


3.26. The product was prepared according to general procedure D using alkyne **3.59** (44 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (42 mg, 71% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.47 (m, 2H), 7.40–7.24 (m, 7H), 6.65 (s, 1H), 6.05 (q, *J* = 6.6 Hz, 1H), 2.87 (s, 3H), 2.72 (s, 3H), 1.40 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 142.9, 141.2, 137.1, 131.5, 129.2, 129.0, 128.7, 128.2, 127.5, 127.4, 70.5, 20.8; **IR** (neat) 3022, 2932, 1698, 1491, 1392, 1185 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{21}NO_2Na$ $(M + Na)^+$ 318.1470, found 318.1469.

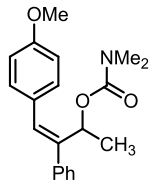


3.22. The product was prepared according to general procedure D using alkyne **3.72** (52 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol),

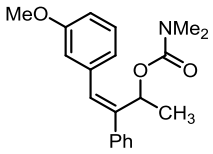
TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (51 mg, 75% yield). **TLC R_f** = 0.3 (25% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.00–7.95 (m, 2H), 7.51–7.45 (m, 4H), 7.40–7.31 (m, 3H), 6.66 (s, 1H), 6.01 (q, *J* = 6.7 Hz, 1H), 2.87 (s, 3H), 2.72 (s, 3H), 2.61 (s, 3H), 1.40 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.0, 144.8, 142.1, 140.6, 135.9, 130.3, 129.4, 128.83, 128.76, 128.3, 127.8, 70.3, 27.0, 20.7; **IR** (neat) 2979, 2931, 1699, 1682, 1267, 1182 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{23}NO_3Na$ $(M + Na)^+$ 360.1576, found 360.1572.



3.23. The product was prepared according to general procedure D using alkyne **3.74** (55 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (51 mg, 72% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.06–8.03 (m, 2H), 7.50–7.46 (m, 2H), 7.46–7.42 (m, 2H), 7.39–7.31 (m, 3H), 6.65 (s, 1H), 5.99 (q, *J* = 6.7 Hz, 1H), 3.92 (s, 3H), 2.86 (s, 3H), 2.69 (s, 3H), 1.43 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 167.2, 156.0, 144.7, 142.0, 140.7, 130.3, 129.9, 129.2, 128.9, 128.8, 128.3, 127.8, 70.4, 52.4, 20.7; **IR** (neat) 2929, 1718, 1698, 1391, 1275, 1178 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{23}NO_4Na$ $(M + Na)^+$ 376.1525, found 376.1522.

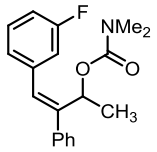


3.24. The product was prepared according to general procedure D using alkyne **3.76** (50 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (33 mg, 50% yield). **TLC R_f** = 0.3 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.50–7.46 (m, 2H), 7.39–7.26 (m, 5H), 6.94–6.88 (m, 2H), 6.65 (s, 1H), 5.89 (t, *J* = 7.34 Hz, 1H), 3.82 (s, 3H), 2.89 (s, 3H), 2.78 (s, 3H), 1.84 (m, 1H), 1.64 (m, 1H), 0.82 (t, *J* = 7.58 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.9, 141.5, 140.5, 132.5, 130.5, 129.7, 128.9, 128.2, 127.3, 114.1, 75.5, 55.6, 27.4, 10.5; **IR** (neat) 2929, 1698, 1573, 1442, 1298, 1033 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{25}NO_3Na$ $(M + Na)^+$ 362.1732, found 362.1730.

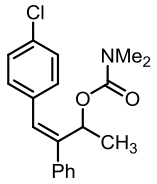


3.25. The product was prepared according to general procedure D using alkyne **3.77** (50 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (35 mg, 54% yield). **TLC R_f** = 0.3 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.50–7.46 (m, 2H), 7.39–7.26 (m,

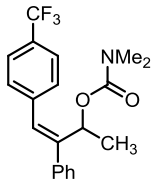
4H), 6.98–6.94 (m, 1H), 6.93–6.90 (m, 1H), 6.85–6.80 (m, 1H), 6.63 (s, 1H), 6.06 (q, *J* = 6.4 Hz 1H), 3.83 (s, 3H), 2.87 (s, 3H), 2.74 (s, 3H), 1.40 (d, *J* = 6.6 Hz 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.8, 143.1, 141.1, 138.5, 131.5, 129.7, 128.9, 128.2, 127.6, 121.6, 114.4, 113.4, 70.6, 55.5, 20.8; **IR** (neat) 2928, 1697, 1488, 1391, 1268, 1182 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{23}NO_3Na$ $(M + Na)^+$ 348.1576, found 348.1577.



3.27. The product was prepared according to general procedure D using alkyne **3.71** (47 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (55 mg, 87% yield). **TLC R_f** = 0.3 (25% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48–7.44 (m, 2H), 7.39–7.29 (m, 4H), 7.17–7.13 (m, 1H), 7.10–7.05 (m, 1H), 6.93 (dt, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 6.58 (s, 1H), 5.99 (q, *J* = 6.8 Hz, 1H), 2.87 (s, 3H), 2.71 (s, 3H), 1.41 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.9 (d, *J* = 245.5 Hz), 156.0, 144.2, 140.8, 139.4 (d, *J* = 7.9 Hz), 130.1 (d, *J* = 8.3 Hz), 130.0 (d, *J* = 1.8 Hz), 128.9, 128.3, 127.7, 124.9, (d, *J* = 3.2 Hz), 116.1 (d, *J* = 21.7 Hz), 114.3 (d, *J* = 21.3 Hz), 70.4, 20.8; **IR** (neat) 2985, 2933, 1698, 1489, 1391, 1180 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{20}FNO_2Na$ $(M + Na)^+$ 336.1376, found 336.1362.

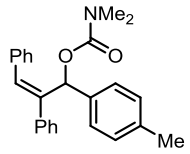


3.28. The product was prepared according to general procedure D using alkyne **3.73** (50 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (52 mg, 79% yield). **TLC R_f** = 0.5 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48–7.44 (m, 2H), 7.38–7.28 (m, 7H), 6.57 (s, 1H), 5.99 (q, *J* = 6.7 Hz, 1H), 2.87 (s, 3H), 2.72 (s, 3H), 1.38 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.0, 143.7, 140.8, 135.6, 133.3, 130.5, 130.1, 128.85, 128.82, 128.3, 127.7, 70.3, 20.7; **IR** (neat) 2977, 2930, 1698, 1490, 1391, 1183 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{20}ClNO_2Na$ $(M + Na)^+$ 352.1080, found 352.1077.

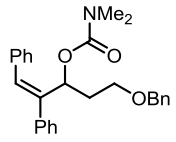


3.29. The product was prepared according to general procedure D using alkyne **3.75** (57 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (60 mg, 83% yield). **TLC R_f** = 0.4 (10% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.65–7.61 (m, 2H), 7.51–7.46 (m, 4H), 7.40–7.31 (m, 3H), 6.63 (s, 1H), 5.95 (q, *J* = 6.7 Hz, 1H), 2.87 (s, 3H), 2.68 (s, 3H), 1.40 (d,

J=6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.0, 144.9, 140.9 (q, *J* = 1.4 Hz), 140.6, 129.7, 129.4, 129.3 (q, *J* = 132.4 Hz), 128.8, 128.4, 127.9, 125.6 (q, *J* = 3.7 Hz), 124.5 (q, *J* = 271.9), 70.4, 20.8; **IR** (neat) 2980, 2934, 1700, 1615, 1393, 1323, 1121 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{20}F_3NO_2Na$ $(M + Na)^+$ 386.1344, found 386.1335.

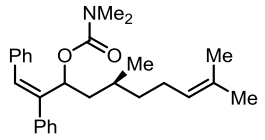


3.30. The product was prepared according to general procedure D using alkyne **3.68** (59 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (60 mg, 81% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.1 Hz, 2H), 7.45–7.38 (m, 4H), 7.34–7.28 (m, 6H), 7.22 (s, 1H), 7.19 (d, *J* = 7.7 Hz, 2H), 7.05 (s, 1H), 2.92 (s, 3H), 2.68 (s, 3H), 2.40 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 140.5, 140.4, 137.4, 137.03, 136.94, 132.4, 129.4, 129.2, 128.78, 128.76, 128.1, 127.6, 127.5, 126.7, 74.6, 21.5; **IR** (neat) 3023, 2923, 1703, 1492, 1389, 1173 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{25}H_{25}NO_2Na$ $(M + Na)^+$ 394.1783, found 394.1784.



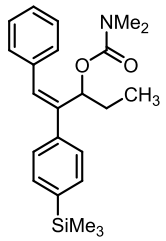
3.31. The product was prepared according to general procedure D using alkyne **3.67** (68 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with

pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (58 mg, 70% yield). **TLC R_f** = 0.6 (20% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48–7.40 (m, 4H), 7.36–7.19 (m, 11H), 6.67 (s, 1H), 6.16 (dd, *J* = 8.9 Hz, *J* = 5.3 Hz, 1H), 4.35 (s, 2H), 3.50–3.40 (m, 2H), 2.84 (s, 3H), 2.64 (s, 3H), 2.22–2.12 (m, 1H), 2.06 (m, 1H), 1.97 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 141.9, 141.2, 138.7, 137.1, 132.3, 129.3, 128.9, 128.6, 128.5, 128.2, 127.9, 127.7, 127.5, 127.3, 73.1, 71.6, 67.0, 35.0; **IR** (neat) 3056, 2927, 1699, 1494, 1391, 1179 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{27}H_{29}NO_3Na$ $(M + Na)^+$ 438.2045, found 438.2048.

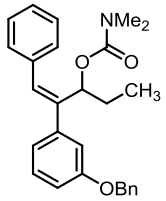


3.32. The product was prepared according to general procedure D using alkyne **3.66** (66 mg, 0.20 mmol), phenylboronic acid (61 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (54 mg, 66% yield). **TLC R_f** = 0.6 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.50–7.44 (m, 2H), 7.44–7.40 (m, 2H), 7.38–7.32 (m, 4H), 7.31–7.27 (m, 1H), 7.27–7.22 (m, 1H), major diastereomer 6.69 (s, 0.52H), minor diastereomer 6.64 (s, 0.46H), 6.06–5.99 (m, 1H), 4.99–4.91 (m, 1H), major diastereomer 2.92– 2.80 (m, 3H), minor diastereomer 2.77-2.58 (m, 3H), 1.97–1.72 (m, 2H), 1.69–1.59 (m, 4H), 1.54–1.49 (m, 3H), 1.49–1.33 (m, 2H), 1.22–0.90 (m, 2H), minor diastereomer 0.73 (d, *J* = 6.6 Hz, 1.3H), major diastereomer 0.68 (d, *J* = 6.7 Hz, 1.59H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 156.2, 143.1, 142.4, 141.28, 141.24, 137.4, 137.3, 132.3, 131.33, 131.28, 131.26, 129.10, 129.09, 128.90, 128.89, 128.6, 128.5, 128.19, 128.16, 127.48,

127.45, 127.3, 127.2, 125.03, 125.01, 73.2, 72.7, 41.9, 41.3, 37.5, 37.0, 29.6, 29.4, 26.01, 26.00, 25.7, 25.4, 19.9, 19.4, 17.9; **IR** (neat) 3022, 2959, 1700, 1493, 1392, 1186 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{27}H_{35}NO_2Na$ $(M + Na)^+$ 428.2566, found 428.2574.

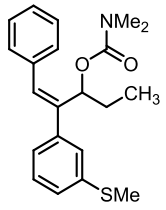


3.33. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 4-trimethylsilylphenylboronic acid (97 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (60 mg, 78% yield). **TLC R_f** = 0.5 (15% EtOAc/Hex); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.52–7.46 (m, 4H), 7.41–7.34 (m, 4H), 7.28–7.23 (m, 1H), 6.73 (s, 1H), 5.86 (dd, *J* = 7.7 Hz, *J* = 6.7 Hz, 1H), 2.89 (s, 3H), 2.75 (s, 3H), 1.89–1.80 (m, 1H), 1.70–1.60 (m, 1H), 0.81 (t, *J* = 7.5 Hz, 3H), 0.29 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.4, 141.76, 141.74, 139.5, 137.4, 133.3, 132.8, 129.2, 128.6, 128.2, 127.3, 75.6, 27.5, 10.5, –0.7; **IR** (neat) 2955, 1703, 1460, 1392, 1248, 1183 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{23}H_{31}NO_2SiNa$ $(M + Na)^+$ 404.2022, found 404.2018.



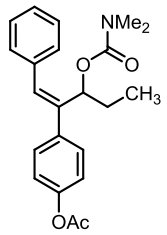
3.34. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 3-benzyloxyphenylboronic acid (114 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020

mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (48 mg, 58% yield). **TLC R_f** = 0.3 (25% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.47–7.43 (m, 2H), 7.41–7.31 (m, 6H), 7.28–7.23 (m, 2H), 7.15–7.13 (m, 1H), 7.12–7.09 (m, 1H), 6.93 (ddd, *J* = 8.3 Hz, *J* = 2.6 Hz, *J* = 0.7 Hz, 1H), 6.72 (s, 1H), 5.82 (dd, *J* = 7.8 Hz, *J* = 6.8 Hz, 1H), 5.09 (s, 2H), 2.89 (s, 3H), 2.73 (s, 3H), 1.89–1.79 (m, 1H), 1.70–1.60 (m, 1H), 0.81 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.7, 156.3, 142.8, 141.7, 137.4, 137.2, 132.7, 129.20, 129.18, 128.9, 128.6, 128.3, 127.8, 127.4, 121.7, 115.7, 113.8, 75.6, 70.3, 27.5, 10.6; **IR** (neat) 2967, 2930, 1698, 1574, 1391, 1182 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{27}H_{29}NO_3Na$ $(M + Na)^+$ 438.2045, found 438.2029.

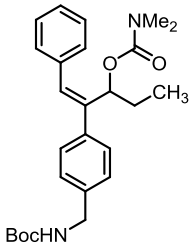


3.35. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 3-methylthiophenylboronic acid (84 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (49 mg, 69% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42–7.35 (m, 5H), 7.30–7.24 (m, 3H), 7.20 (dt, *J* = 7.1 Hz, *J* = 1.8 Hz, 1H), 6.70 (s, 1H), 5.83 (dd, *J* = 8.0 Hz, *J* = 6.7 Hz, 1H), 2.89 (s, 3H), 2.75 (s, 3H), 2.51 (s, 3H), 1.88–1.79 (m, 1H), 1.69–1.60 (m, 1H), 0.82 (t, *J* = 7.5 Hz,

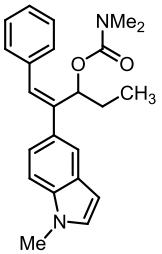
3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 142.0, 141.5, 138.2, 137.1, 132.9, 129.2, 128.7, 128.6, 127.4, 127.2, 125.8, 125.6, 75.5, 27.5, 16.2, 10.5; **IR** (neat) 2966, 1698, 1584, 1443, 1390, 1182 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{25}NO_2SNa$ $(M + Na)^+$ 378.1504, found 378.1510.



3.36. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 4-acetoxyphenylboronic acid (90 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (47 mg, 64% yield). **TLC R_f** = 0.3 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.47 (m, 2H), 7.41–7.33 (m, 4H), 7.28–7.23 (m, 1H), 7.10–7.05 (m, 2H), 6.70 (s, 1H), 5.83 (dd, *J* = 8.0 Hz, *J* = 6.6 Hz, 1H), 2.87 (s, 3H), 2.72 (s, 3H), 2.32 (s, 3H), 1.91–1.79 (m, 1H), 1.71–1.60 (m, 1H), 0.82 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 169.8, 156.3, 150.2, 141.1, 139.0, 137.2, 132.8, 129.9, 129.2, 128.6, 127.4, 121.3, 75.6, 27.5, 21.5, 10.6; **IR** (neat) 2926, 1760, 1698, 1503, 1186, 1166 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{25}NO_4Na$ $(M + Na)^+$ 390.1681 found, 390.1684.

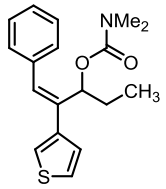


3.37. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 4-(tert-butoxycarbonylaminomethyl)phenylboronic acid (130 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (57 mg, 65% yield). **TLC R_f** = 0.3 (25% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.2 Hz, 2H), 7.42–7.33 (m, 4H), 7.29–7.23 (m, 3H), 6.69 (s, 1H), 5.83 (t, *J* = 7.2 Hz, 1H), 4.88 (s, 1H), 4.34 (d, *J* = 5.5 Hz, 2H), 2.88 (s, 3H), 2.74 (s, 3H), 1.88–1.78 (m, 1H), 1.66–1.58 (m, 1H), 1.48 (s, 9H), 0.80 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 141.5, 140.4, 137.2, 132.6, 129.18, 129.11, 128.6, 127.3, 75.6, 44.7, 28.8, 27.5, 10.5; **IR** (neat) 3339, 2970, 2932, 1699, 1507, 1175 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{26}H_{34}N_2O_4Na$ $(M + Na)^+$ 461.2416, found 461.2411.



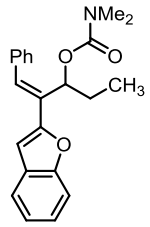
3.38. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 1-methyl-5-indolylboronic acid (87 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica

plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (45 mg, 62% yield). **TLC R_f** = 0.2 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.75–7.74 (m, 1H), 7.44–7.34 (m, 5H), 7.31–7.28 (m, 1H), 7.26–7.22 (m, 1H), 7.06 (d, *J* = 3.1 Hz, 1H), 6.74 (s, 1H), 6.49 (dd, *J* = 3.2 Hz, *J* = 0.7 Hz, 1H), 5.88 (t, *J* = 7.3 Hz, 1H), 3.80 (s, 3H), 2.89 (s, 3H), 2.74 (s, 3H), 1.91–1.81 (m, 1H), 1.73–1.64 (m, 1H), 0.81 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.5, 142.7, 137.9, 136.4, 132.7, 132.0, 129.5, 129.2, 128.5, 127.0, 123.0, 121.2, 108.8, 101.4, 76.2, 33.2, 27.6, 10.6; **IR** (neat) 2964, 2931, 2695, 1488, 1391, 1186 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{23}H_{26}N_2O_2Na$ $(M + Na)^+$ 385.1892, found 385.1900.

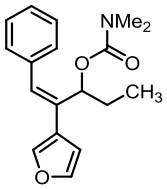


3.39. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 3-thienylboronic acid (64 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (47 mg, 75% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45–7.34 (m, 4H), 7.33–7.28 (m, 2H), 7.28–7.24 (m, 2H), 6.95 (s, 1H), 5.89 (dd, *J* = 8.1 Hz, *J* = 6.9 Hz, 1H), 2.88 (s, 3H), 2.85 (s, 3H), 1.98–1.87 (m, 1H), 1.77–1.68 (m, 1H), 0.83 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 140.8, 137.2, 136.3, 131.1, 129.2, 128.7, 128.0, 127.4, 125.0, 122.5, 75.3, 27.7,

10.6; **IR** (neat) 2969, 1702, 1491, 1390, 1183 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{18}H_{21}NO_2SNa$ $(M + Na)^+$ 338.1191, found 338.1184.

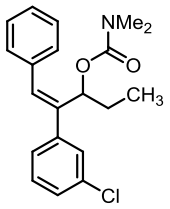


3.40. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 2-benzofuranylboronic acid (81 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (51 mg, 73% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.57–7.50 (m, 4H), 7.47 (d, *J* = 8.2 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.31–7.23 (m, 2H), 7.20 (t, *J* = 7.6 Hz, 1H), 6.96 (s, 1H), 6.03 (dd, *J* = 8.6 Hz, *J* = 6.4 Hz, 1H), 2.93 (s, 3H), 2.88 (s, 3H), 2.18–2.08 (m, 1H), 1.96–1.86 (m, 1H), 0.90 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 154.4, 154.3, 136.6, 130.8, 130.5, 129.5, 129.4, 128.8, 127.8, 124.8, 123.1, 121.2, 111.2, 105.3, 74.0, 28.1, 10.8; **IR** (neat) 3057, 2967, 1699, 1451, 1390, 1181 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{23}NO_3Na$ $(M + Na)^+$ 372.1576, found 372.1582.

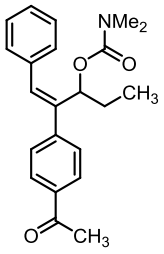


3.41. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 3-furanboronic acid (56 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with

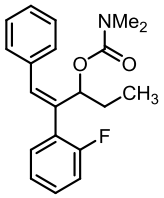
pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (34 mg, 57% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.43–7.40 (m, 2H), 7.39–7.34 (m, 2H), 7.27–7.23 (m, 2H), 6.88 (s, 1H), 6.65–6.63 (m, 1H), 5.87 (dd, *J* = 8.3 Hz, *J* = 6.6 Hz, 1H), 2.90 (s, 6H), 2.00–1.89 (m, 1H), 1.78–1.69 (m, 1H), 0.84 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 142.8, 140.5, 137.1, 132.6, 129.2, 129.1, 128.7, 127.3, 124.2, 109.8, 75.2, 27.7, 10.6; **IR** (neat) 2971, 1698, 1491, 1394, 1264, 1188 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{18}H_{21}NO_3Na$ $(M + Na)^+$ 322.1419, found 322.1426.



3.42. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 3-chlorophenylboronic acid (78 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (60 mg, 87% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.49 (m, 1H), 7.42–7.34 (m, 5H), 7.31–7.26 (m, 3H), 6.71 (s, 1H), 5.83 (dd, *J* = 8.1 Hz, *J* = 6.7 Hz, 1H), 2.89 (s, 3H), 2.76 (s, 3H), 1.88–1.78 (m, 1H), 1.67–1.58 (m, 1H), 0.82 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.2, 143.1, 140.7, 136.8, 134.0, 133.3, 129.5, 129.2, 129.1, 128.7, 127.59, 127.58, 127.1, 75.3, 27.5, 10.5; **IR** (neat) 2968, 2933, 1699, 1472, 1390, 1182 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{22}ClNO_2Na$ $(M + Na)^+$ 366.1237, found 366.1227.

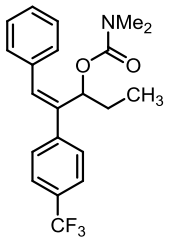


3.43. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 4-acetylphenylboronic acid (82 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (56 mg, 80% yield). **TLC R_f** = 0.2 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.60 (d, *J* = 8.3 Hz, 2H), 7.43–7.35 (m, 4H), 7.31–7.27 (m, 1H), 6.77 (s, 1H), 5.87 (dd, *J* = 7.7 Hz, *J* = 6.8 Hz, 1H), 2.88 (s, 3H), 2.74 (s, 3H), 2.63 (s, 3H), 1.93–1.80 (m, 1H), 1.71–1.59 (m, 1H), 0.82 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.2, 156.2, 146.4, 141.2, 136.8, 136.2, 133.7, 129.2, 129.1, 128.7, 128.4, 127.7, 75.4, 27.7, 27.0, 10.5; **IR** (neat) 2963, 2925, 1698, 1681, 1602, 1265, 1180 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{22}H_{25}NO_3Na$ $(M + Na)^+$ 374.1732, found 374.1729.



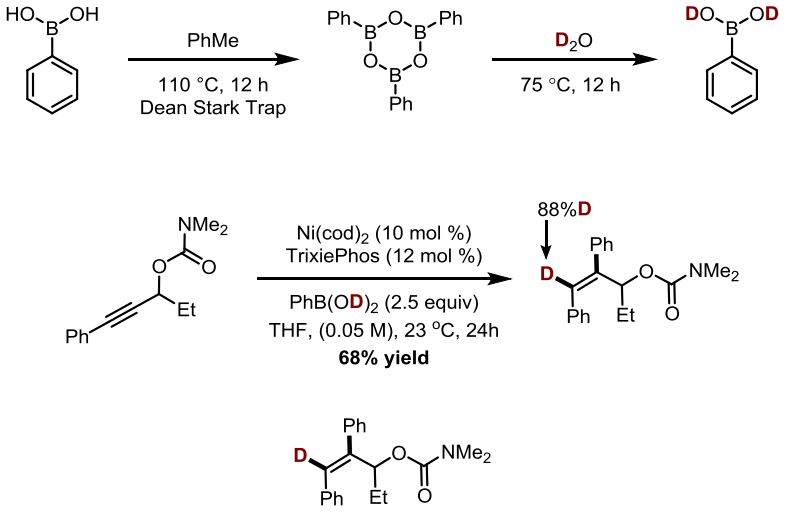
3.44. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 2-fluorophenylboronic acid (70 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column

chromatography to afford the title compound as a pale yellow oil (47 mg, 72% yield). **TLC R_f** = 0.4 (12% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.41–7.43 (m, 2H), 7.40–7.35 (m, 2H), 7.33–7.26 (m, 3H), 7.14–7.06 (m, 2H), 6.60 (s, 1H), 5.87 (dd, *J* = 8.4 Hz, *J* = 6.0 Hz, 1H), 2.89 (s, 3H), 2.68 (s, 3H), 1.81–1.58 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 160.6 (d, *J* = 245.5 Hz), 156.4, 134.1, (*J* = 1.4 Hz), 132.3 (*J* = 3.7 Hz), 131.9, 129.3, 129.21, 129.18, 128.7, 127.5, 123.6 (d, *J* = 3.7), 115.7 (d, *J* = 23.1 Hz), 75.3, 27.3, 10.5; **IR** (neat) 2931, 1698, 1487, 1391, 1182 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{20}H_{22}FNO_2$ $(M)^+$ 327.1635, found 327.1630.



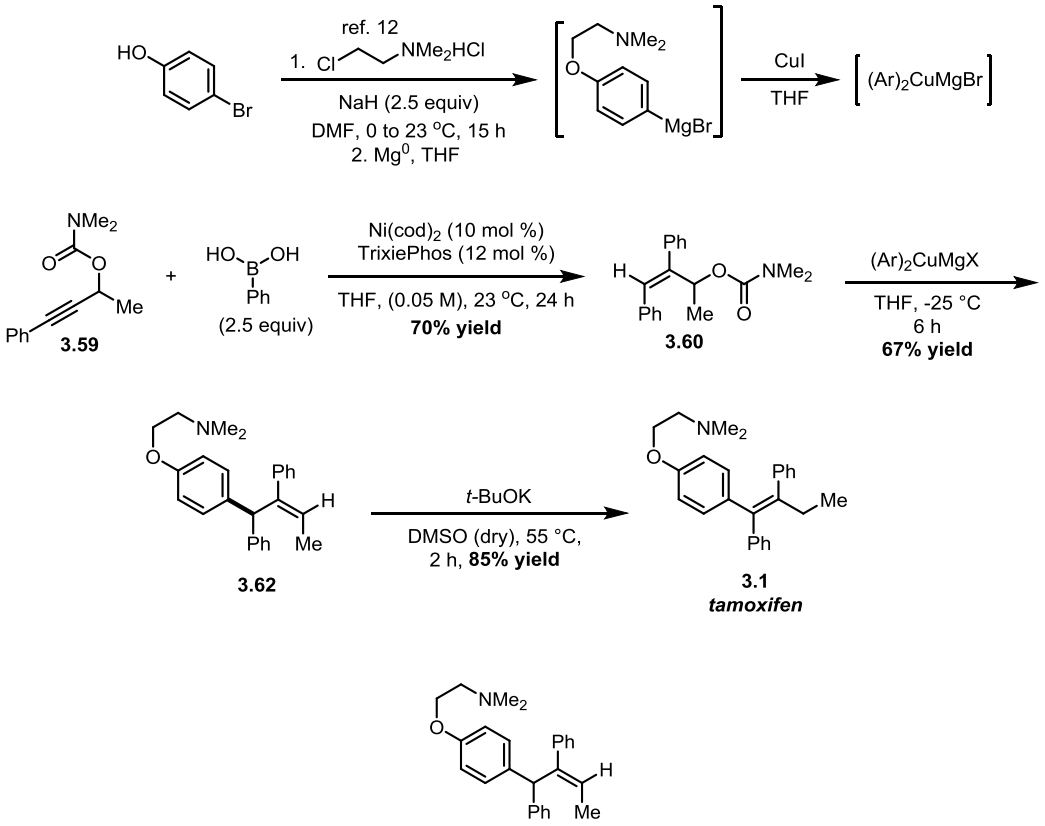
3.45. The product was prepared according to general procedure D using alkyne **3.18** (46 mg, 0.20 mmol), 4-(trifluoromethyl)phenylboronic acid (95 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (63 mg, 83% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.63–7.58 (m, 4H), 7.43–7.36 (m , 4H), 7.31–7.26 (m, 1H), 6.73 (s, 1H), 5.85 (t, *J* = 7.3 Hz, 1H), 2.89 (s, 3H), 2.73 (s, 3H), 1.88–1.80 (m, 1H), 1.68–1.59 (m, 1H), 0.83 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.2, 145.1 (q, *J* = 1.4 Hz), 140.9, 136.7, 133.7, 129.6 (q, *J* = 32.4 Hz), 129.23, 129.16, 128.7, 127.7, 125.2 (q, *J* = 3.7 Hz), 124.5 (q, *J* = 127.4 Hz), 75.4, 27.6, 10.5; **IR** (neat) 2969, 2934, 1699, 1322, 1164, 1122 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{21}H_{22}F_3NO_2Na$ $(M + Na)^+$ 400.1500, found 400.1489.

Deuterium labeling experiments



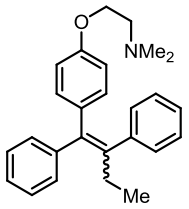
D-3.19. The product was prepared according to general procedure D using alkyne **3.18** (44 mg, 0.20 mmol), phenylboronic acid-D_2 (62 mg, 0.50 mmol), $Ni(cod)_2$ (5.5 mg, 0.020 mmol), TrixiePhos (8.0 mg, 0.020 mmol). The reaction mixture was eluted through a silica plug (with pure Et_2O) and concentrated under reduced pressure. Purified by flash column chromatography to afford the title compound as a pale yellow oil (42 mg, 68% yield). **TLC R_f** = 0.4 (15% EtOAc/hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51–7.46 (m, 2H), 7.43–7.23 (m, 8H), 6.71 (s, (12% H incorporation), 1H), 5.84 (t, *J* = 6.9 Hz, 1H), 2.88 (s, 3H), 2.7 (s, 3H), 1.85 (m, 1H), 1.65 (m, 3H), 0.82 (t, *J* = 7.8 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.3, 141.9, 141.4, 137.3, 132.6, 129.2, 128.9, 128.6, 128.2, 127.5, 127.3, 75.6, 27.5, 10.5; **IR** (neat) 2966, 2932, 1698, 1493, 1390, 1179 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{22}DNO_2Na$ $(M + Na)^+$ 333.1689, found 333.1677.

Synthesis of tamoxifen

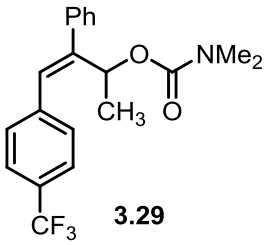


3.62. The Grignard reagent was prepared by adding to Taillefer's procedure.[3] A 25 mL round bottom flask was filled with aryl bromide (0.80 g, 3.3 mmol), magnesium turnings (160 mg, 6.6 mmol, 2 equiv), THF (3.0 mL), and 1,2-dibromoethane (70 µL). The mixture was stirred at room temperature for 3 hours, resulting in a solution of ~ 0.55 M of Grignard reagent. To a 7 dram vial was added CuI (76 mg, 0.40 mmol) and Grignard reagent (1.5 mL, 0.80 mmol) at 0 °C, and the mixture was stirred for 30 minutes. The mixture was then cooled to -25 °C and a solution of allylic carbamate (30 mg, 0.10 mmol, 0.10 M in THF) was added dropwise over 40 minutes. The mixture was stirred for 6 hours at –25 °C then quenched with saturated aqueous ammonium chloride, extracted with EtOAc, dried over Mg_2SO_4, and concentrated under reduced pressure. The product was purified by reverse-phase HPLC with H_2O/CH_3CN

(ZORBAX SB-C18 column, gradient elution of 40–70% CH3CN w/ 0.1% TFA) to afford the compound as a colorless oil (24 mg, 65% yield). **TLC R_f** = 0.3 (15% EtOAc/hexanes w/ 5% NEt_3); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37–7.28 (m, 5H), 7.27–7.16 (m, 7H), 6.78 (d, *J* = 8.7 Hz, 2H), 5.24 (q, *J* = 7.0 Hz, 1H), 5.08 (s, 1H), 4.30 (t, *J* = 3.8 Hz, 2H), 3.53 (t, *J* = 3.9 Hz, 2H), 2.99 (s, 6H), 1.58 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.7, 144.2, 142.9, 141.8, 136.8, 131.2, 129.8, 129.1, 128.5, 128.3, 126.8, 126.6, 126.5, 114.3, 62.6, 58.9, 57.2, 44.3, 15.3; **IR** (neat) 3027, 1609, 1509, 1238, 1175 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{26}H_{29}NOH$ $(M + H)^+$ 372.2327, found 372.2328.



3.1. The product was prepared according to a modified procedure reported by Taillefer.[3] **3.62** (9.1 mg, 0.024 mmol) was dissolved in dry DMSO (0.8 mL) and *t*-BuOK (14 mg, 0.12 mmol, 5 equiv) was added. The mixture was then heated at 50 °C for 5 h. The crude reaction mixture was then filtered through a pad of silica and eluted with Et_2O then concentrated under reduced pressure. The product was purified by flash column chromatography to afford the title compound as a colorless oil (7.6 mg, 84% yield, 1:1 *E:Z*). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38–7.33 (m, 2H), 7.30–7.23 (m, 4H), 7.21–7.08 (m, 12H), 7.02–6.97 (m, 2H), 6.93–6.87 (m, 4H), 6.80–6.75 (m, 2H), 6.59–6.55 (m, 2H), 4.10 (t, *J* = 6.1 Hz, 1H), 3.94 (t, *J* = 6.0 Hz, 1H), 2.76 (t, *J* = 5.8 Hz, 1H), 2.65 (t, *J* = 5.8 Hz, 1H), 2.52 (q, *J* = 7.4 Hz, 1H), 2.47 (q, *J* = 7.4 Hz, 1H), 2.36 (s, 6H), 2.29 (s, 6H), 0.94 (q, *J* = 8.6 Hz, 6H).



3.29

X-ray Data Collection, Structure Solution and Refinement for erj34.

A colorless crystal of approximate dimensions 0.220 x 0.246 x 0.338 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (15 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was $2/m$ and the systematic absences were consistent with the monoclinic space group $P2_1/n$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model. The fluorine atoms were disordered and included using multiple components with partial site-occupancy-factors (0.3333 each).

At convergence, wR2 = 0.1015 and Goof = 1.045 for 292 variables refined against 3679 data (0.80Å), R1 = 0.0375 for those 3245 data with $I > 2.0\sigma(I)$.

References.

1. APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

3. Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

4. Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014

5. International Tables for Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

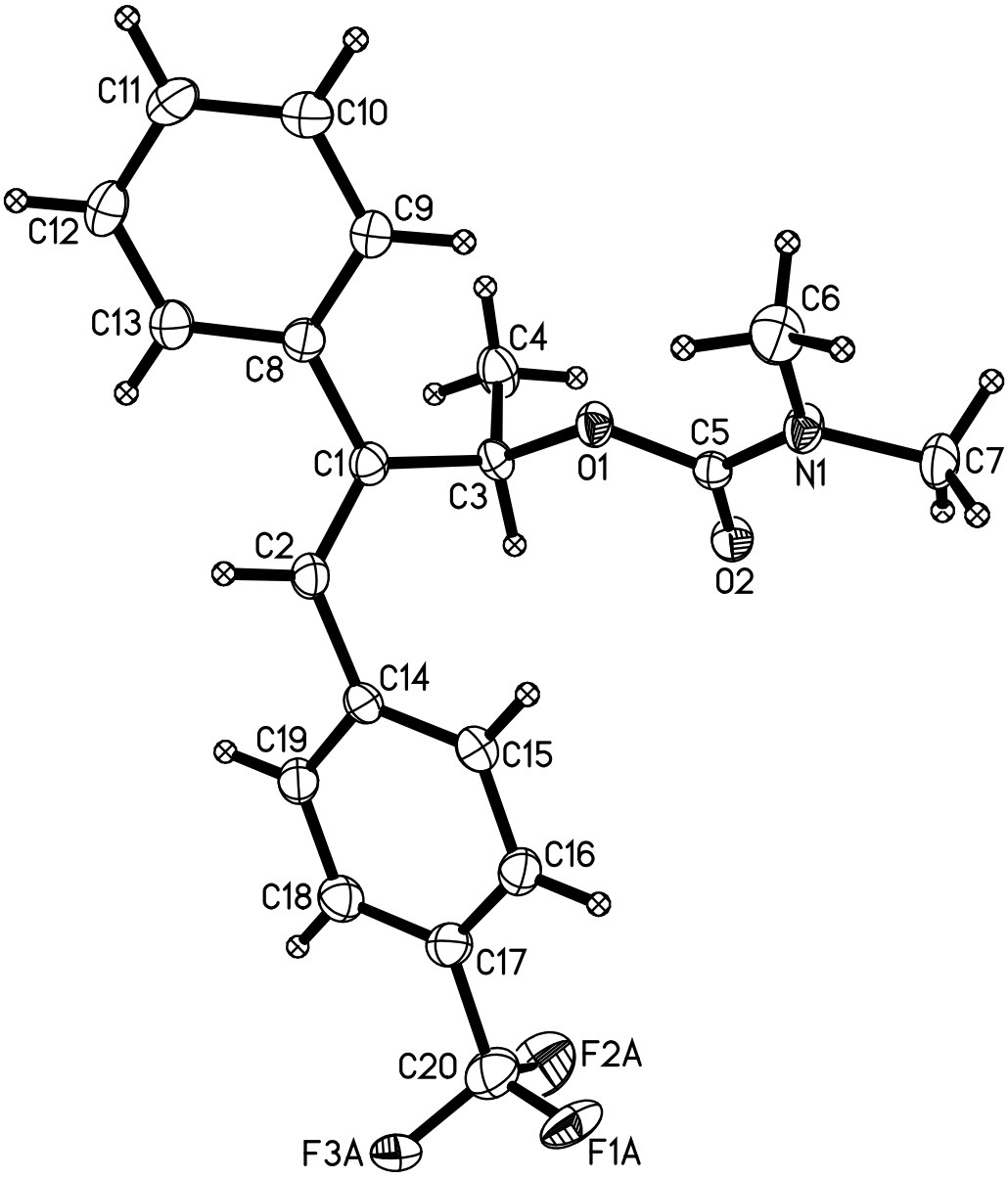
Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.


C11
C10
C12
C9
C13
C8
C4
C6
C1
C3
O1
C5
N1
C7
O2
C2
C14
C19
C15
C16
C18
C17
C20
F2A
F3A
F1A

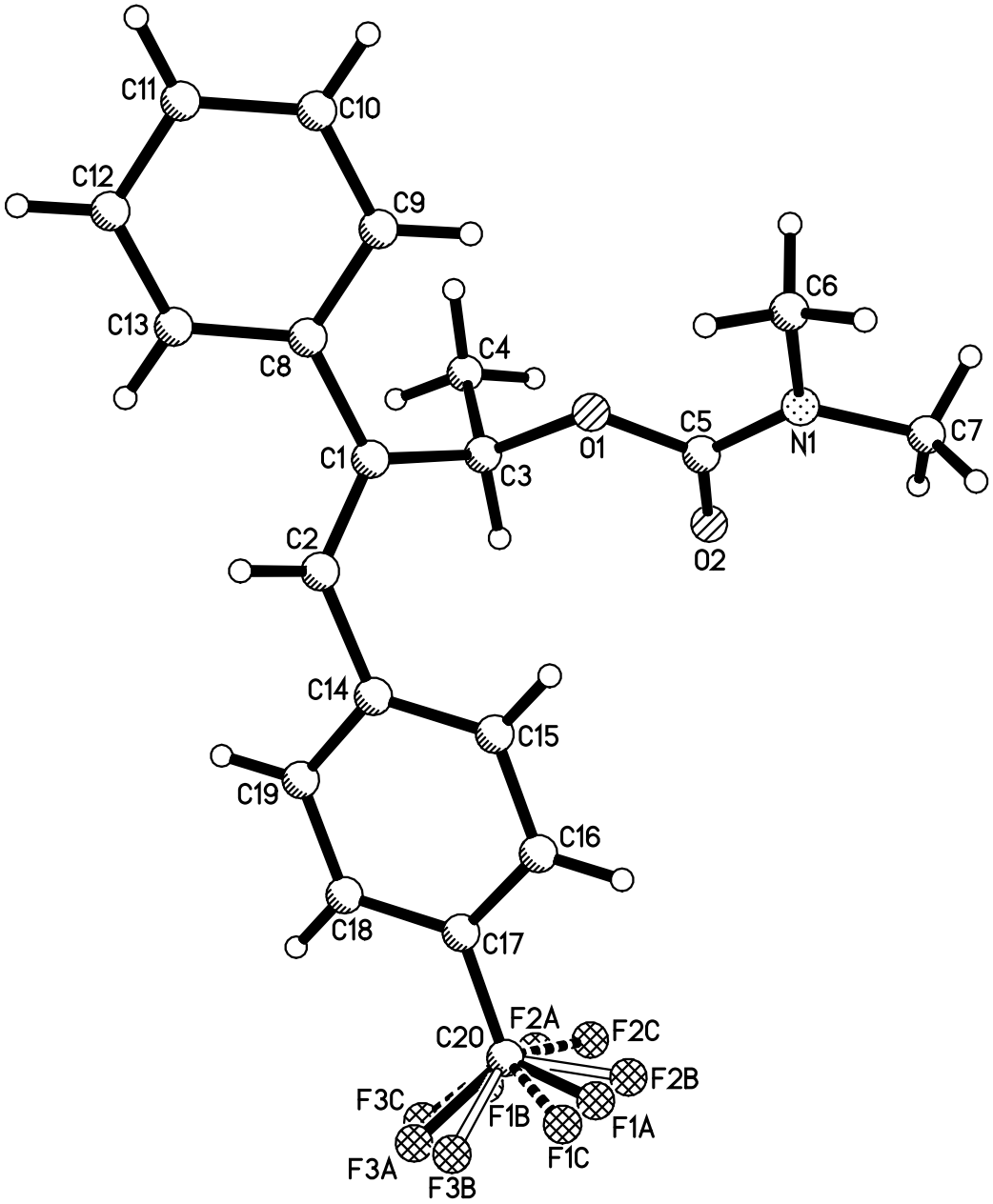

C11
C10
C12
C9
C13
C8
C4
C6
C1
C3
O1
C5
N1
C7
O2
C2
C14
C15
C19
C16
C18
C17
C20
F2A
F2C
F2B
F3C
F1B
F1A
F3A
F1C
F3B

Table 1. Crystal data and structure refinement for erj34.

Identification code	erj34 (Mikhail Konev)	
Empirical formula	C_{20} H_{20} F_3 N O_2	
Formula weight	363.37	
Temperature	128(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 6.6889(4) Å	□= 90°.
	b = 14.0475(8) Å	□= 91.9731(8)°.
	c = 19.3273(11) Å	□ = 90°.
Volume	1814.96(18) $Å^3$	
Z	4	
Density (calculated)	1.330 Mg/m^3	
Absorption coefficient	0.106 mm^{-1}	
F(000)	760	
Crystal color	colorless	
Crystal size	0.338 x 0.246 x 0.220 mm^3	
Theta range for data collection	1.792 to 26.372°	
Index ranges	$-8 \le h \le 8$, $-17 \le k \le 17$, $-24 \le l \le 24$	
Reflections collected	19096	
Independent reflections	3679 [R(int) = 0.0235]	
Completeness to theta = 25.500°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8621 and 0.8095	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3679 / 0 / 292	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I) = 3245 data]	R1 = 0.0375, wR2 = 0.0973	
R indices (all data, 0.80 Å)	R1 = 0.0431, wR2 = 0.1015	
Largest diff. peak and hole	0.318 and -0.333 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for erj34. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	9927(1)	8274(1)	7389(1)	19(1)
O(2)	7940(1)	9055(1)	6600(1)	22(1)
N(1)	11313(2)	8980(1)	6491(1)	22(1)
C(1)	8539(2)	7125(1)	8157(1)	19(1)
C(2)	7506(2)	6349(1)	7962(1)	21(1)
C(3)	8166(2)	8064(1)	7789(1)	18(1)
C(4)	7753(2)	8883(1)	8274(1)	28(1)
C(5)	9594(2)	8798(1)	6807(1)	17(1)
C(6)	13268(2)	8680(1)	6771(1)	32(1)
C(7)	11285(2)	9583(1)	5880(1)	27(1)
C(8)	9972(2)	7086(1)	8766(1)	20(1)
C(9)	11803(2)	7559(1)	8769(1)	30(1)
C(10)	13130(2)	7502(1)	9335(1)	34(1)
C(11)	12658(2)	6976(1)	9912(1)	27(1)
C(12)	10859(2)	6497(1)	9916(1)	28(1)
C(13)	9523(2)	6554(1)	9352(1)	25(1)
C(14)	6092(2)	6266(1)	7356(1)	20(1)
C(15)	6700(2)	6449(1)	6685(1)	22(1)
C(16)	5423(2)	6293(1)	6117(1)	24(1)
C(17)	3499(2)	5960(1)	6217(1)	24(1)
C(18)	2852(2)	5791(1)	6880(1)	26(1)
C(19)	4146(2)	5939(1)	7445(1)	24(1)
C(20)	2111(2)	5775(1)	5607(1)	36(1)
F(1A)	2869(11)	5712(10)	5051(4)	36(2)
F(2A)	776(14)	6494(8)	5547(5)	51(2)
F(3A)	940(20)	5007(8)	5707(5)	52(3)
F(1B)	333(13)	6077(14)	5647(4)	63(3)
F(2B)	2780(20)	6117(12)	4998(6)	61(3)
F(3B)	1980(30)	4798(4)	5477(7)	81(3)
F(1C)	2990(20)	5301(13)	5102(8)	73(5)

F(2C)	1560(30)	6630(7)	5327(11)	131(6)
F(3C)	493(19)	5367(16)	5773(5)	79(6)

Table 3. Bond lengths [Å] and angles [°] for erj34.

O(1)-C(5)	1.3566(14)
O(1)-C(3)	1.4617(13)
O(2)-C(5)	1.2183(14)
N(1)-C(5)	1.3448(15)
N(1)-C(7)	1.4524(16)
N(1)-C(6)	1.4600(16)
C(1)-C(2)	1.3371(17)
C(1)-C(8)	1.4938(16)
C(1)-C(3)	1.5152(16)
C(2)-C(14)	1.4831(16)
C(3)-C(4)	1.5154(17)
C(8)-C(9)	1.3937(18)
C(8)-C(13)	1.3966(17)
C(9)-C(10)	1.3881(18)
C(10)-C(11)	1.3816(19)
C(11)-C(12)	1.379(2)
C(12)-C(13)	1.3880(18)
C(14)-C(15)	1.3965(17)
C(14)-C(19)	1.3966(18)
C(15)-C(16)	1.3857(17)
C(16)-C(17)	1.3887(18)
C(17)-C(18)	1.3866(19)
C(17)-C(20)	1.4984(18)
C(18)-C(19)	1.3855(18)
C(20)-F(1A)	1.207(7)
C(20)-F(1B)	1.268(7)
C(20)-F(3C)	1.275(9)
C(20)-F(1C)	1.333(11)
C(20)-F(2A)	1.351(9)
C(20)-F(3A)	1.351(8)
C(20)-F(2B)	1.362(11)
C(20)-F(2C)	1.363(9)
C(20)-F(3B)	1.397(6)

C(5)-O(1)-C(3)	115.85(9)
C(5)-N(1)-C(7)	119.52(10)
C(5)-N(1)-C(6)	123.09(10)
C(7)-N(1)-C(6)	117.10(10)
C(2)-C(1)-C(8)	120.27(11)
C(2)-C(1)-C(3)	120.12(11)
C(8)-C(1)-C(3)	119.49(10)
C(1)-C(2)-C(14)	126.59(11)
O(1)-C(3)-C(1)	107.50(9)
O(1)-C(3)-C(4)	109.99(9)
C(1)-C(3)-C(4)	113.73(10)
O(2)-C(5)-N(1)	125.09(11)
O(2)-C(5)-O(1)	123.74(10)
N(1)-C(5)-O(1)	111.17(10)
C(9)-C(8)-C(13)	117.76(11)
C(9)-C(8)-C(1)	121.66(11)
C(13)-C(8)-C(1)	120.56(11)
C(10)-C(9)-C(8)	120.86(12)
C(11)-C(10)-C(9)	120.61(13)
C(12)-C(11)-C(10)	119.30(12)
C(11)-C(12)-C(13)	120.37(12)
C(12)-C(13)-C(8)	121.10(12)
C(15)-C(14)-C(19)	118.50(11)
C(15)-C(14)-C(2)	121.30(11)
C(19)-C(14)-C(2)	120.06(11)
C(16)-C(15)-C(14)	121.04(12)
C(15)-C(16)-C(17)	119.43(12)
C(18)-C(17)-C(16)	120.49(12)
C(18)-C(17)-C(20)	119.49(12)
C(16)-C(17)-C(20)	120.02(12)
C(19)-C(18)-C(17)	119.71(12)
C(18)-C(19)-C(14)	120.82(12)
F(3C)-C(20)-F(1C)	111.0(7)
F(1A)-C(20)-F(2A)	105.9(5)
F(1A)-C(20)-F(3A)	109.4(5)
F(2A)-C(20)-F(3A)	102.9(5)

F(1B)-C(20)-F(2B)	105.7(6)
F(3C)-C(20)-F(2C)	106.0(7)
F(1C)-C(20)-F(2C)	105.6(7)
F(1B)-C(20)-F(3B)	106.8(5)
F(2B)-C(20)-F(3B)	102.1(5)
F(1A)-C(20)-C(17)	116.5(4)
F(1B)-C(20)-C(17)	116.8(4)
F(3C)-C(20)-C(17)	112.9(5)
F(1C)-C(20)-C(17)	112.8(5)
F(2A)-C(20)-C(17)	109.1(5)
F(3A)-C(20)-C(17)	111.9(3)
F(2B)-C(20)-C(17)	113.9(5)
F(2C)-C(20)-C(17)	108.1(4)
F(3B)-C(20)-C(17)	110.2(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj34. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	17(1)	22(1)	20(1)	5(1)	3(1)	0(1)
O(2)	17(1)	24(1)	24(1)	4(1)	0(1)	2(1)
N(1)	18(1)	28(1)	20(1)	6(1)	4(1)	1(1)
C(1)	20(1)	20(1)	18(1)	0(1)	4(1)	-1(1)
C(2)	26(1)	17(1)	20(1)	3(1)	1(1)	0(1)
C(3)	18(1)	18(1)	20(1)	1(1)	5(1)	-3(1)
C(4)	36(1)	19(1)	28(1)	-1(1)	10(1)	-2(1)
C(5)	19(1)	14(1)	17(1)	-2(1)	2(1)	0(1)
C(6)	16(1)	45(1)	35(1)	8(1)	3(1)	2(1)
C(7)	29(1)	32(1)	21(1)	6(1)	7(1)	-2(1)
C(8)	25(1)	18(1)	18(1)	-1(1)	1(1)	1(1)
C(9)	35(1)	34(1)	22(1)	7(1)	-3(1)	-12(1)
C(10)	32(1)	40(1)	28(1)	4(1)	-6(1)	-13(1)
C(11)	32(1)	30(1)	19(1)	-2(1)	-4(1)	3(1)
C(12)	34(1)	31(1)	18(1)	4(1)	4(1)	2(1)
C(13)	26(1)	28(1)	22(1)	2(1)	3(1)	-2(1)
C(14)	26(1)	12(1)	22(1)	0(1)	-1(1)	0(1)
C(15)	23(1)	18(1)	25(1)	1(1)	2(1)	-1(1)
C(16)	30(1)	21(1)	21(1)	1(1)	2(1)	2(1)
C(17)	26(1)	21(1)	25(1)	0(1)	-4(1)	2(1)
C(18)	22(1)	27(1)	29(1)	2(1)	0(1)	-2(1)
C(19)	28(1)	21(1)	22(1)	2(1)	2(1)	-1(1)
C(20)	33(1)	43(1)	30(1)	4(1)	-8(1)	-1(1)
F(1A)	37(3)	55(6)	16(3)	-10(5)	1(2)	2(6)
F(2A)	39(5)	61(6)	51(3)	10(3)	-18(3)	18(4)
F(3A)	80(8)	43(5)	30(4)	6(3)	-26(4)	-33(4)
F(1B)	29(3)	124(11)	38(4)	-20(6)	-11(2)	16(6)
F(2B)	62(5)	94(8)	26(2)	20(5)	-13(2)	-30(6)
F(3B)	130(7)	36(3)	72(5)	-13(3)	-64(5)	-2(5)
F(1C)	52(3)	108(10)	58(6)	-57(7)	-23(3)	30(6)
F(2C)	160(12)	80(5)	144(12)	32(8)	-122(9)	-4(9)

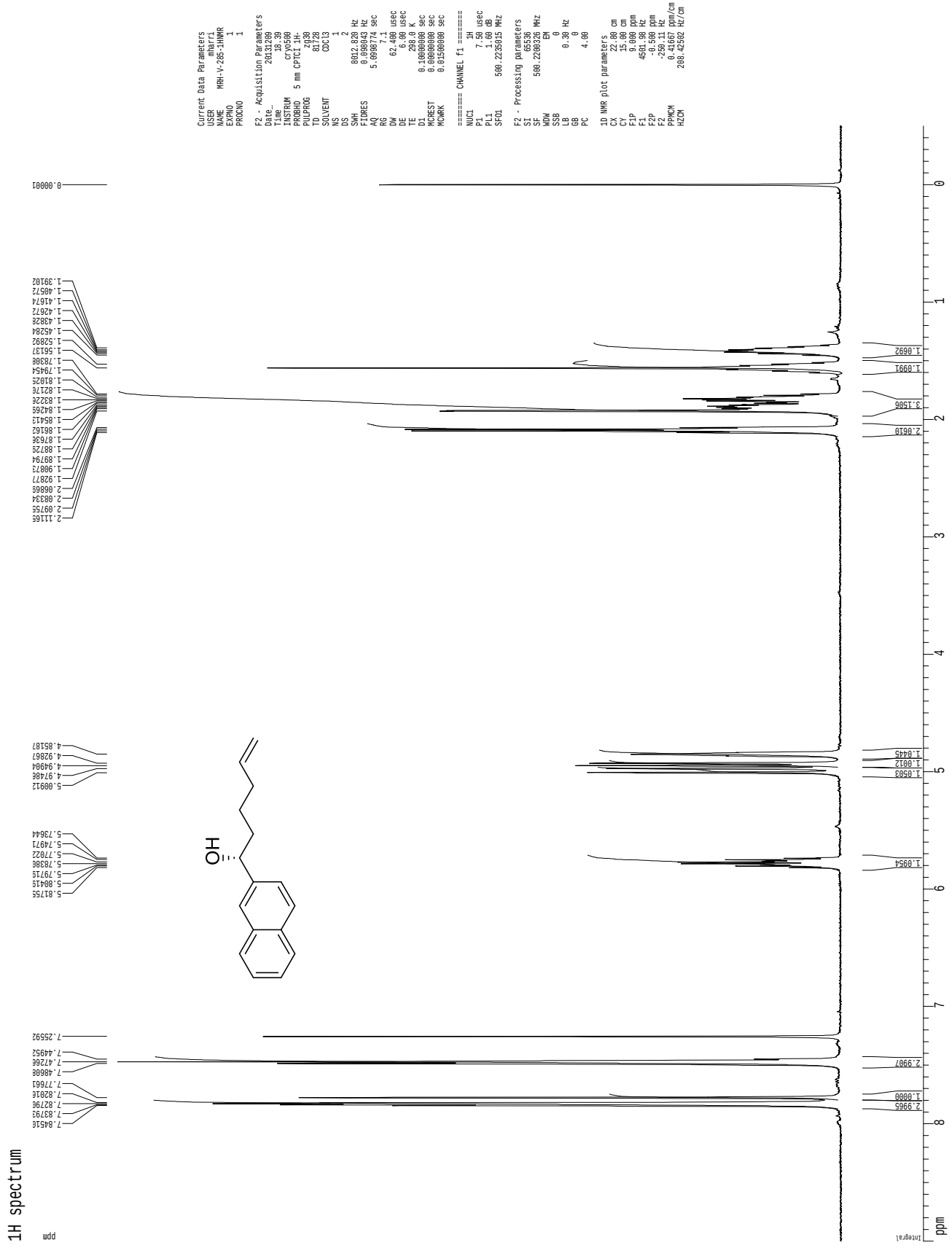
F(3C)	43(5)	146(15)	49(4)	-34(7)	2(3)	-49(8)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj34.

	x	y	z	U(eq)
H(2A)	7697	5796	8238	25
H(3A)	6988	7988	7461	22
H(4A)	6601	8727	8553	41
H(4B)	7462	9459	8003	41
H(4C)	8928	8993	8580	41
H(6A)	14169	8571	6390	48
H(6B)	13122	8090	7035	48
H(6C)	13824	9179	7076	48
H(7A)	11745	9217	5484	41
H(7B)	12172	10130	5963	41
H(7C)	9919	9809	5781	41
H(9A)	12148	7925	8377	36
H(10A)	14374	7827	9327	41
H(11A)	13562	6945	10301	33
H(12A)	10532	6126	10307	33
H(13A)	8282	6226	9364	30
H(15A)	8011	6684	6617	26
H(16A)	5859	6412	5663	29
H(18A)	1526	5575	6947	31
H(19A)	3705	5815	7898	28

References

[i] Panteleev, J.; Huang, R. Y.; Lui, E. K. J.; Lautens, M. *Org. Lett.* **2011**, *13*, 5314.
[2] Oestreich, M.; Fröhlich, R.; Hoppe, D. *J. Org. Chem*. **1999**, *64*, 8616.
[3] Danoun, G.; Tlili, A.; Monnier, F.; Taillefer, M. *Angew. Chem. Int. Ed*. **2012**, *51*, 12815.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PROCNO 1
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PULPROG zg30
TD 65728
SOLVENT CDCl3
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DS 2
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FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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PL1 1.00 dB
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F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
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CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.464 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
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1.41674
1.42672
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1.45284
1.52892
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1.78308
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1.81025
1.82176
1.83226
1.84265
1.85413
1.86162
1.87636
1.88725
1.89794
1.90873
1.92877
2.06865
2.08334
2.09755
2.11165
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4.92867
4.94904
4.97486
5.00912
5.73644
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7.47266
7.48608
7.76681
7.82016
7.82796
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7.84516
1.06920
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3.15066
2.06180
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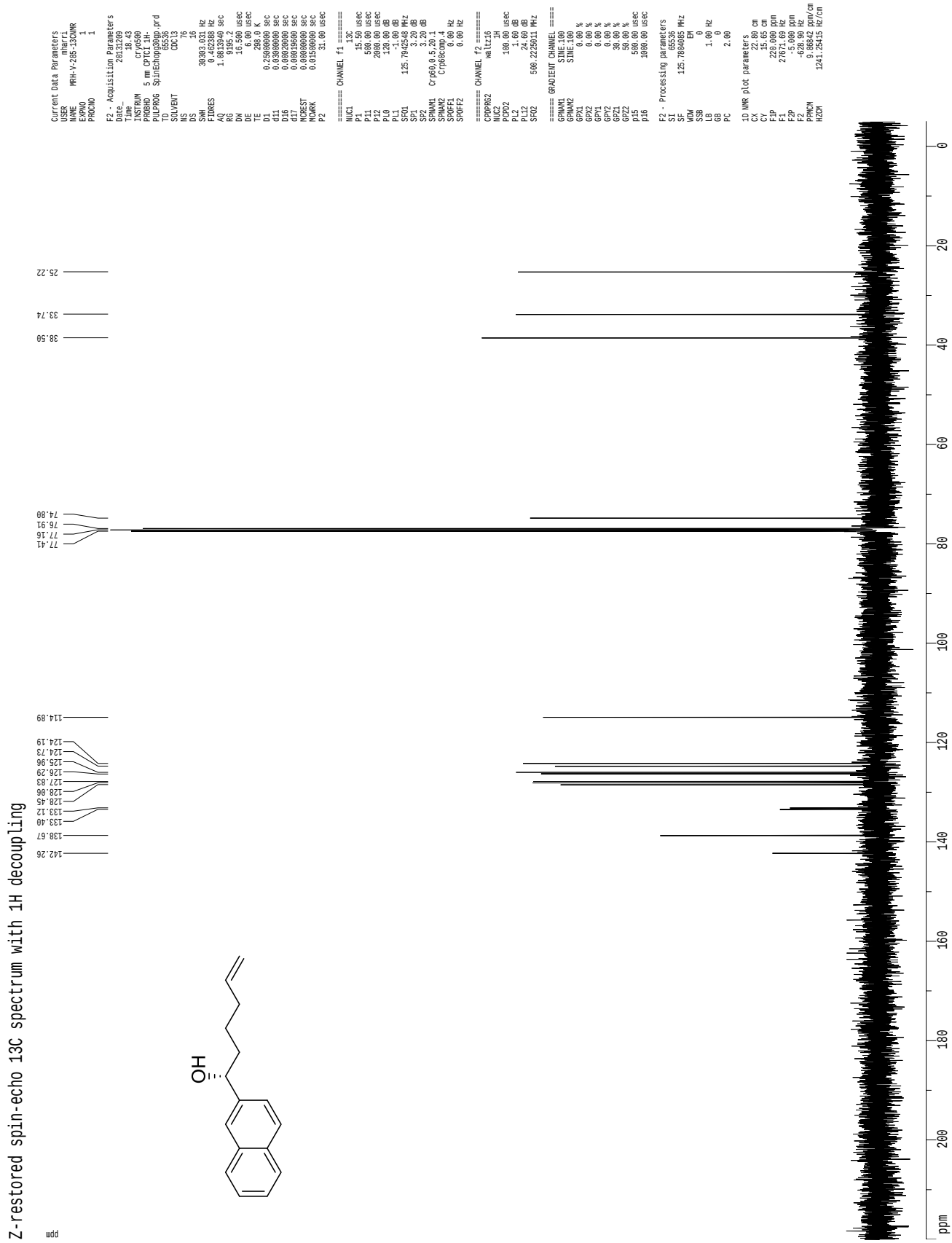
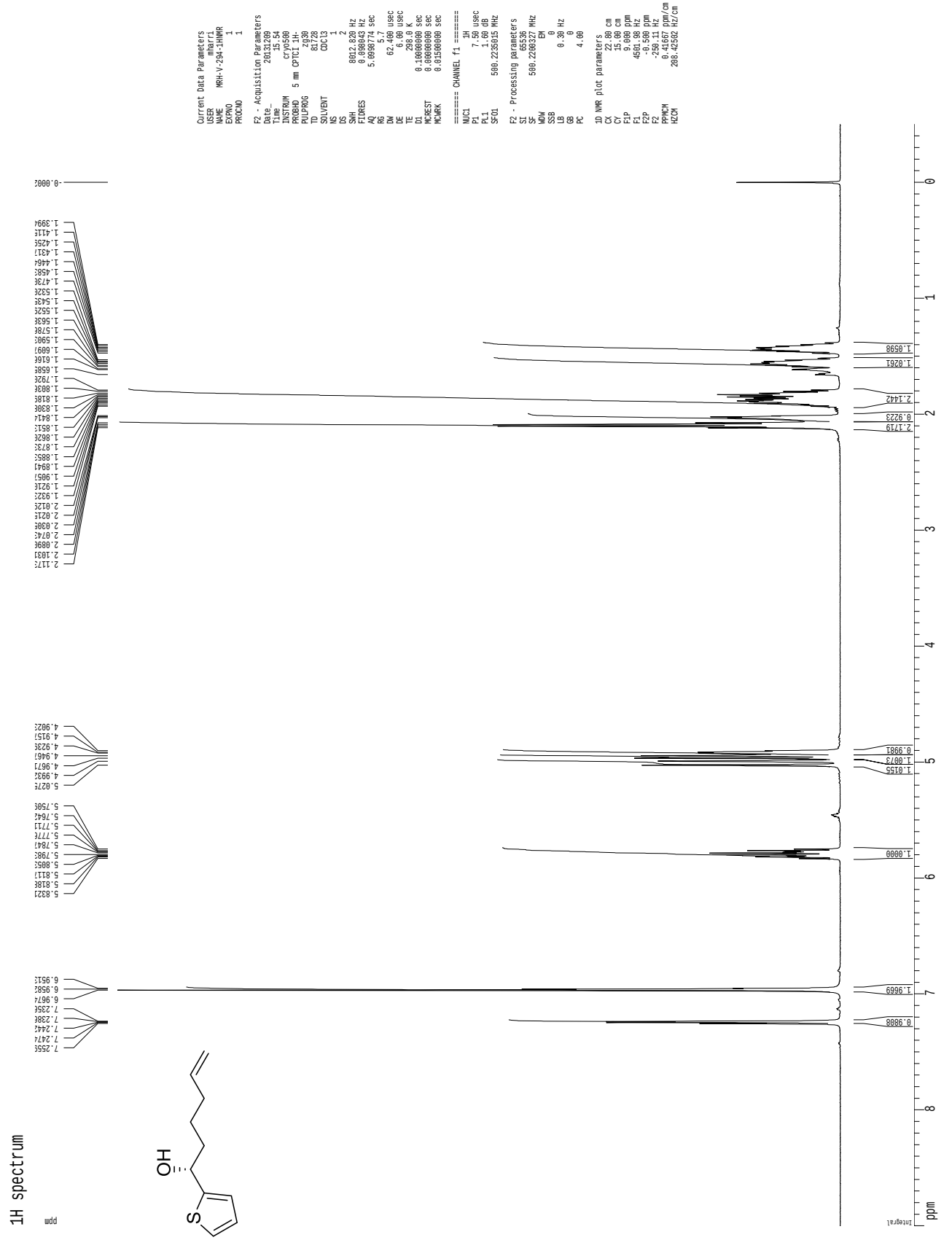
Z-restored spin-echo 13C spectrum with 1H decoupling

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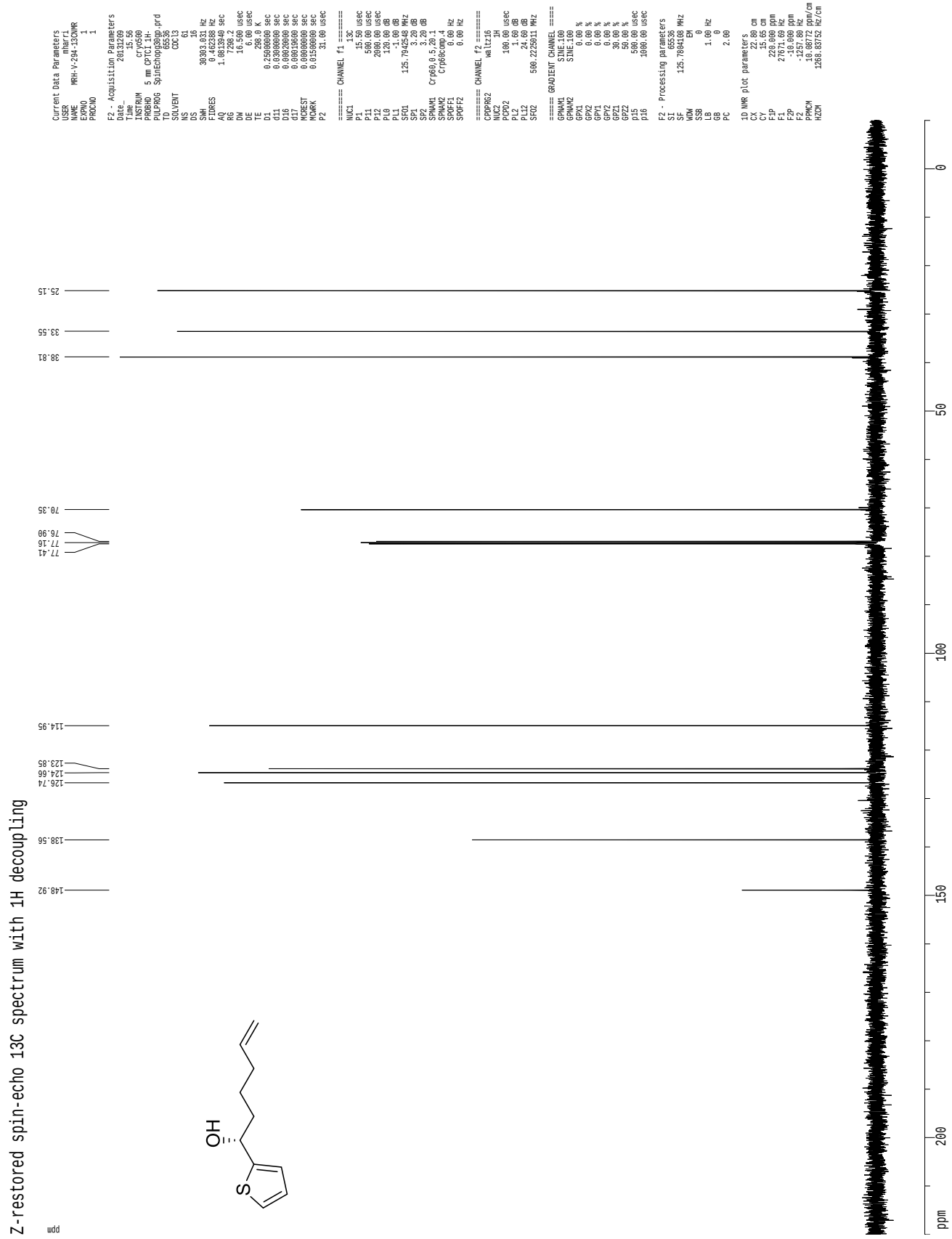
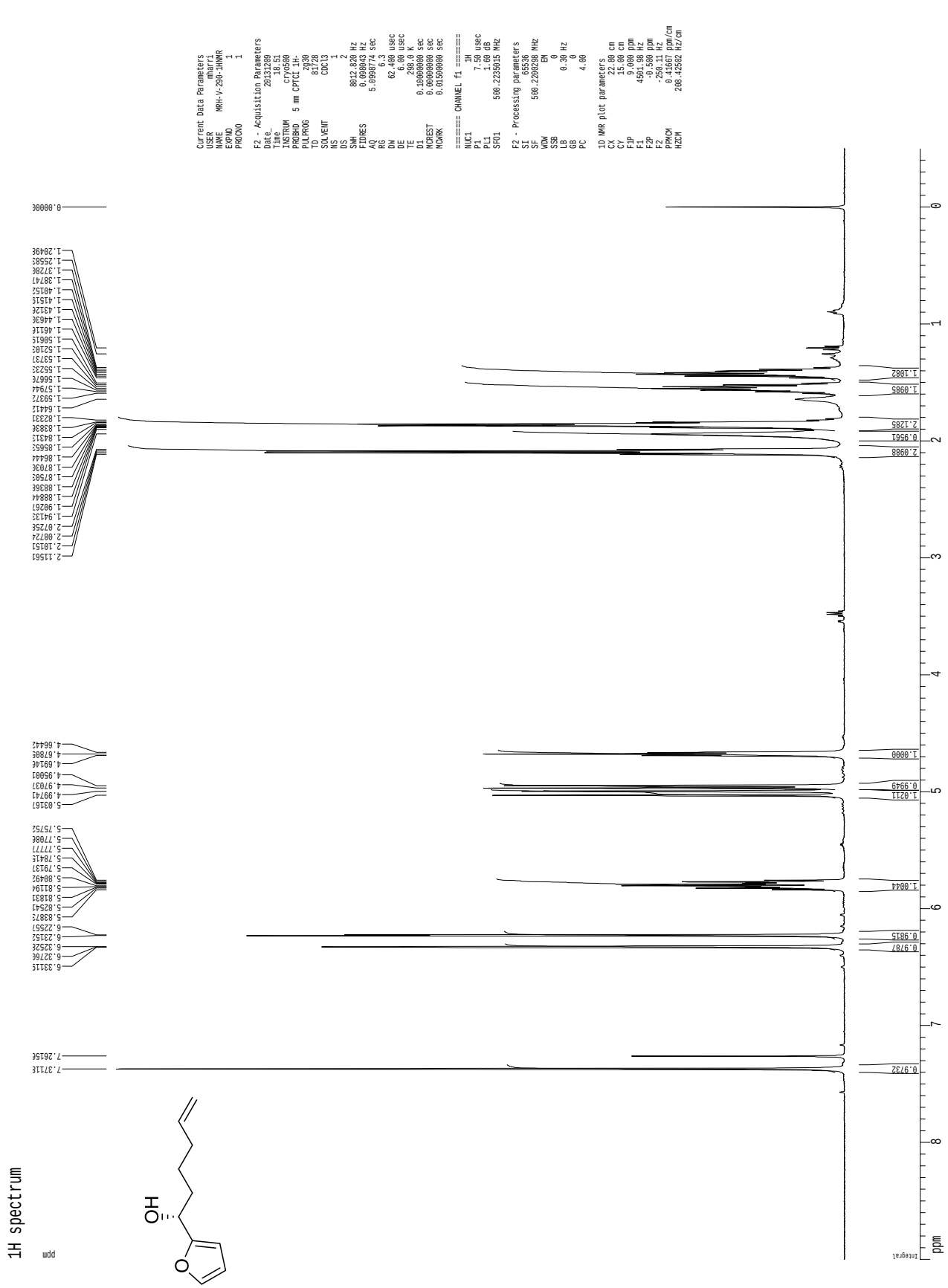
OH
S
Integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

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TD 81728
SOLVENT CDCl3
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RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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LB 0.30 Hz
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PC 4.00
1D NMR plot parameters
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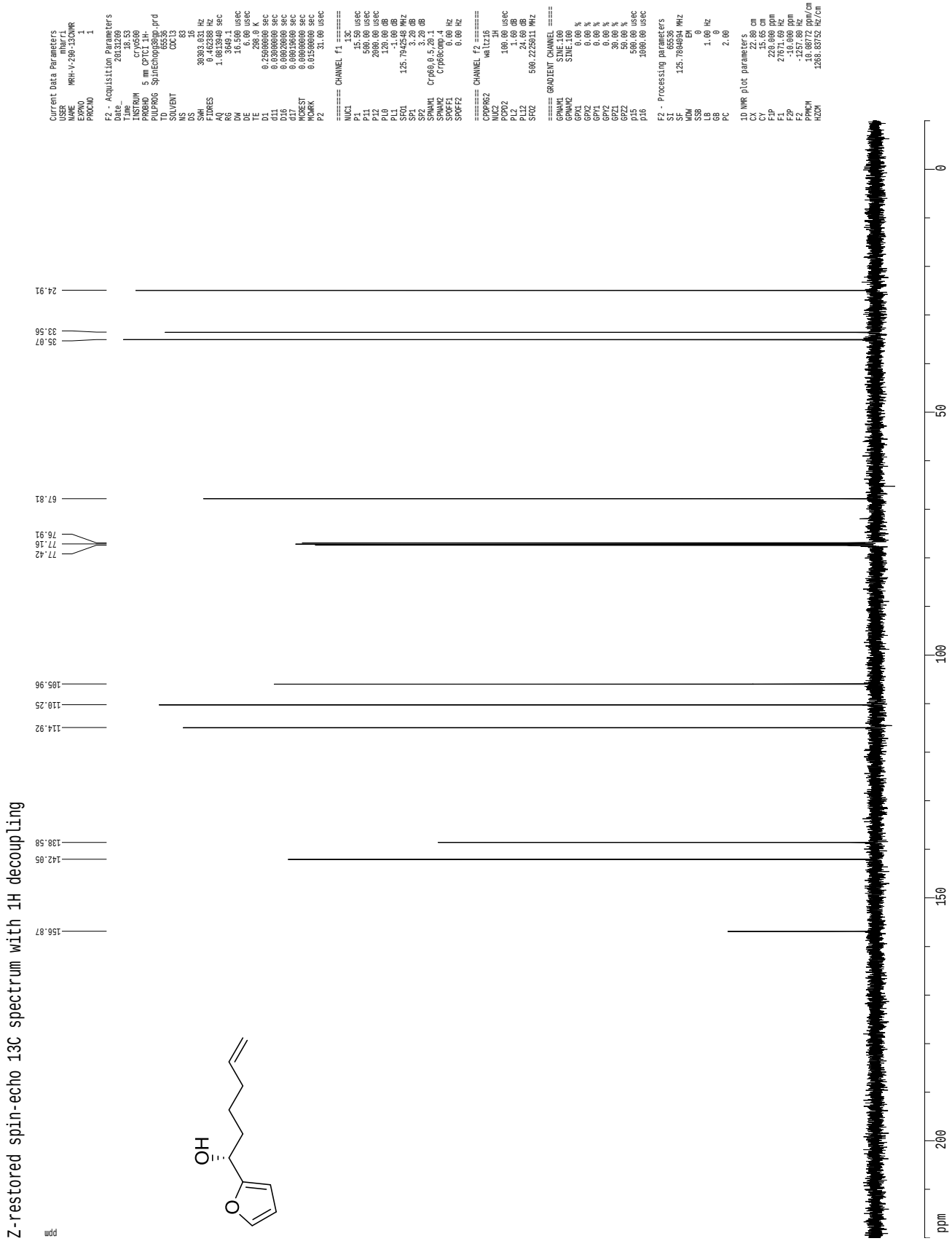
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
ppm
Integral
OH
O


Z-restored spin-echo 13C spectrum with 1H decoupling
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110.25
114.92
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142.05
156.87
OH
O
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PROCNO 1
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PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
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DS 16
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FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3649.1
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019060 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
====== CHANNEL f1 ======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
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SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
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PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
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GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
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WDW EM
SSB 0
LB 1.00 Hz
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PC 2.00
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F2P -10.000 ppm
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150
200
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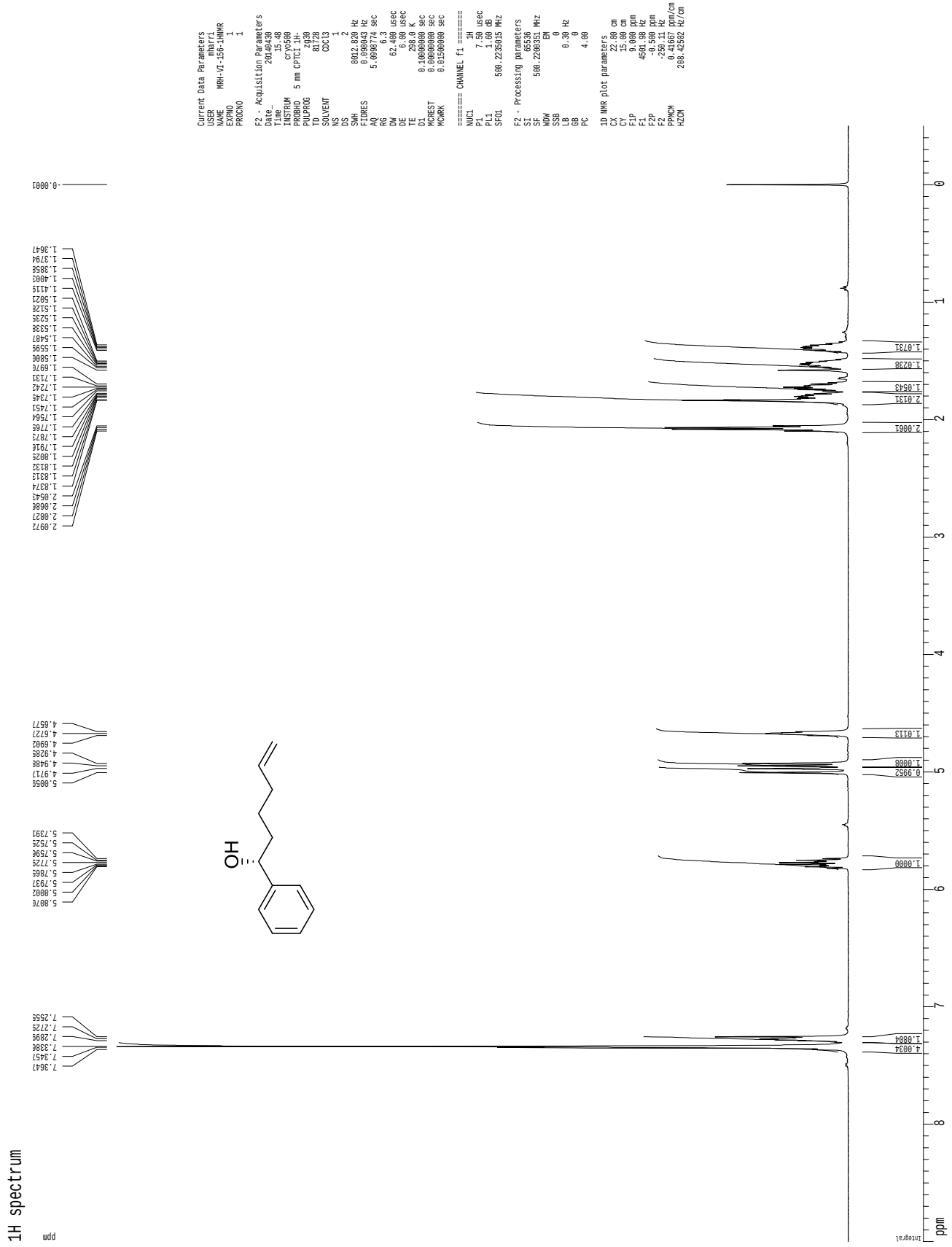
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TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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NUC1 1H
P1 7.50 usec
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SFO1 500.2235015 MHz
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LB 0.30 Hz
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CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.459 ppm
F2 -229.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
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7.3386
7.2899
7.2726
7.2556
5.8076
5.8002
5.7937
5.7866
5.7726
5.7596
5.7526
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2.0543
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1.7345
1.7242
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1.6976
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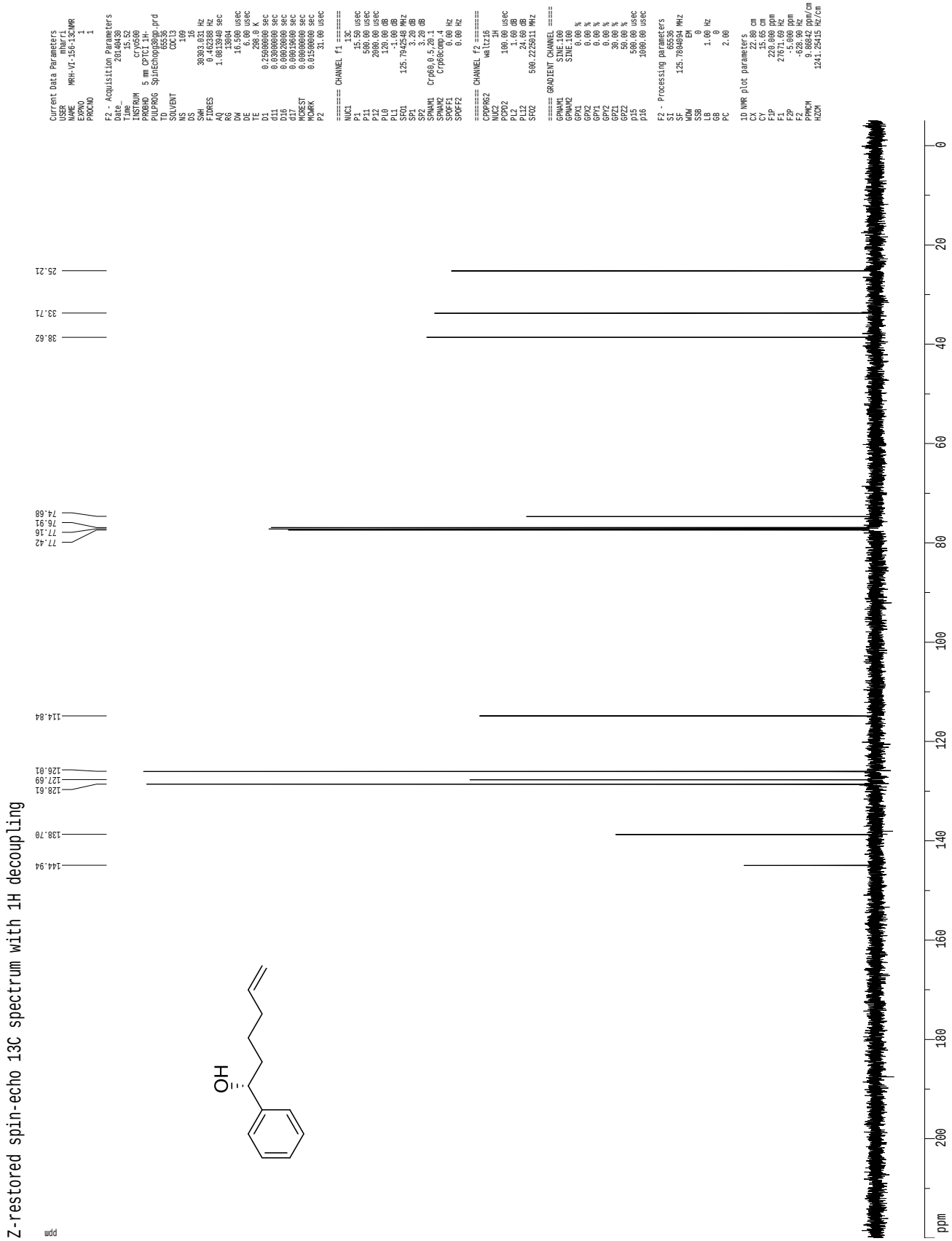
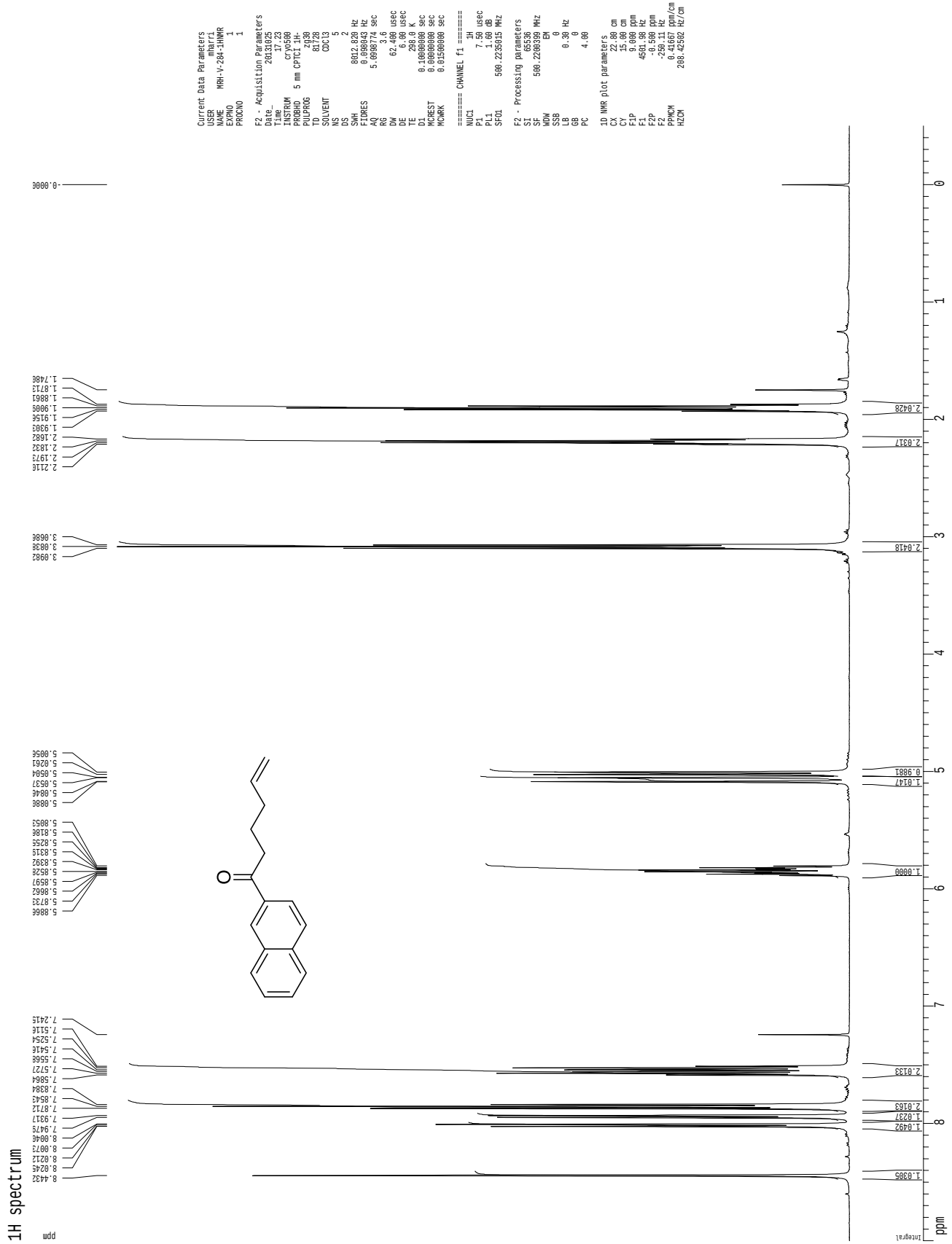
Z-restored spin-echo 13C spectrum with 1H decoupling

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PULPROG zg30
TD 81728
SOLVENT CDCl3
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DS 2
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FIDRES 0.098043 Hz
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RG 3.6
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
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MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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PL1 1.00 dB
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F2 - Processing parameters
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SF 500.2200390 MHz
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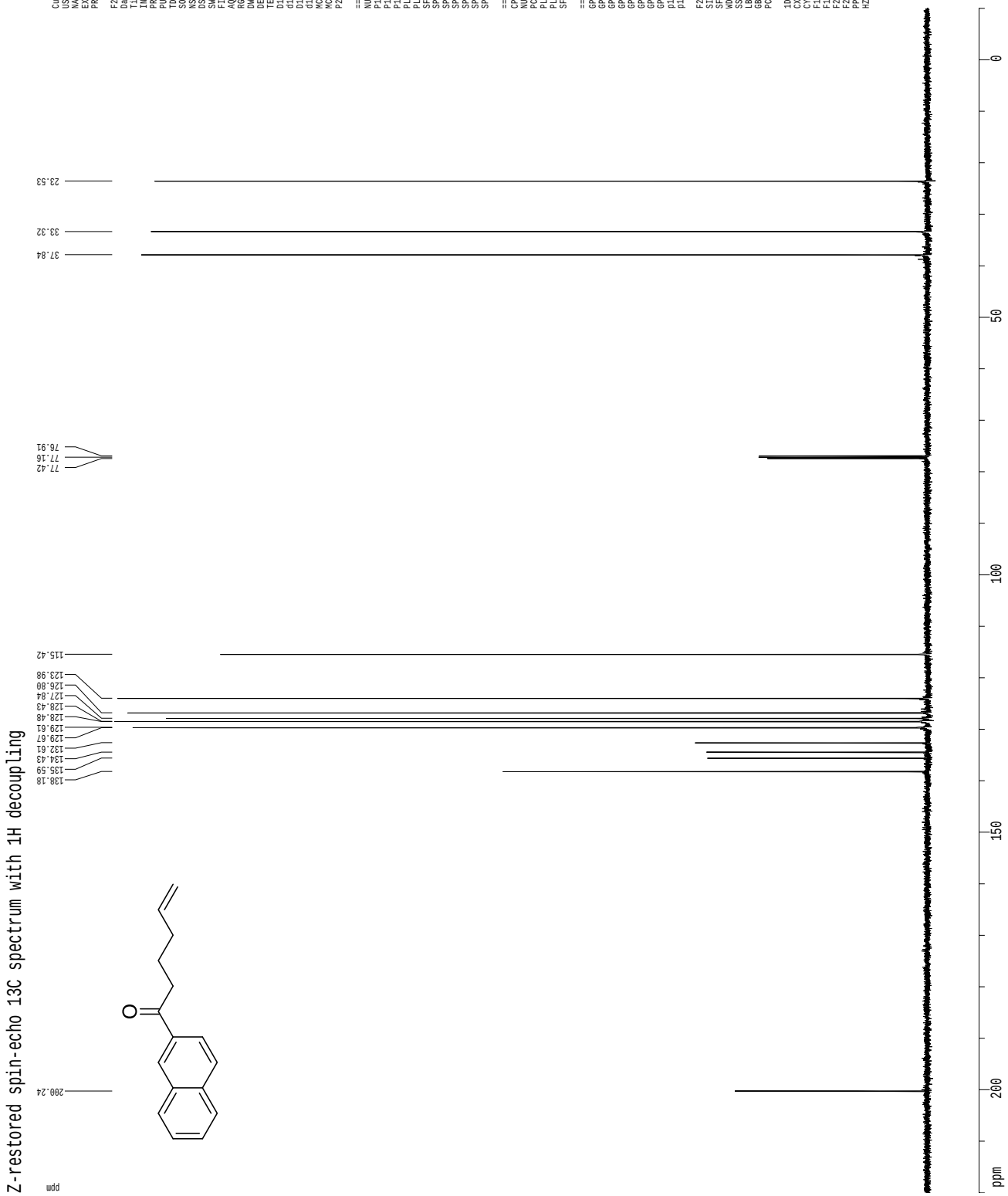
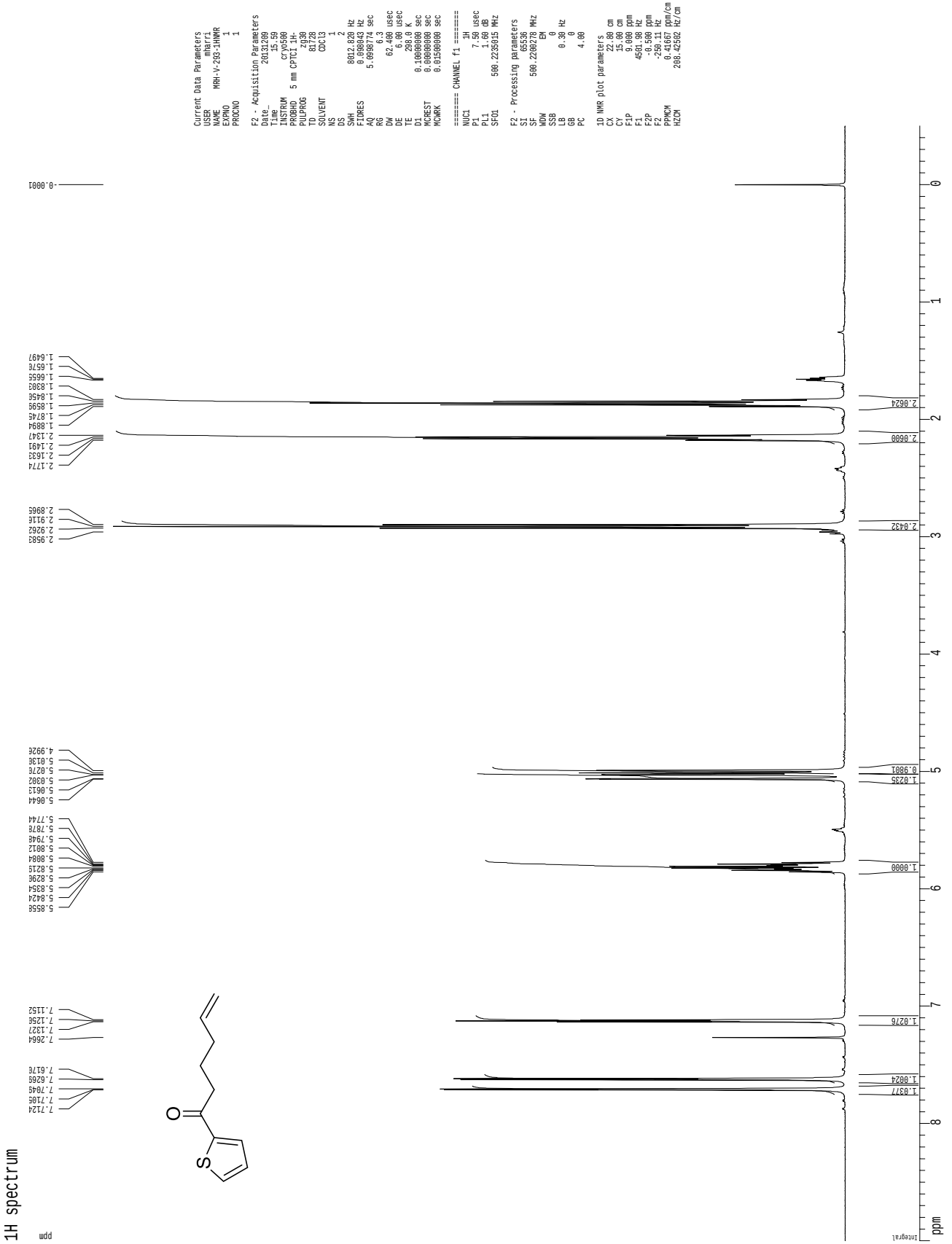
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

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Integral
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PROCNO 1
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Time 15.59
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PULPROG zg30
TD 65728
SOLVENT CDCl3
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DS 2
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FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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NUC1 1H
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LB 0.30 Hz
GB 0
PC 4.00
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CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
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1.8303
1.8456
1.8596
1.8746
1.8894
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7.7124
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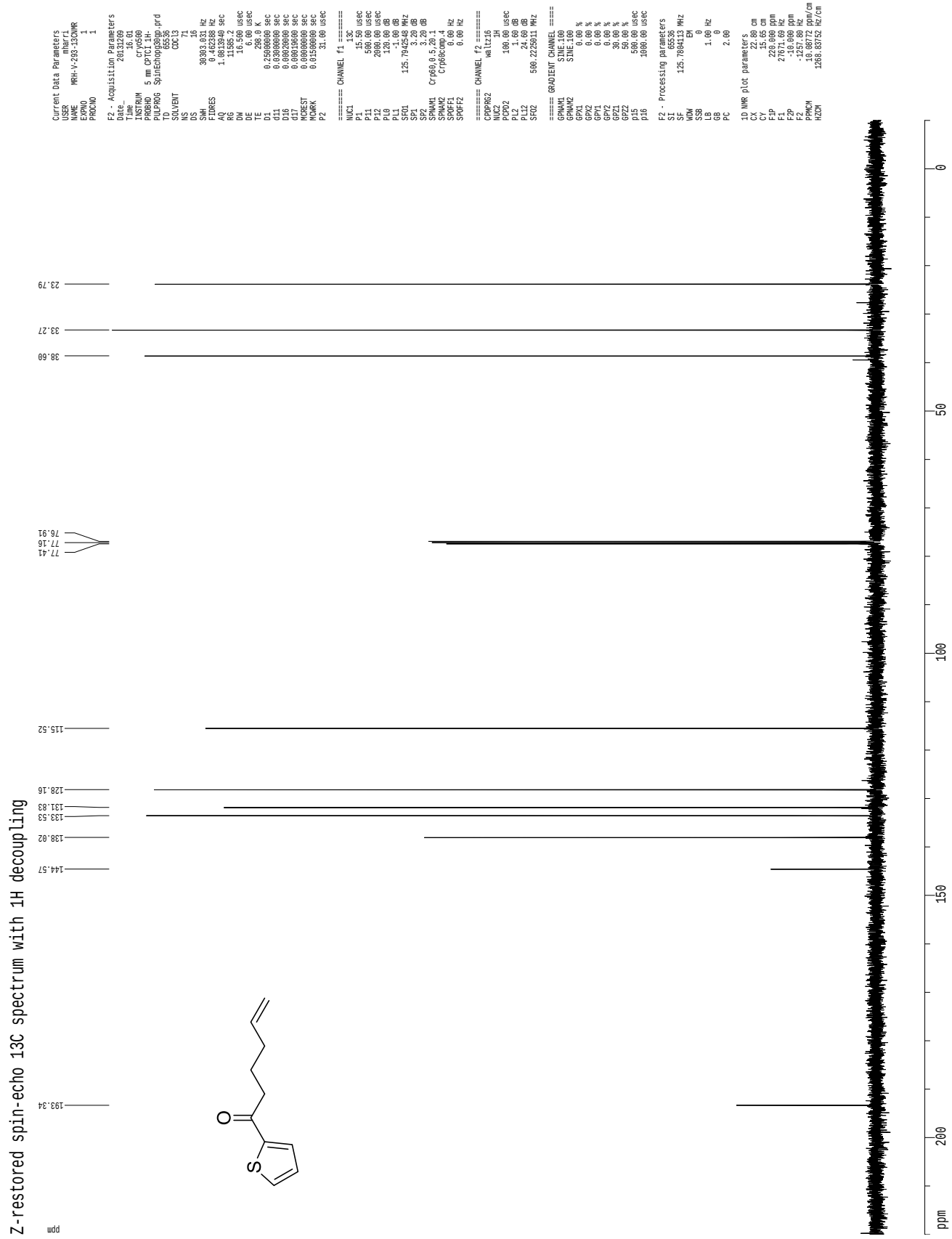
O
S


Z-restored spin-echo 13C spectrum with 1H decoupling
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115.52
128.16
131.83
133.53
138.02
144.57
193.34
O
S
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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 16.01
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TD 65536
SOLVENT CDCl3
NS 16
DS 16
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RG 11585.2
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TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
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P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
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SPOFF2 0.00 Hz
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CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
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GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm
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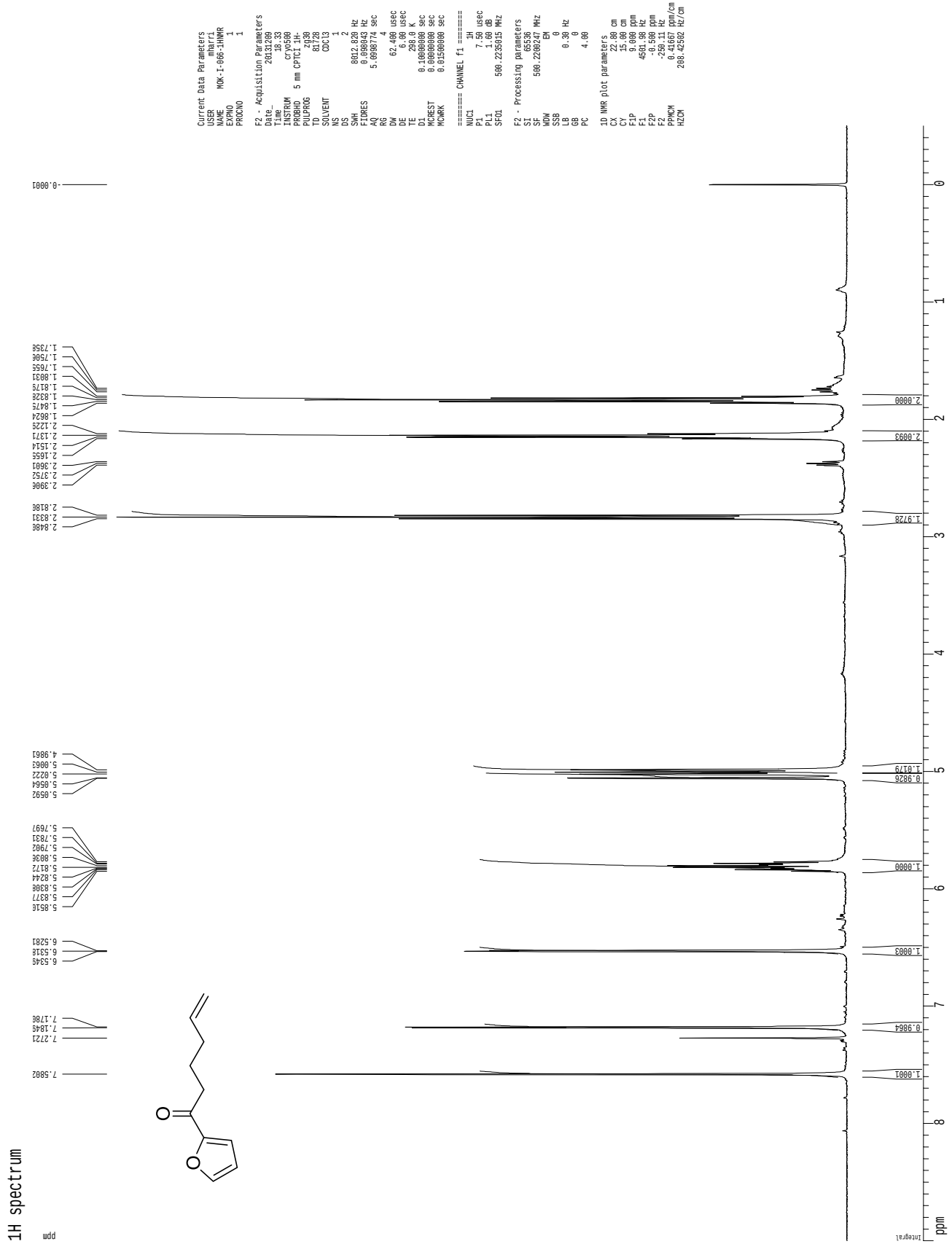
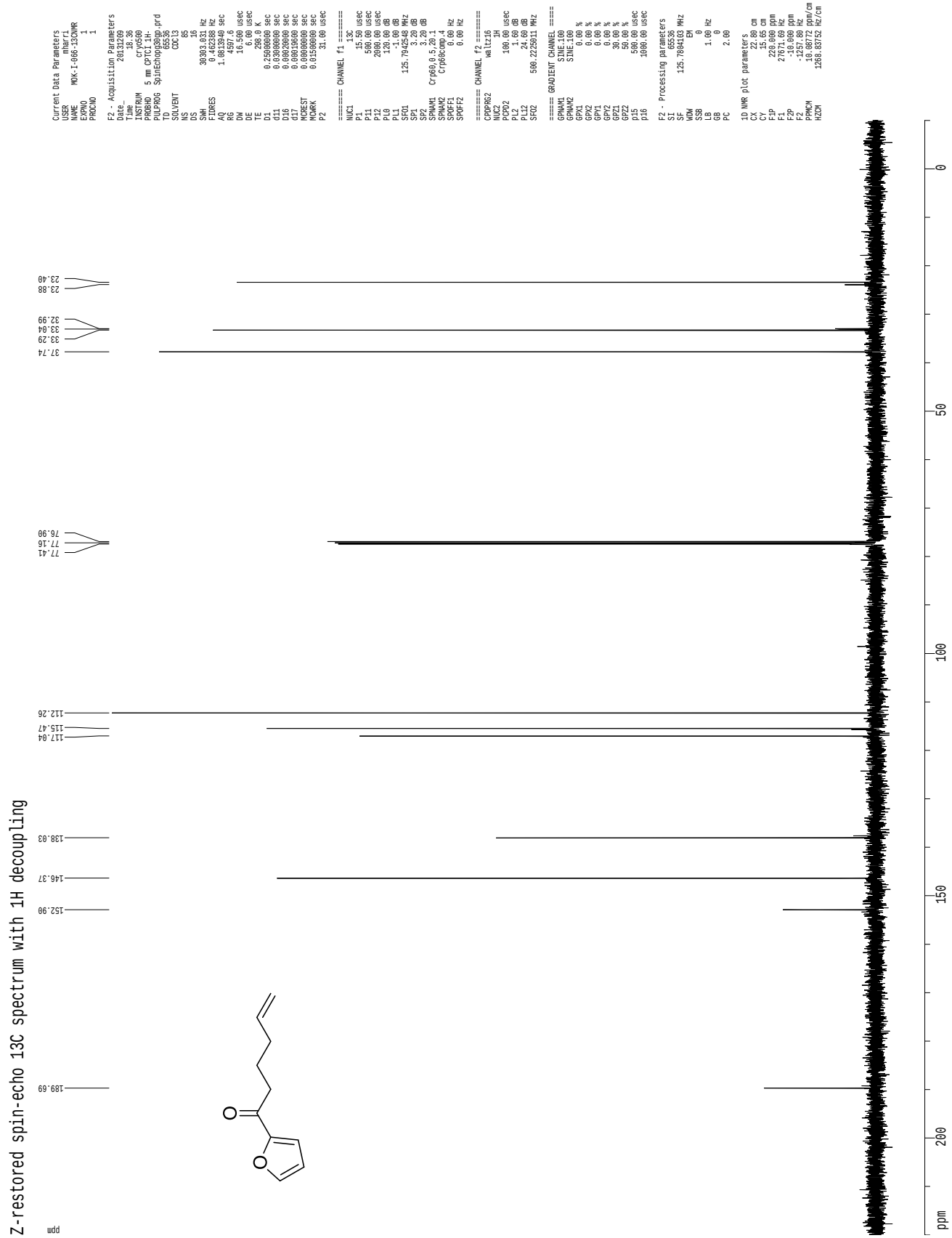
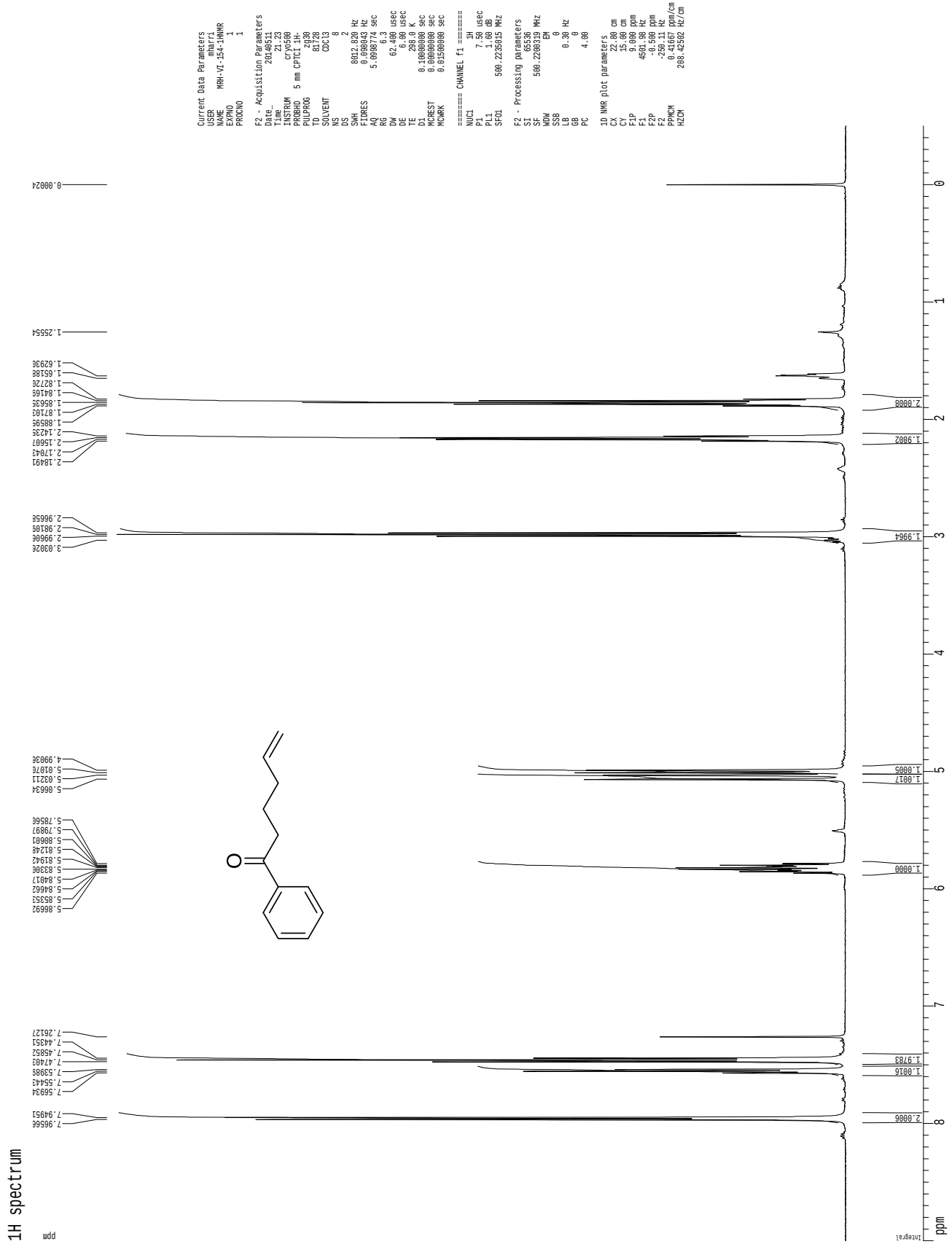
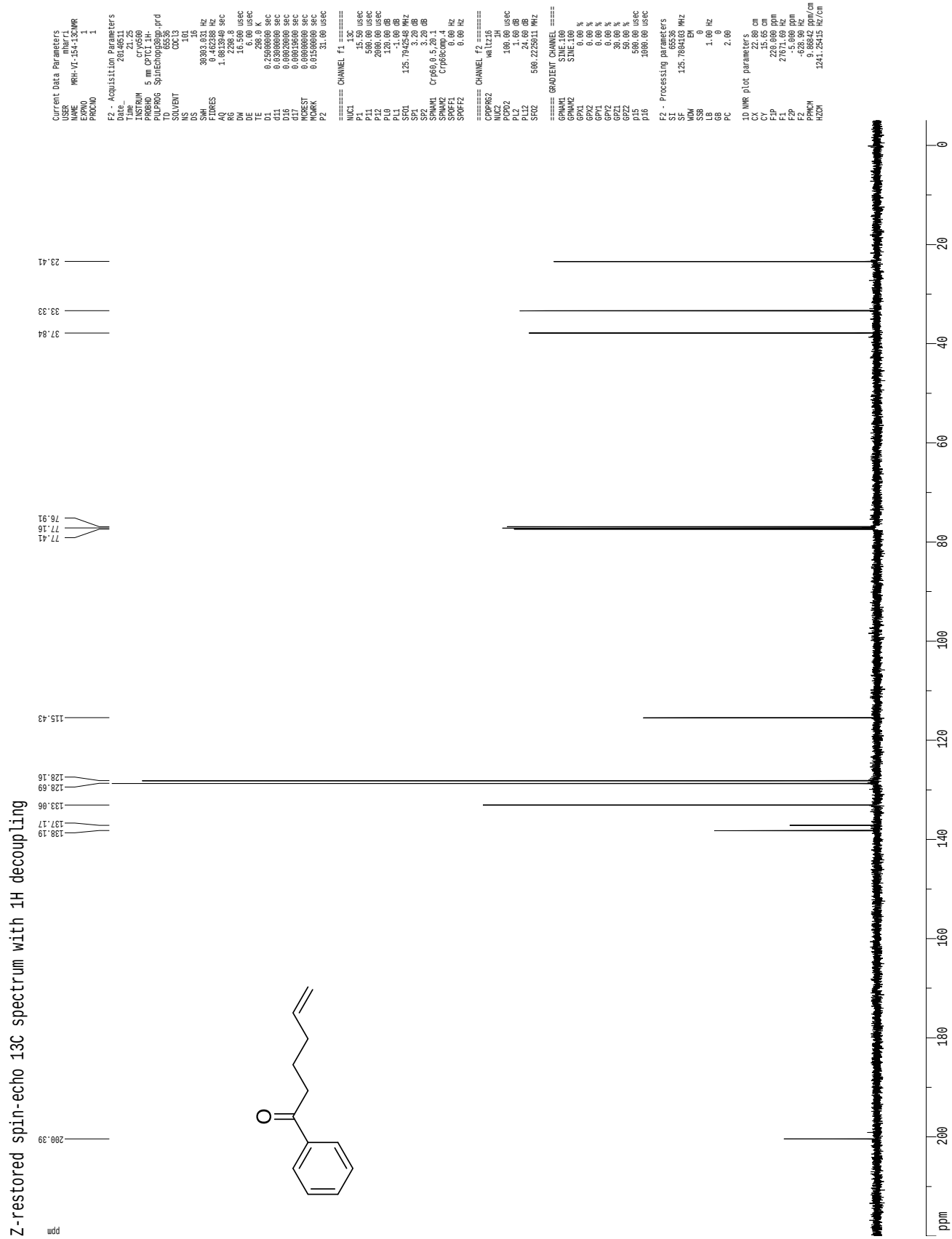
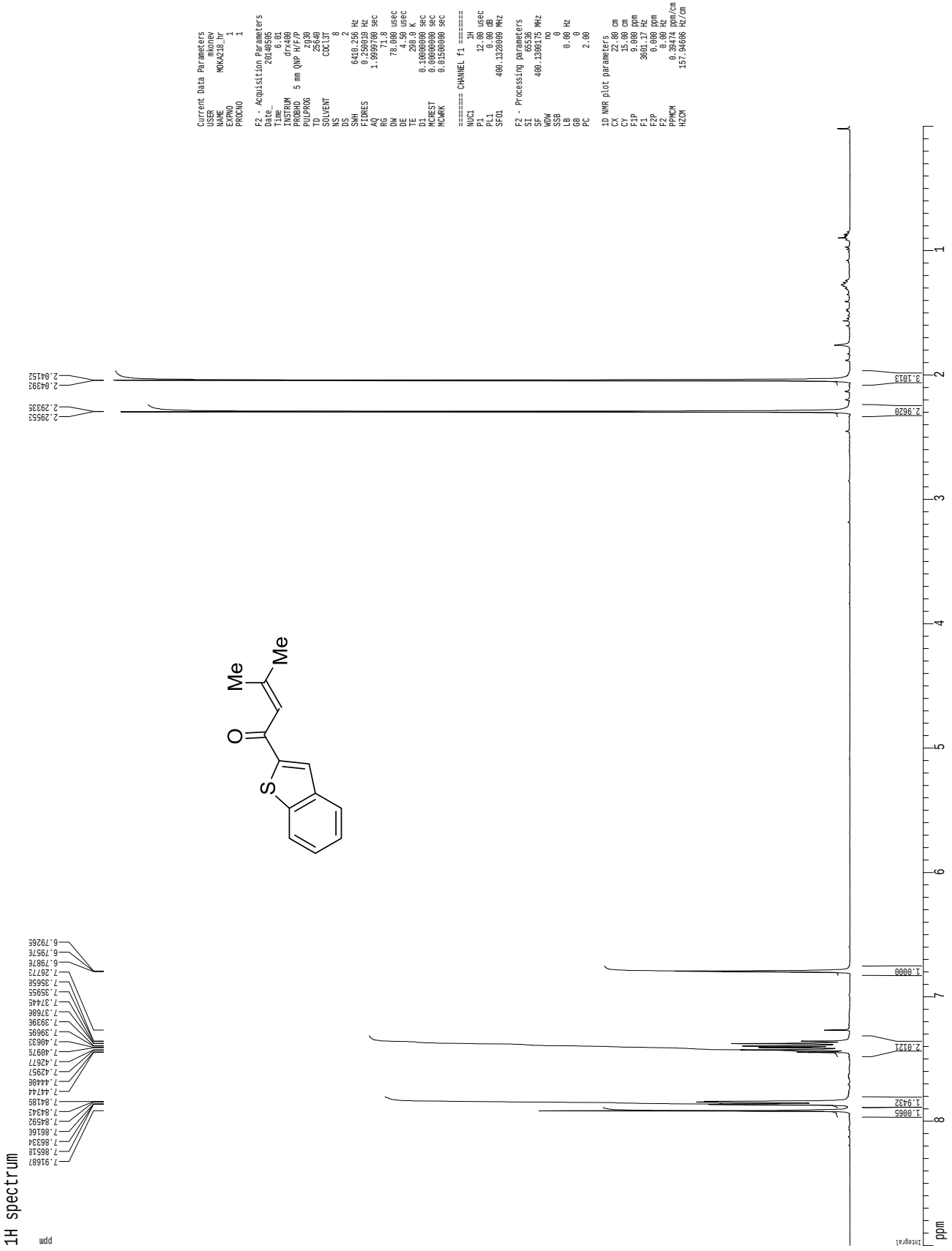
50
100
150
200
ppm

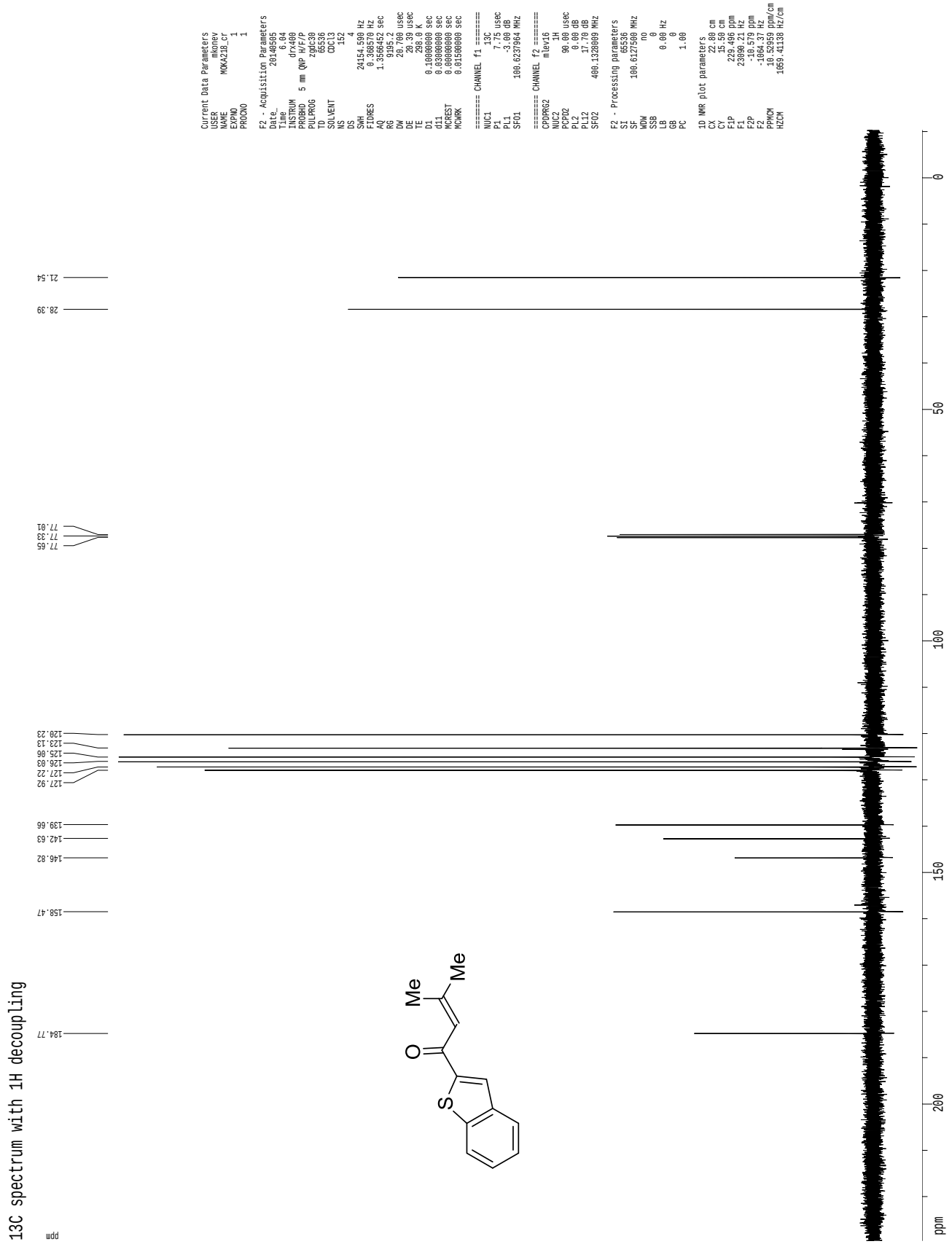

1H spectrum
ppm
Integral
Current Data Parameters
USER mharri
NAME MOK-I-066-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131209
Time 18:33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200247 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -298.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0001
7.7358
7.7506
7.7655
1.8031
1.8179
1.8328
1.8475
1.8624
2.1225
2.1371
2.1514
2.1656
2.3601
2.3752
2.3906
2.8186
2.8331
2.8486
4.9861
5.0063
5.0222
5.0564
5.0592
5.7697
5.7831
5.7902
5.8036
5.8172
5.8244
5.8308
5.8377
5.8516
6.5281
6.5318
6.5349
7.1786
7.1849
7.2721
7.5802
2.0000
2.0093
1.9728
1.0179
0.9826
1.0000
1.0003
0.9864
1.0001


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
189.69
152.90
146.37
138.03
117.04
115.47
112.26
77.41
77.16
76.90
37.74
33.29
33.04
32.99
23.88
23.40
0
50
100
150
200
O
O

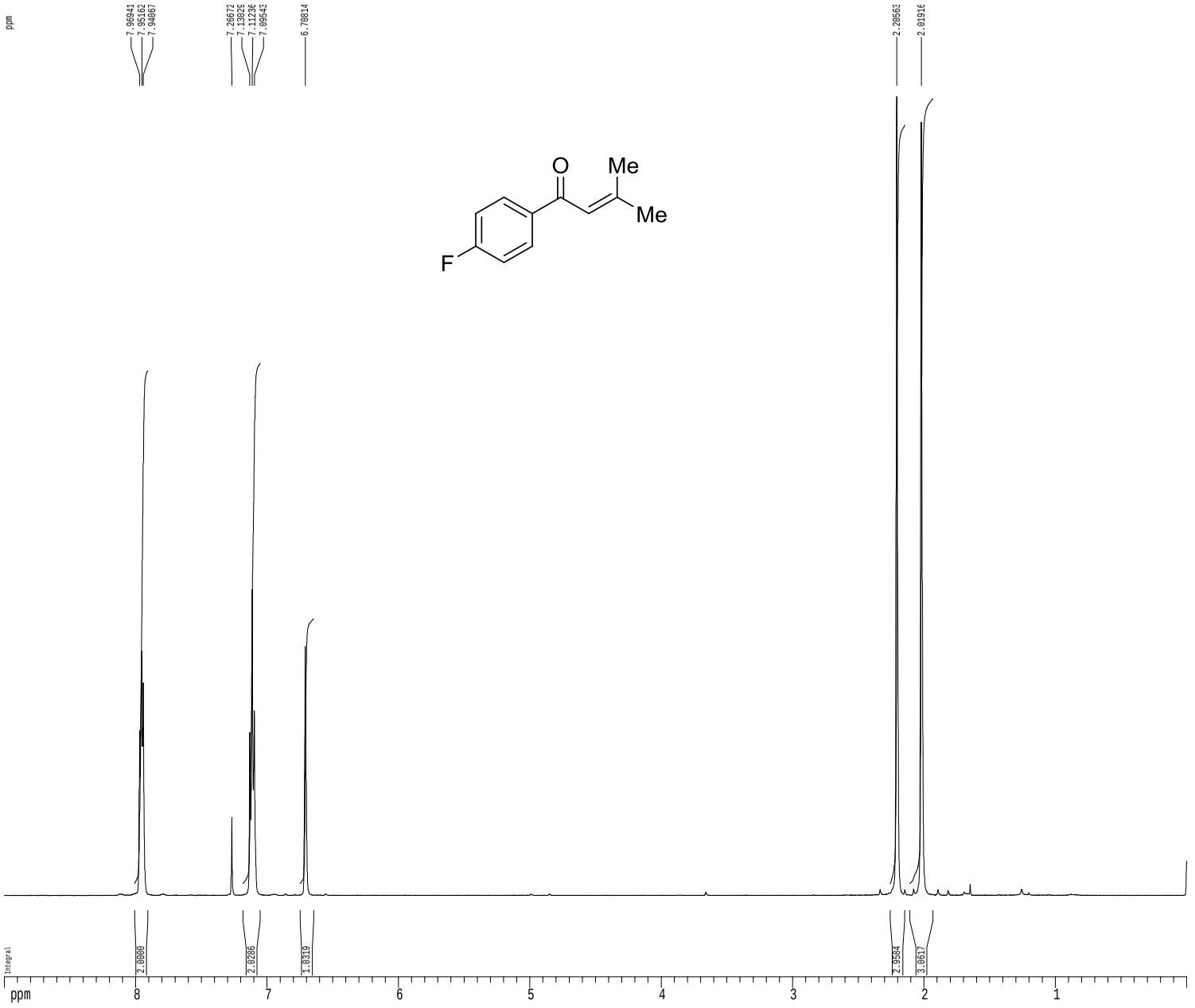

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-154-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140511
Time 21.23
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200319 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.460 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00024
1.25554
1.62936
1.65188
1.82726
1.84166
1.85636
1.87103
1.88596
2.14236
2.15607
2.17043
2.18491
2.96658
2.98109
2.99606
3.03026
4.99036
5.01076
5.03211
5.06634
5.78566
5.79897
5.80601
5.81246
5.81942
5.83306
5.84017
5.84662
5.85353
5.86692
7.26127
7.44351
7.45852
7.47403
7.53985
7.55442
7.56934
7.94951
7.96566
2.0008
1.9783
1.0016
1.0000
1.0017
1.0005
1.9964
1.9802
2.0008
integral
ppm
0
1
2
3
4
5
6
7
8
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
200.39
138.19
137.17
133.06
128.69
128.16
115.43
77.41
77.16
76.91
37.84
33.33
23.41
Current Data Parameters
USER mmarri
NAME MRH-VI-154-13C.NMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140511
Time 21.25
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 192
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2208.8
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
====== CHANNEL f1 ======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884103 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PPMCM 9.88842 ppm/cm
HZCM 1241.25415 Hz/cm
0
20
40
60
80
100
120
140
160
180
200
ppm


1H spectrum
Current Data Parameters
USER mickey
NAME MOKA218_1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140505
Time 6.01
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250078 Hz
AQ 1.997098 sec
RG 71.13
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
Me
Me
O
S
2.04152
2.04392
2.29535
2.29336
3.1013
2.9620
1.0000
2.0121
1.9432
1.0065
7.91687
7.86518
7.86334
7.86166
7.84592
7.84343
7.47444
7.44408
7.42957
7.42677
7.40979
7.40633
7.39696
7.39396
7.37686
7.37445
7.35655
7.35658
7.26773
6.79876
6.79576
6.79265
integral
ppm
1
2
3
4
5
6
7
8
ppm


13C spectrum with 1H decoupling
Current Data Parameters
USER mkonev
NAME MOKA210_cr
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140505
Time 6.04
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 152
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.35566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.76 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127600 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 229.496 ppm
F1 23090.21 Hz
F2P -10.579 ppm
F2 -1064.37 Hz
PPMCM 10.52959 ppm/cm
HZCM 1059.41138 Hz/cm
21.54
28.39
77.01
77.33
77.65
120.23
123.13
125.06
126.03
127.22
127.92
139.66
142.63
146.82
158.47
184.77
Me
Me
O
S
ppm
0
50
100
150
200

1H spectrum



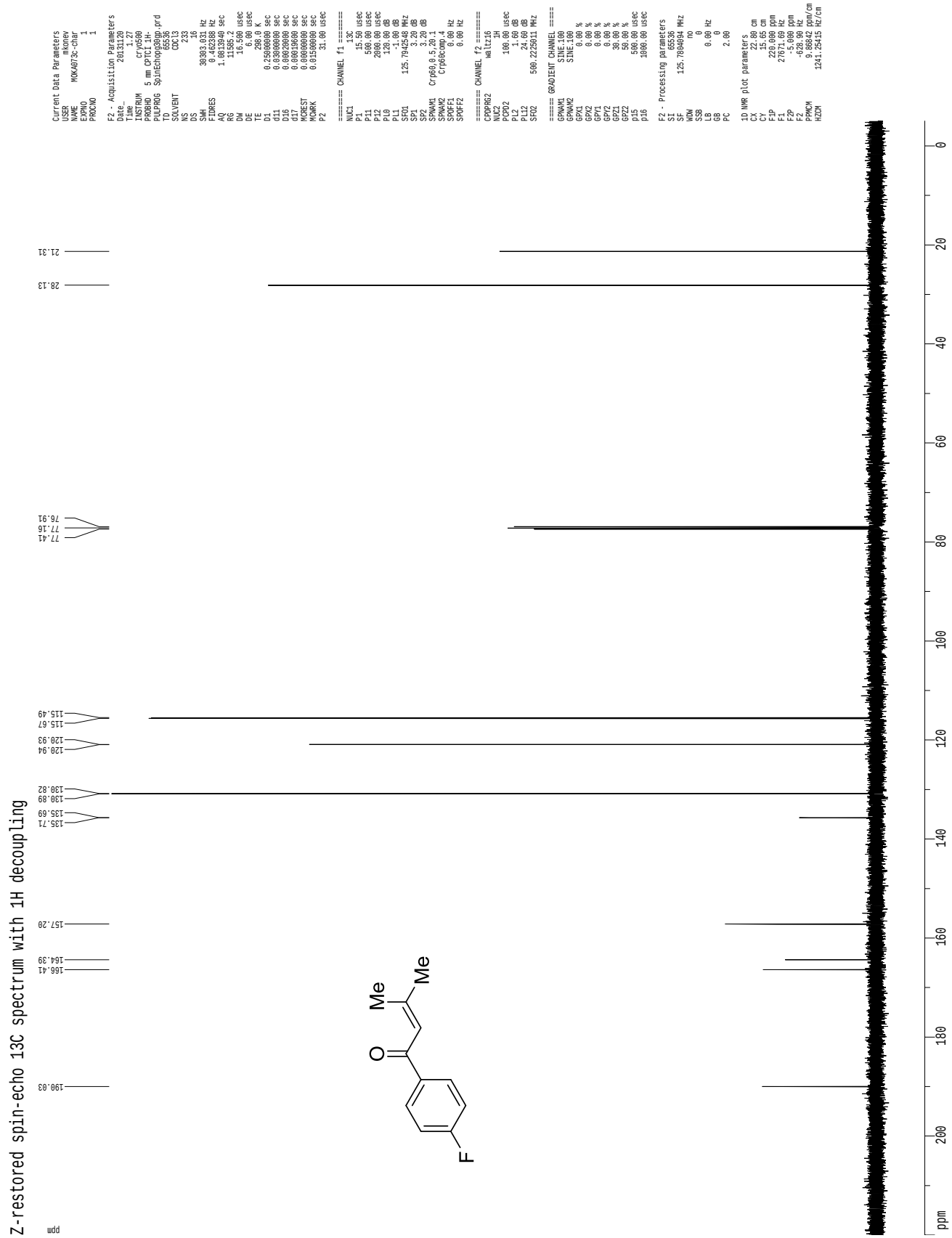
Current Data Parameters
USER mkonev
NAME MOKA073h-char
EXPNO 1
PROCNO 1

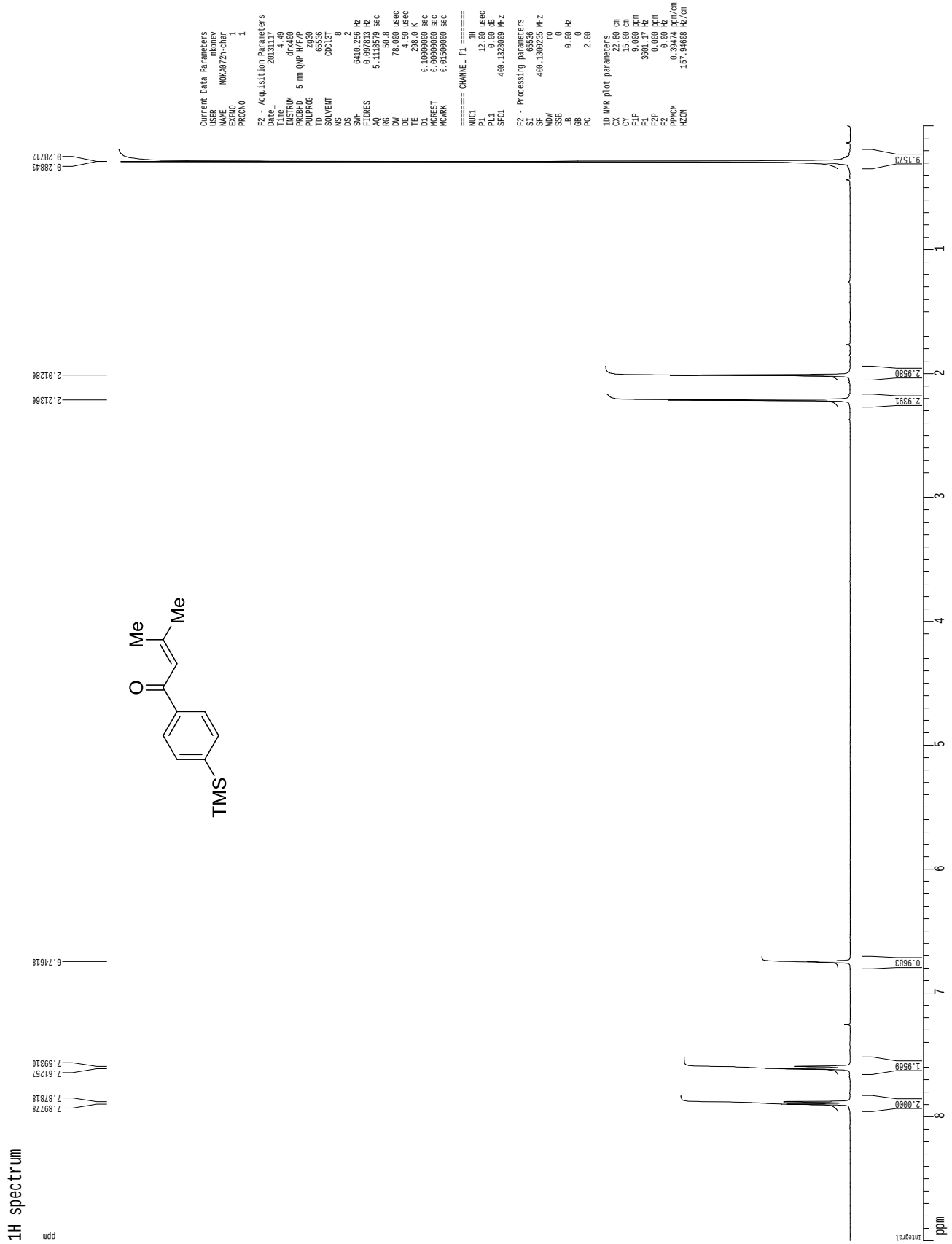
F2 - Acquisition Parameters
Date_ 20131120
Time 1.24
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

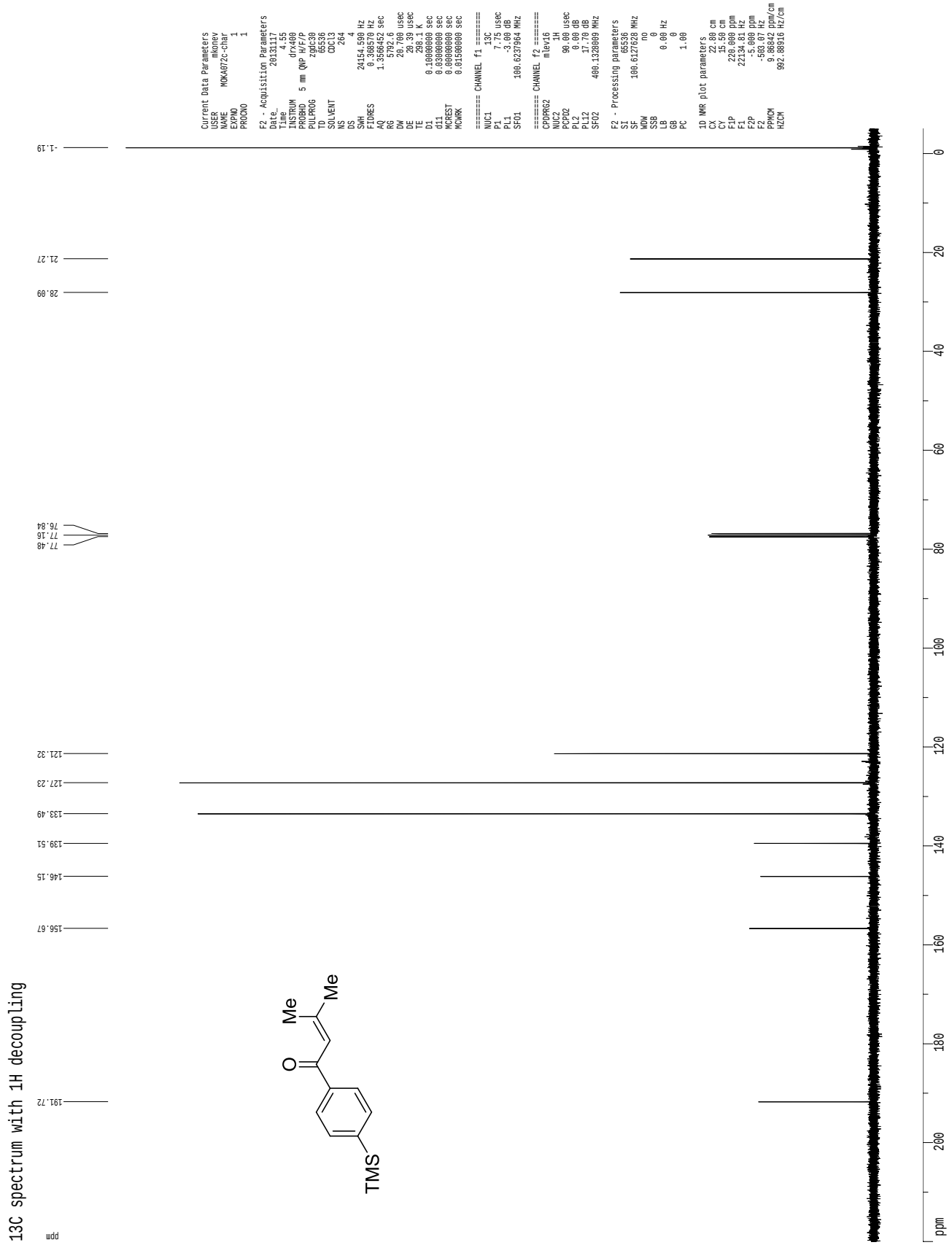
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

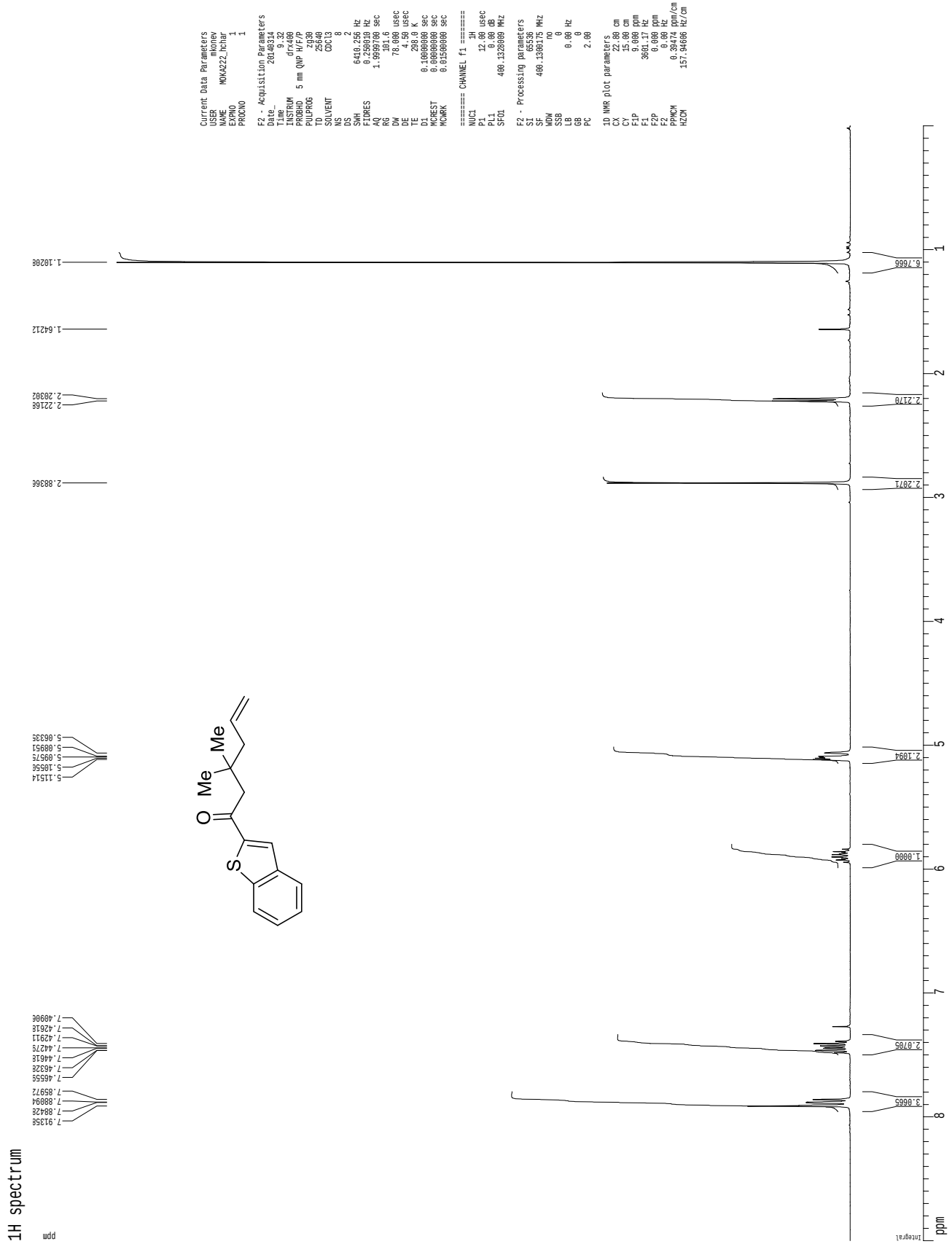
F2 - Processing parameters
SI 65536
SF 500.2200276 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

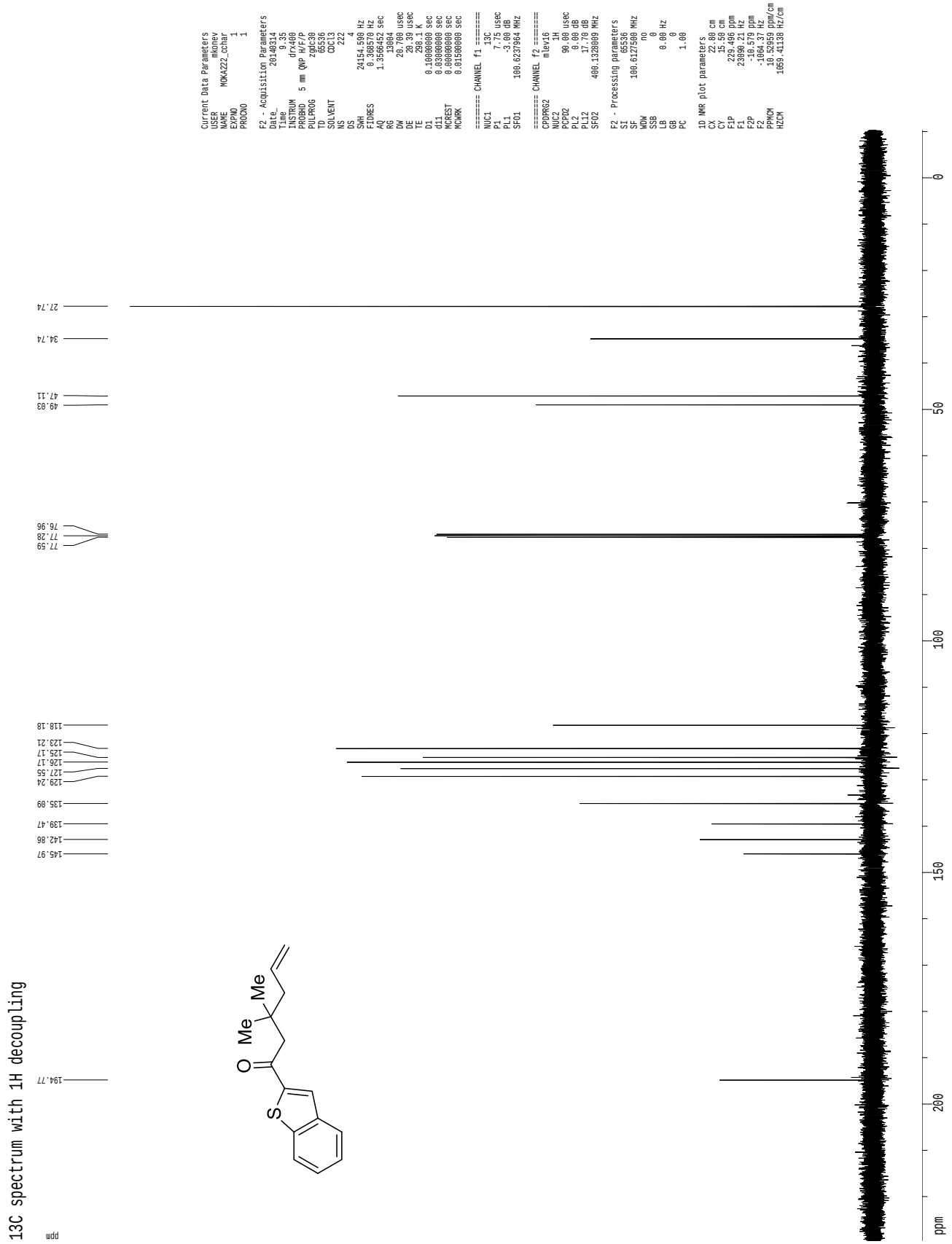
1D NMR plot parameters
CX 22.15 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40632 ppm/cm
HZCM 203.25119 Hz/cm

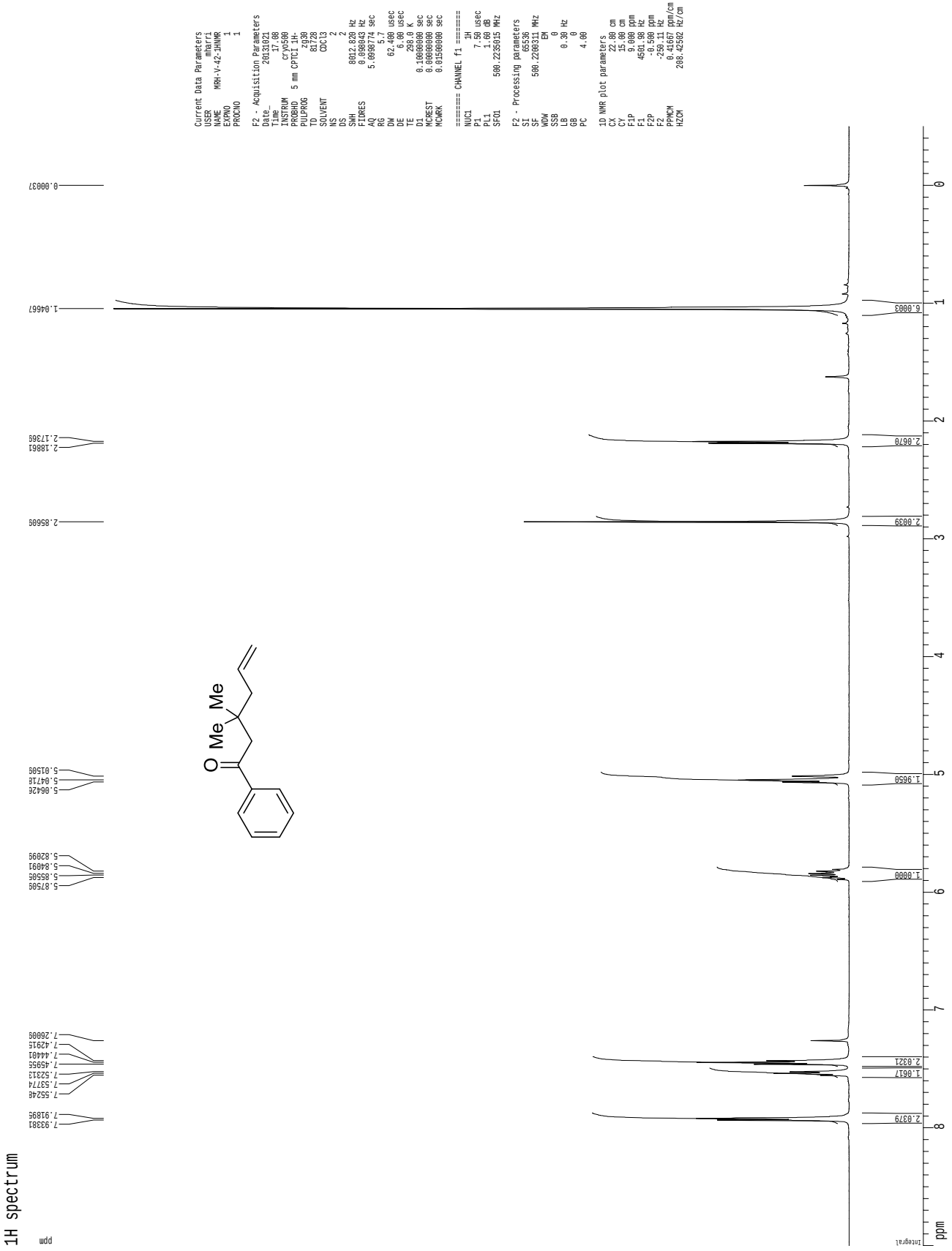

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Me
Me
O
F
21.31
28.13
76.91
77.16
77.41
115.49
115.67
120.93
120.94
130.82
130.89
135.69
135.71
157.20
164.39
166.41
190.03
Current Data Parameters
USER mkonev
NAME MOKA073c-Char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.27
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 233
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7894094 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PPMCM 9.88842 ppm/cm
HZCM 1241.25415 Hz/cm
0
20
40
60
80
100
120
140
160
180
200


Current Data Parameters
USER mickey
NAME MOKA072h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131117
Time 4.49
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097753 Hz
AQ 5.111616 sec
RG 50.8
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300235 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94608 Hz/cm
0.28712
0.28843
2.01286
2.21366
6.74618
7.59316
7.61257
7.87818
7.89778
9.1573
2.9580
2.9391
0.9883
1.9569
2.0000
Integral
ppm
1
2
3
4
5
6
7
8
Me
Me
O
TMS
1H spectrum
ppm

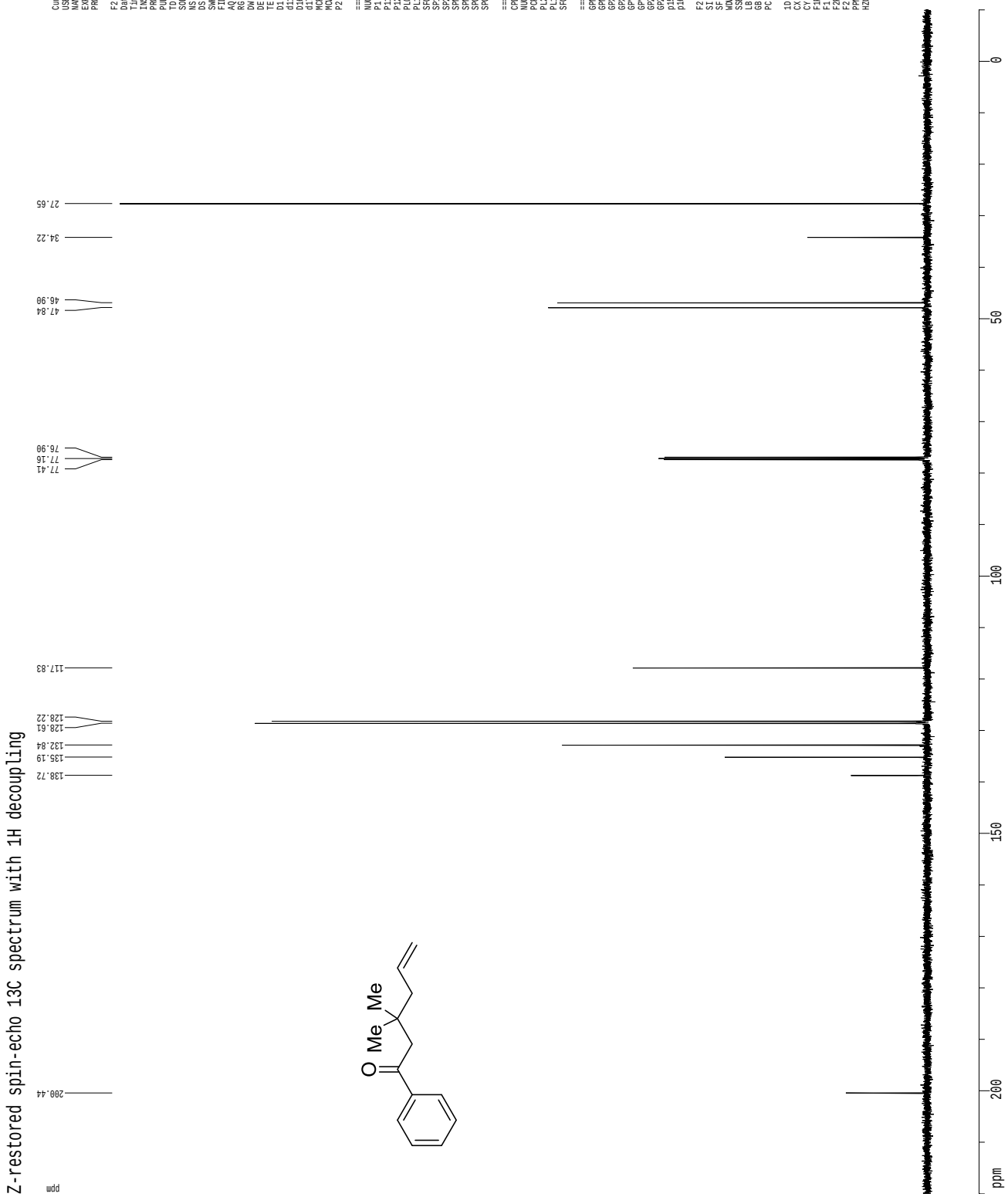

13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER mkonev
NAME MOKA0726-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131117
Time 4.55
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 264
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 5792.6
DW 20.700 usec
DE 20.39 usec
TE 298.1 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.76 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127620 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -5.000 ppm
F2 -503.07 Hz
PPMCM 9.86042 ppm/cm
HZCM 992.88916 Hz/cm
-1.19
21.27
28.09
76.84
77.16
77.48
121.32
127.23
133.49
139.51
146.15
156.67
191.72
Me
Me
O
TMS
0
20
40
60
80
100
120
140
160
180
200
ppm

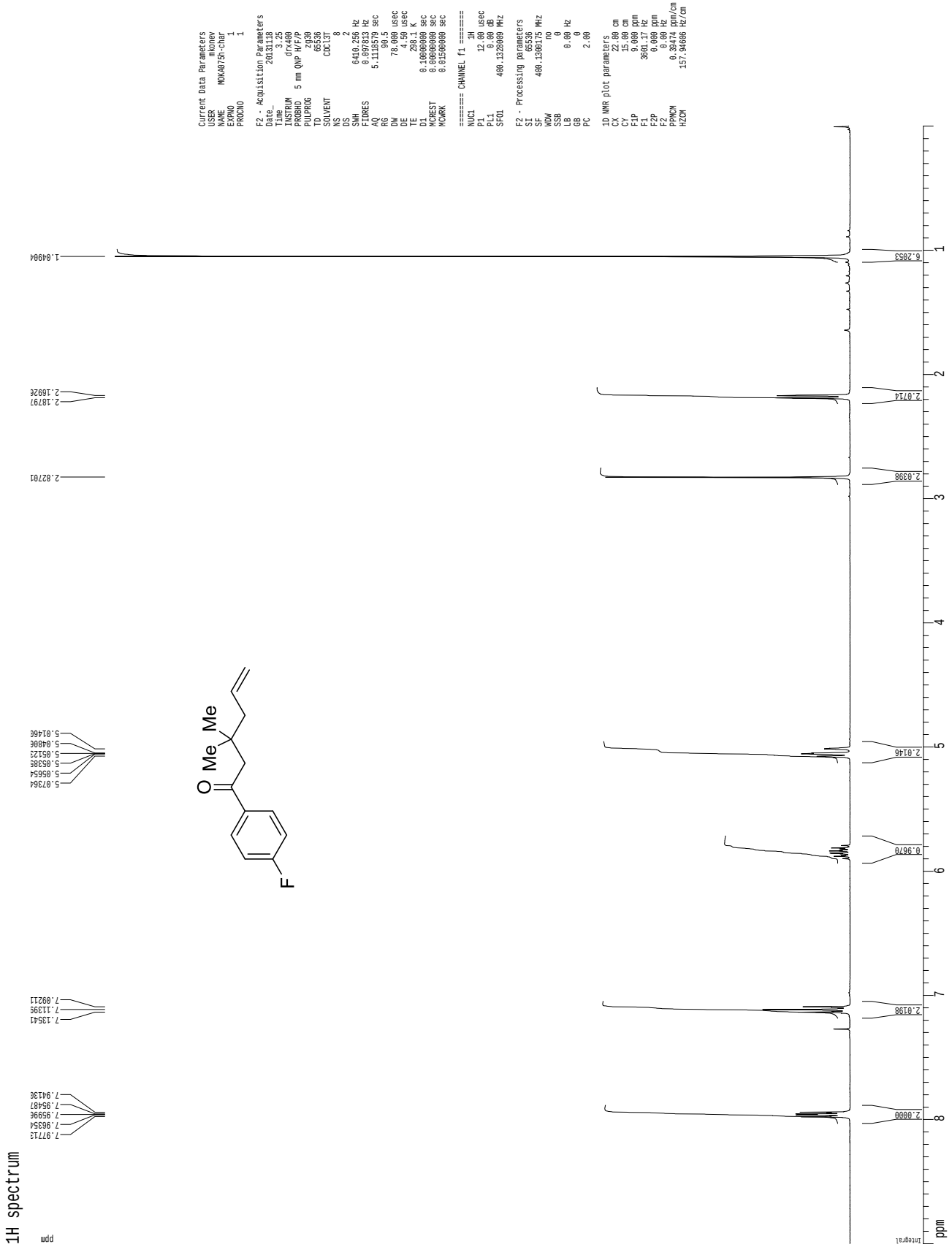

1H spectrum
Current Data Parameters
USER mickey
NAME MONA222_hcmar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140314
Time 9.32
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250078 Hz
AQ 1.9961088 sec
RG 101.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
O
Me
Me
S
1.10208
1.64212
2.20302
2.22168
2.88366
5.06335
5.08951
5.09575
5.10656
5.11514
7.40906
7.42618
7.42911
7.44275
7.46418
7.46528
7.46556
7.85972
7.88094
7.88428
7.91358
6.7666
2.2170
2.2071
2.1094
1.0000
2.0705
3.0665
Integral
ppm


13C spectrum with 1H decoupling
Current Data Parameters
USER mkonev
NAME MOKA222_cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140314
Time 9.35
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 222
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.35566452 sec
RG 13004
DW 20.700 usec
DE 20.39 usec
TE 298.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.76 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127600 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 229.496 ppm
F1 23090.21 Hz
F2P -10.579 ppm
F2 -1064.37 Hz
PPMCM 10.52959 ppm/cm
HZCM 1059.41138 Hz/cm
194.77
145.97
142.86
139.47
135.09
129.24
127.55
126.17
126.17
125.17
123.21
118.18
77.59
77.28
76.96
49.03
47.11
34.74
27.74
ppm
0
50
100
150
200
O
Me
Me
S

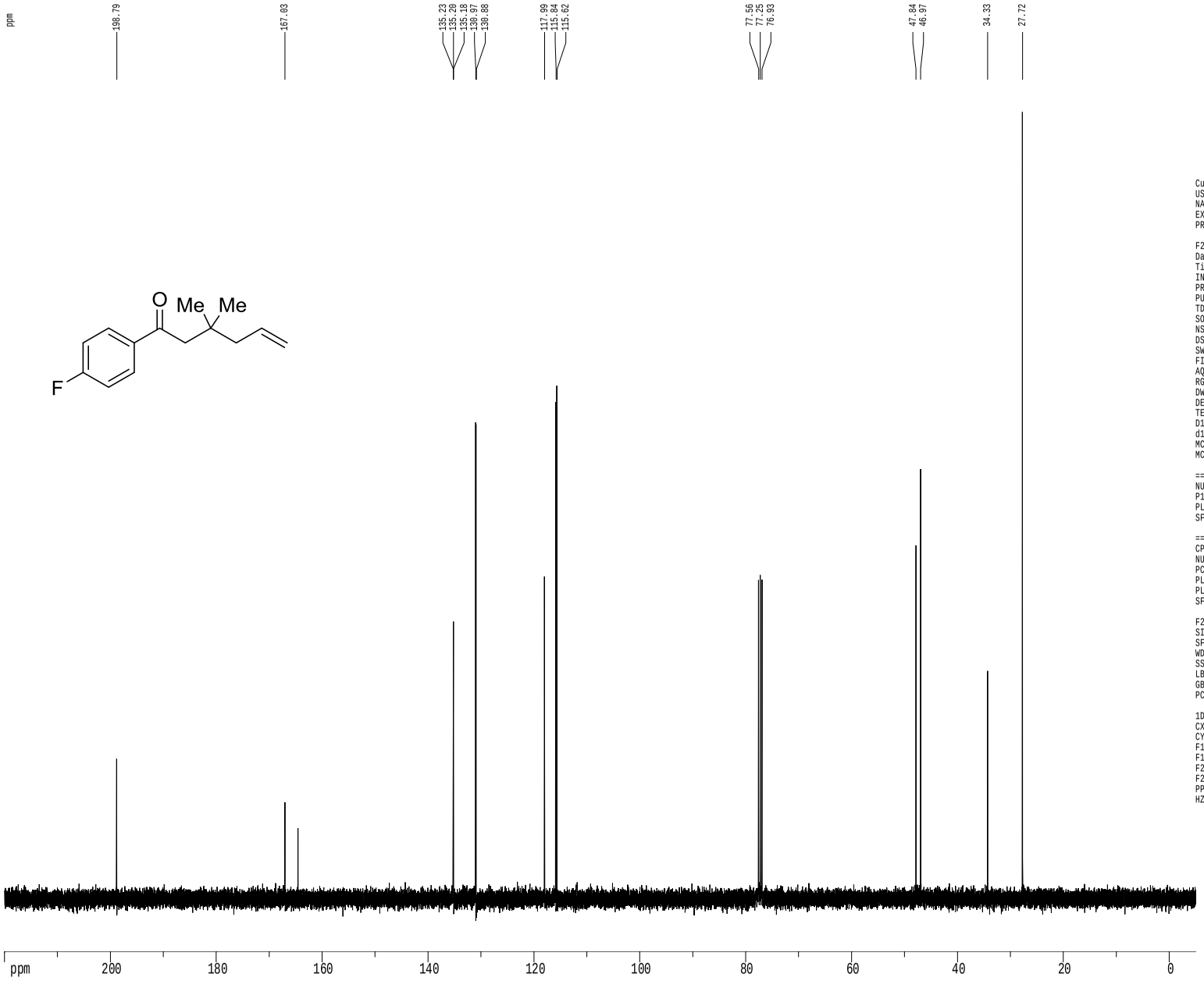

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-V-42-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131021
Time 17.08
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200311 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.580 ppm
F2 -290.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00037
1.04667
2.17365
2.18861
2.85605
5.01509
5.04718
5.06426
5.82095
5.84091
5.85506
5.87509
7.26009
7.42915
7.44401
7.46955
7.52313
7.53774
7.55248
7.91895
7.93381
6.0003
2.0670
2.0839
1.9650
1.0000
2.0321
1.0617
2.0379
0
1
2
3
4
5
6
7
8
ppm
Integral
O
Me
Me
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER mckay
NAME MCKA075h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131118
Time 3.25
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097753 Hz
AQ 5.111575 sec
RG 90.1
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
O
Me
Me
F
1.04904
2.16926
2.18797
2.82701
5.01466
5.04806
5.05123
5.05386
5.06654
5.07364
7.09211
7.11399
7.13541
7.94136
7.95487
7.95996
7.96354
7.97713
6.2053
2.0714
2.0398
2.0146
0.9670
2.0198
2.0000
Integral
ppm

13C spectrum with 1H decoupling



Current Data Parameters
USER mkonev
NAME MOKA075c-char
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131118
Time 3.28
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.8 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

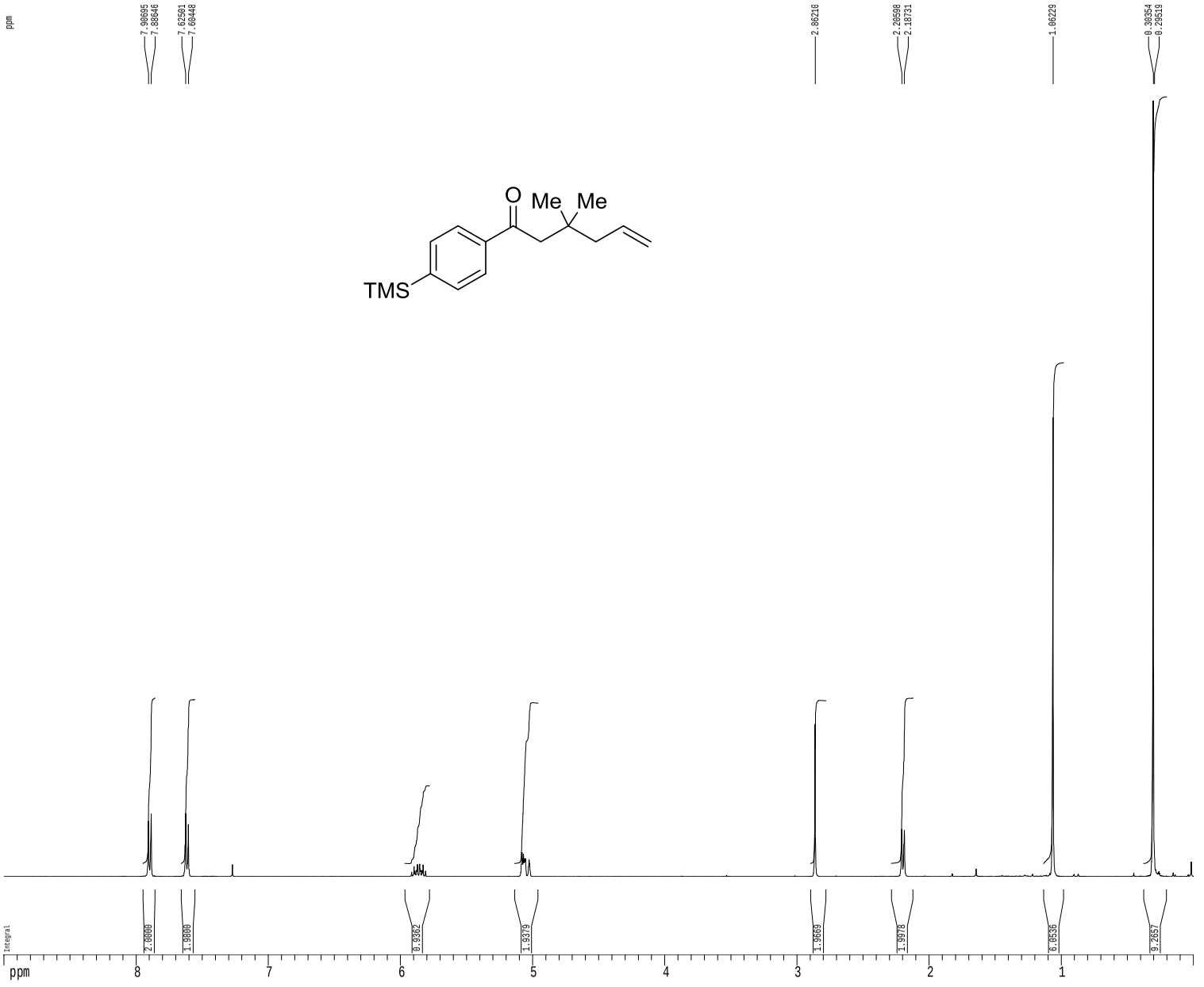
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 9.86842 ppm/cm
HZCM 992.88898 Hz/cm

1H spectrum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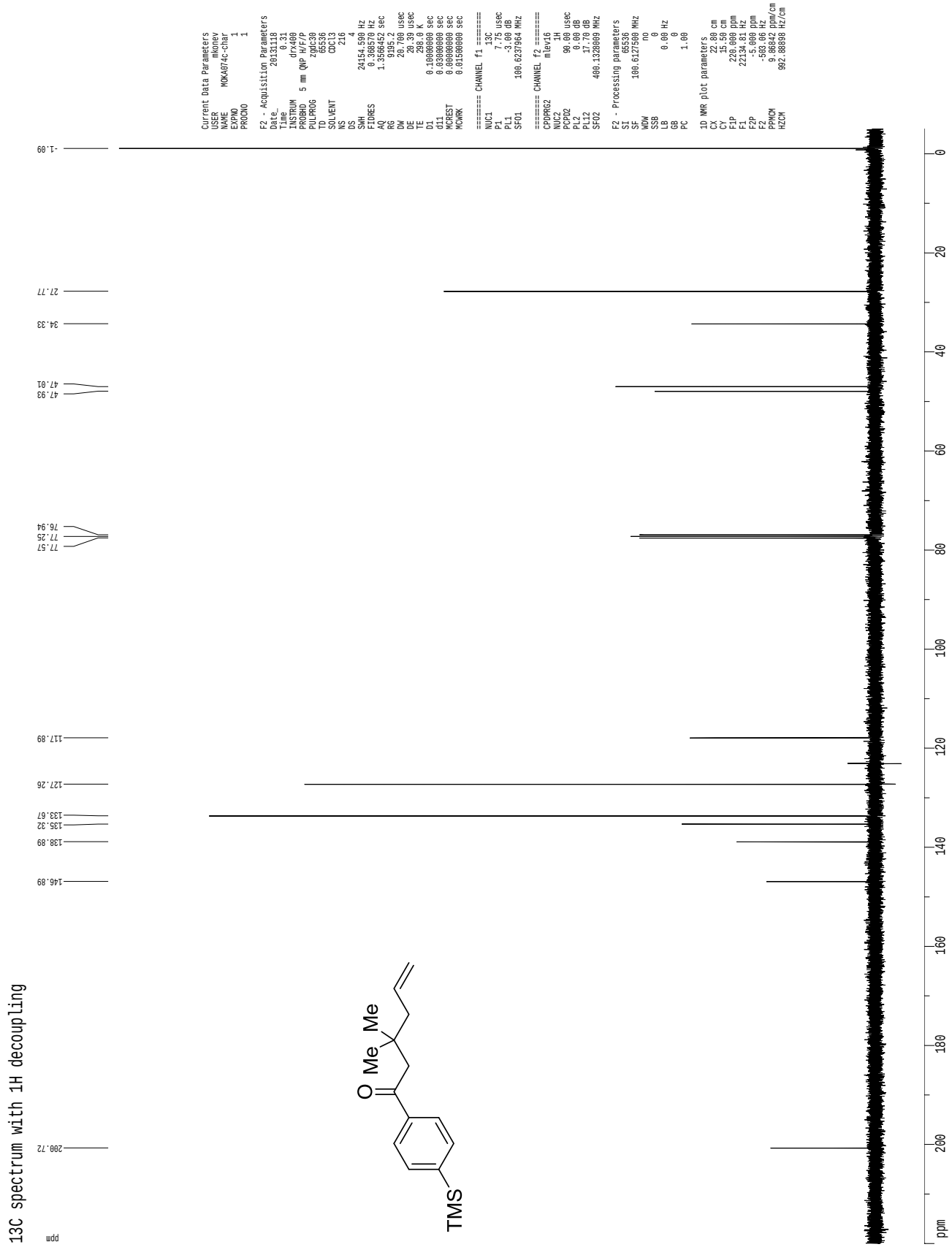
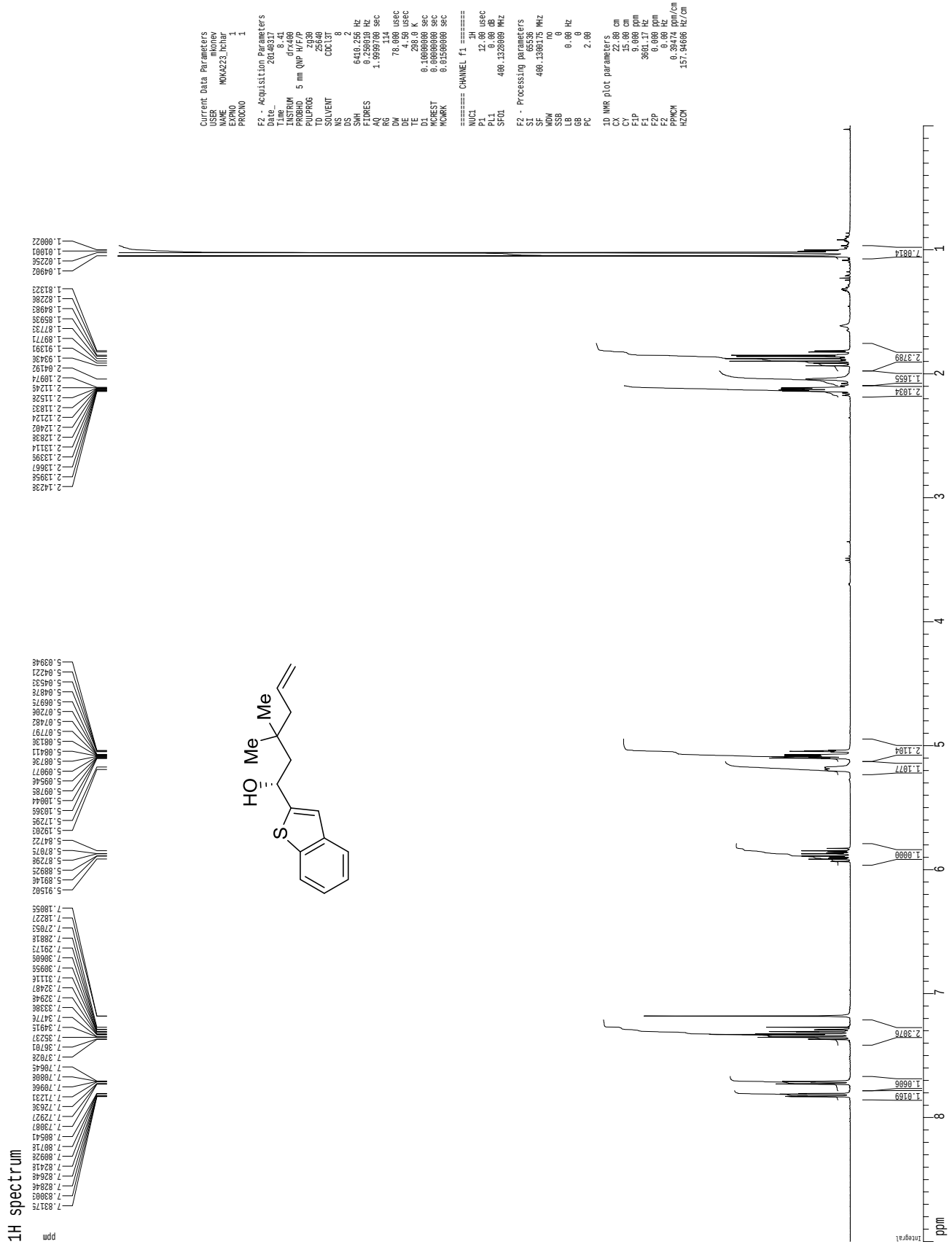
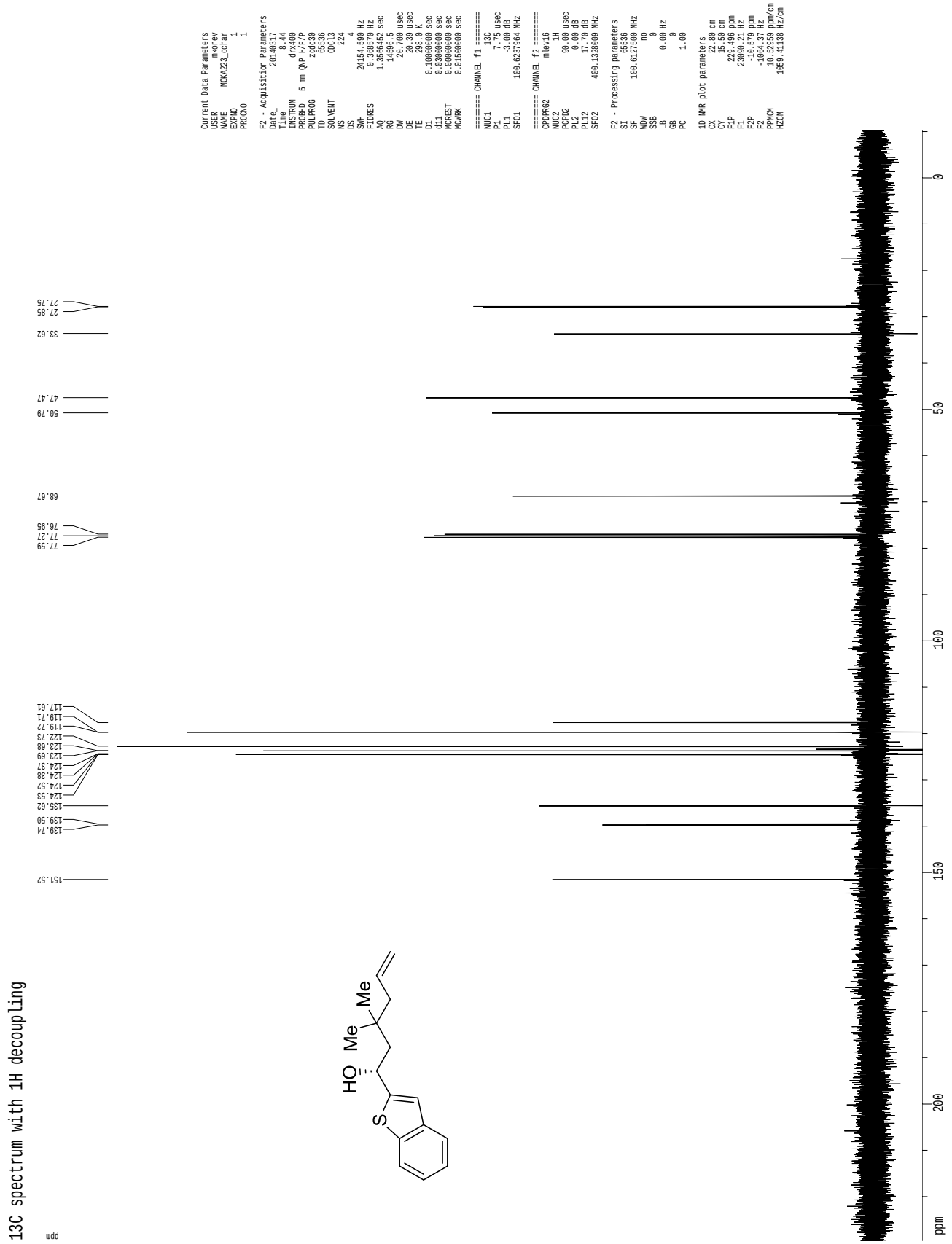
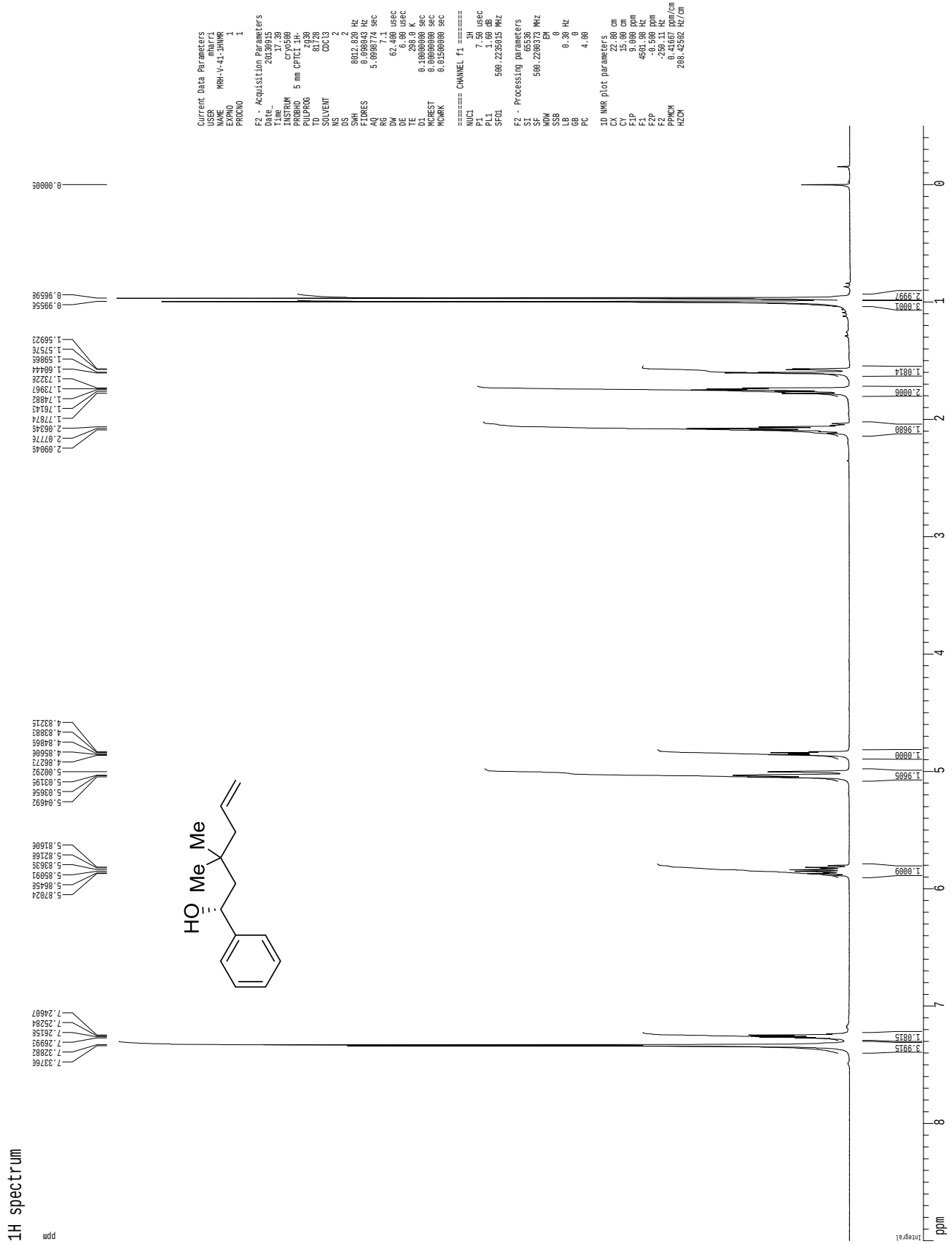
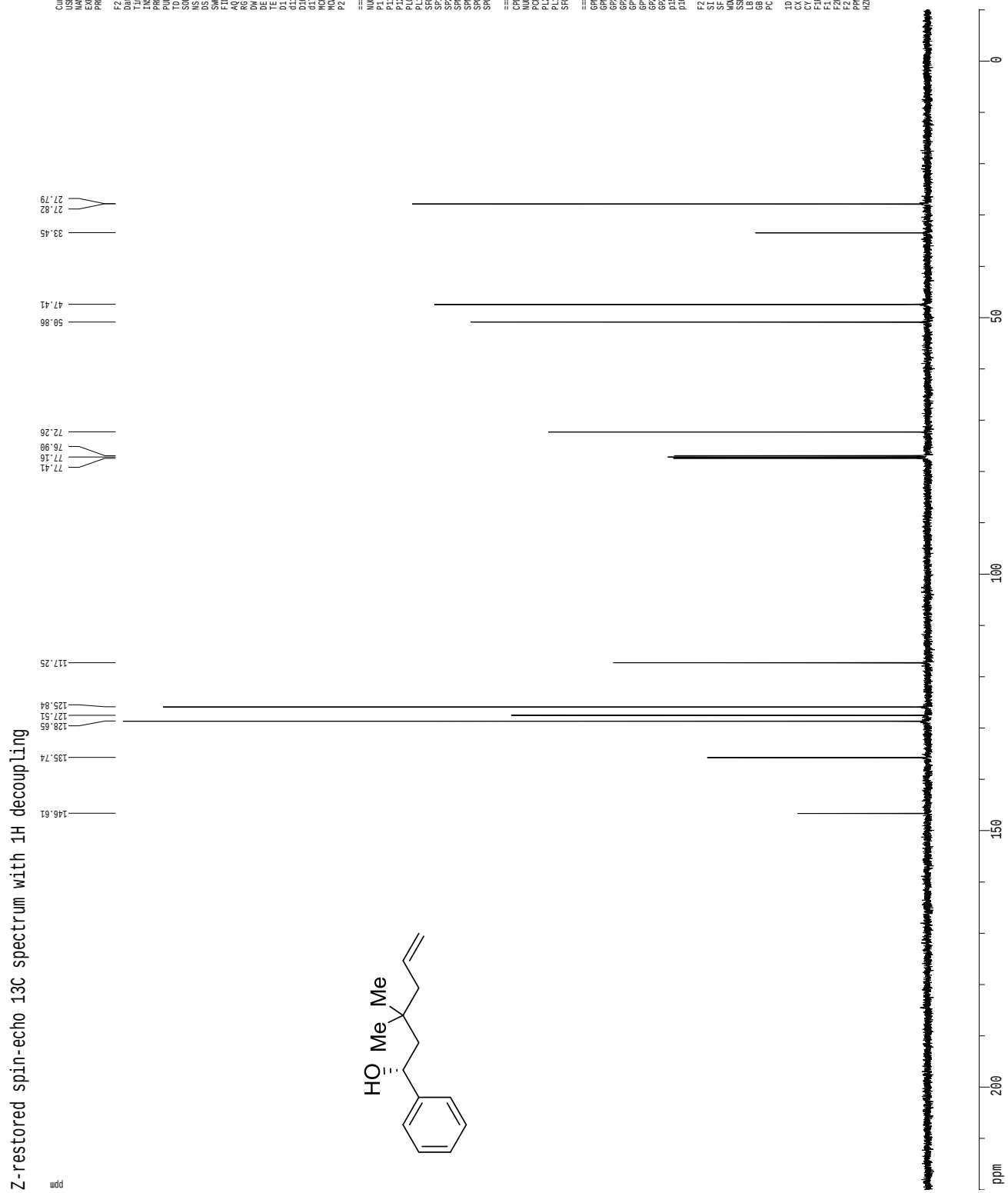
7.90695
7.88646
7.62501
7.60448
2.86218
2.20598
2.18731
1.86229
0.30354
0.29519
O
Me
Me
TMS
Integral
2.0000
1.9800
0.9362
1.9379
1.9669
1.9978
6.0536
9.2657
ppm
8
7
6
5
4
3
2
1

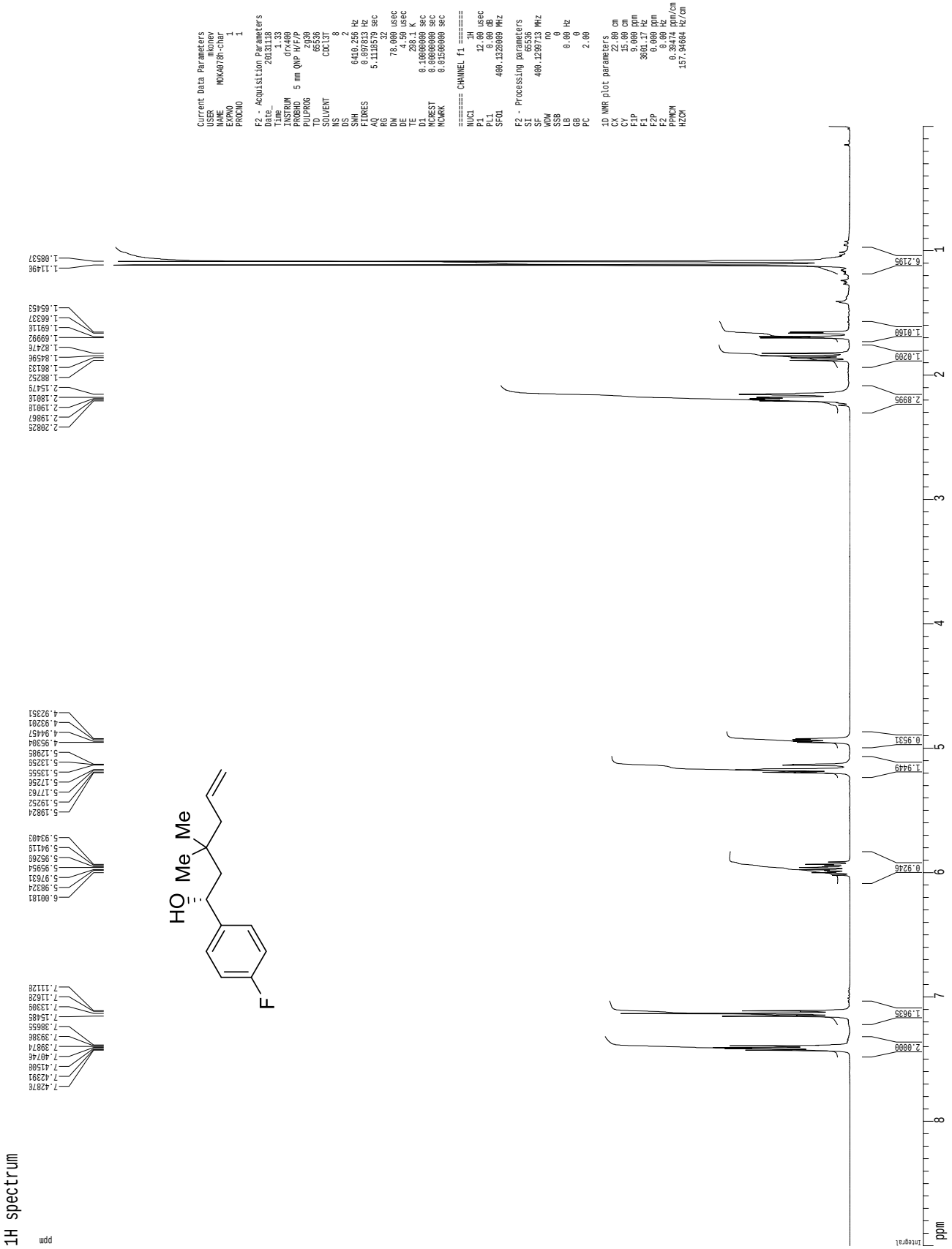

13C spectrum with 1H decoupling
ppm
-1.09
27.77
34.33
47.01
47.93
76.94
77.25
77.57
117.89
127.26
133.67
135.32
138.89
146.89
200.72
O
Me
Me
TMS
Current Data Parameters
USER mkonev
NAME MOKA074c-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131118
Time 0.31
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 216
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127600 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 9.86042 ppm/cm
HZCM 992.88898 Hz/cm
0
20
40
60
80
100
120
140
160
180
200
ppm


1H spectrum
Current Data Parameters
USER mickey
NAME MONA223_1cmar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140317
Time 8.41
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250098 Hz
AQ 1.9999700 sec
RG 114
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
HO
Me
Me
S
ppm
Integral

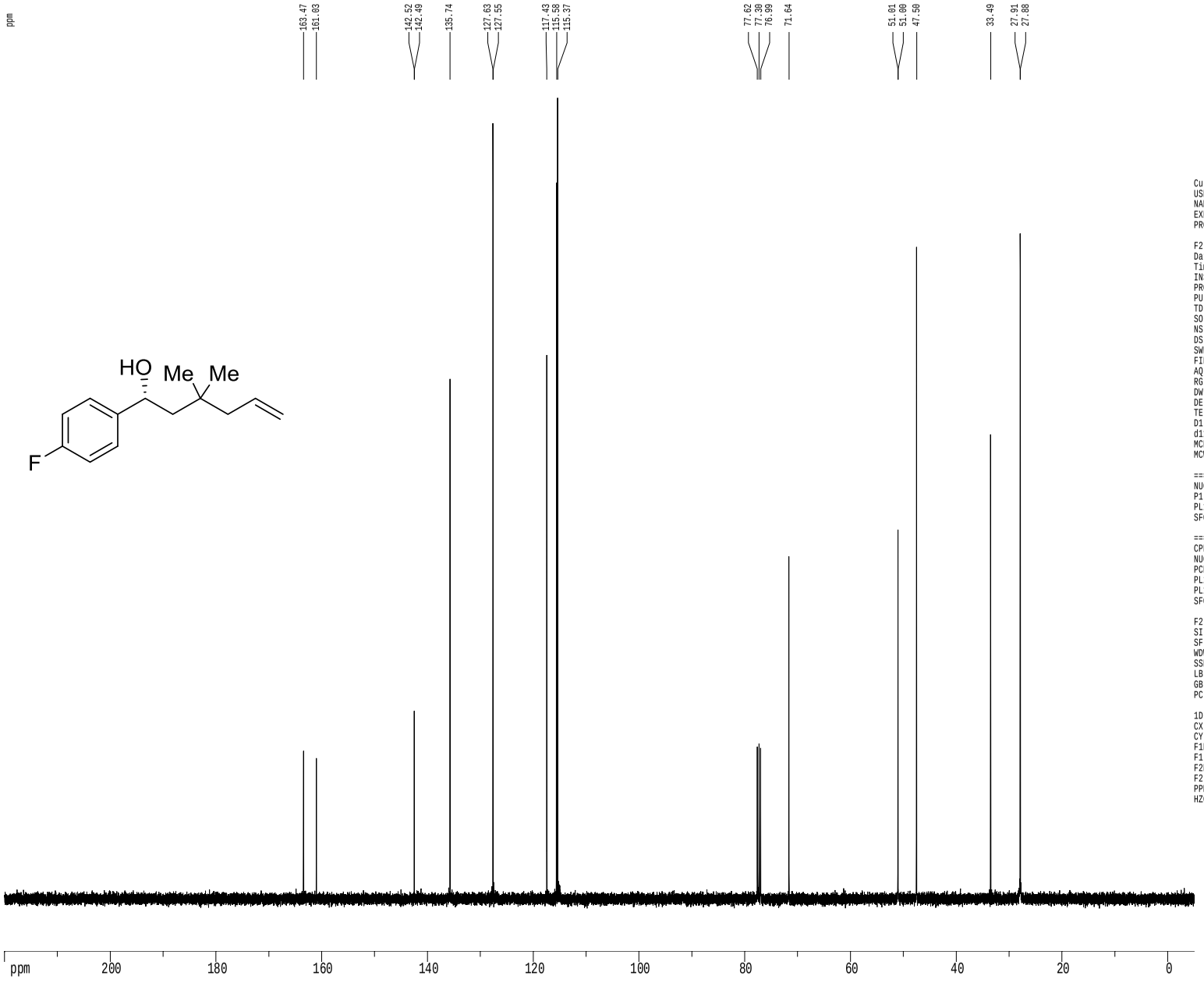

13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER mkonev
NAME MOKA223_cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140317
Time 8.44
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 224
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 14596.5
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127560 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 229.496 ppm
F1 23090.21 Hz
F2P -10.579 ppm
F2 -1064.37 Hz
PPMCM 10.52959 ppm/cm
HZCM 1059.41138 Hz/cm
151.52
139.74
139.50
135.62
124.53
124.52
124.38
124.37
123.69
123.68
122.73
119.72
119.71
117.61
77.59
77.27
76.95
68.67
50.79
47.47
33.62
27.85
27.75
HO
Me
Me
S
0
50
100
150
200
ppm


1H spectrum
Current Data Parameters
USER mharri
NAME MRH-V-41-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130915
Time 17.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200373 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.696 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00005
0.96598
0.99556
1.56923
1.57576
1.59865
1.60444
1.73228
1.79967
1.74882
1.76143
1.77874
2.06345
2.07776
2.09045
4.83215
4.83883
4.84865
4.85606
4.86273
5.00292
5.03196
5.03656
5.04692
5.81606
5.82168
5.83635
5.85091
5.86456
5.87024
7.24607
7.25284
7.26158
7.26992
7.28282
7.33766
2.9997
3.0001
1.0814
2.0006
1.9880
1.0000
1.9605
1.0009
1.0815
3.9915
integral
ppm
0
1
2
3
4
5
6
7
8
HO
Me
Me
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER mharri
NAME MRH-V-41-13CNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130915
Time 17.41
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoDig90gp.prd
TD 65536
SOLVENT CDCl3
NS 192
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm
27.79
27.82
33.45
47.41
50.86
72.26
76.90
77.16
77.41
117.25
125.84
127.51
128.65
135.74
146.61
HO
Me
Me
0
50
100
150
200


1H spectrum
ppm
Integral
HO
Me
Me
F
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
1.08537
1.11496
1.65452
1.66337
1.69116
1.69992
1.82476
1.84596
1.86133
1.88252
2.15479
2.18016
2.19018
2.19867
2.20825
4.92351
4.93201
4.94457
4.95304
5.12986
5.13259
5.13656
5.17256
5.17763
5.19252
5.19824
5.93403
5.94119
5.95266
5.95954
5.97631
5.98324
6.00181
7.11128
7.11628
7.13306
7.15485
7.38655
7.39388
7.39874
7.40746
7.41568
7.42391
7.42876
6.2195
1.0160
1.0209
2.8995
0.9531
1.9449
0.9246
1.9635
2.0000

13C spectrum with 1H decoupling



Current Data Parameters
USER mkonev
NAME MOKA078c-char
EXPNO 1
PROCNO 1

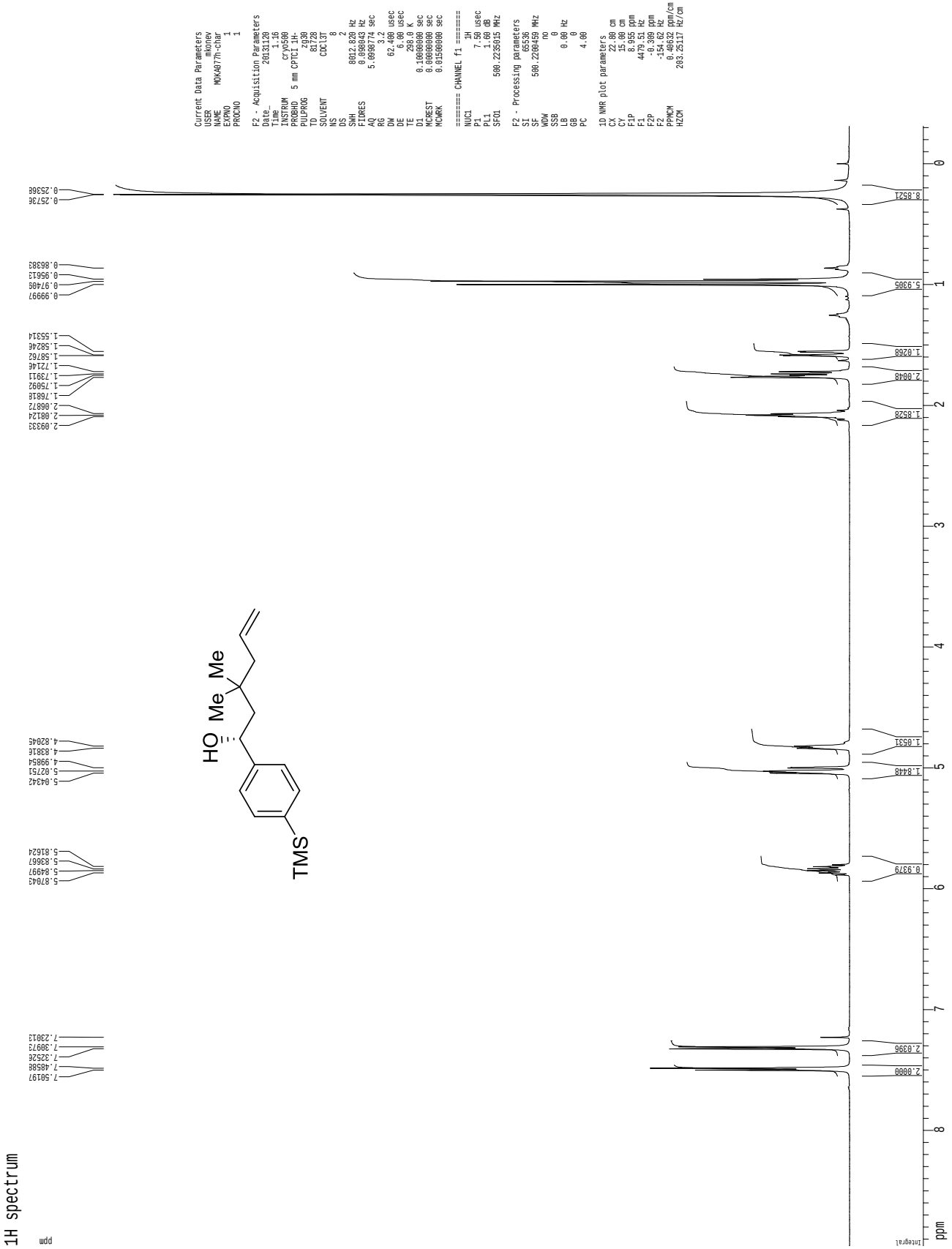
F2 - Acquisition Parameters
Date_ 20131118
Time 1.38
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 187
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.8 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

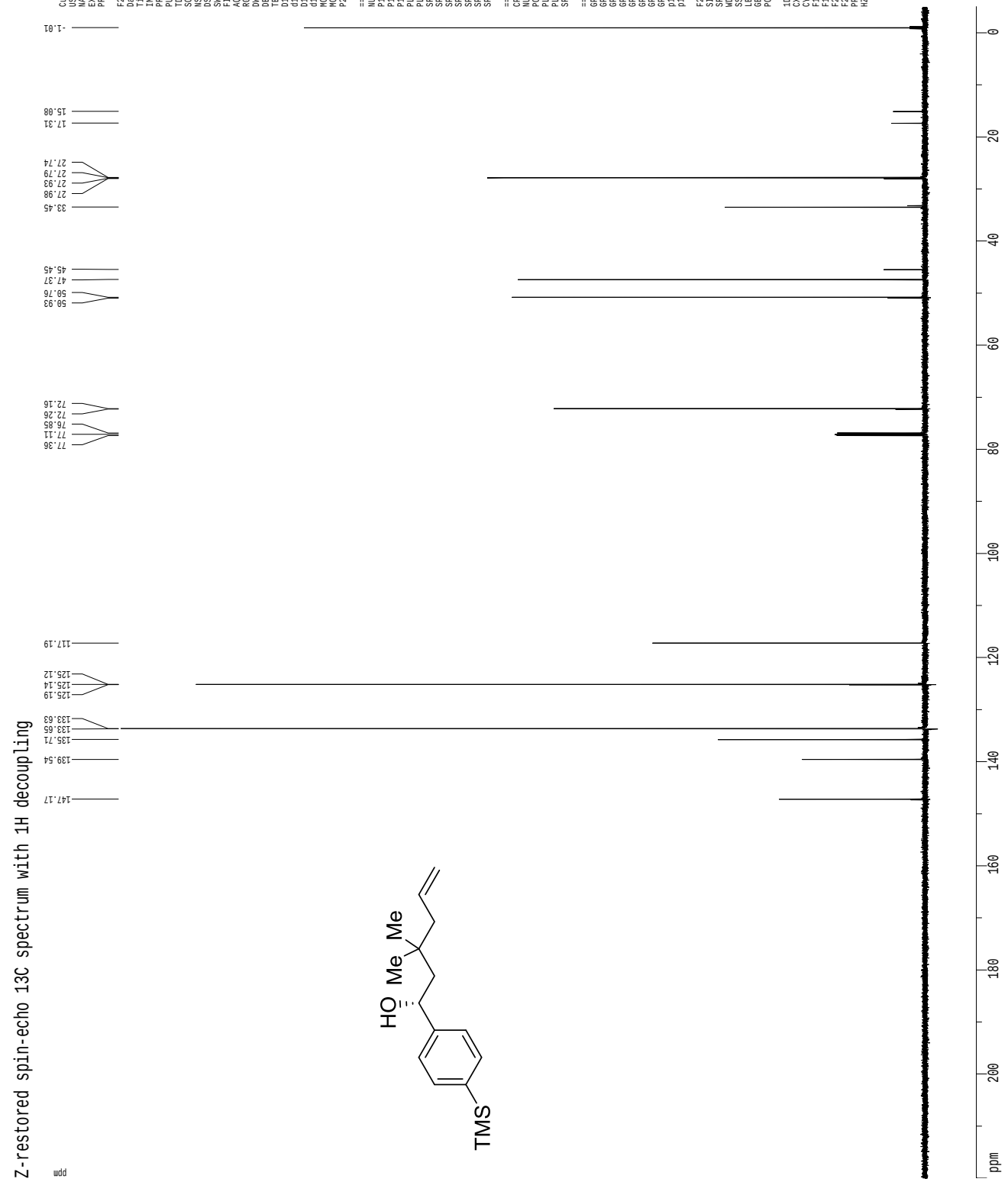
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 9.86842 ppm/cm
HZCM 992.88898 Hz/cm


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK4077h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 3.2
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200459 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.955 ppm
F1 4479.51 Hz
F2P -0.400 ppm
F2 -154.62 Hz
PPMCM 0.40632 ppm/cm
HZCM 203.25117 Hz/cm
HO
Me
Me
Me
TMS
integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER mkonev
NAME MOKA077C-Char 1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131120
Time 1.19
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoDig30gp.prd
TD 65536
SOLVENT CDCl3
NS 128
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec

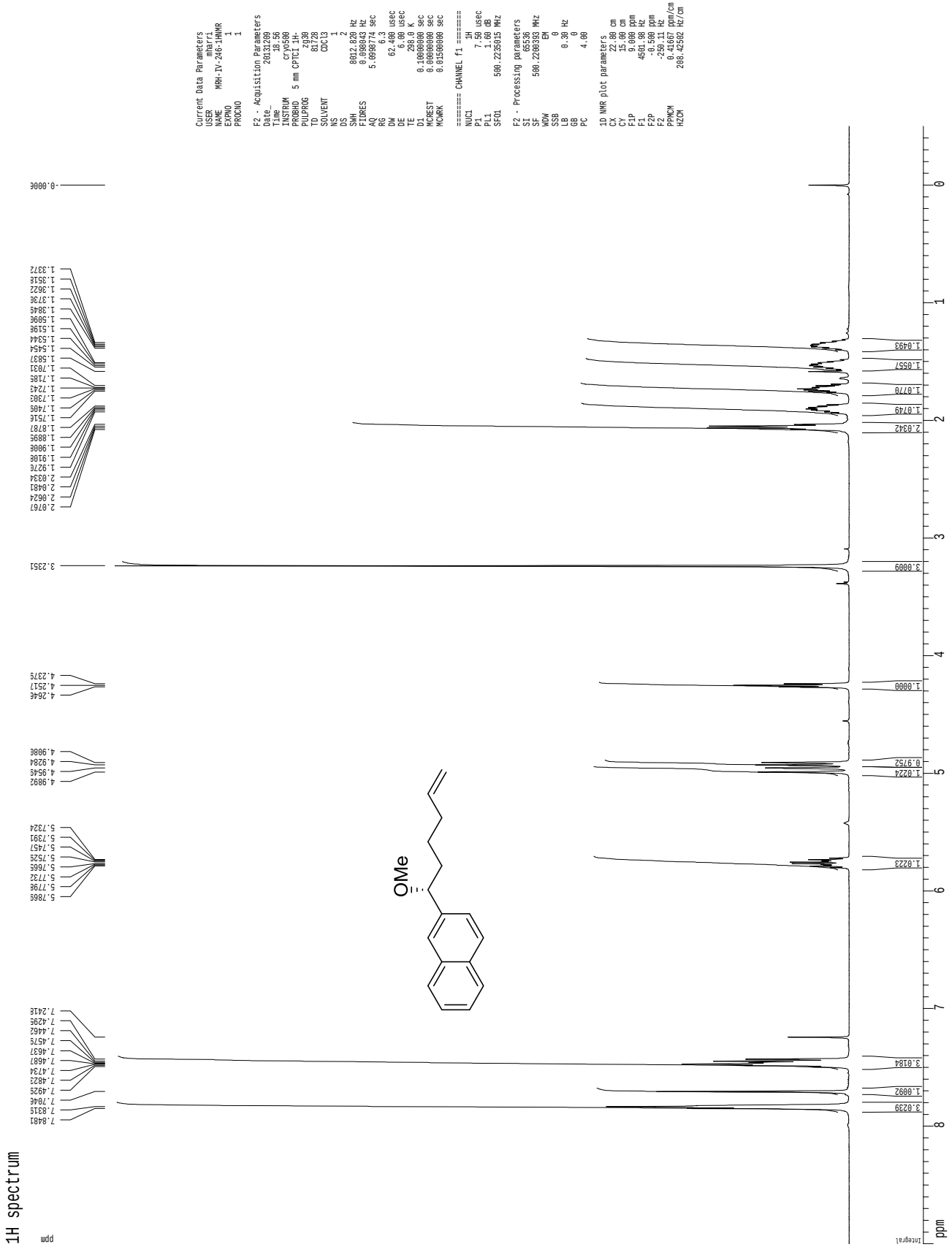
====== CHANNEL f1 ======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz

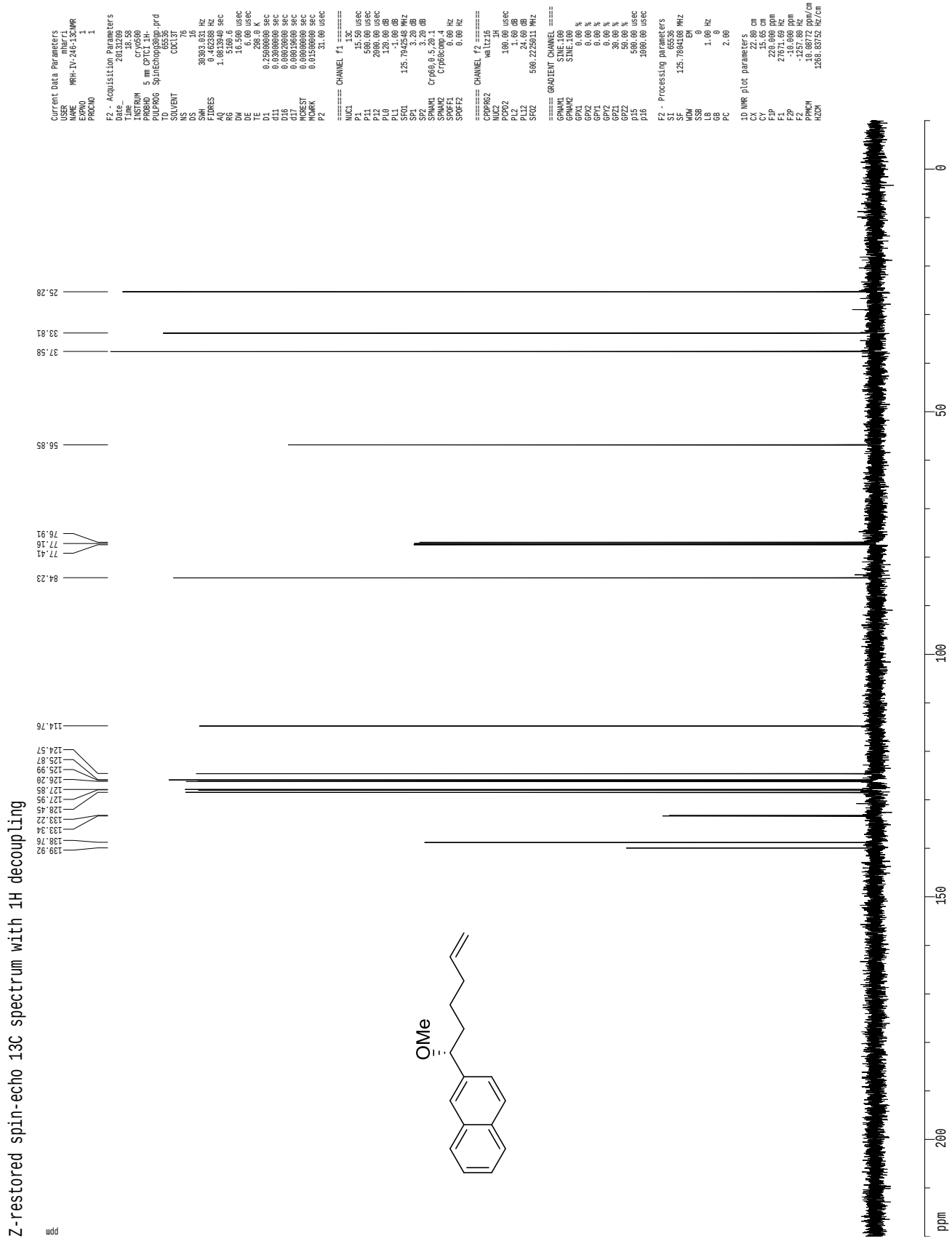
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

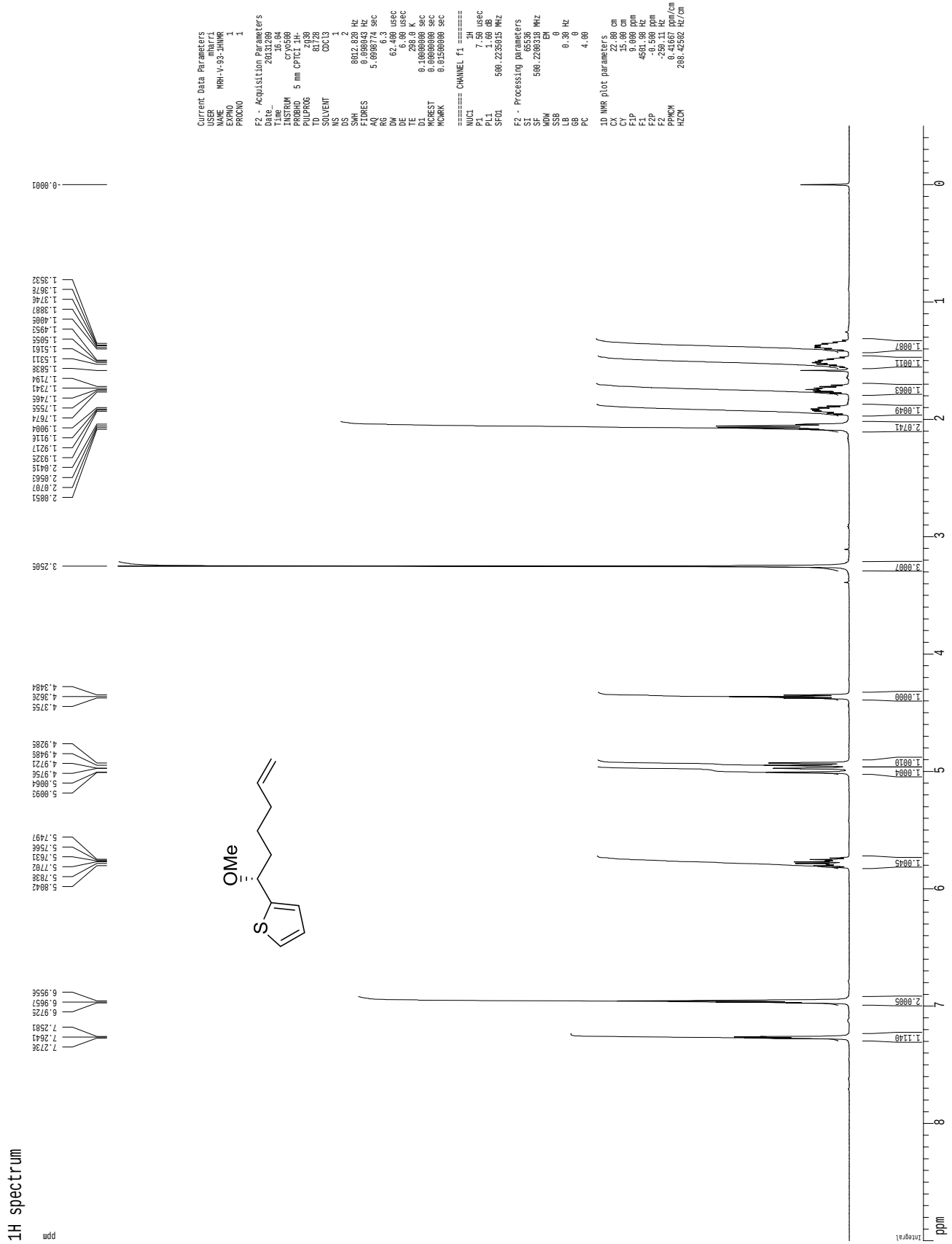
F2 - Processing parameters
SI 65536
SF 125.7884190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PPMCM 9.88842 ppm/cm
HZCM 1241.25415 Hz/cm

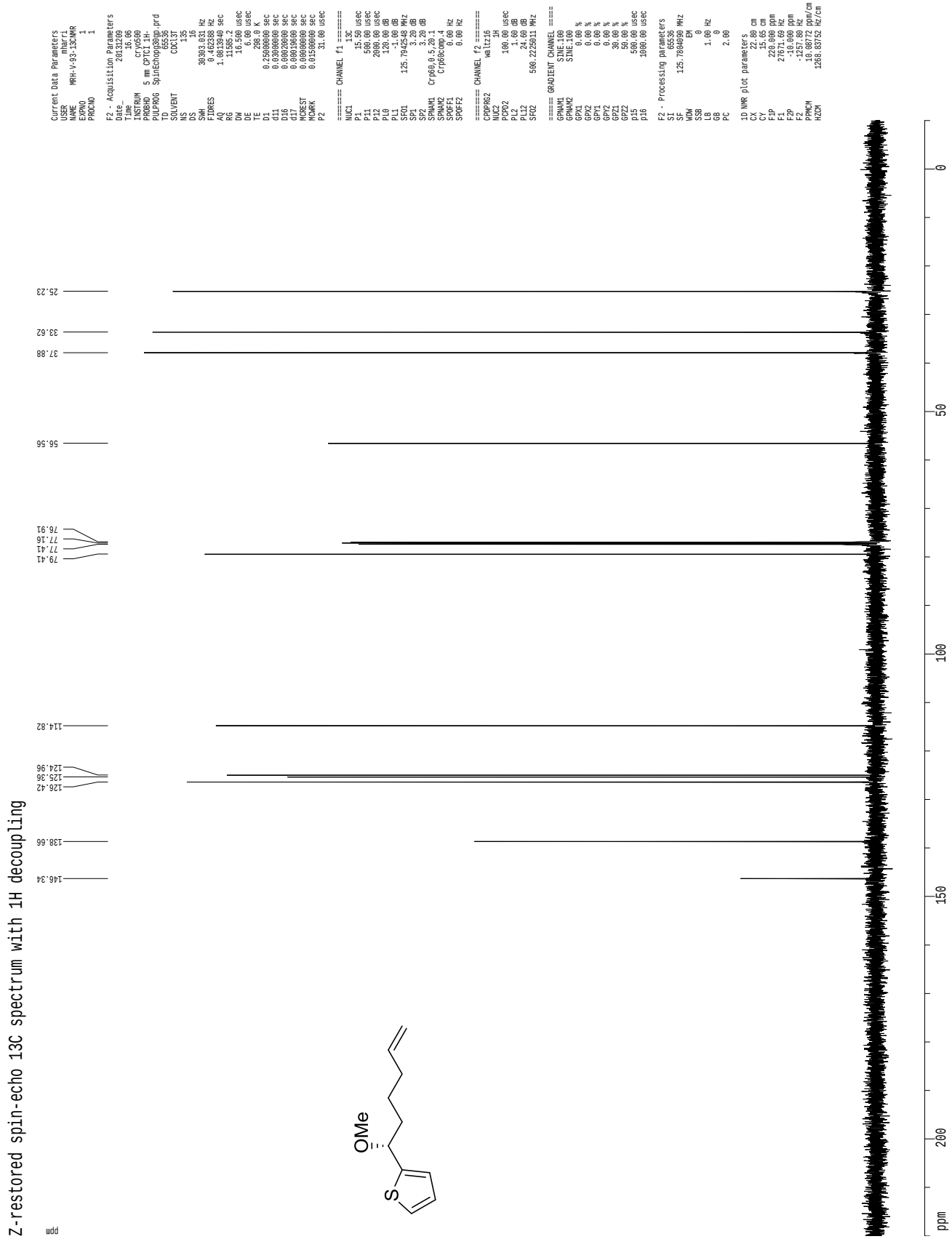

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-IV-246-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131209
Time 18.56
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200393 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.464 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
OMe
integral
ppm

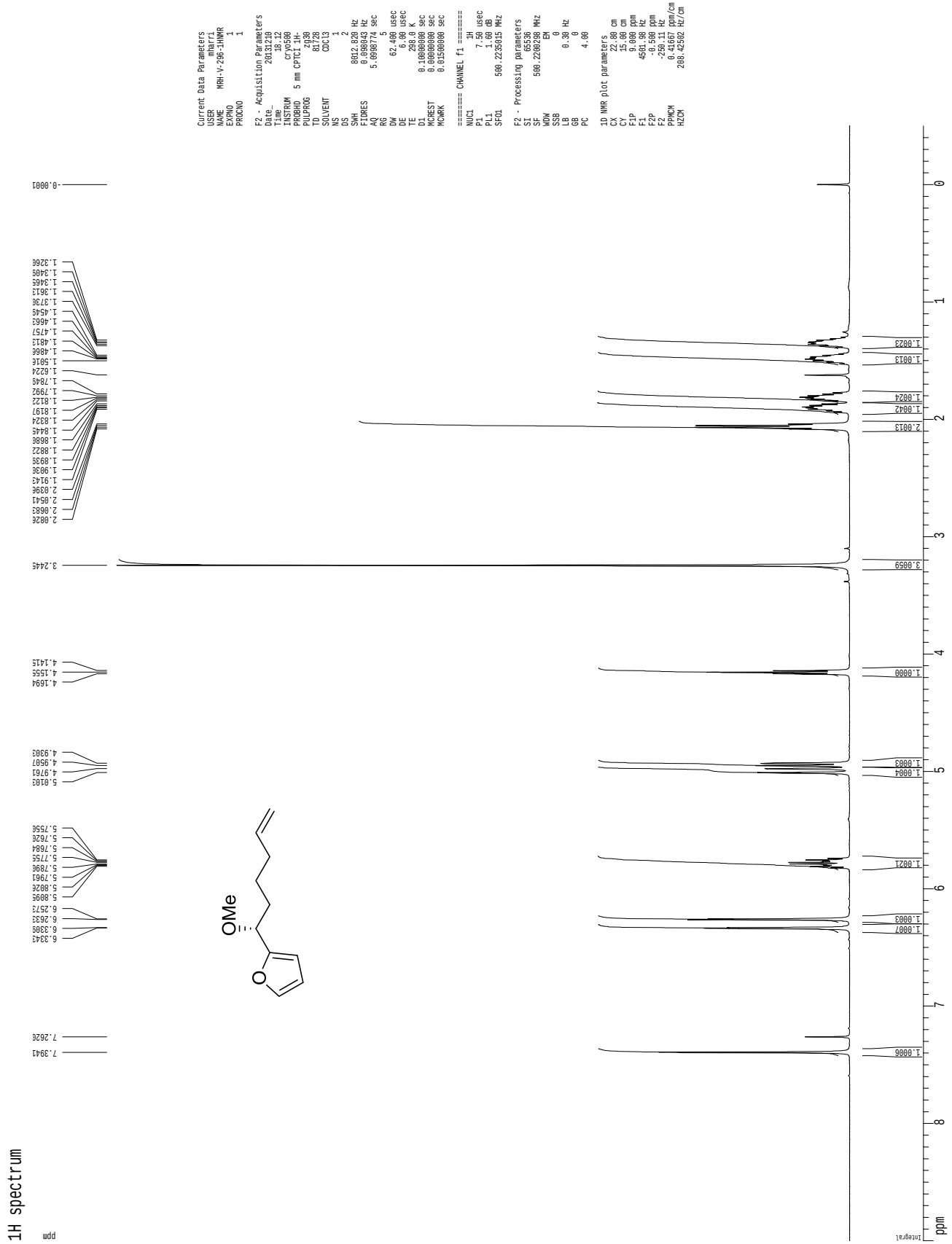
Z-restored spin-echo 13C spectrum with 1H decoupling



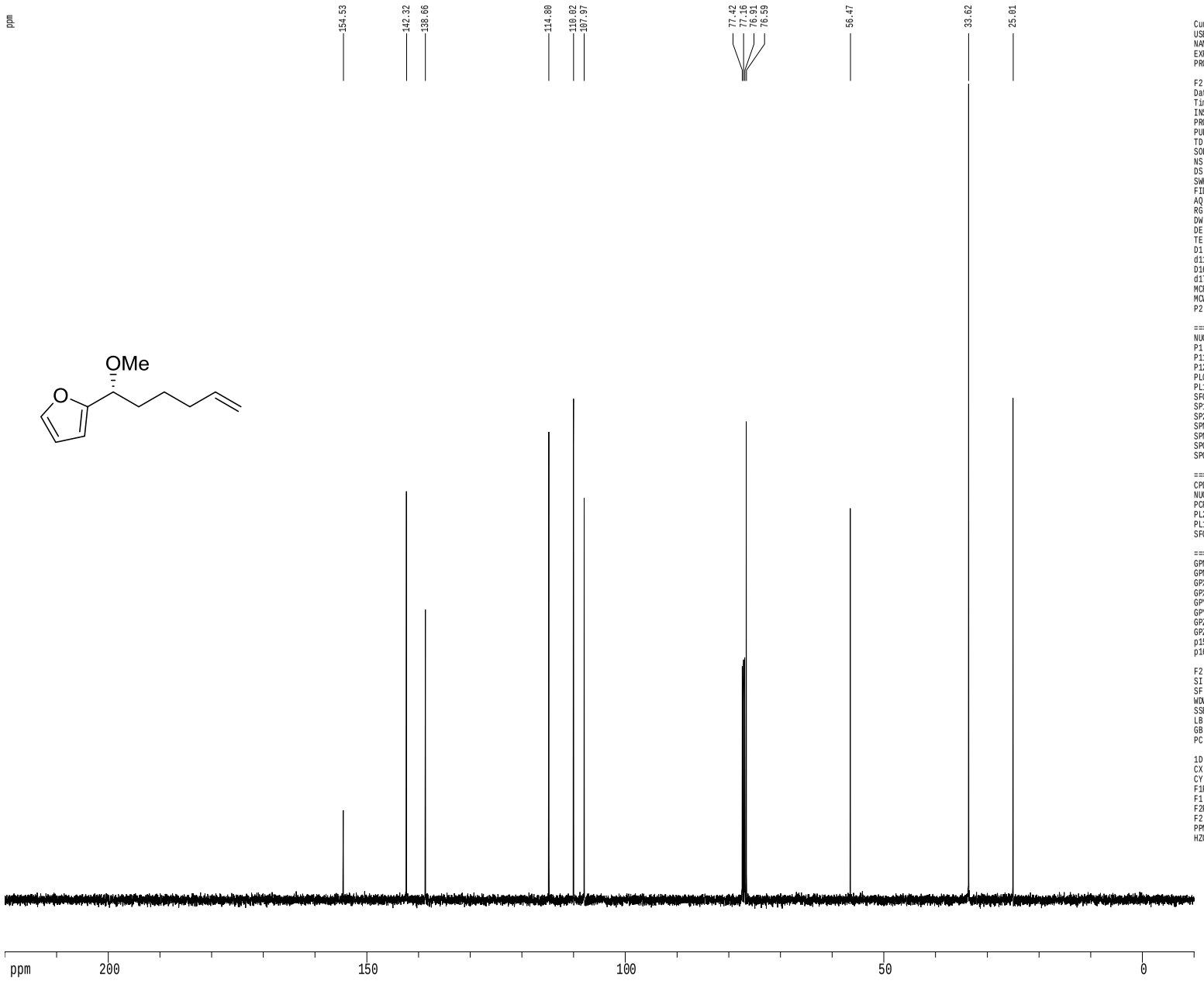

1H spectrum
ppm
Integral
Current Data Parameters
USER mharri
NAME MRH-V-93-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131209
Time 16.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200318 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.465 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
OMe
S
0.0001
1.3532
1.3678
1.3746
1.3887
1.4006
1.4953
1.5055
1.5163
1.5311
1.5838
1.7194
1.7341
1.7465
1.7555
1.7674
1.9004
1.9116
1.9217
1.9325
2.0415
2.0563
2.0707
2.0851
3.2506
4.3484
4.3626
4.3756
4.9285
4.9485
4.9721
4.9756
5.0064
5.0093
5.7497
5.7566
5.7633
5.7702
5.7838
5.8042
6.9556
6.9657
6.9726
7.2581
7.2641
7.2736
2.0741
1.0049
1.0053
1.0011
1.0087
3.0007
1.0000
1.0004
1.0010
1.0045
2.0005
1.1140
0
1
2
3
4
5
6
7
8
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
ppm
Integral
OMe
O

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER mharri
NAME MRH-V-296-13CNMR
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131210
Time 18.15
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 72
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3251
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec

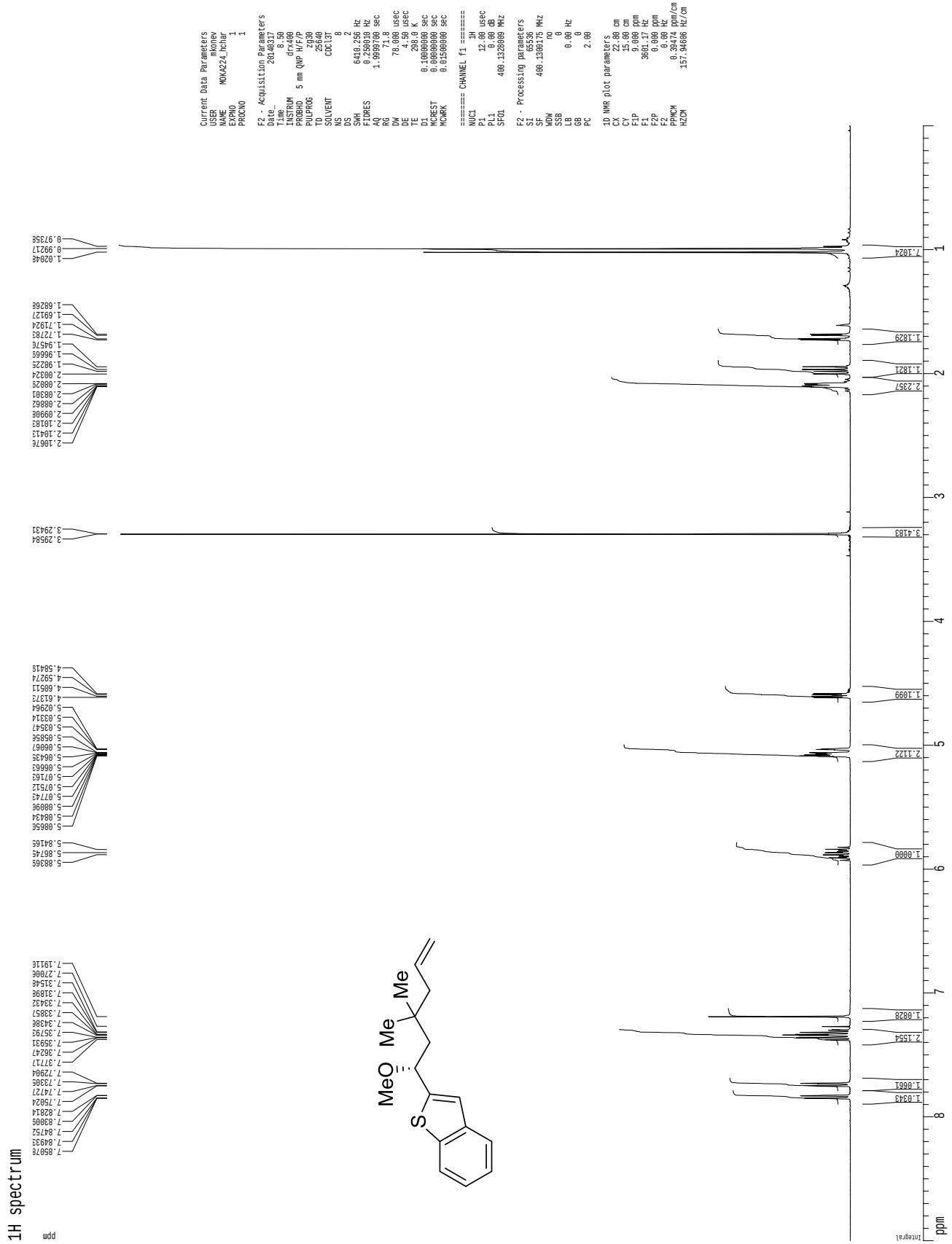
======= CHANNEL f1 =======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOF1 0.00 Hz
SPOF2 0.00 Hz

======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

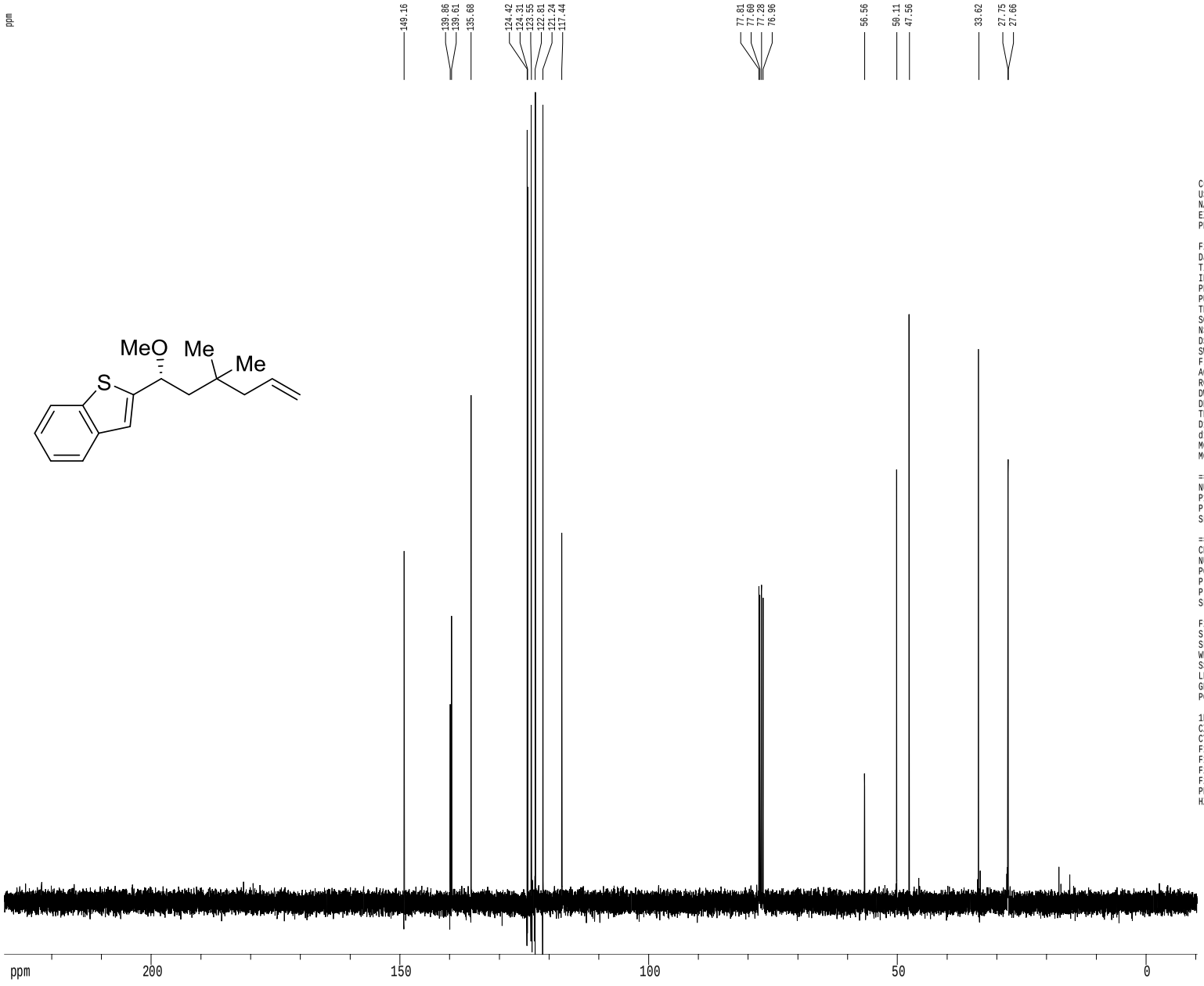
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm


1H spectrum
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
MeO
Me
Me
S
ppm

13C spectrum with 1H decoupling



Current Data Parameters
USER mkonev
NAME MOKA224_cchar
EXPNO 1
PROCNO 1

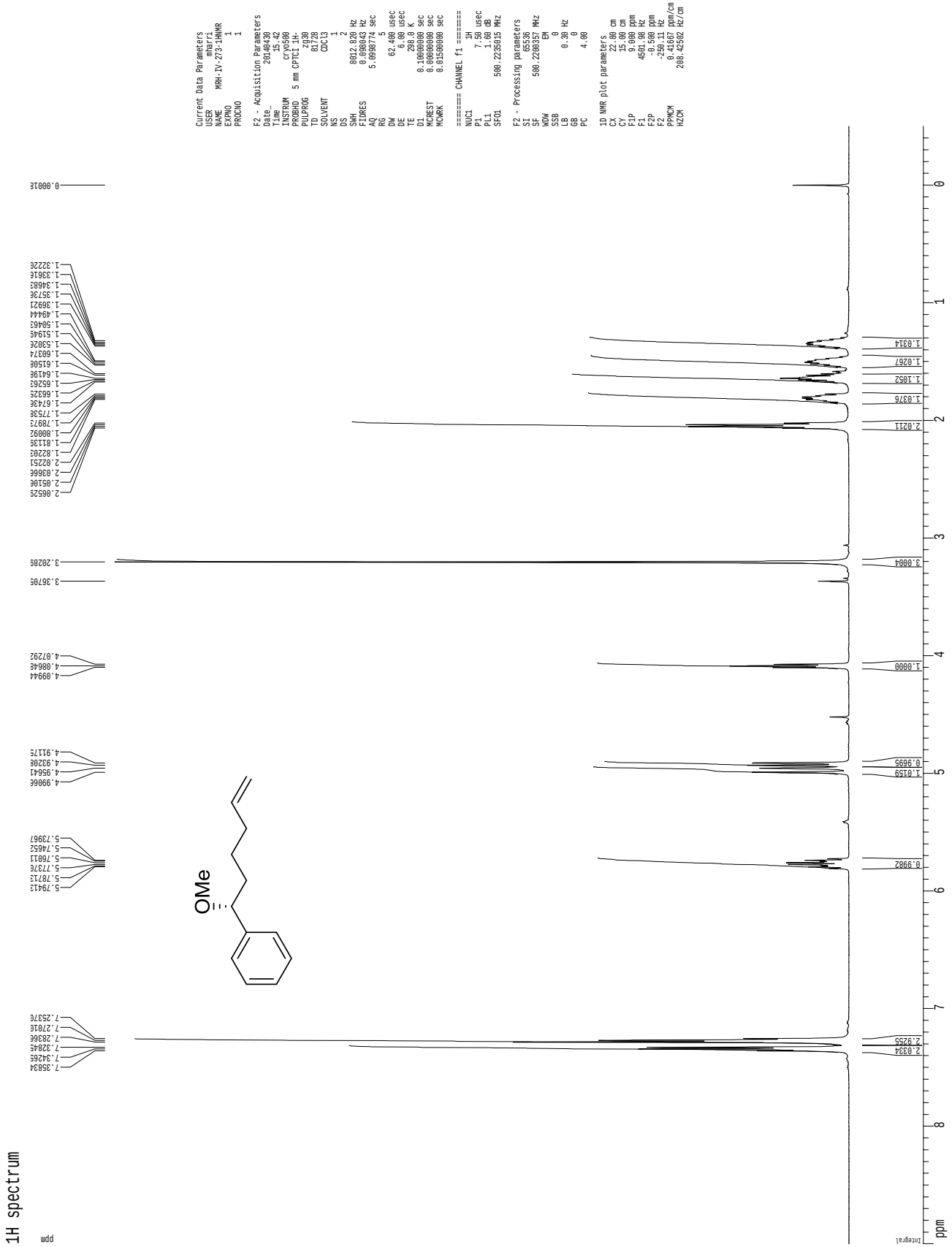
F2 - Acquisition Parameters
Date_ 20140317
Time 8.54
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.8 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

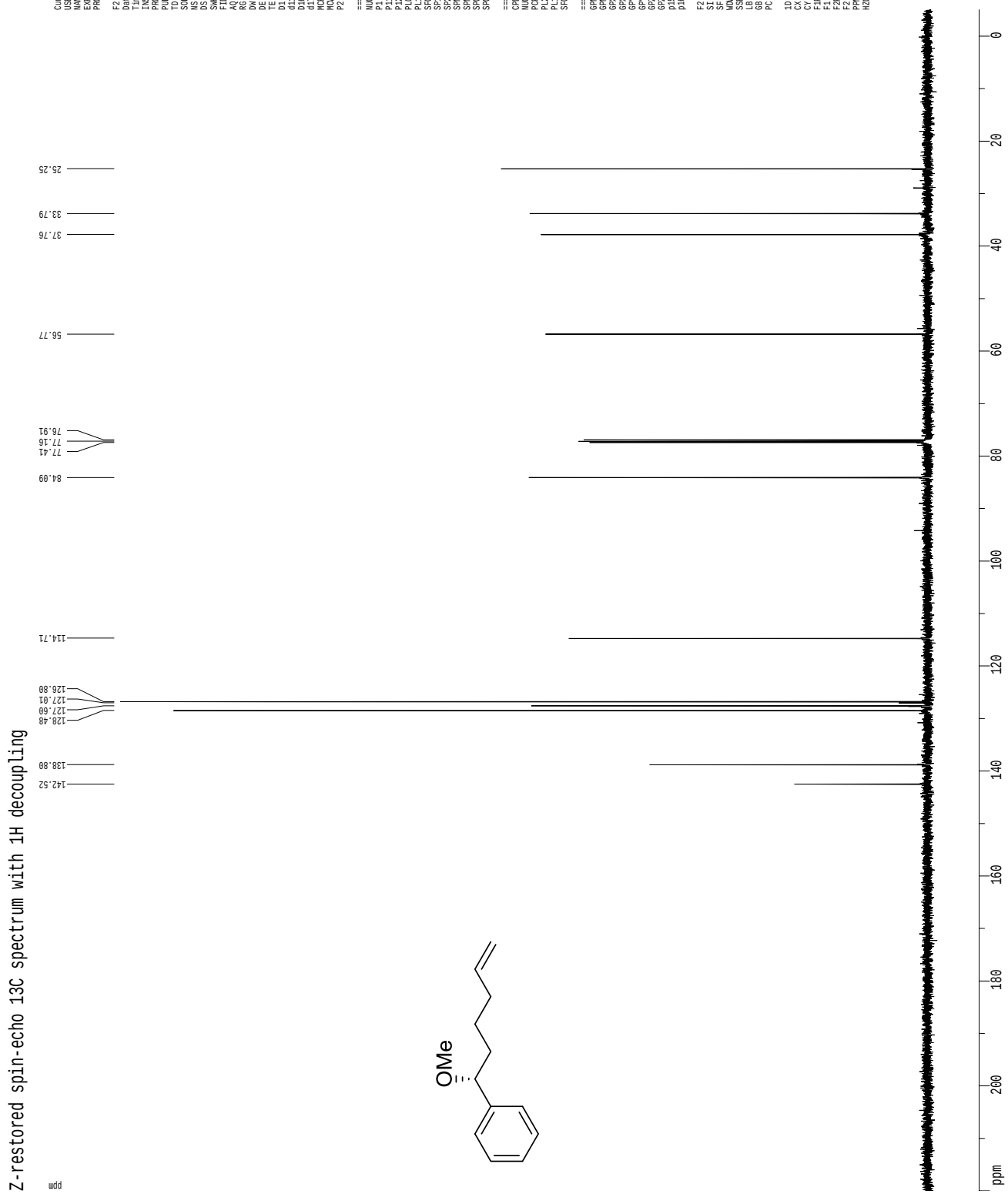
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

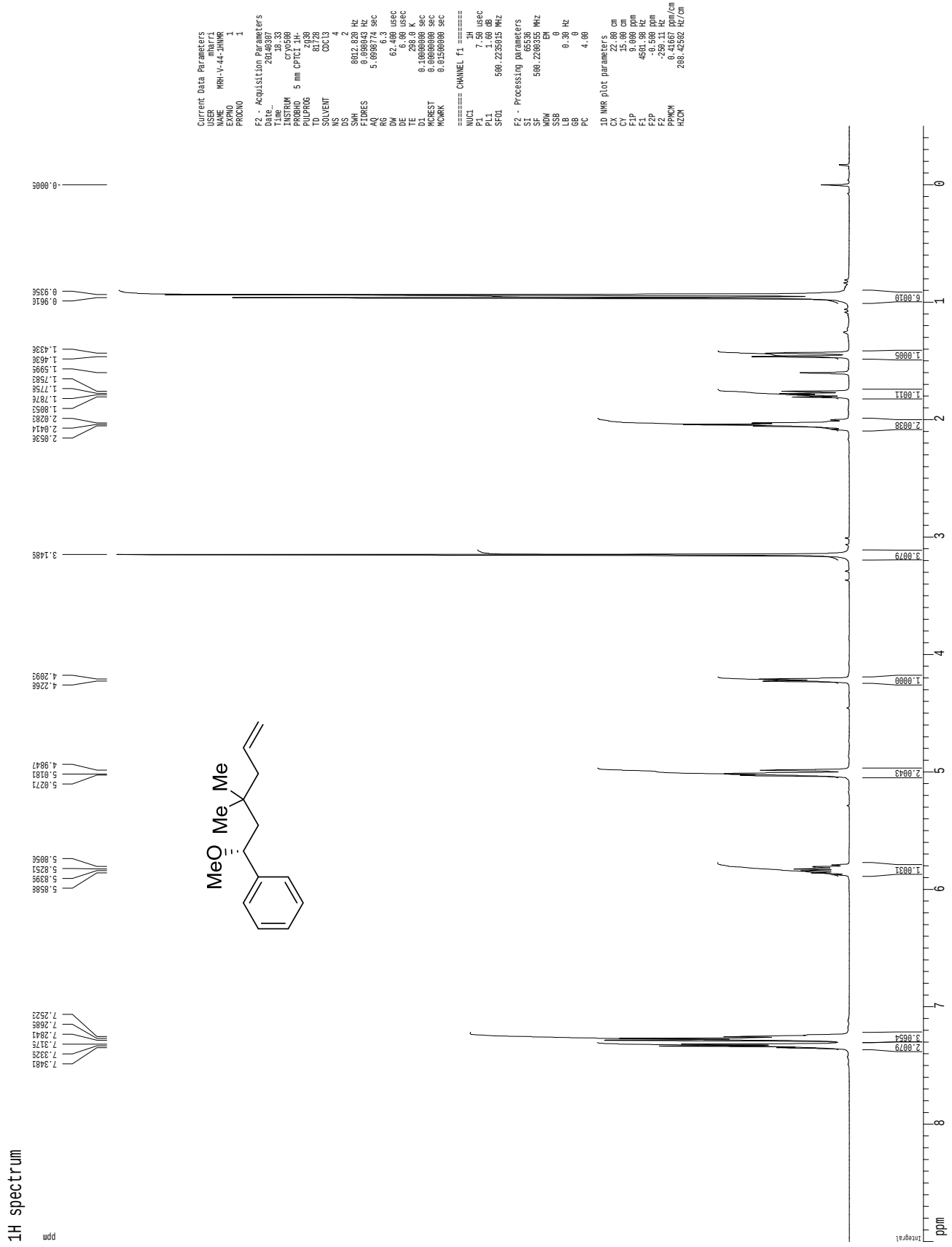
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 229.496 ppm
F1 23090.21 Hz
F2P -10.579 ppm
F2 -1064.37 Hz
PPMCM 10.52959 ppm/cm
HZCM 1059.41138 Hz/cm

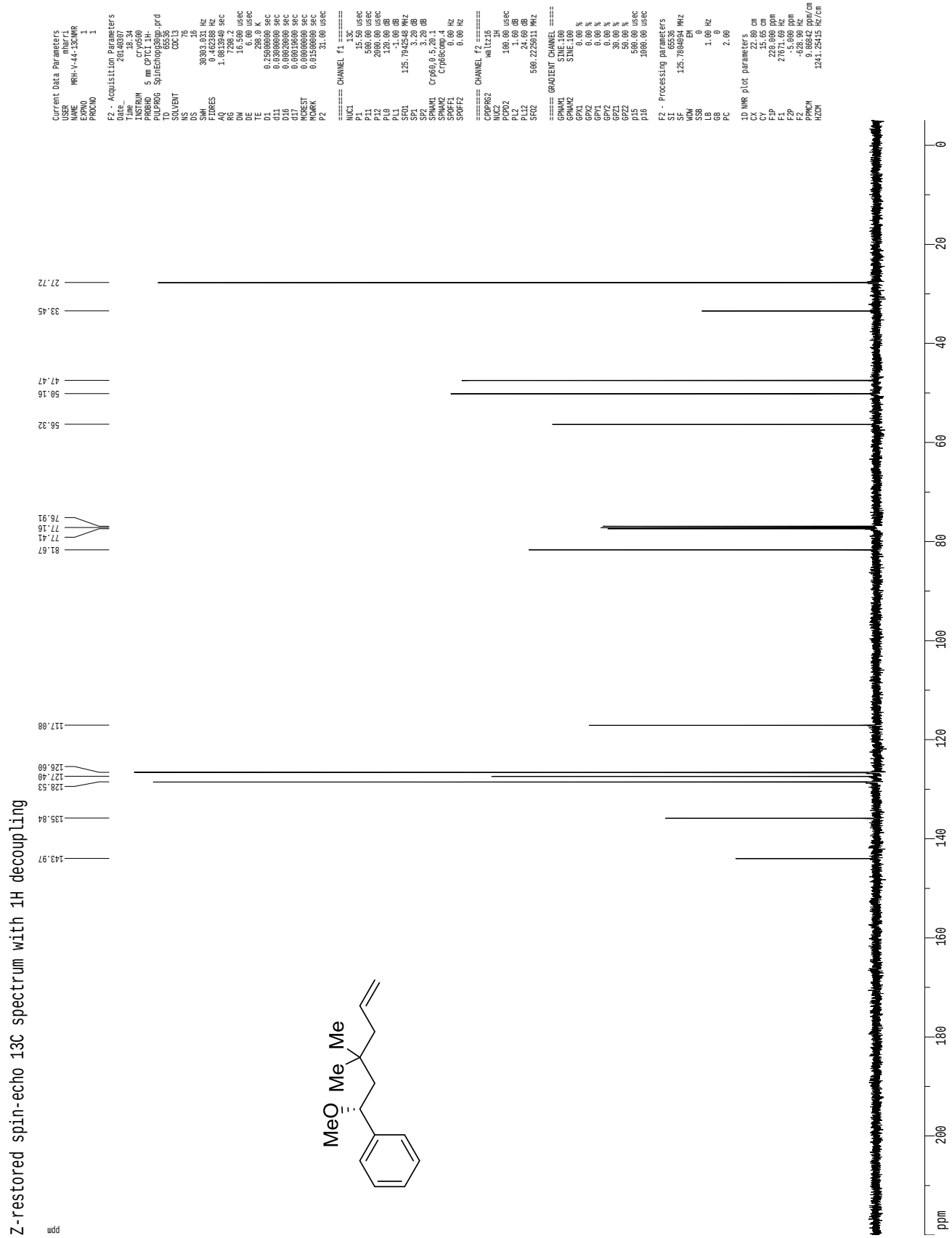

1H spectrum
ppm
OMe

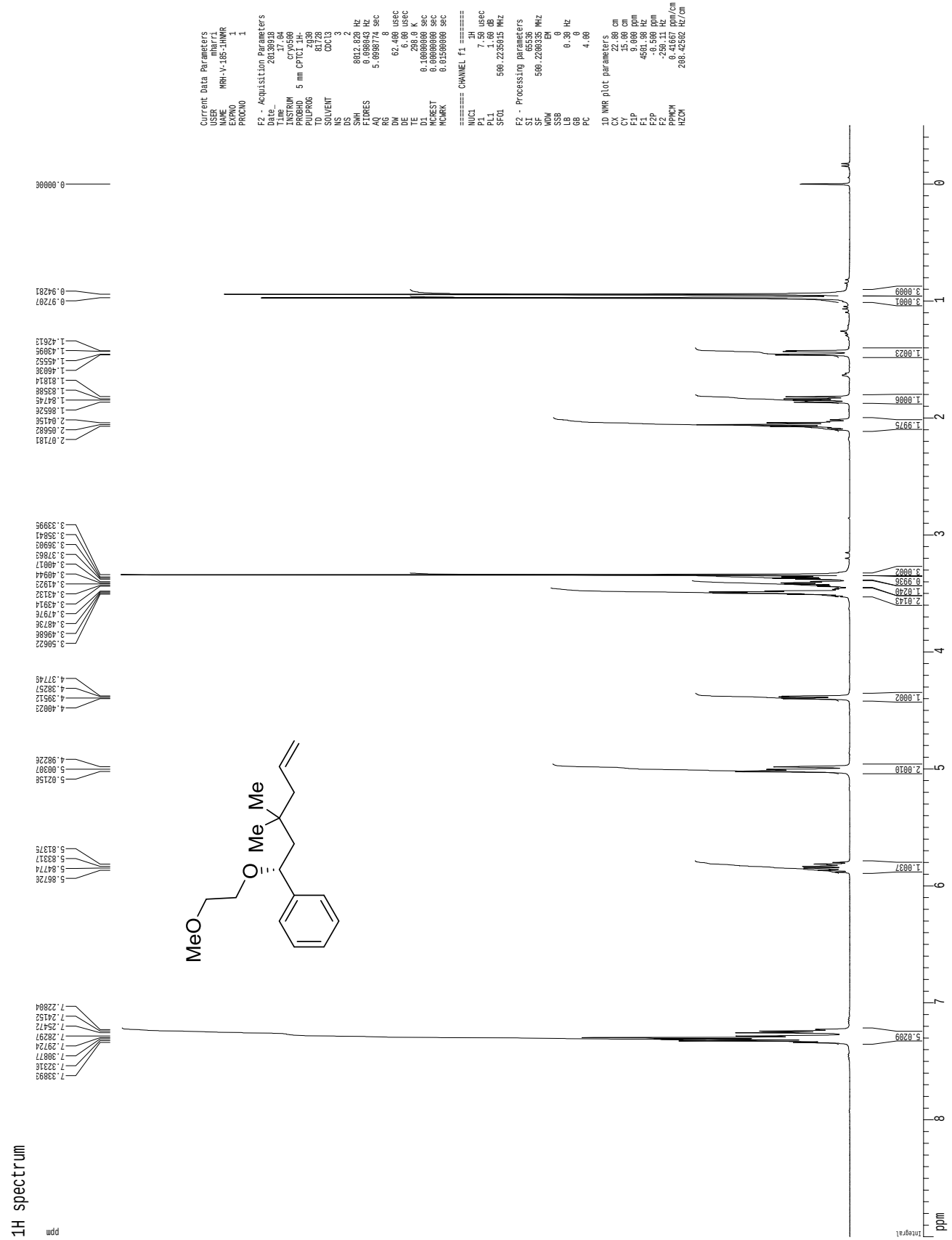
Z-restored spin-echo 13C spectrum with 1H decoupling



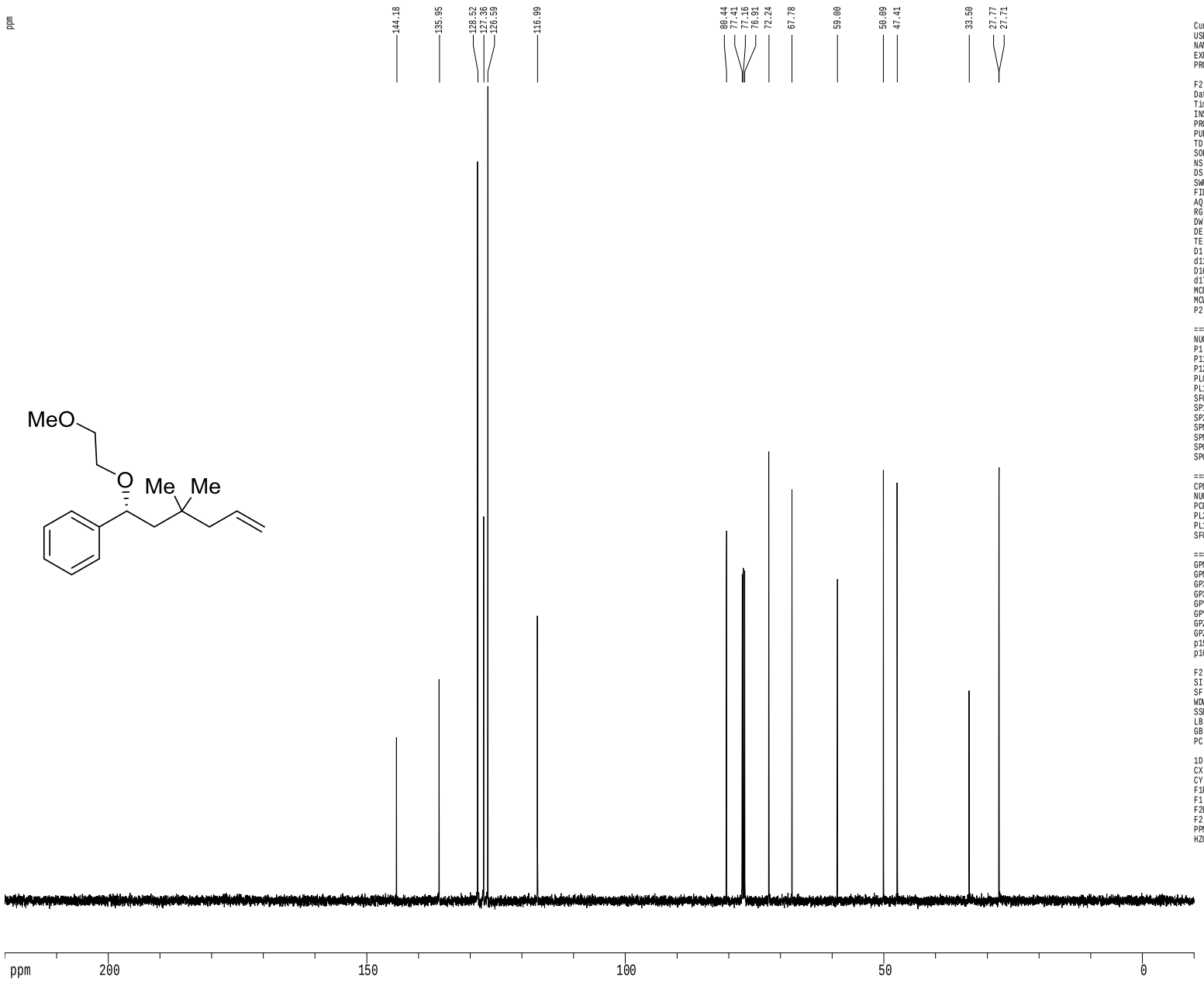

1H spectrum
ppm
Integral
Current Data Parameters
USER mharri
NAME MRH-V-44-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140307
Time 18:33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200355 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
MeO
Me
Me
0.0005
0.9356
0.9616
1.4336
1.4638
1.5995
1.7583
1.7756
1.7876
1.8052
2.0283
2.0414
2.0536
3.1485
4.2268
4.2093
4.9847
5.0181
5.0273
5.8056
5.8251
5.8399
5.8588
7.2523
7.2685
7.2841
7.3175
7.3328
7.3481
6.0810
1.0005
1.0011
2.0038
3.0079
1.0000
2.0043
1.0031
3.0654
2.0079

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER mharri
NAME MRH-V-185-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130918
Time 17.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200335 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
MeO
O
Me
Me
integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER mharri
NAME MRH-V-185-13CNMR
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130918
Time 17.06
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 96
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec

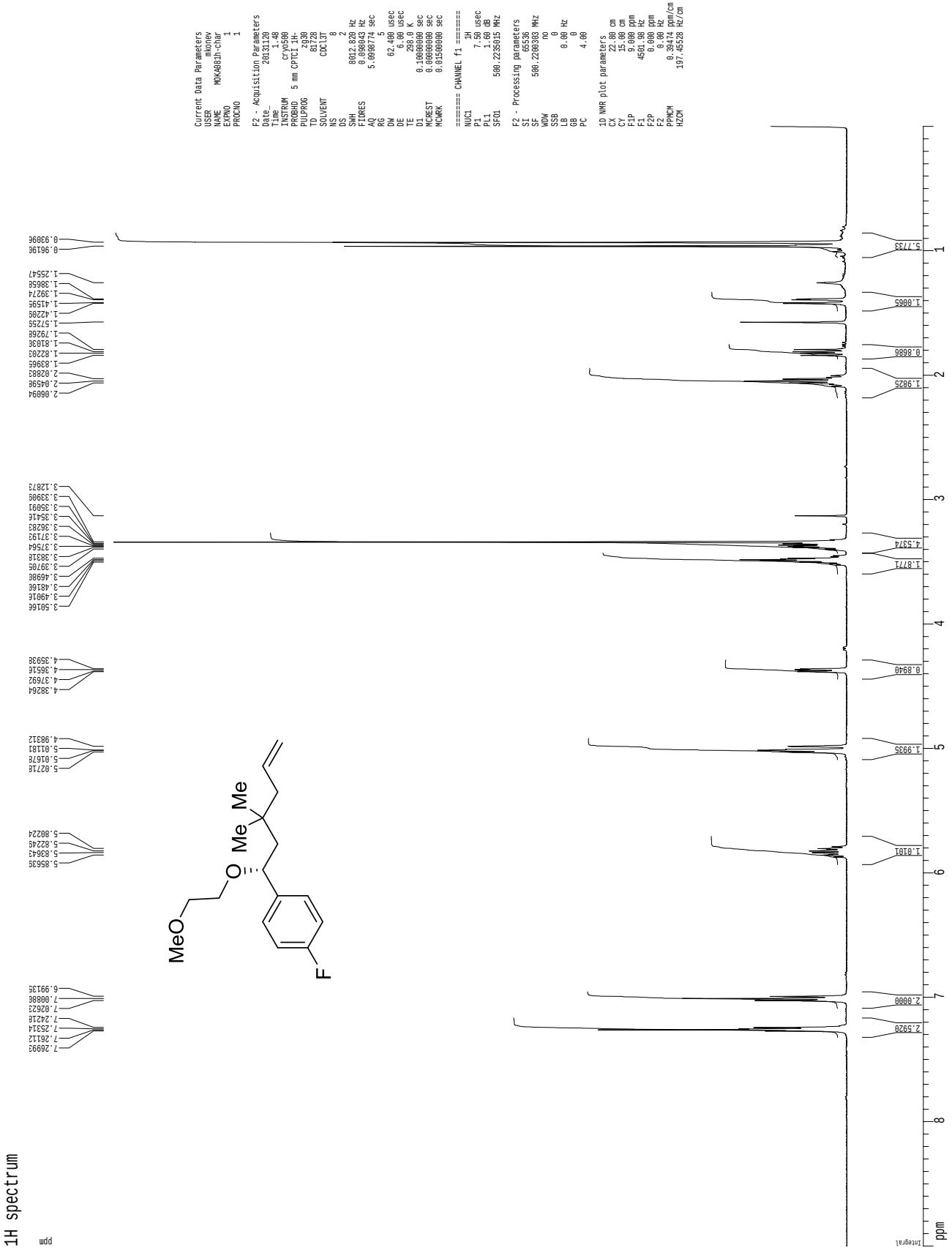
======= CHANNEL f1 =======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

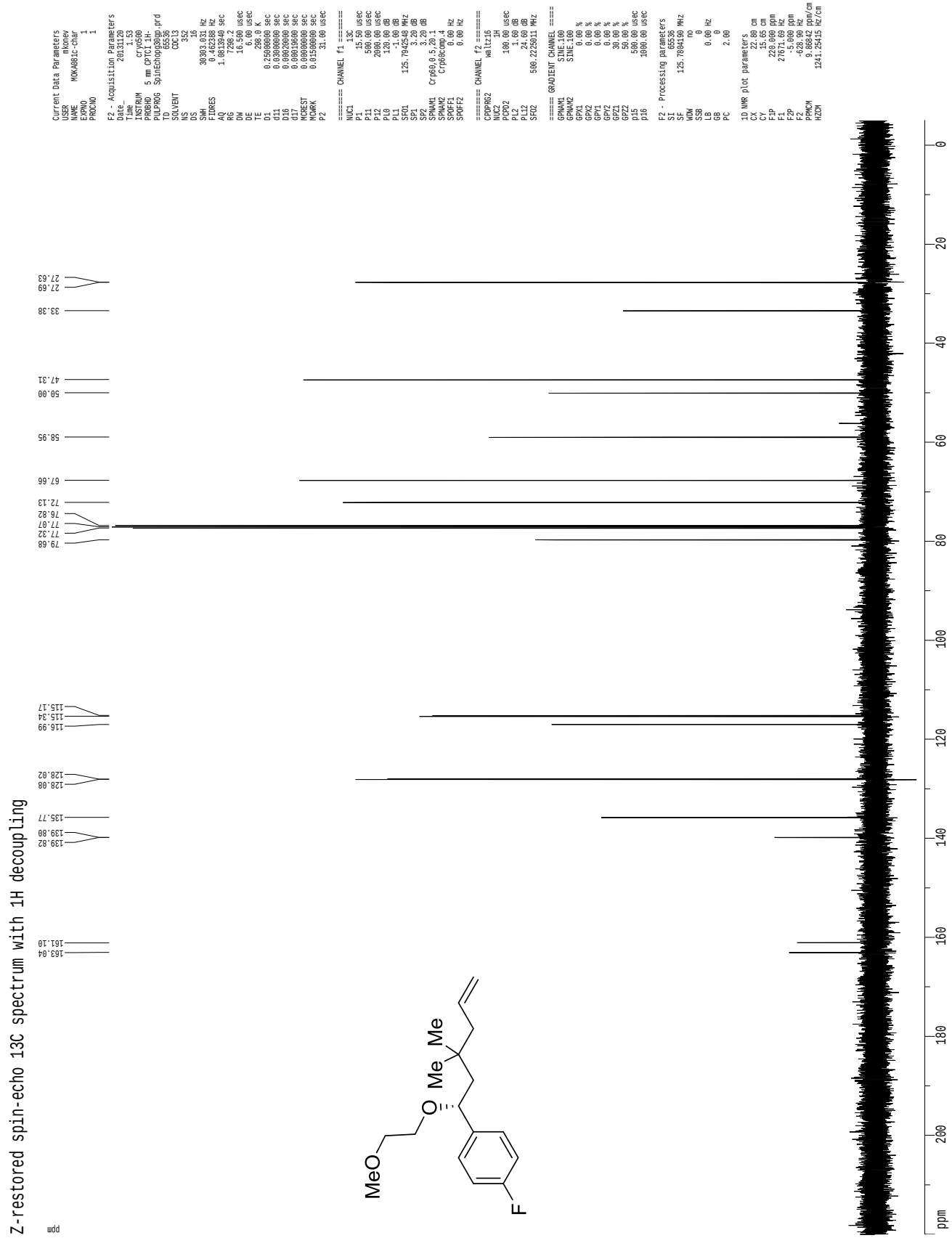
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

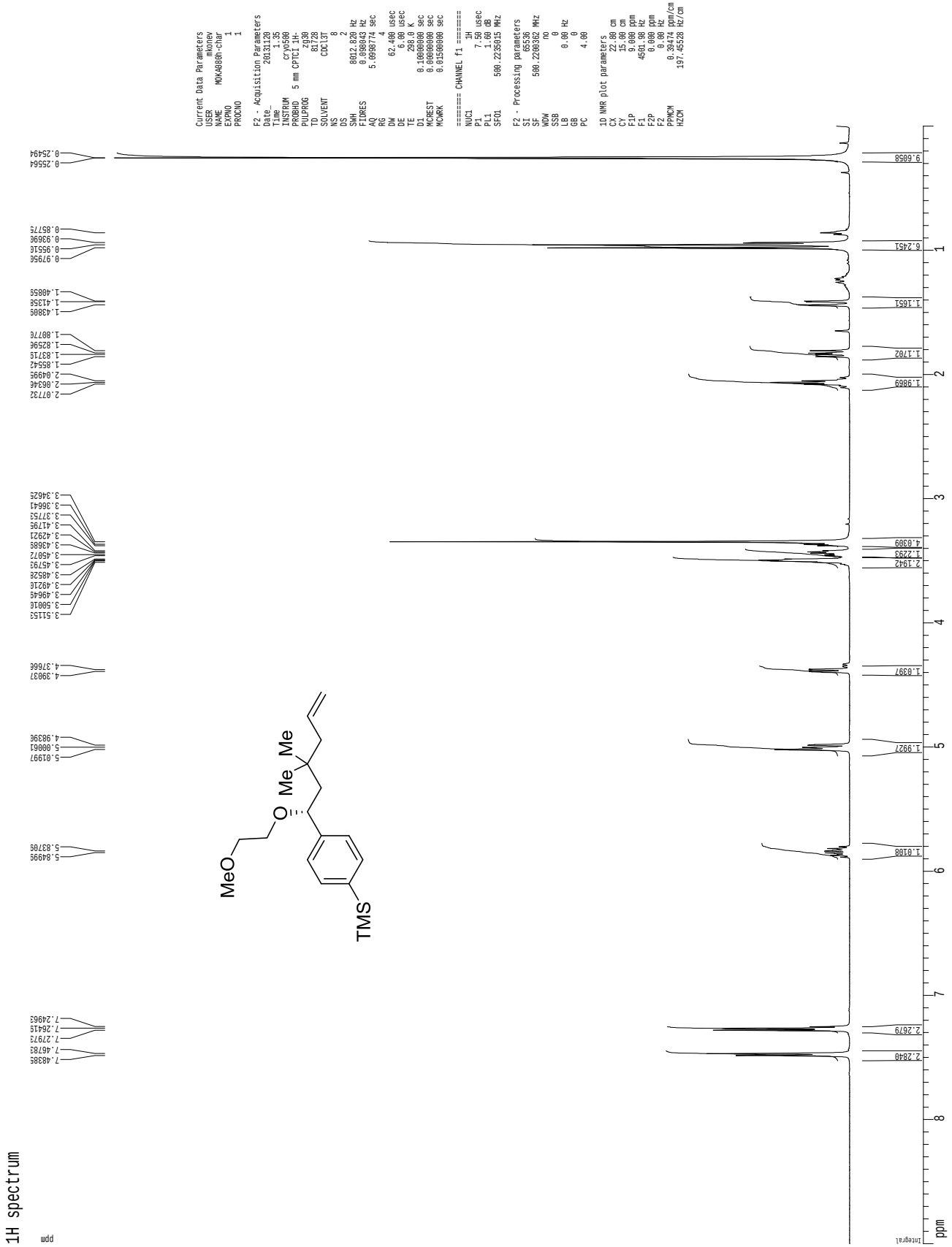
F2 - Processing parameters
SI 65536
SF 125.7804099 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm

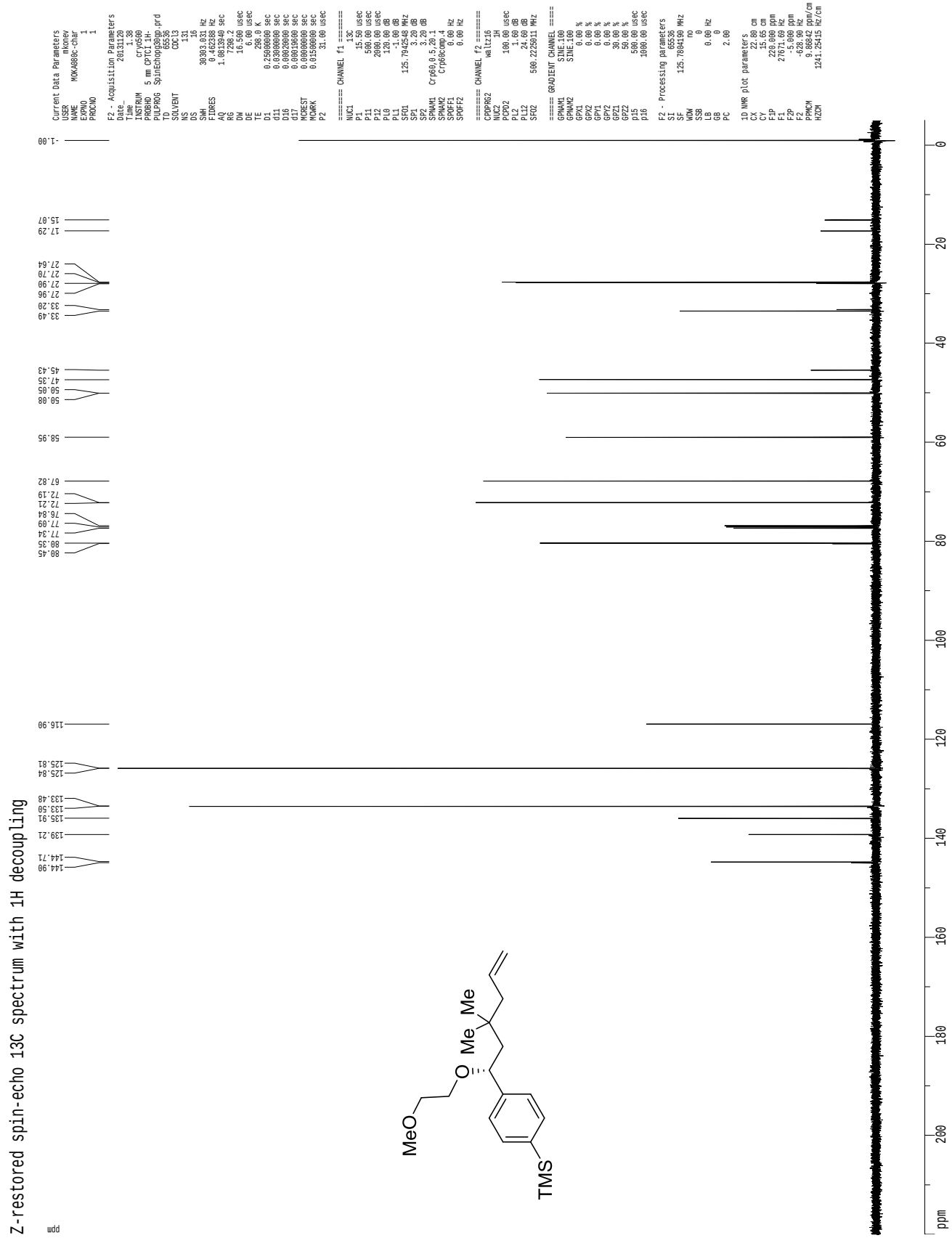

1H spectrum
Current Data Parameters
USER mkonev
NAME MOKA081h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200383 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
MeO
O
Me
Me
F
integral
ppm

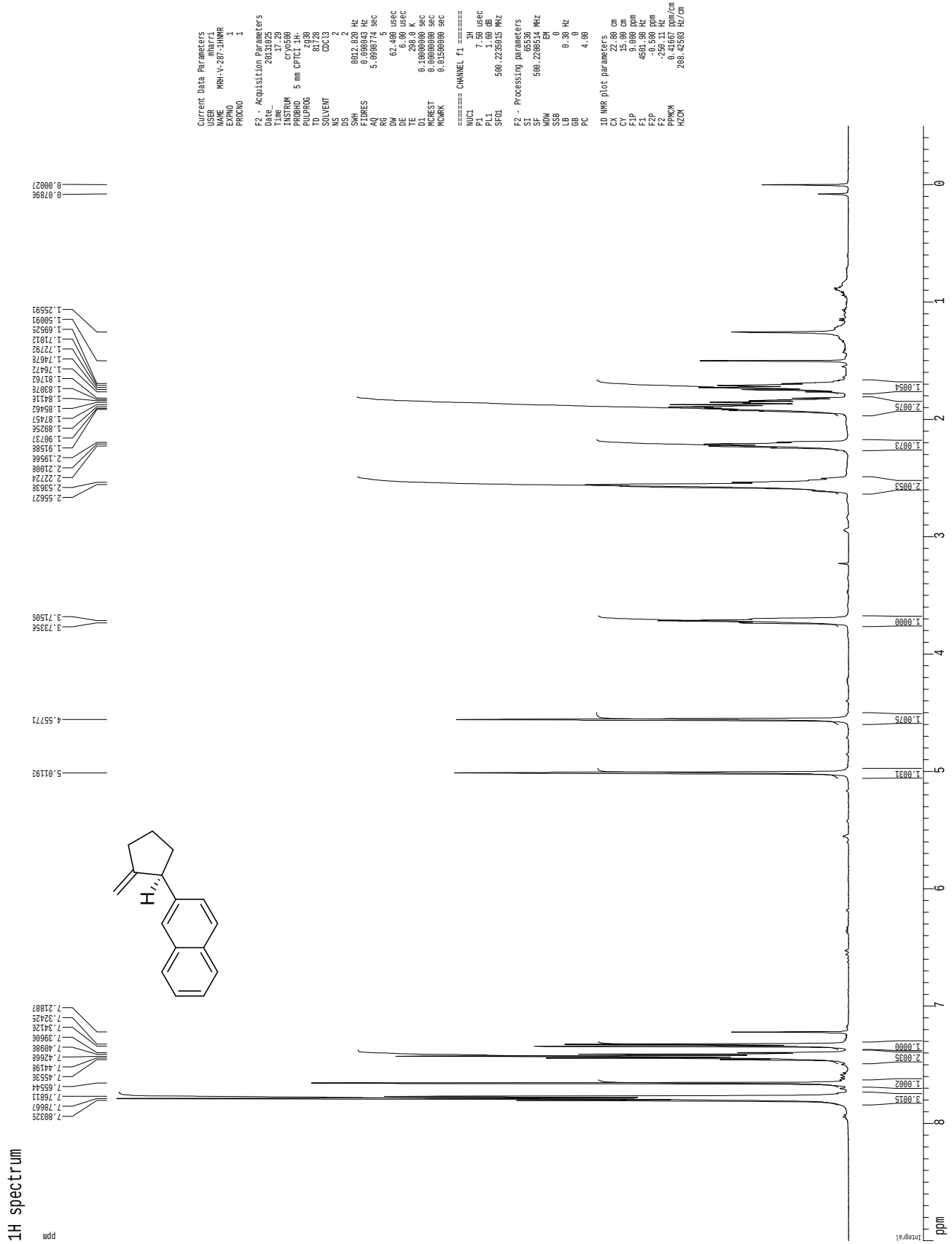
Z-restored spin-echo 13C spectrum with 1H decoupling

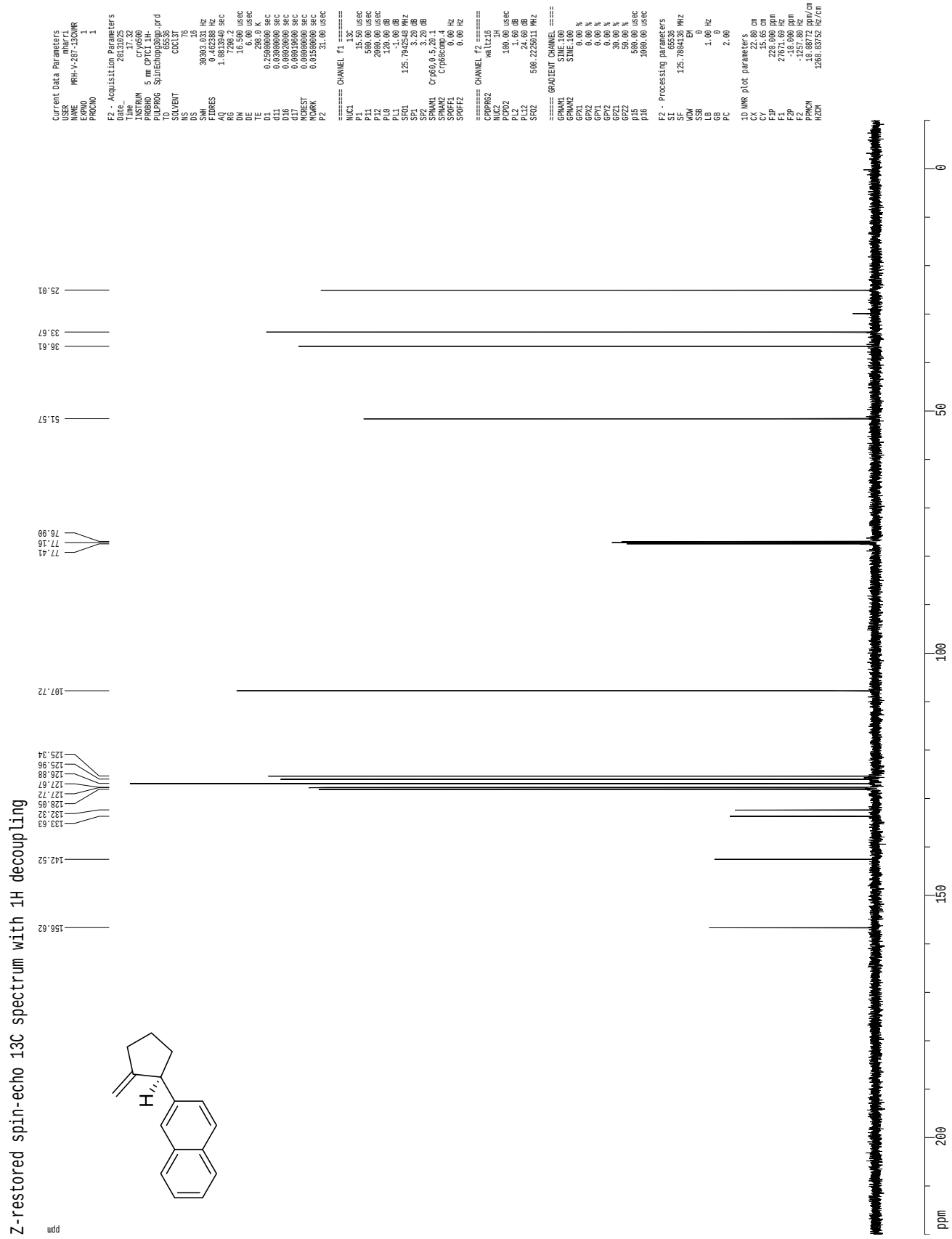


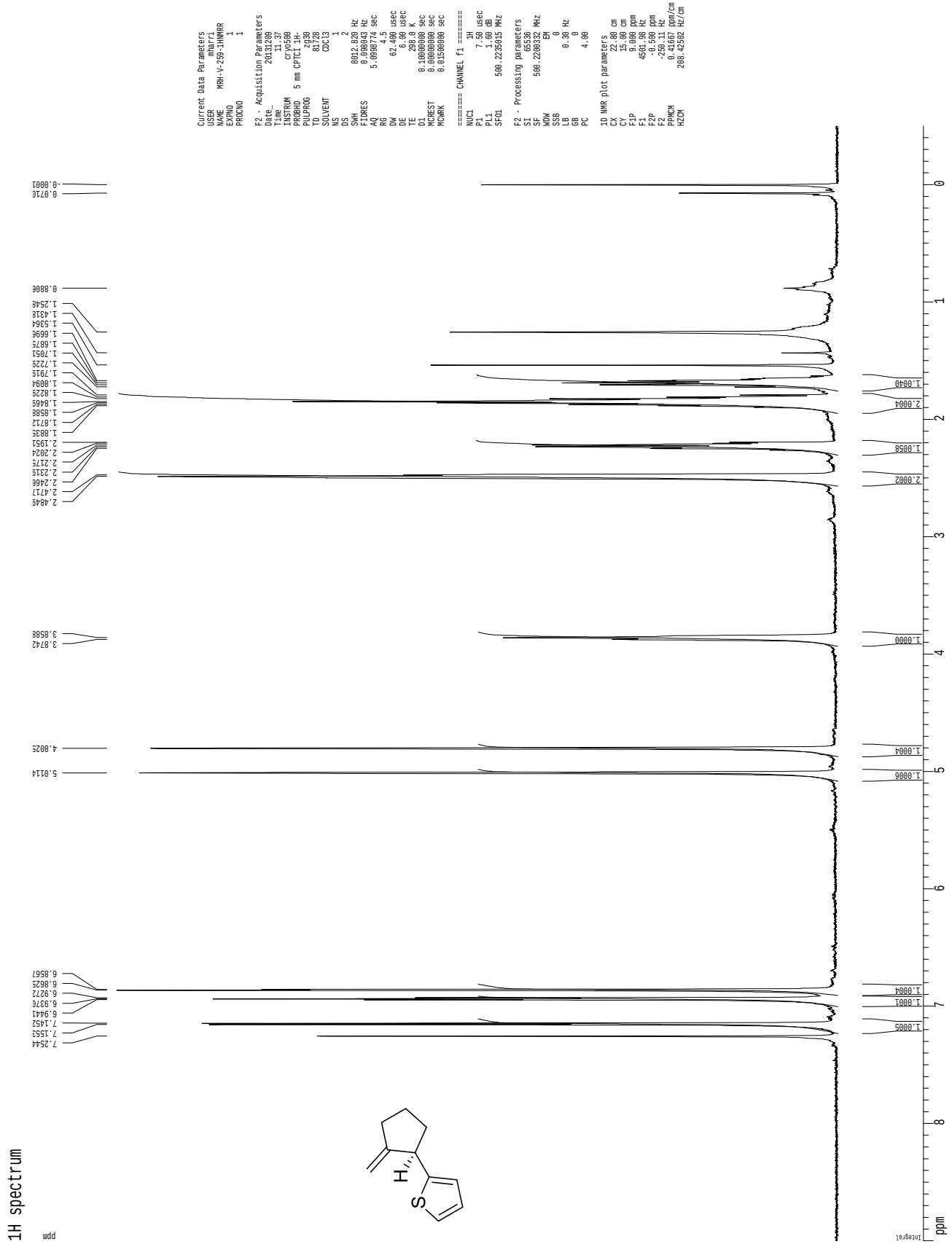

1H spectrum
Current Data Parameters
USER mkonev
NAME MOXA088h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.35
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200362 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
MeO
O
Me
Me
TMS
ppm
Integral

Z-restored spin-echo 13C spectrum with 1H decoupling

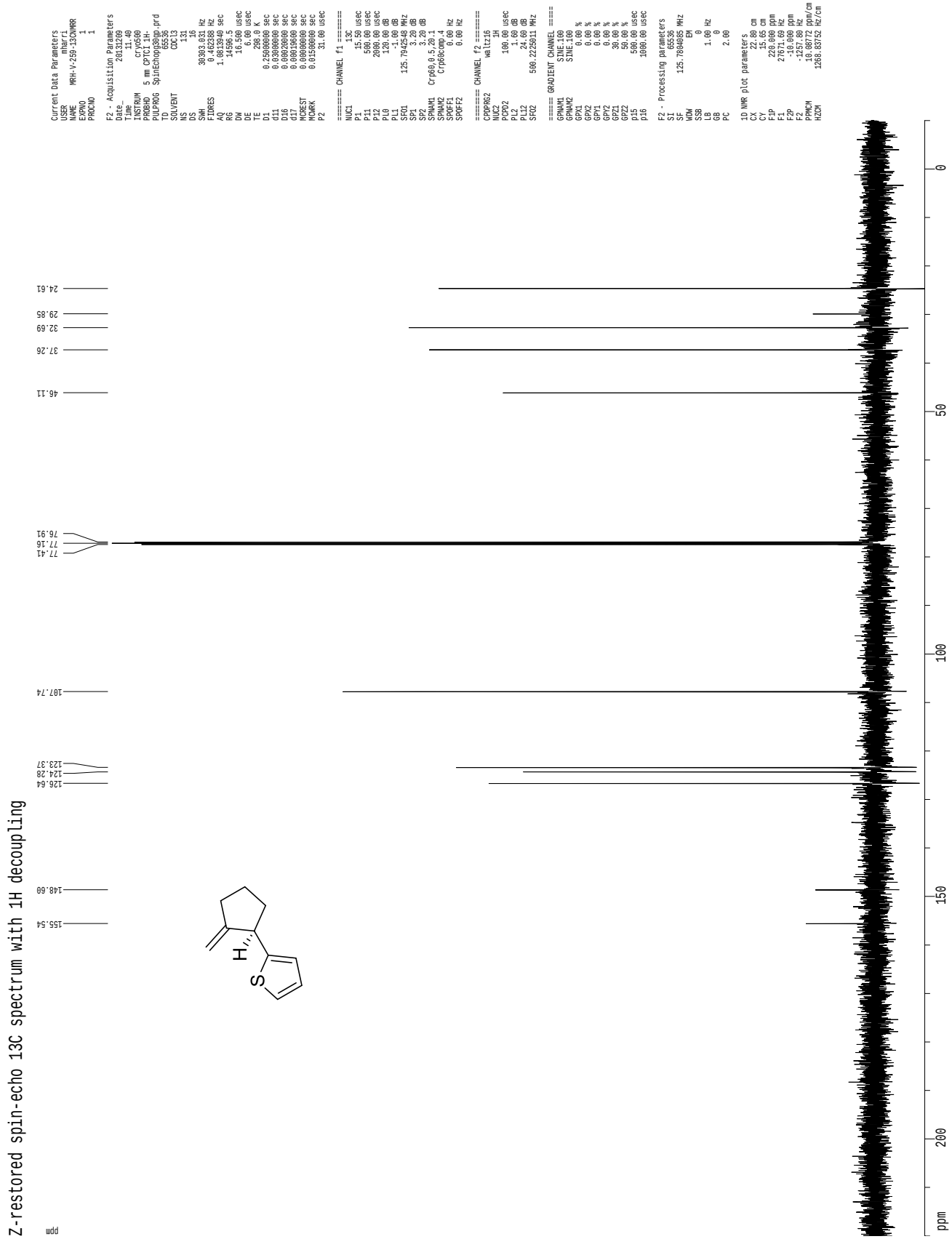


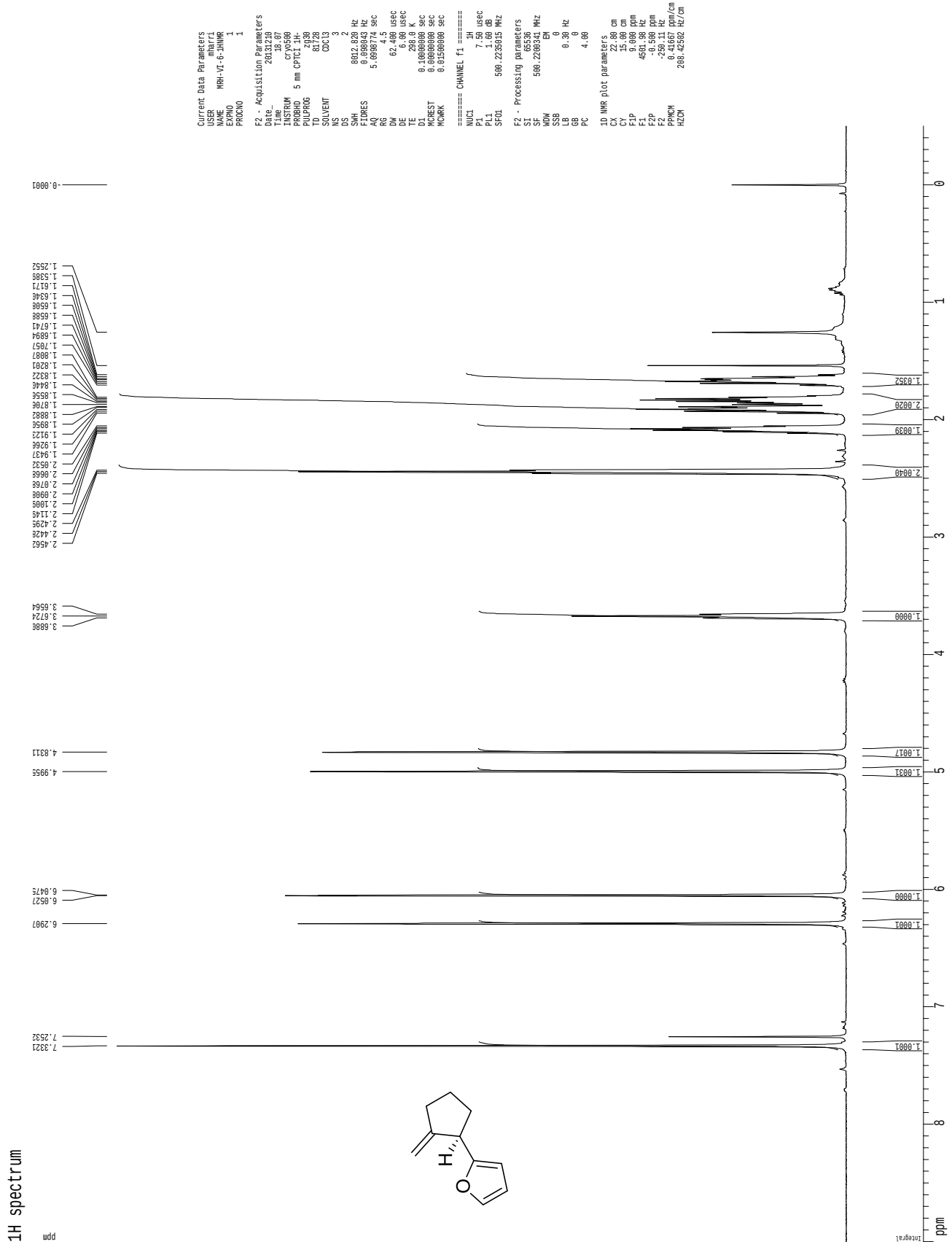

1H spectrum
ppm
Integral
Current Data Parameters
USER mharri
NAME MRH-V-287-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 17.29
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200514 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm
0.00027
0.07896
1.25591
1.50091
1.69526
1.71012
1.72792
1.74678
1.76472
1.81762
1.83078
1.84116
1.85462
1.87457
1.89256
1.90737
1.91588
1.95966
2.21008
2.22724
2.53638
2.55627
3.71506
3.73356
4.55771
5.01193
7.21887
7.32425
7.34126
7.39606
7.40986
7.42666
7.44198
7.45536
7.65544
7.76811
7.78667
7.80325
1.0054
2.0075
1.0073
2.0053
1.0000
1.0075
1.0031
1.0000
2.0035
1.0002
3.0015

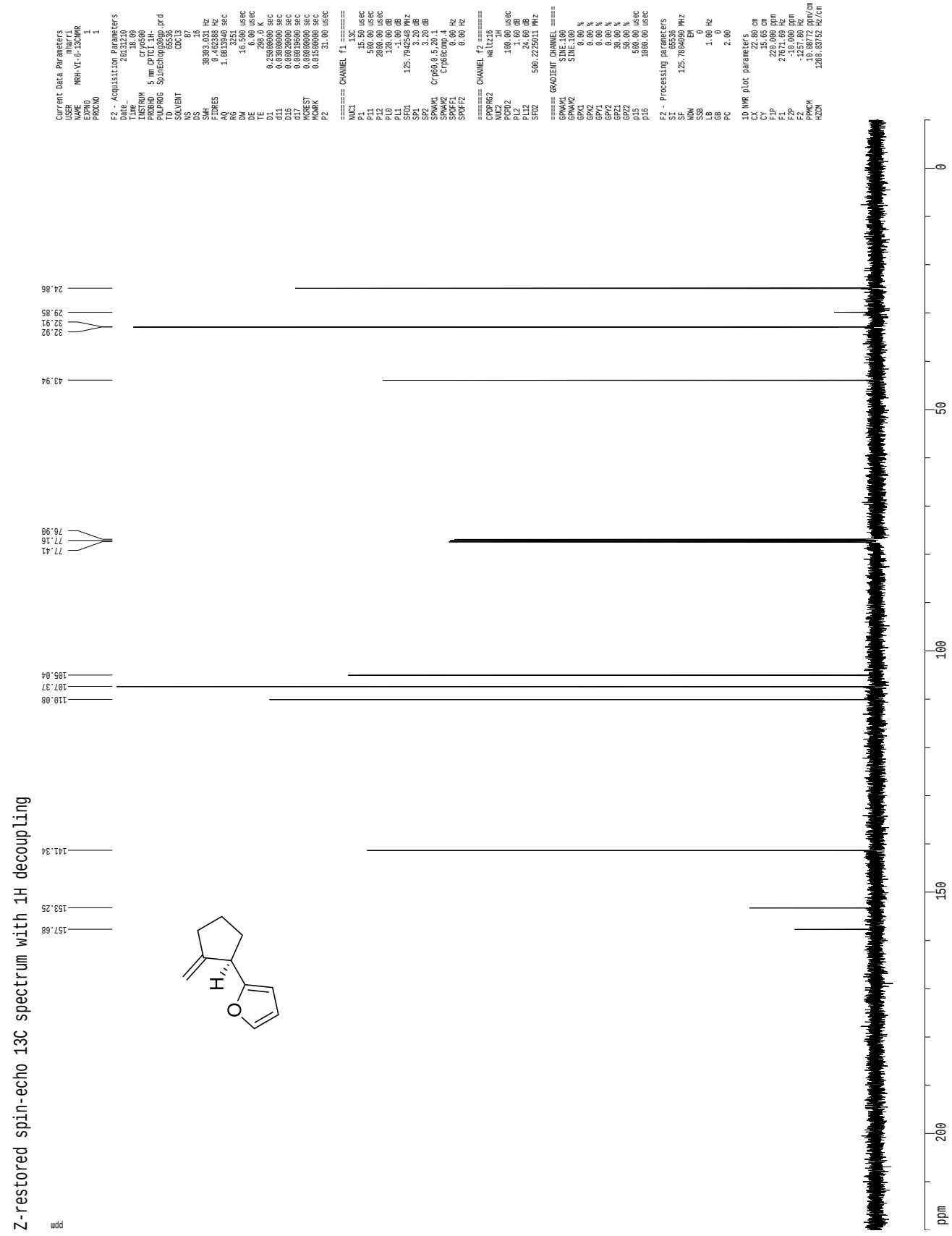

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
25.01
33.67
36.61
51.57
76.90
77.16
77.41
107.72
125.34
125.96
126.88
127.67
127.72
128.05
132.32
133.63
142.52
156.62
H
Current Data Parameters
USER mhart1
NAME MRH-V-287-13CHMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 17.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
NS 16
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
====== CHANNEL f1 ======
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884136 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm
0
50
100
150
200
ppm

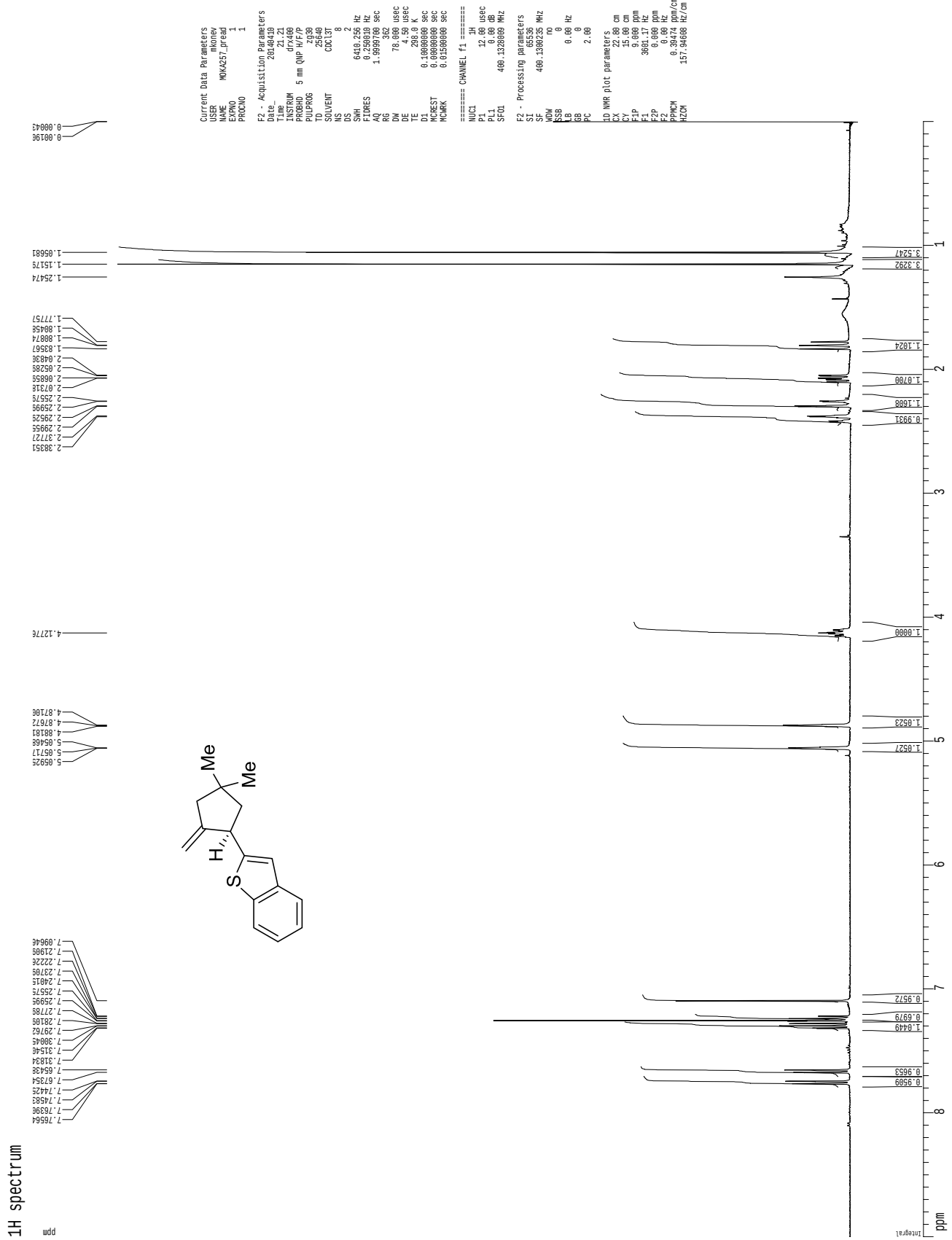

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-V-259-1HNMRR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131209
Time 11.37
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200332 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.498 ppm
F2 -249.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0001
0.0716
0.8806
1.2548
1.4318
1.5364
1.6696
1.6875
1.7051
1.7226
1.7914
1.8094
1.8226
1.8466
1.8588
1.8712
1.8836
2.1951
2.2024
2.2176
2.2314
2.2466
2.4717
2.4848
3.8588
3.8742
4.8025
5.0114
6.8567
6.8626
6.9272
6.9376
6.9441
7.1452
7.1552
7.2544
1.0040
2.0004
1.0058
2.0002
1.0000
1.0004
1.0006
1.0004
1.0001
1.0005
Integral
ppm
H
S
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

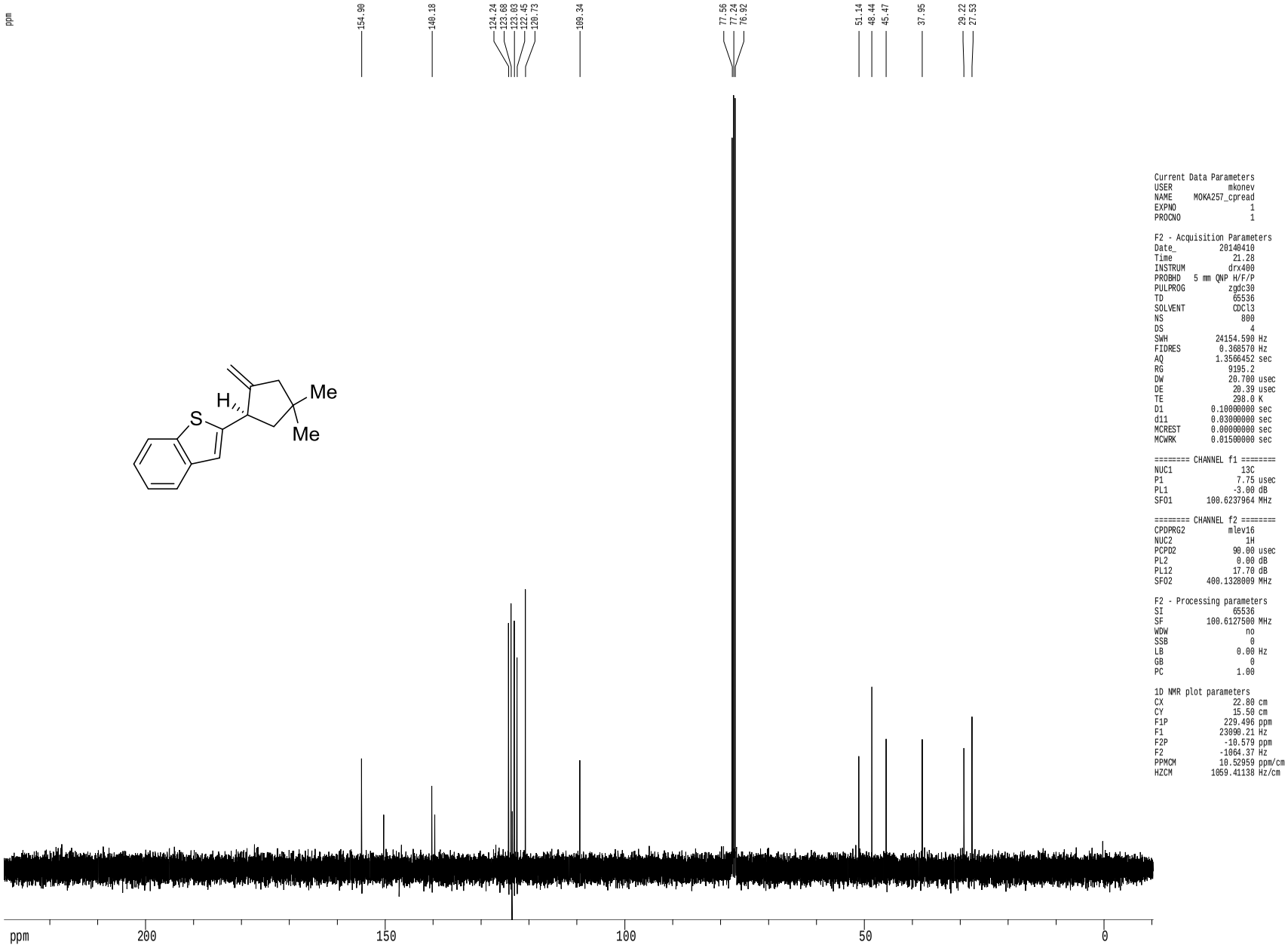


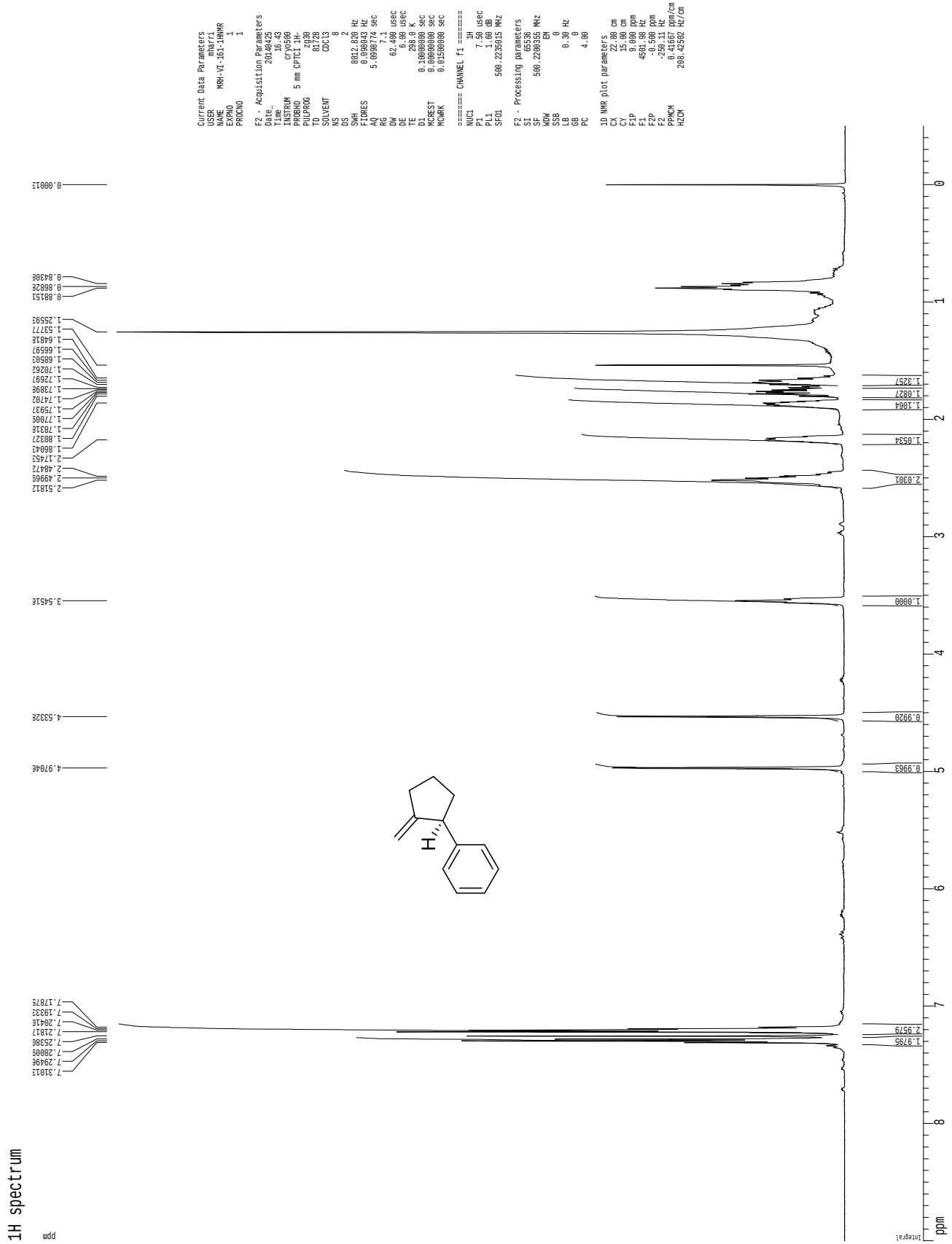

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-6-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131210
Time 18.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 82728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200341 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0001
1.2552
1.5389
1.6172
1.6346
1.6508
1.6588
1.6741
1.6894
1.7057
1.8087
1.8201
1.8322
1.8446
1.8556
1.8706
1.8882
1.8956
1.9123
1.9266
1.9437
2.0532
2.0668
2.0768
2.0908
2.1008
2.1145
2.4295
2.4428
2.4562
3.6564
3.6724
3.6888
4.8311
4.9956
6.0475
6.0527
6.2907
7.2532
7.3321
1.0352
2.0020
1.0839
2.0040
1.0000
1.0017
1.0031
1.0000
1.0001
1.0001
Integral
ppm
0
1
2
3
4
5
6
7
8
ppm
H
O


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
24.86
29.85
32.91
32.92
43.94
76.90
77.16
77.41
105.04
107.37
110.08
141.34
153.25
157.68
0
50
100
150
200
H
O

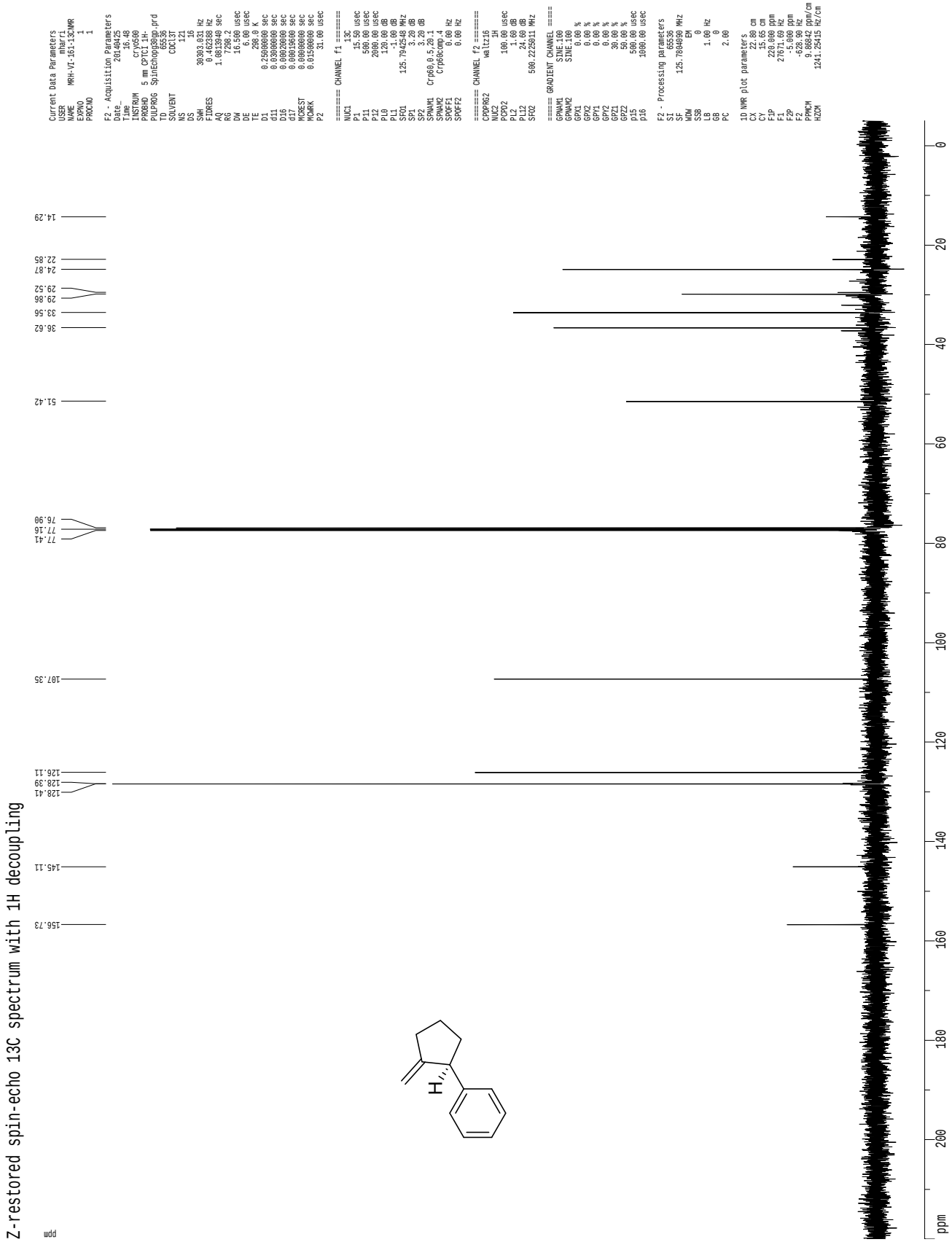

1H spectrum
Me
Me
H
S
ppm
Integral

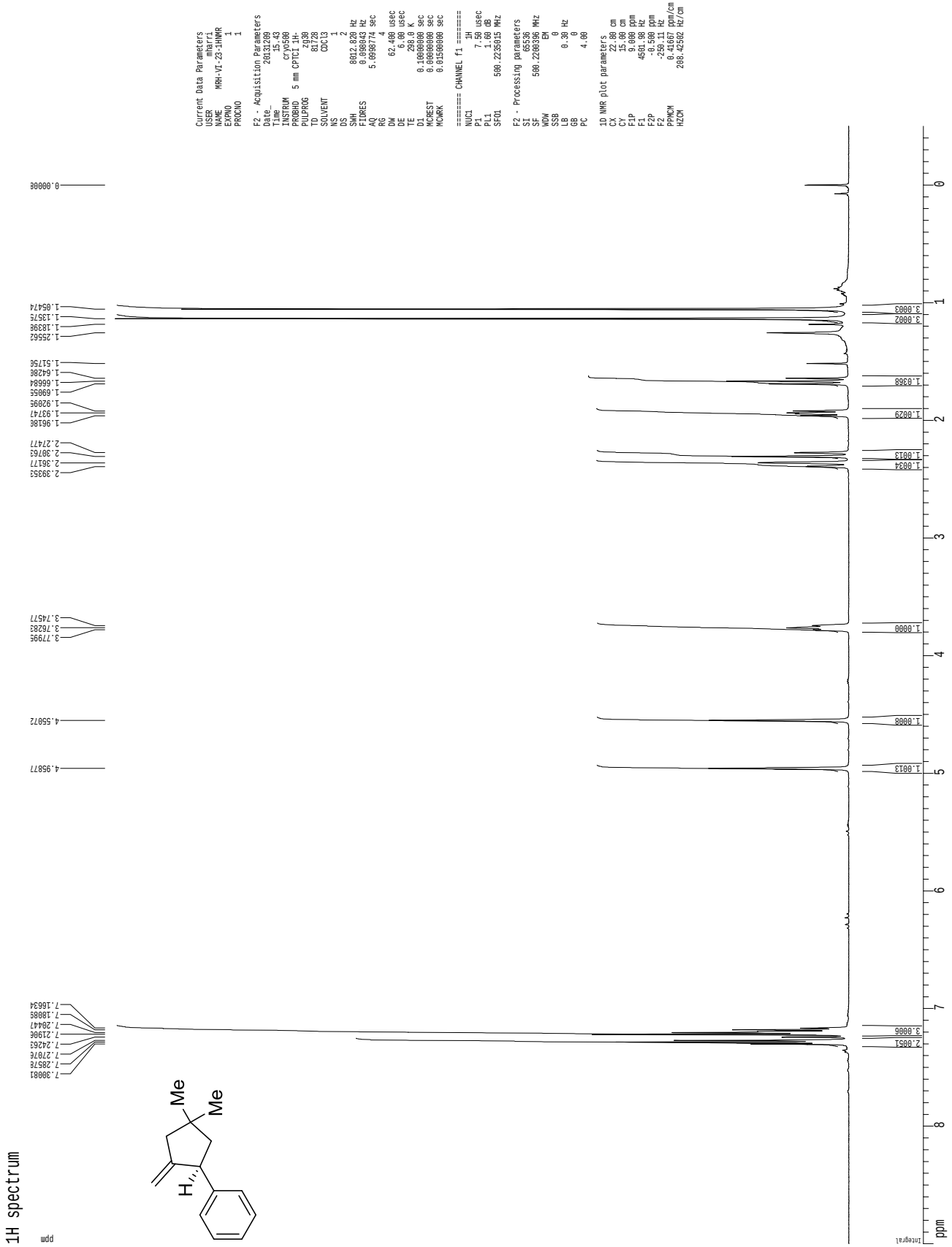
13C spectrum with 1H decoupling



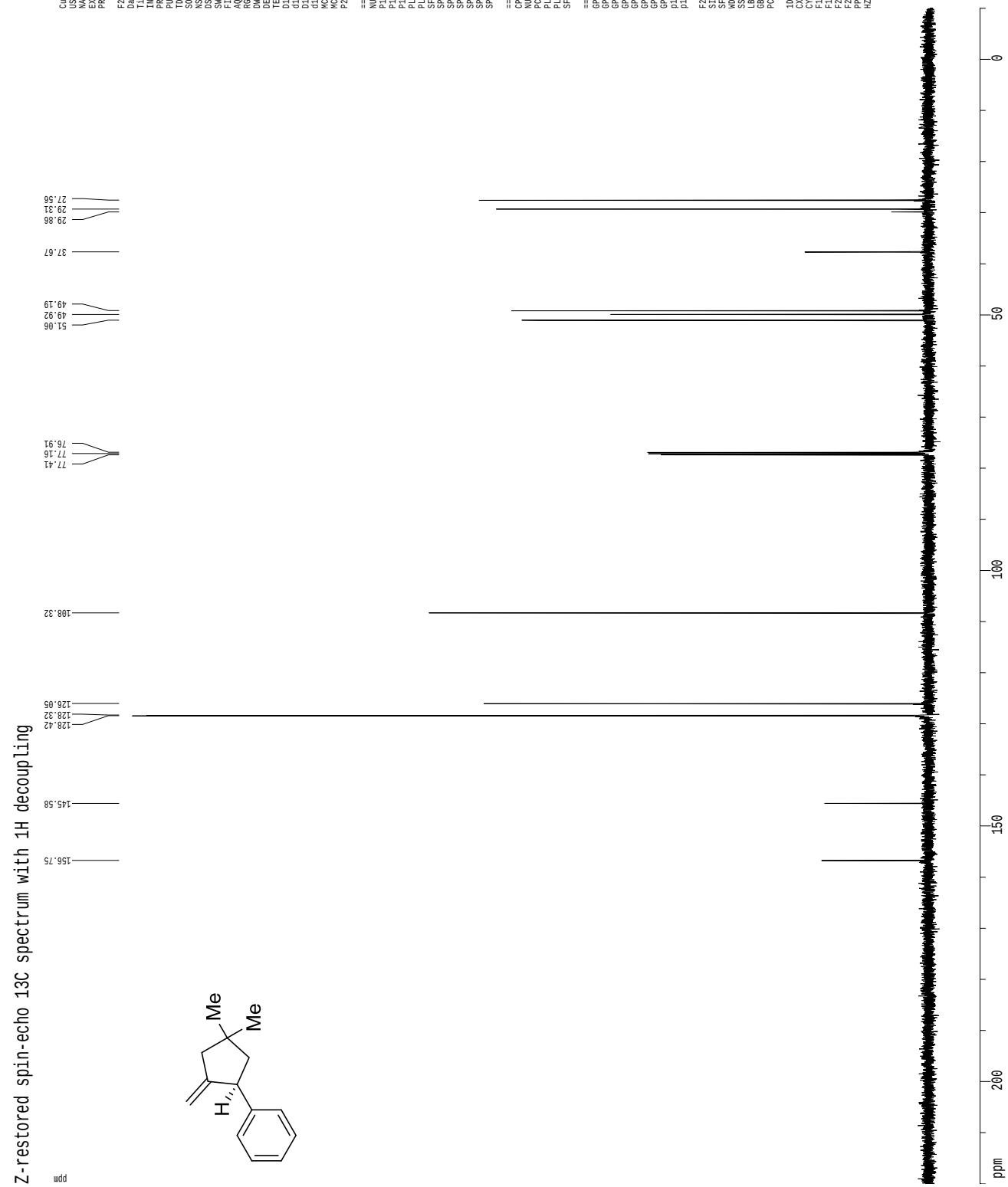

Current Data Parameters
USER mharri
NAME MRH-VI-161-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140425
Time 16.43
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200355 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.460 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00013
0.84308
0.86828
0.88154
1.25592
1.53777
1.64818
1.66597
1.68585
1.70262
1.72697
1.73898
1.74702
1.75937
1.77009
1.78318
1.80327
1.86043
2.17453
2.48472
2.49969
2.51812
3.54516
4.53328
4.97046
7.17875
7.19333
7.20416
7.21817
7.25386
7.28005
7.29496
7.31013
1.3257
1.0827
1.1064
1.0534
2.0301
1.0000
0.9920
0.9963
1.9795
2.9579
Integral
ppm
1H spectrum
ppm

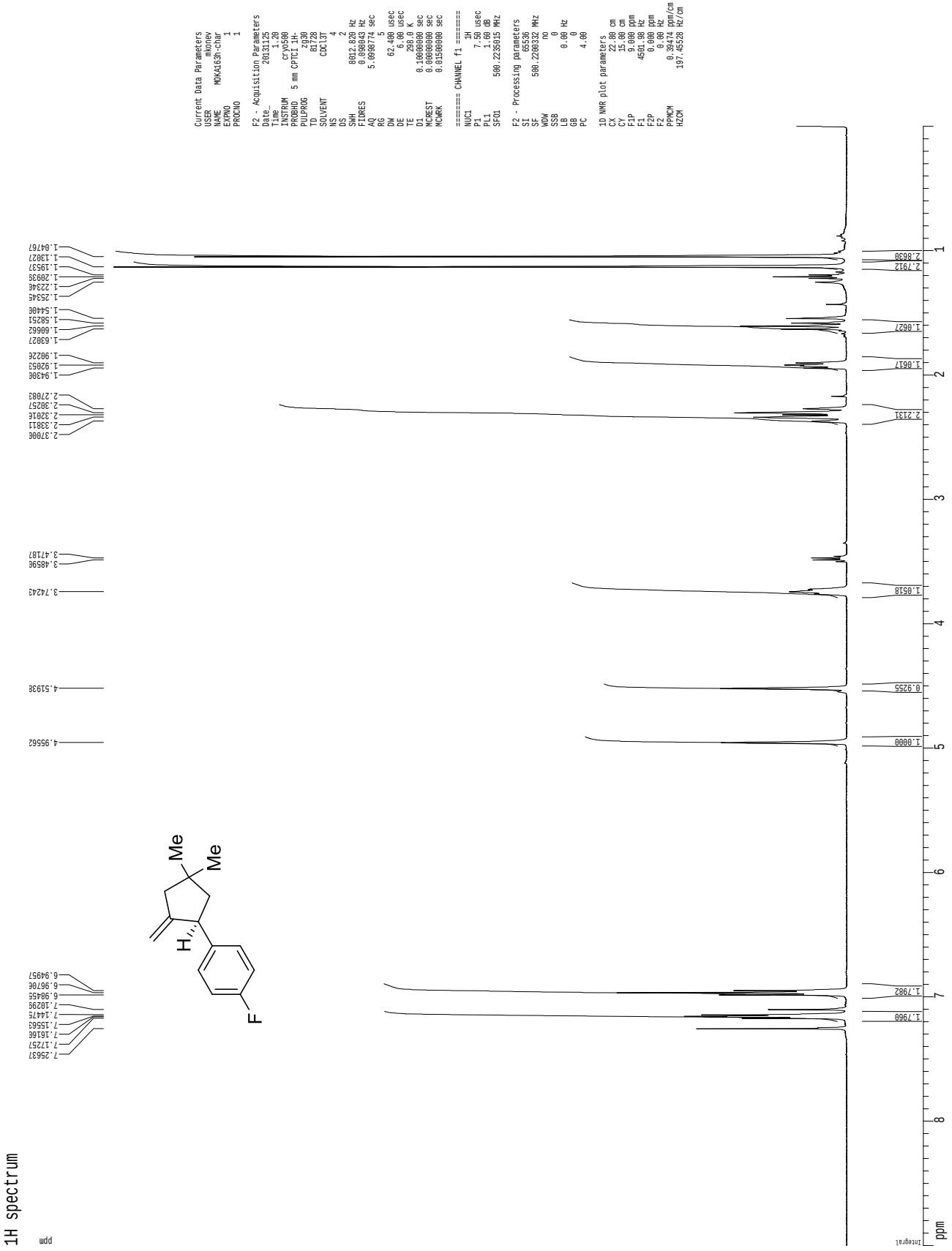
Z-restored spin-echo 13C spectrum with 1H decoupling



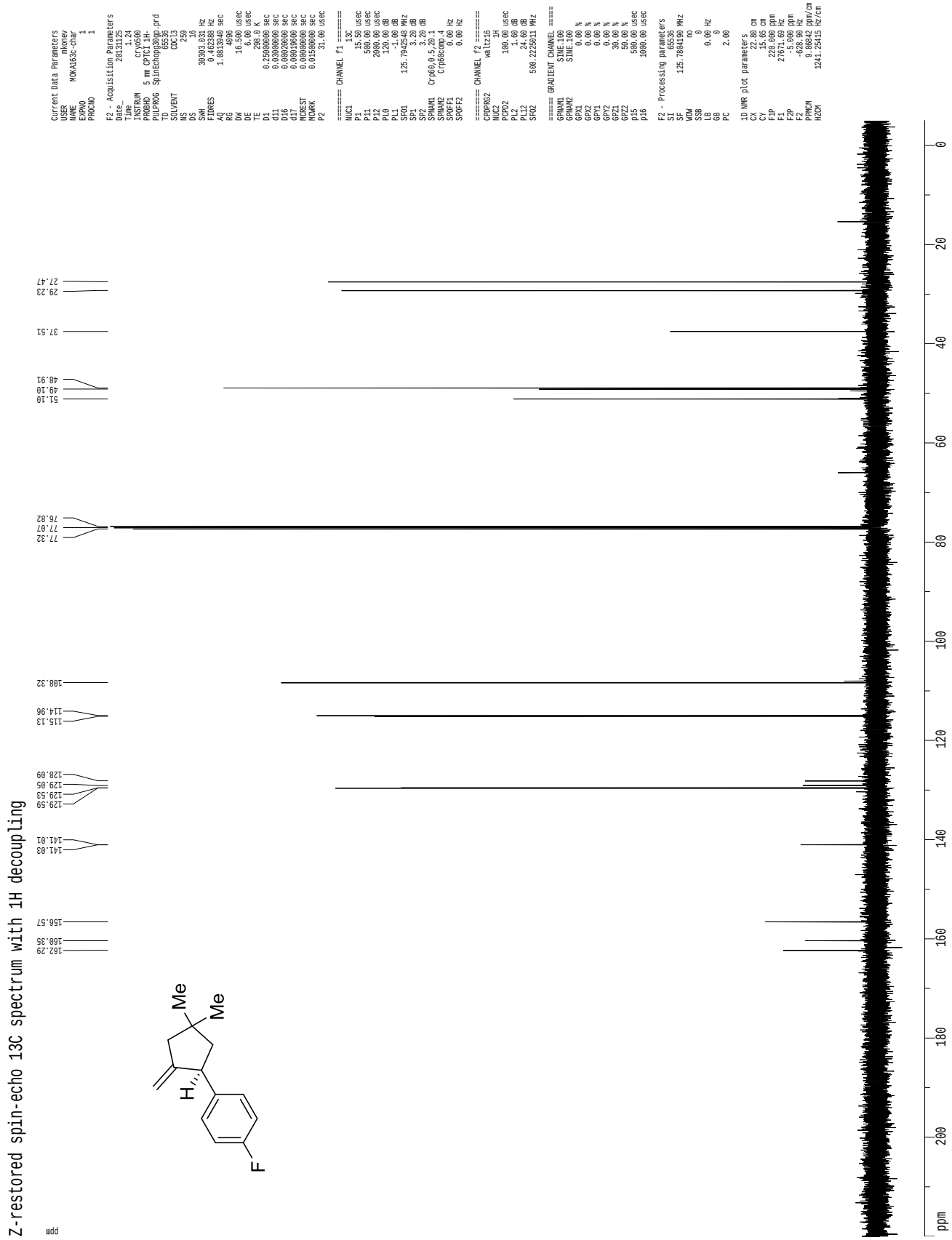

1H spectrum
Me
Me
H
ppm
Integral

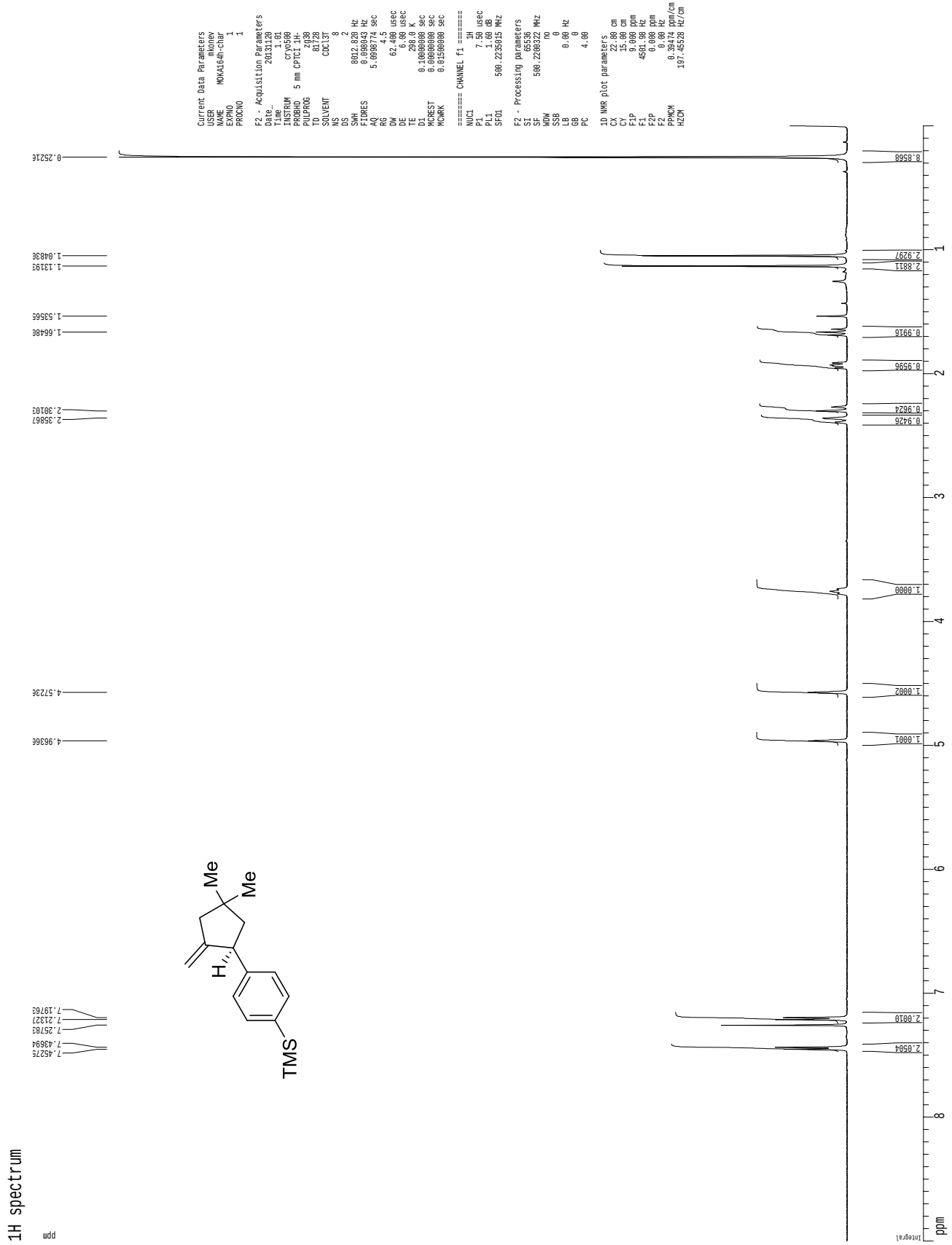
Z-restored spin-echo 13C spectrum with 1H decoupling

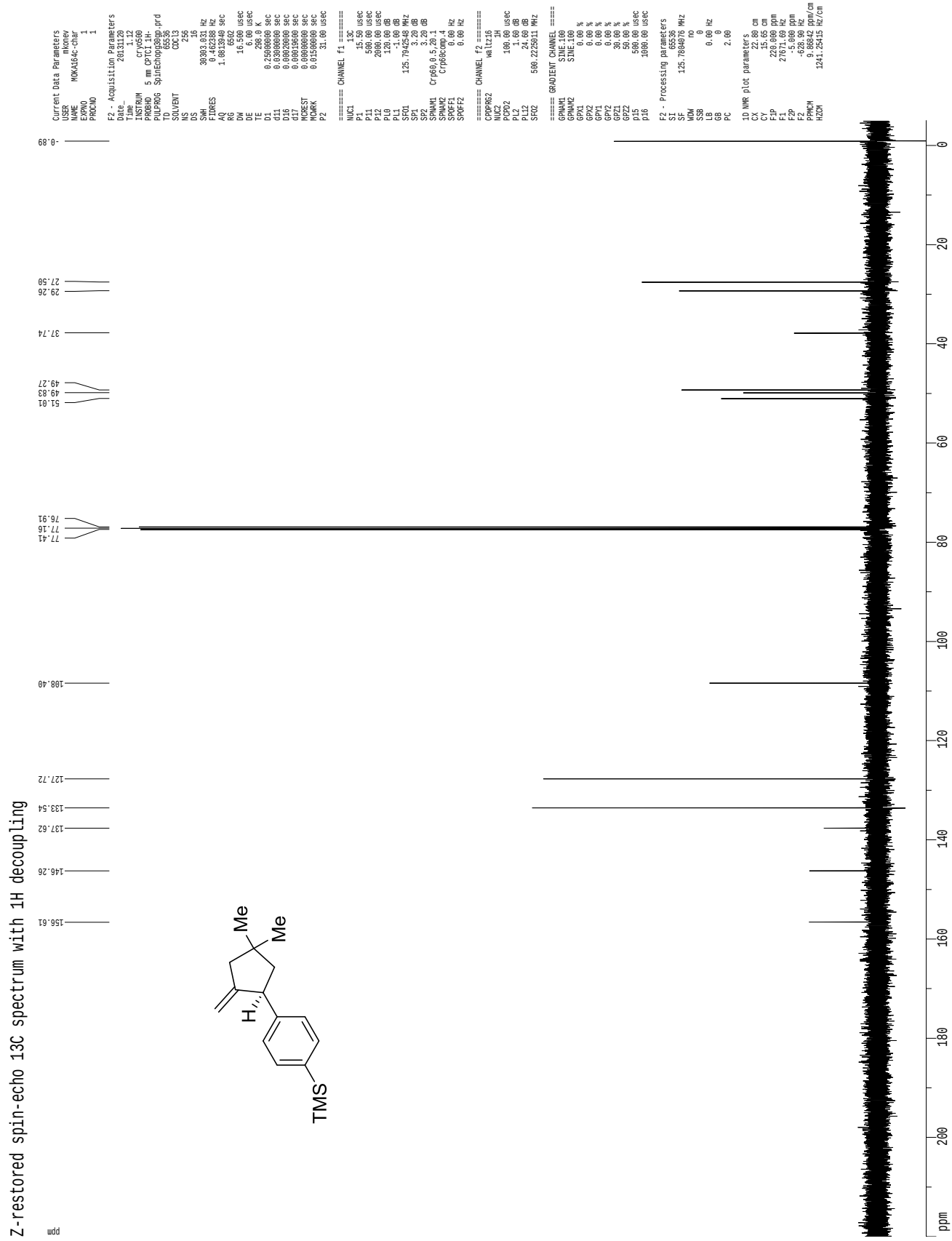


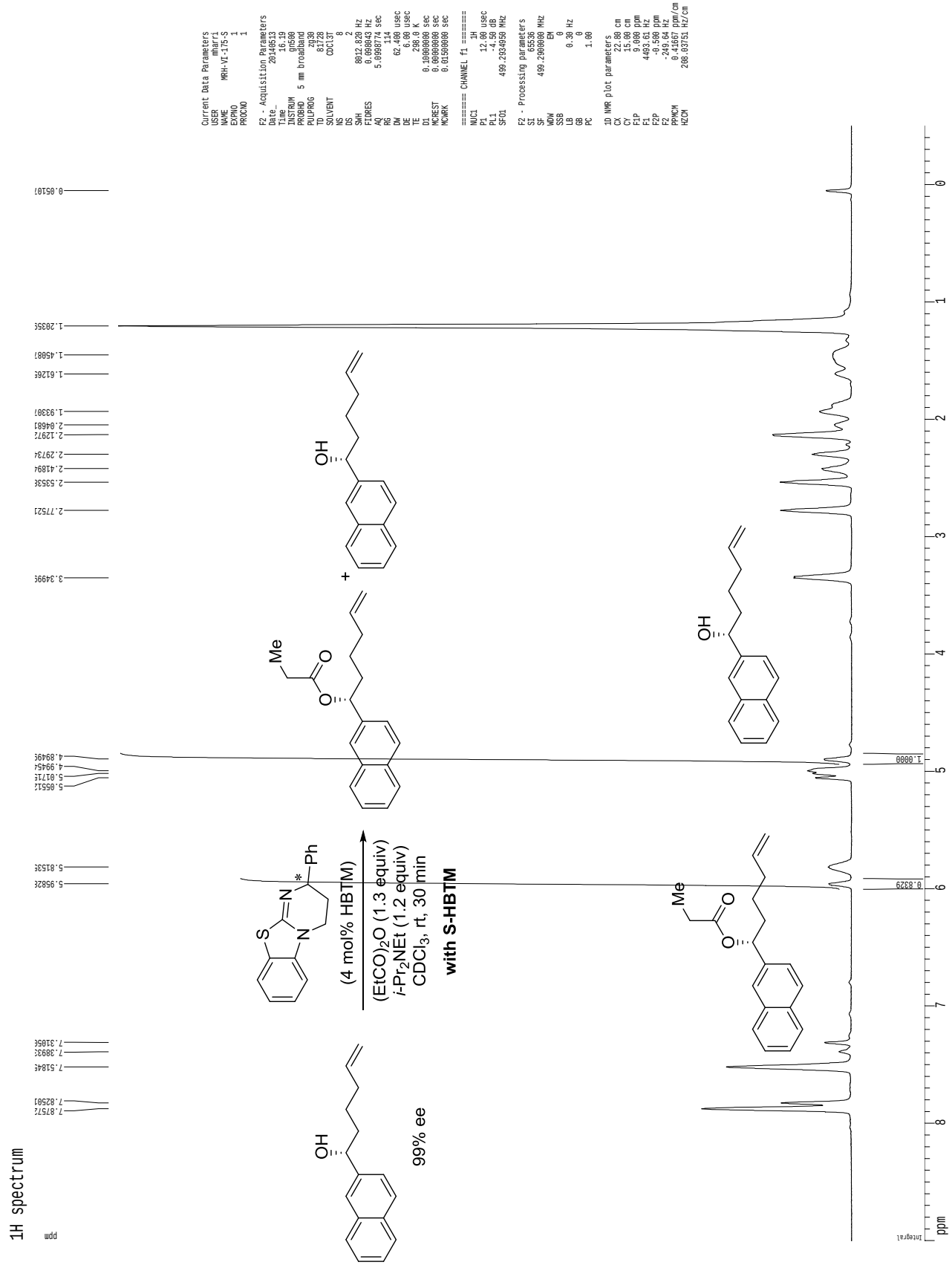

Current Data Parameters
USER mkonev
NAME MOKA163h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131125
Time 1.20
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200332 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
1.04767
1.13027
1.19537
1.20939
1.22346
1.25345
1.54406
1.58251
1.60662
1.63027
1.90226
1.92053
1.94306
2.27083
2.30257
2.32016
2.38311
2.37006
3.47187
3.48596
3.74243
4.51938
4.95562
7.25637
7.17257
7.16166
7.15563
7.14475
7.10299
6.98455
6.96706
6.94957
2.8630
2.7912
1.0627
1.0617
2.2131
1.0518
0.9255
1.0000
1.7982
1.7960
Me
Me
H
F
1
2
3
4
5
6
7
8
ppm
Integral
1H spectrum
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

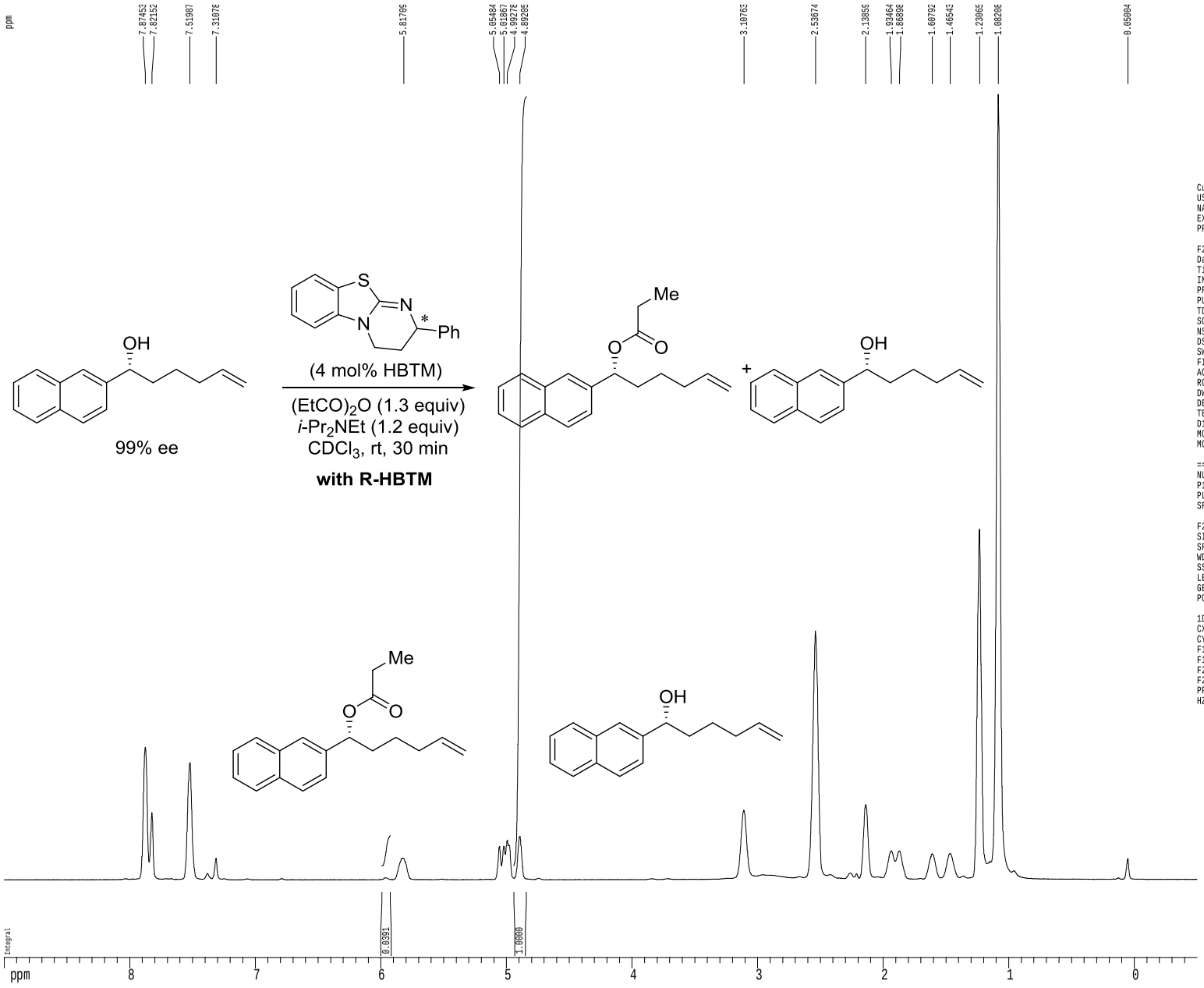



Me
Me
H
TMS
1H spectrum
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Me
Me
H
TMS


1H spectrum
ppm
Integral
Current Data Parameters
USER mharri
NAME MRH-VI-175-S
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140513
Time 13.13
INSTRUM d500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098843 Hz
AQ 5.0896774 sec
RG 144
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -4.50 dB
SFO1 499.2934950 MHz
F2 - Processing parameters
SI 65536
SF 499.2900000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 4003.65 Hz
F2P -0.560 ppm
F2 -249.64 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.03751 Hz/cm
0.05107
1.20359
1.45087
1.61266
1.93307
2.04681
2.12972
2.29734
2.41894
2.53536
2.77521
3.34995
4.89498
4.99454
5.01718
5.05512
5.81536
5.95826
7.31056
7.38932
7.51844
7.82501
7.87572
1.0000
0.8329
(4 mol% HBTM)
(EtCO)2O (1.3 equiv)
i-Pr2NEt (1.2 equiv)
CDCl3, rt, 30 min
with S-HBTM
99% ee

1H spectrum

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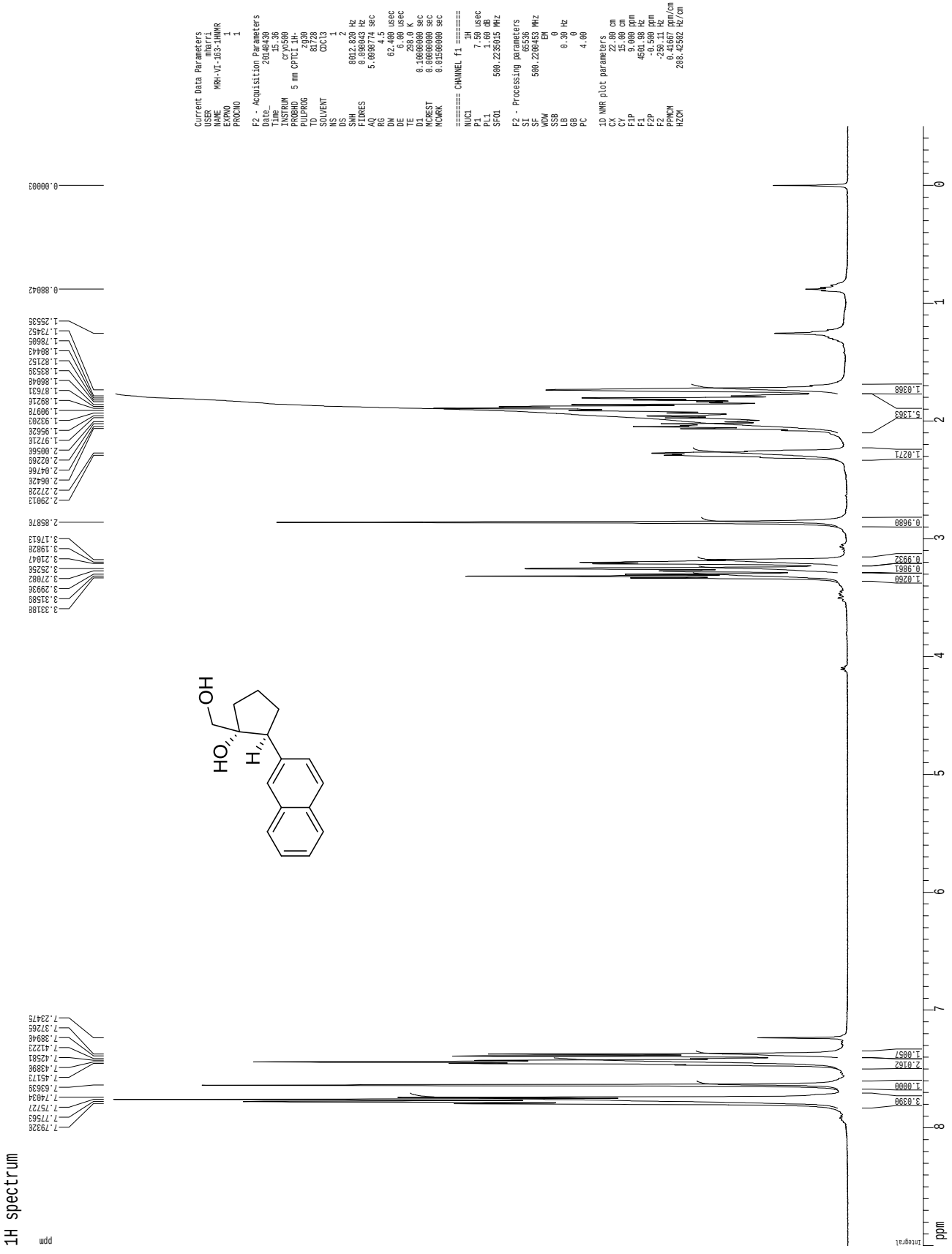
Current Data Parameters
USER                mharri
NAME          MRH-VI-175-R
EXPNO                   1
PROCNO                  1

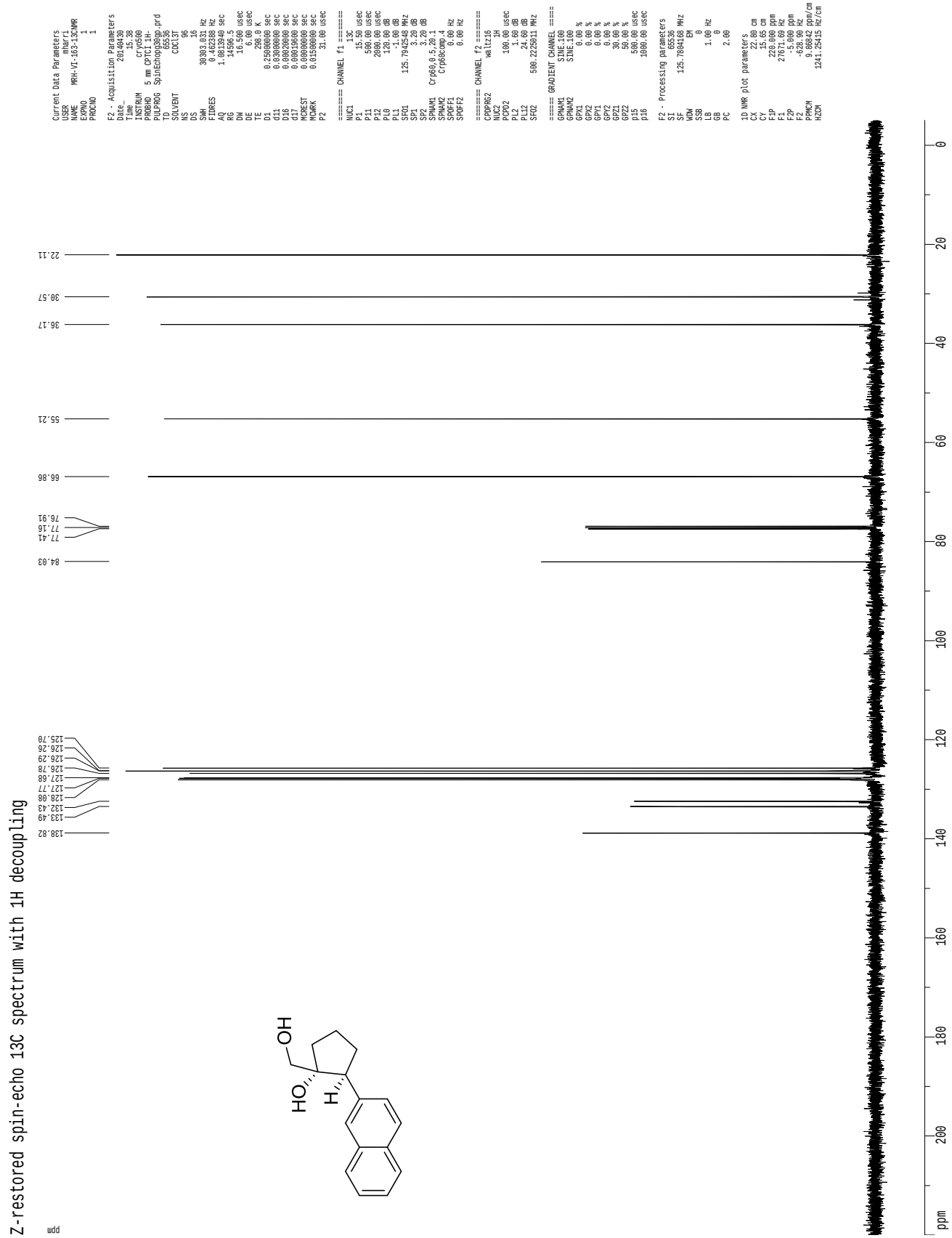
F2 - Acquisition Parameters
Date_            20140513
Time                16.22
INSTRUM             gn500
PROBHD   5 mm broadband
PULPROG              zg30
TD                  81728
SOLVENT           CDCl3T
NS                      8
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                   71.8
DW                 62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

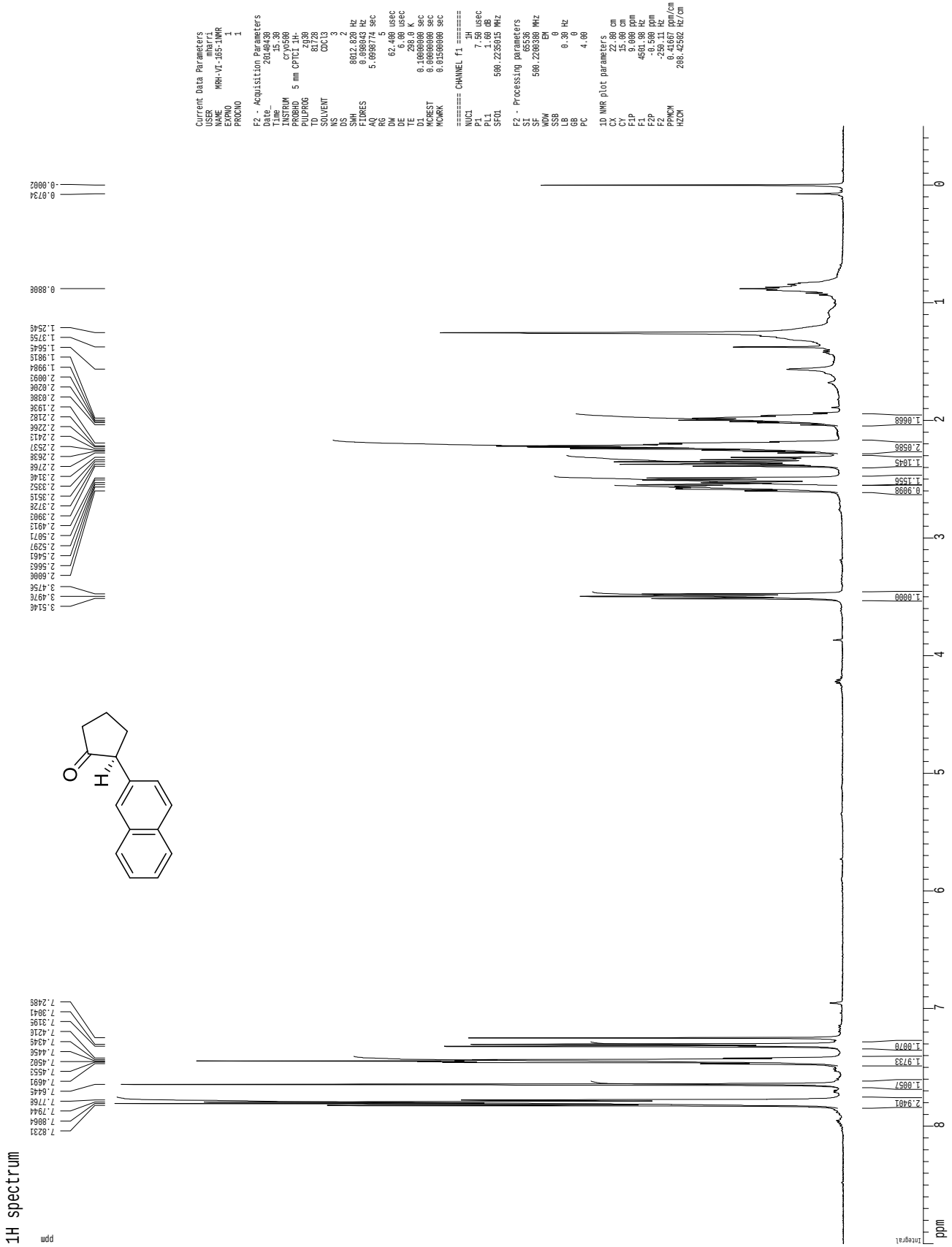
======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -4.50 dB
SFO1           499.2934950 MHz

F2 - Processing parameters
SI                    65536
SF             499.2900000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

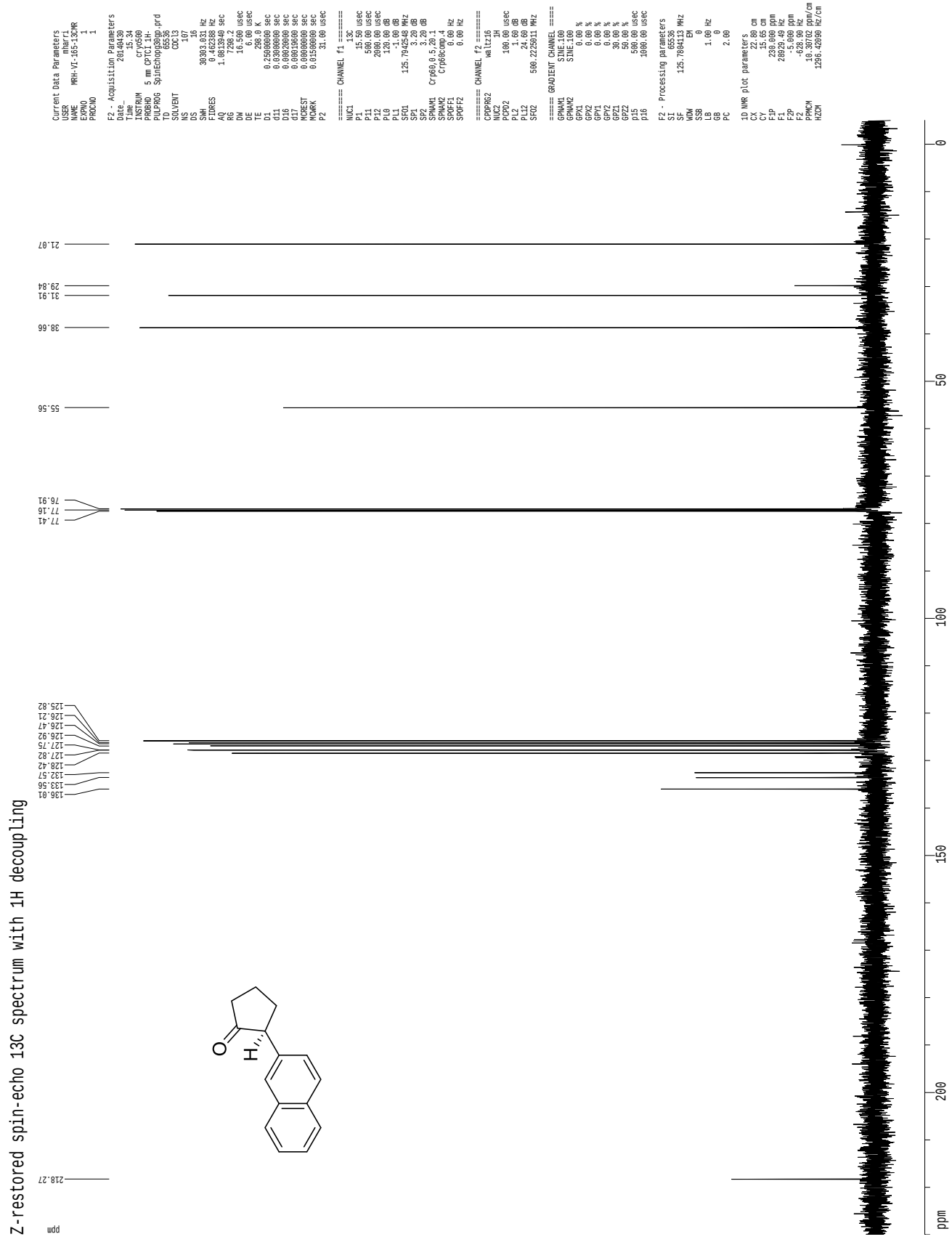
1D NMR plot parameters
CX                    22.80 cm
CY                    15.00 cm
F1P                    9.000 ppm
F1                  4493.61 Hz
F2P                   -0.500 ppm
F2                  -249.64 Hz
PPMCM              0.41667 ppm/cm
HZCM             208.03751 Hz/cm
```

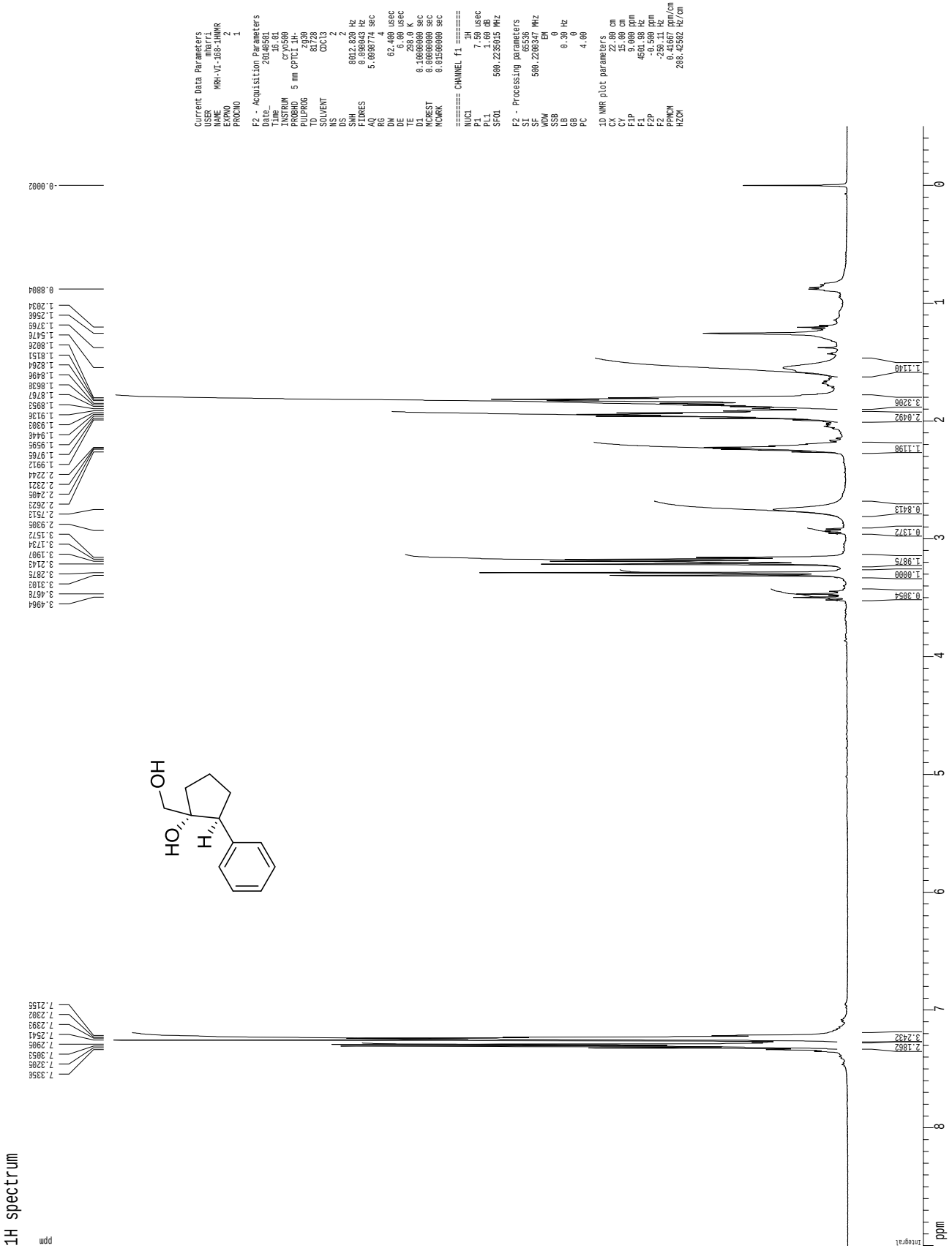

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-163-1HNMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140430
Time 15:36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200453 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -298.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
OH
HO
H
integral
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
wdd
ppm
22.11
30.57
36.17
55.21
66.86
76.91
77.16
77.41
84.03
125.70
126.26
126.29
126.78
127.68
127.77
128.08
132.43
133.49
138.82
OH
HO
H

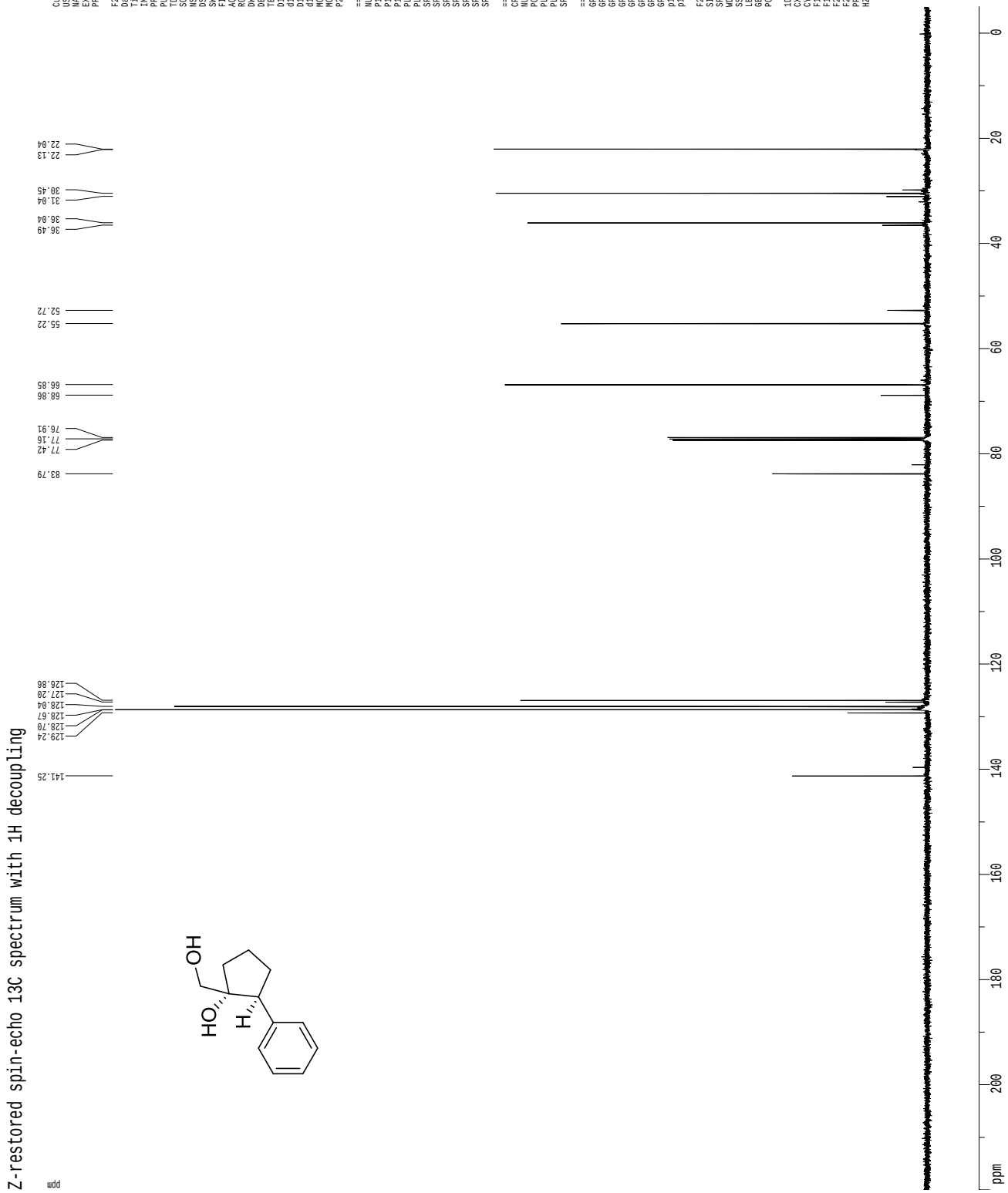

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-165-1NMR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140430
Time 15.30
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200388 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -299.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0734
0.0002
0.8808
1.2548
1.3759
1.5645
1.9815
1.9984
2.0093
2.0206
2.0386
2.1936
2.2182
2.2266
2.2413
2.2537
2.2638
2.2768
2.3146
2.3352
2.3519
2.3728
2.3903
2.4913
2.5071
2.5297
2.5461
2.5663
2.6006
3.4756
3.4976
3.5146
7.2485
7.3041
7.3195
7.4216
7.4348
7.4456
7.4502
7.4553
7.4691
7.6445
7.7766
7.7944
7.8064
7.8233
1.0668
2.0586
1.1845
1.1556
0.9098
1.0000
1.0070
1.9733
1.0057
2.9401
integral
ppm
ppm

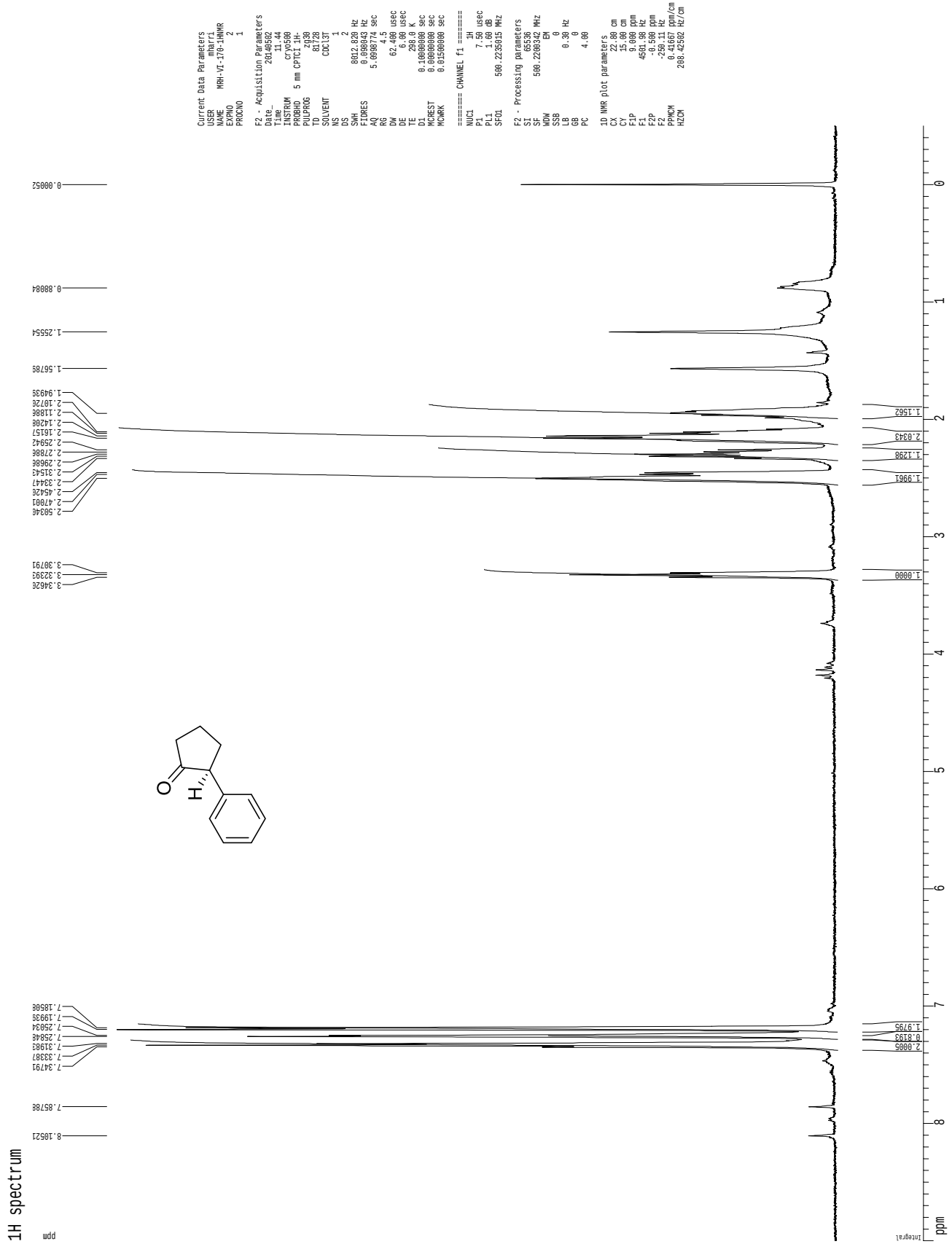
Z-restored spin-echo 13C spectrum with 1H decoupling



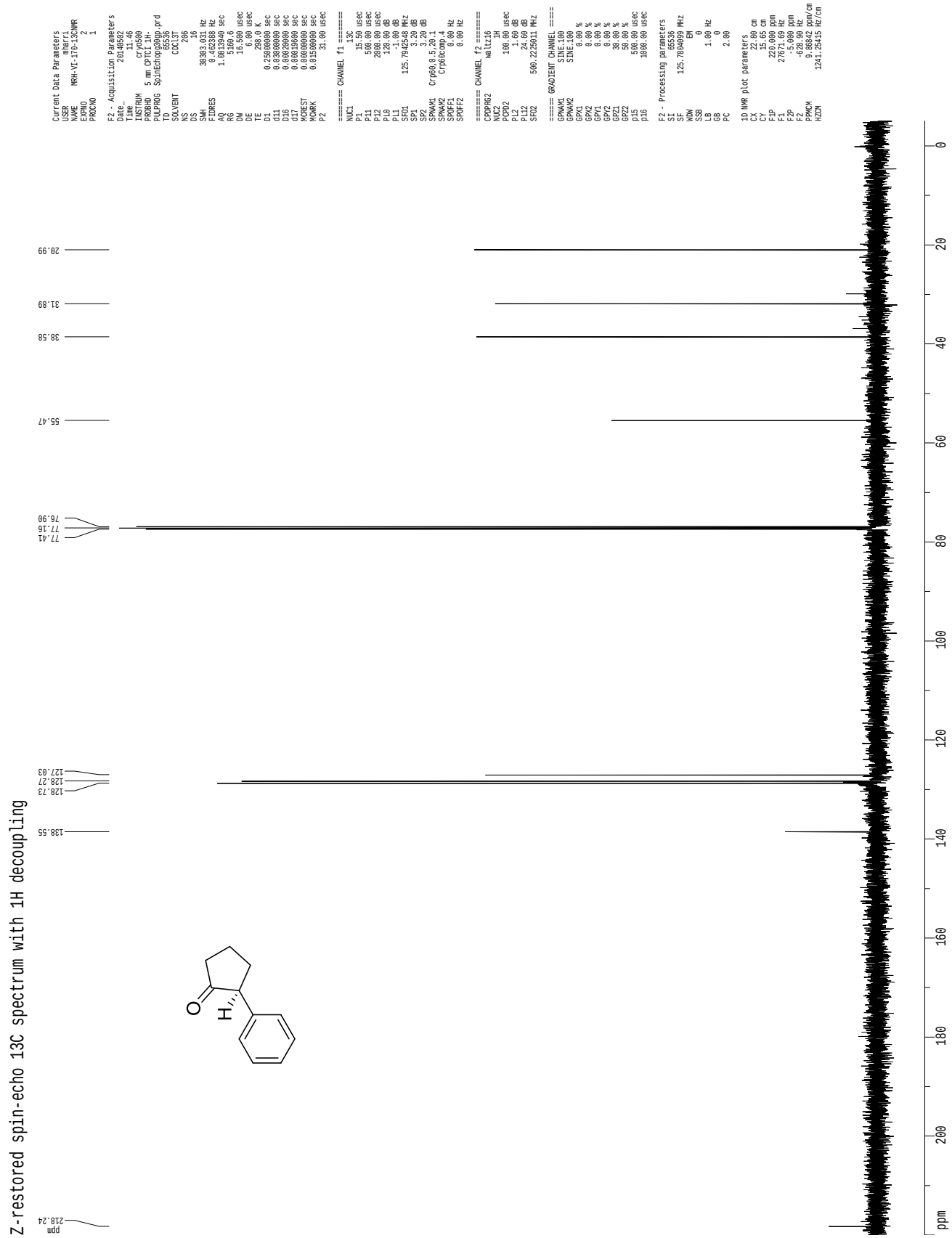

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-168-1HNMR
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140501
Time 16.01
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200347 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.598 ppm
F2 -298.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0002
0.8804
1.2034
1.2566
1.3765
1.5476
1.8026
1.8151
1.8264
1.8496
1.8638
1.8767
1.8952
1.9136
1.9303
1.9446
1.9596
1.9766
1.9912
2.2244
2.2321
2.2405
2.2623
2.7512
2.9906
3.1572
3.1734
3.1907
3.2143
3.2875
3.3103
3.4678
3.4964
7.3356
7.3526
7.3053
7.2906
7.2641
7.2393
7.2302
7.2155
1.1140
3.3206
2.0492
1.1198
0.8413
0.1372
1.9875
1.0000
0.3854
3.2432
2.1862
ppm
Integral
OH
HO
H

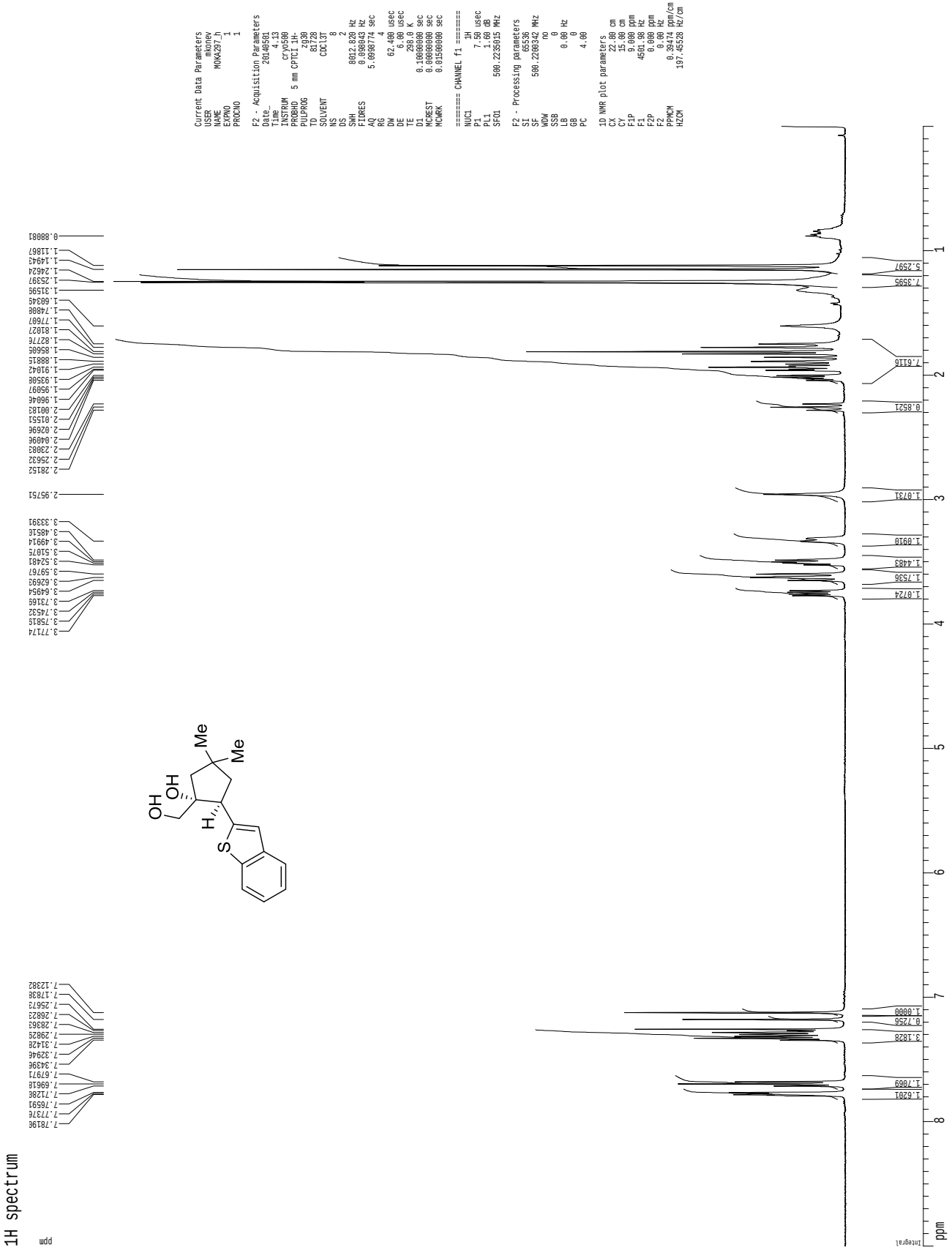
Z-restored spin-echo 13C spectrum with 1H decoupling



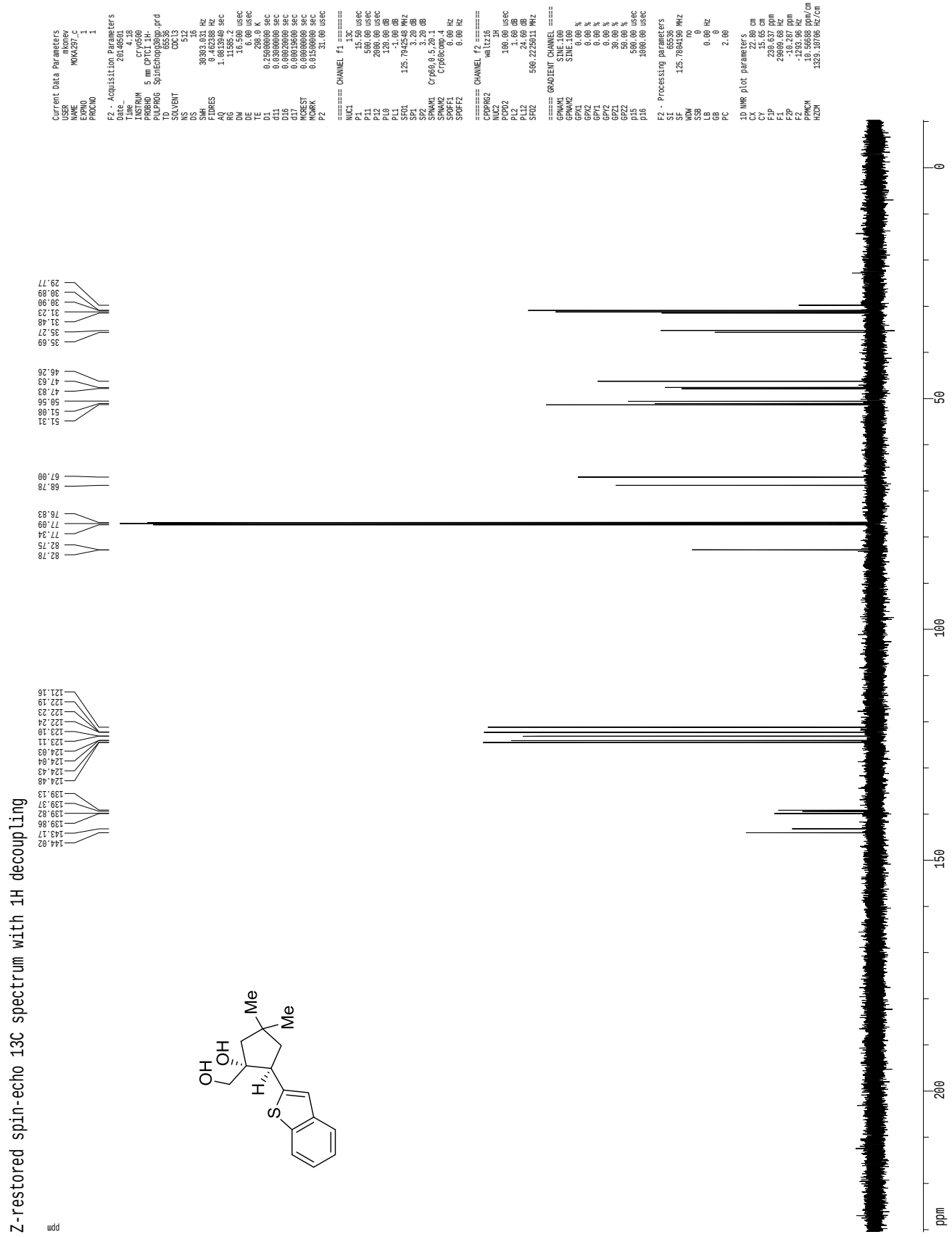

1H spectrum
Current Data Parameters
USER mharri
NAME MRH-VI-170-1HNMR
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140502
Time 11.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200342 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.698 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00052
0.88084
1.25554
1.56785
1.94935
2.10726
2.11886
2.14208
2.16157
2.25942
2.27886
2.29686
2.31543
2.33447
2.48426
2.47901
2.50346
3.30791
3.32293
3.34626
7.18508
7.19935
7.25034
7.25648
7.31983
7.33387
7.34791
7.85786
8.10521
1.1562
2.0343
1.1298
1.9961
1.0000
1.9795
0.8193
2.0005
integral
ppm
O
H

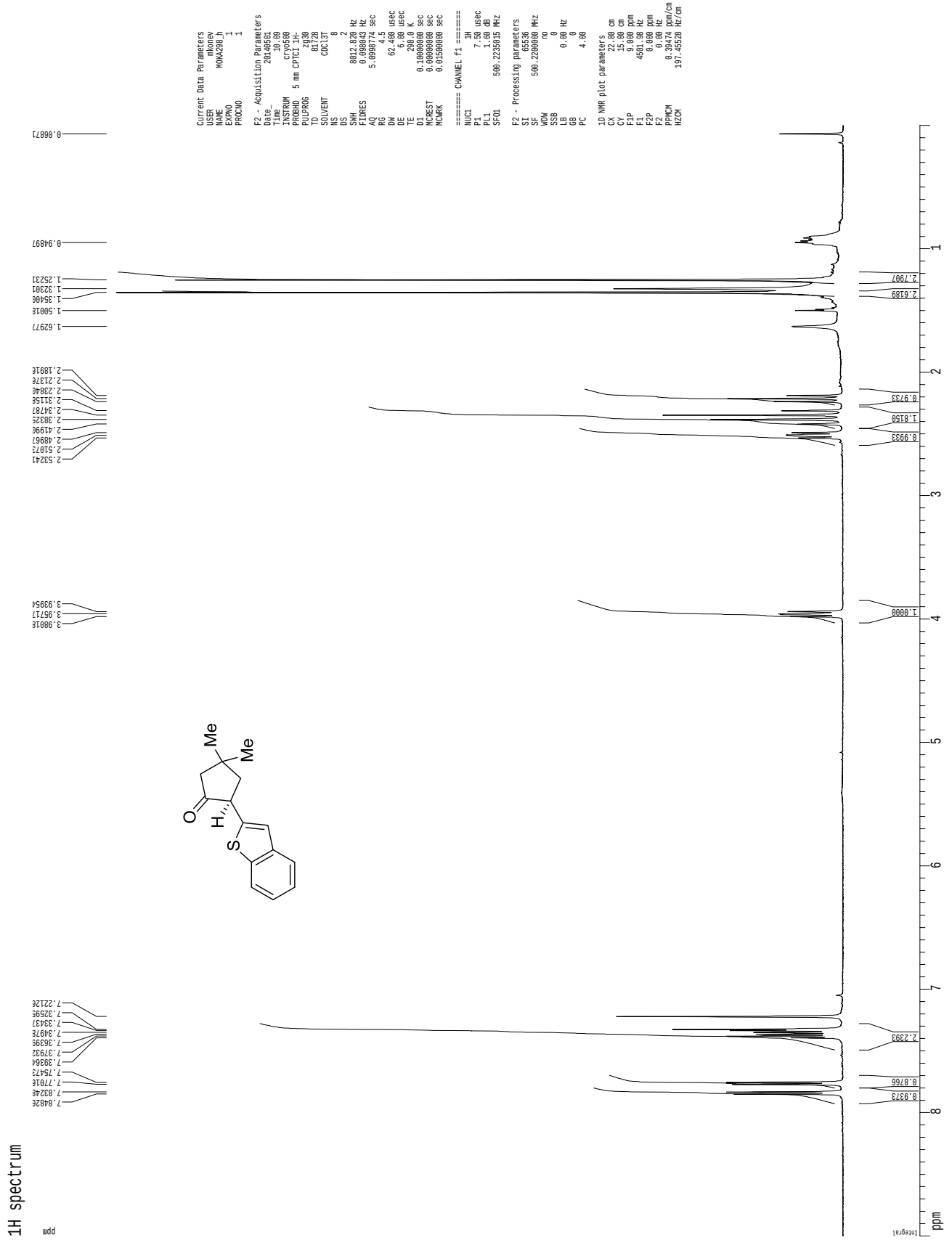
Z-restored spin-echo 13C spectrum with 1H decoupling

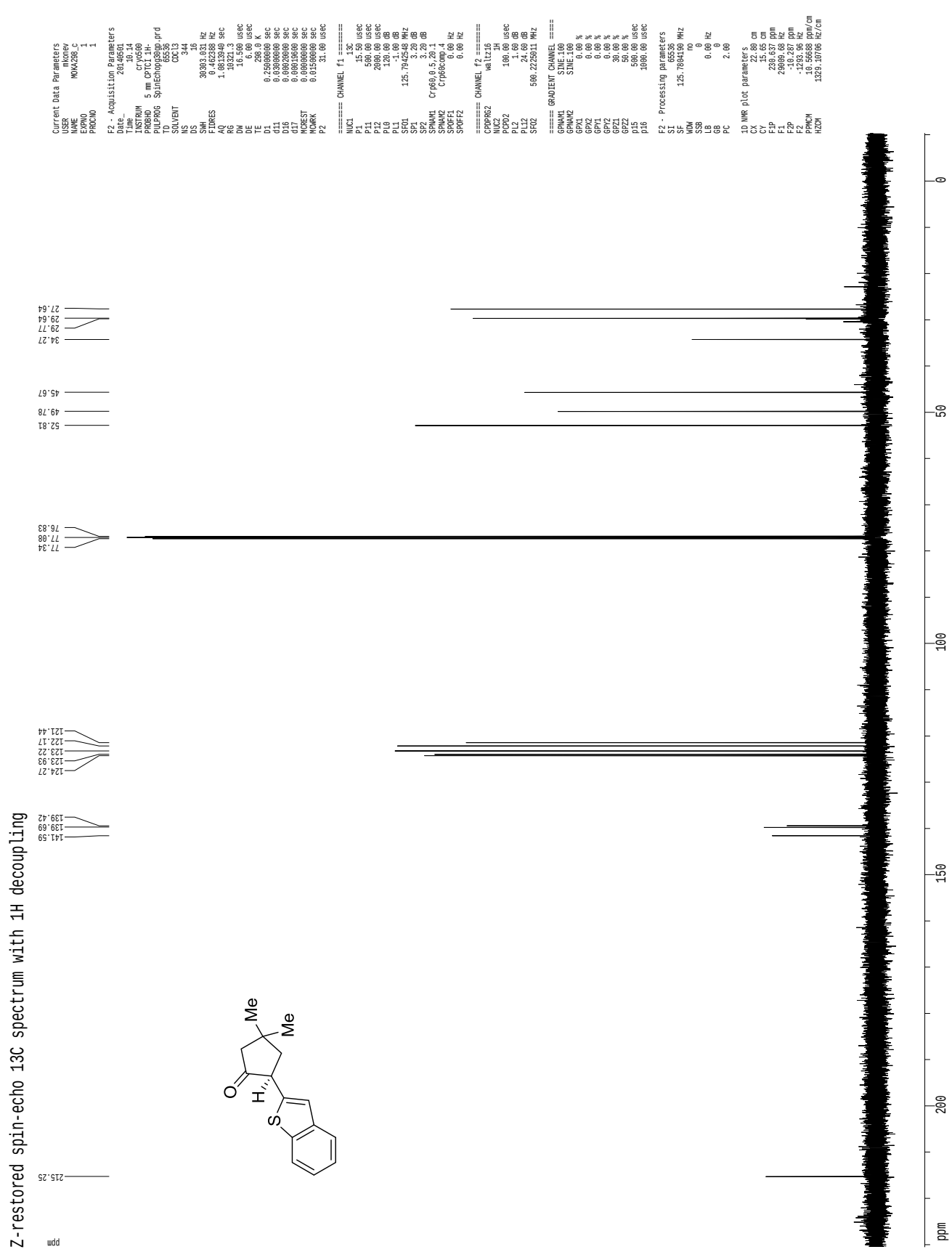


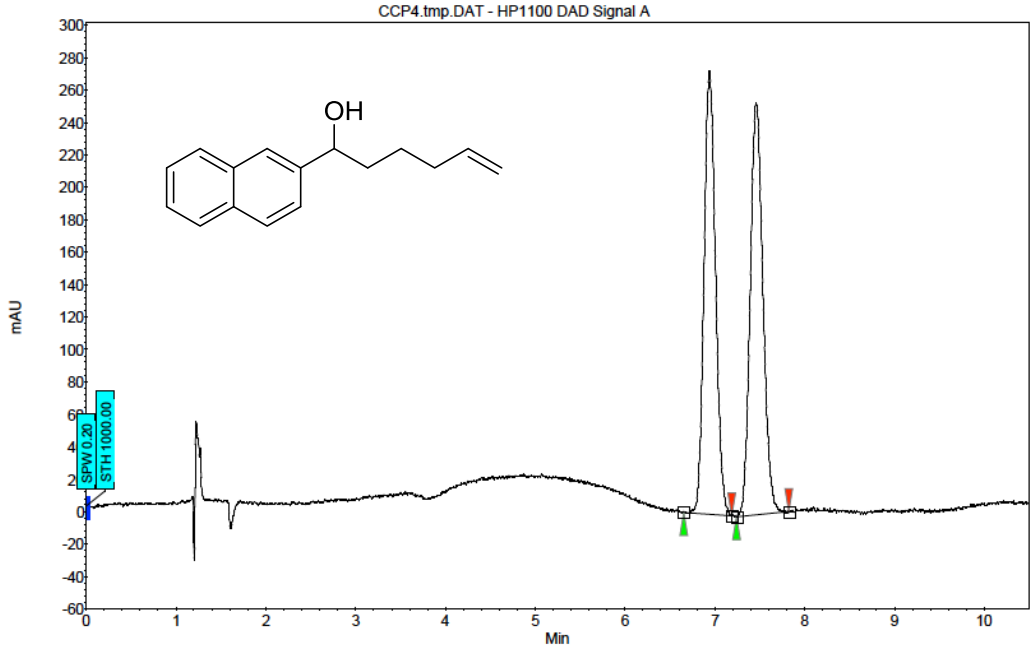

1H spectrum
Current Data Parameters
USER mkoney
NAME MOKA297_h
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140501
Time 4.13
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200342 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
Integral
ppm
Me
Me
OH
OH
H
S

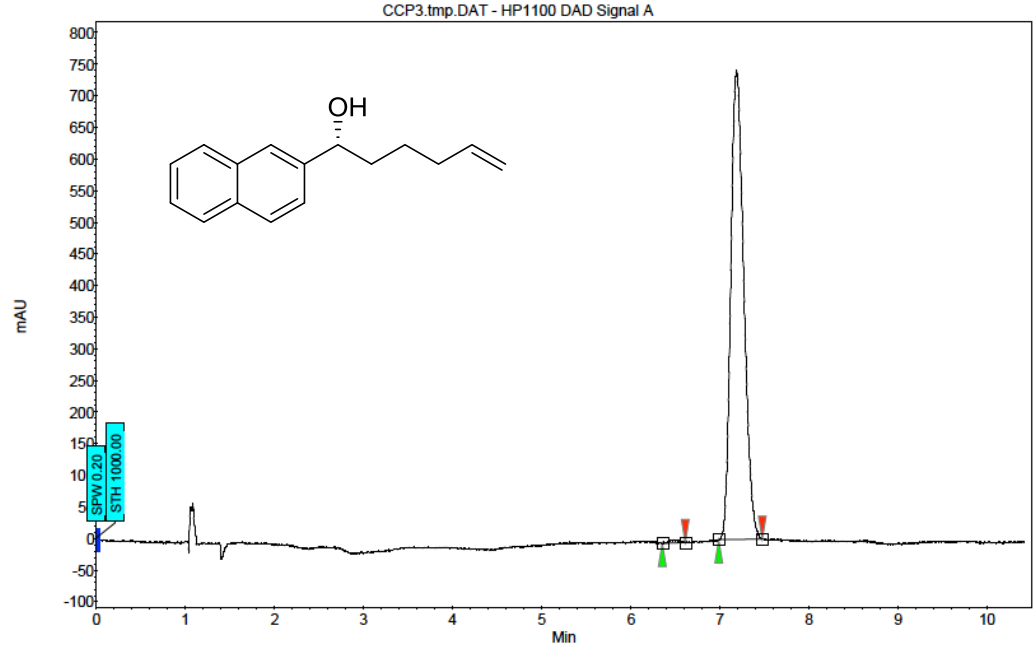
Z-restored spin-echo 13C spectrum with 1H decoupling



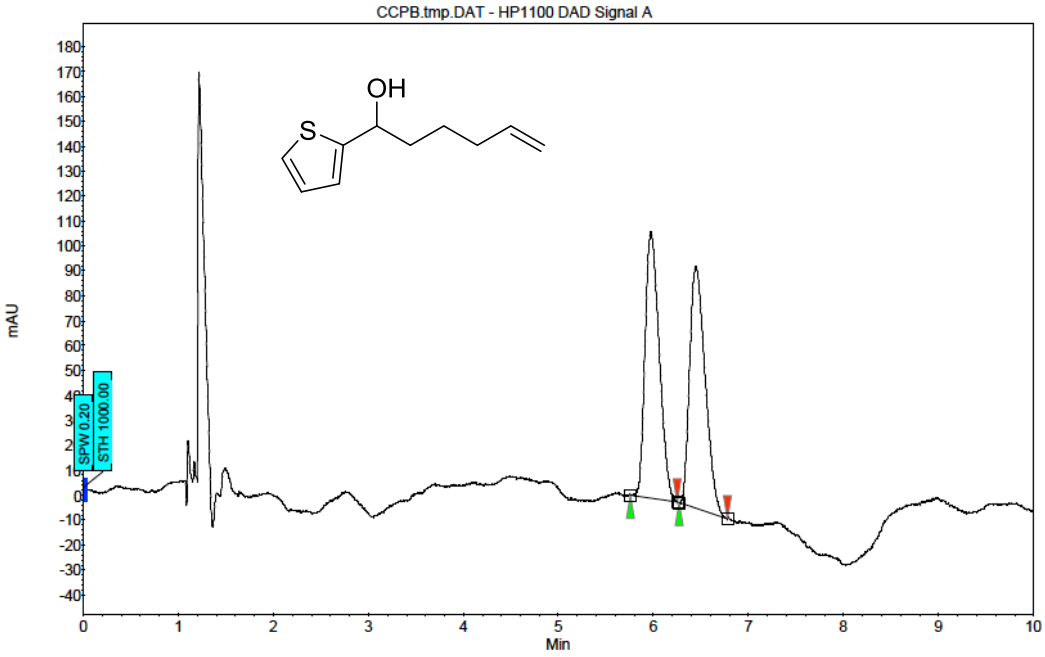

1H spectrum
ppm
Integral
Current Data Parameters
USER mkonev
NAME MOKA298_h
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140501
Time 10.09
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
0.06871
0.94897
1.25231
1.32301
1.35406
1.50018
1.62977
2.18916
2.21376
2.23846
2.31158
2.34787
2.38326
2.41996
2.48967
2.51073
2.53241
3.98018
3.99571
3.99354
7.22126
7.32596
7.33437
7.34978
7.36395
7.37932
7.39364
7.75473
7.77016
7.83248
7.84826
2.7987
2.6189
0.9733
1.8156
0.9933
1.0000
2.2393
0.8766
0.9373
Me
Me
O
H
S

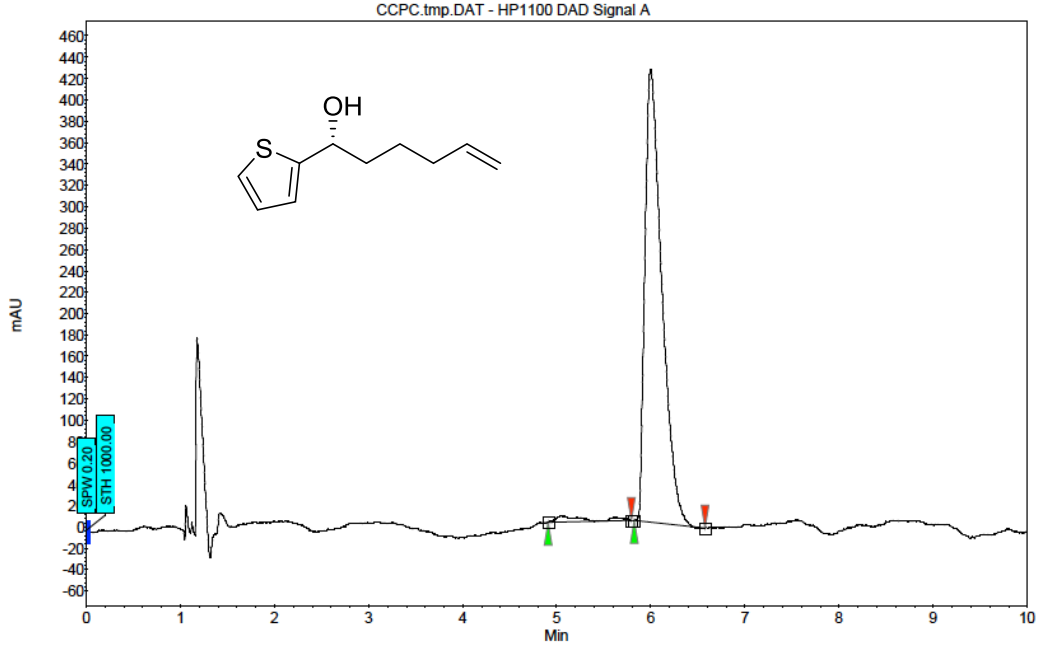

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
215.25
141.59
139.69
139.42
124.27
123.93
123.22
122.17
121.44
77.34
77.08
76.83
52.81
49.78
45.67
34.27
29.77
29.64
27.64
Me
Me
O
H
S
0
50
100
150
200
ppm



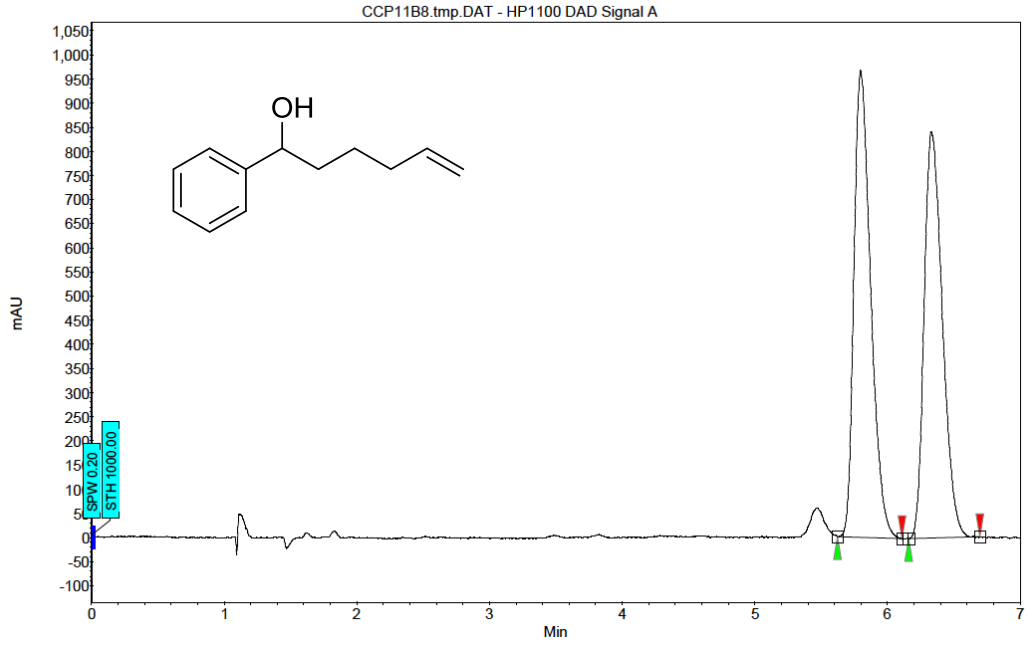


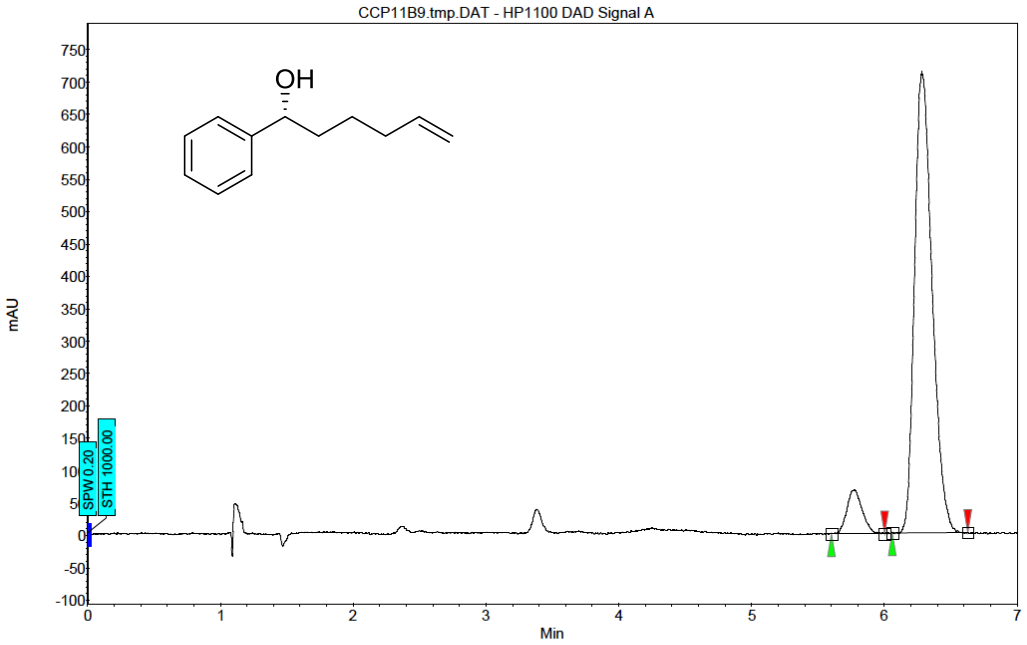
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	6.36	6.50	6.61	0.00	0.44	5.2	0.6	0.442
1	UNKNOWN	6.99	7.19	7.48	0.00	99.56	740.8	125.2	99.558
Total						100.00	746.0	125.8	100.000



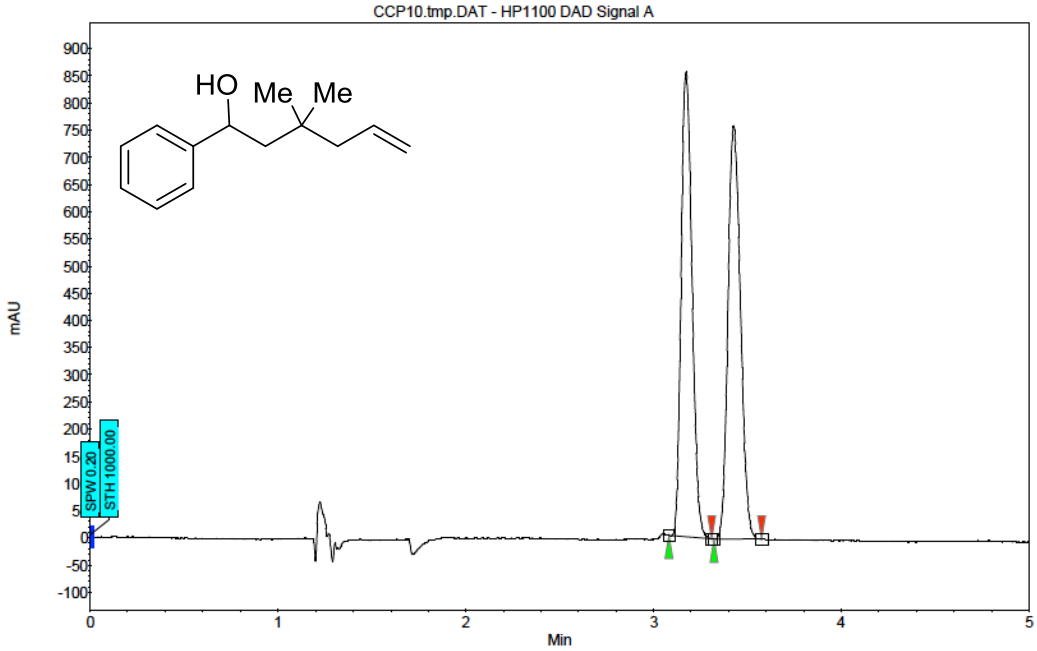


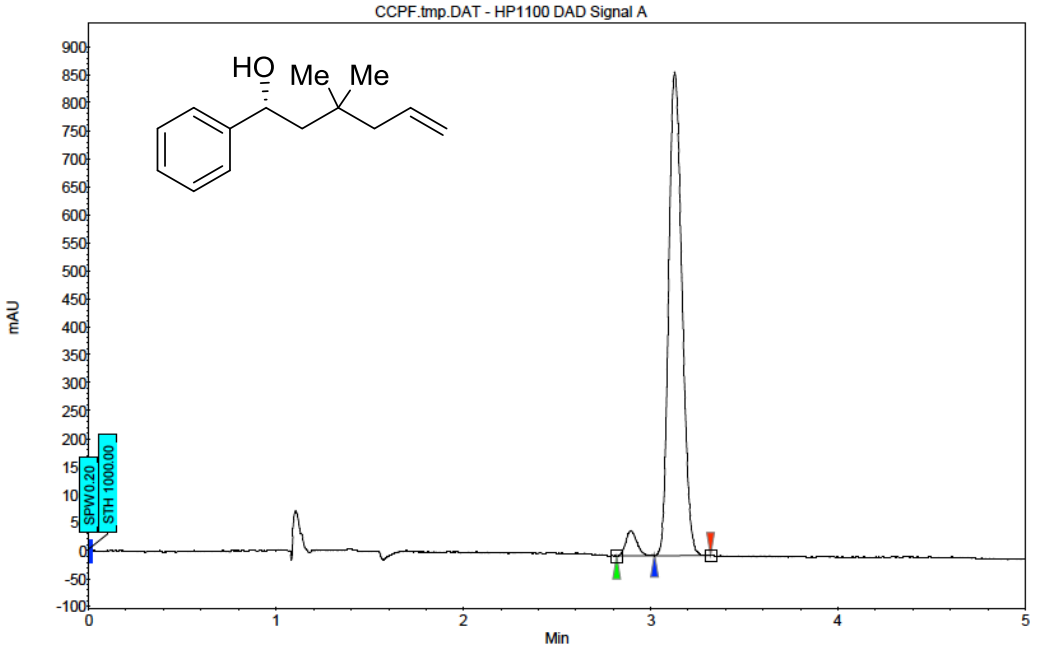
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	4.91	5.05	5.80	0.00	2.20	6.1	2.0	2.200
1	UNKNOWN	5.82	6.00	6.58	0.00	97.80	423.3	88.0	97.800
Total						100.00	429.3	90.0	100.000



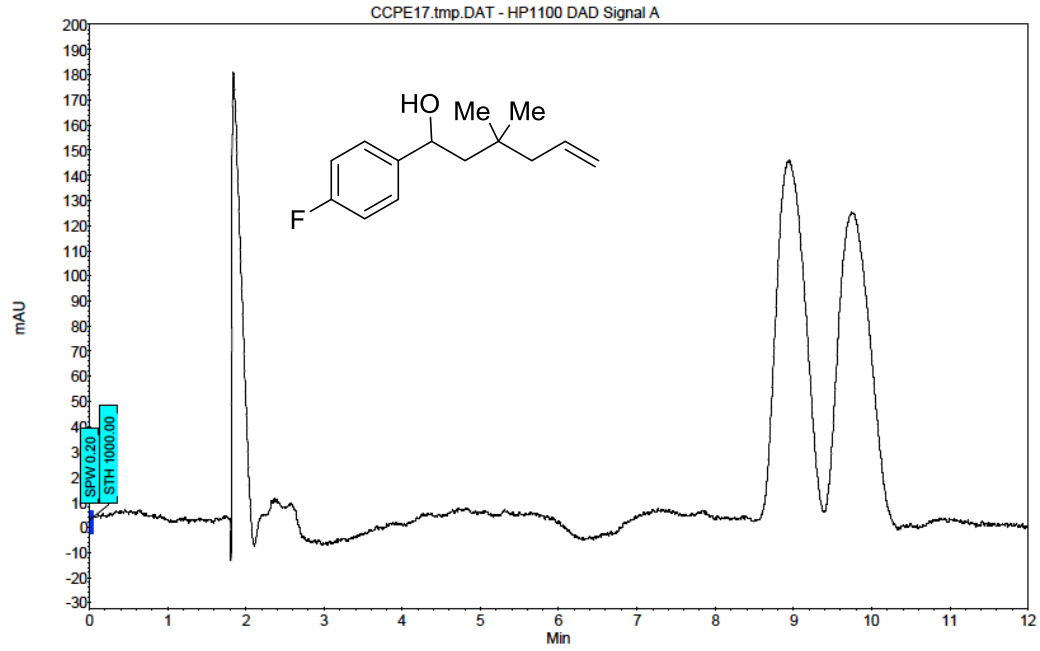


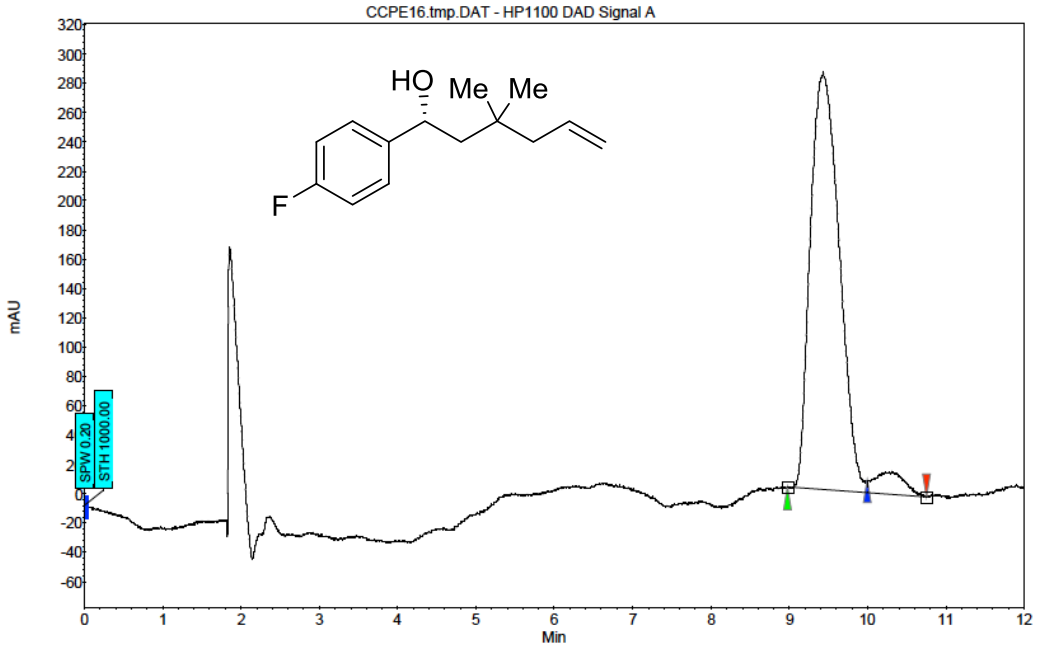
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.60	5.78	6.00	0.00	7.72	67.9	9.1	7.716
2	UNKNOWN	6.06	6.28	6.63	0.00	92.28	712.1	109.2	92.284
Total						100.00	780.0	118.3	100.000



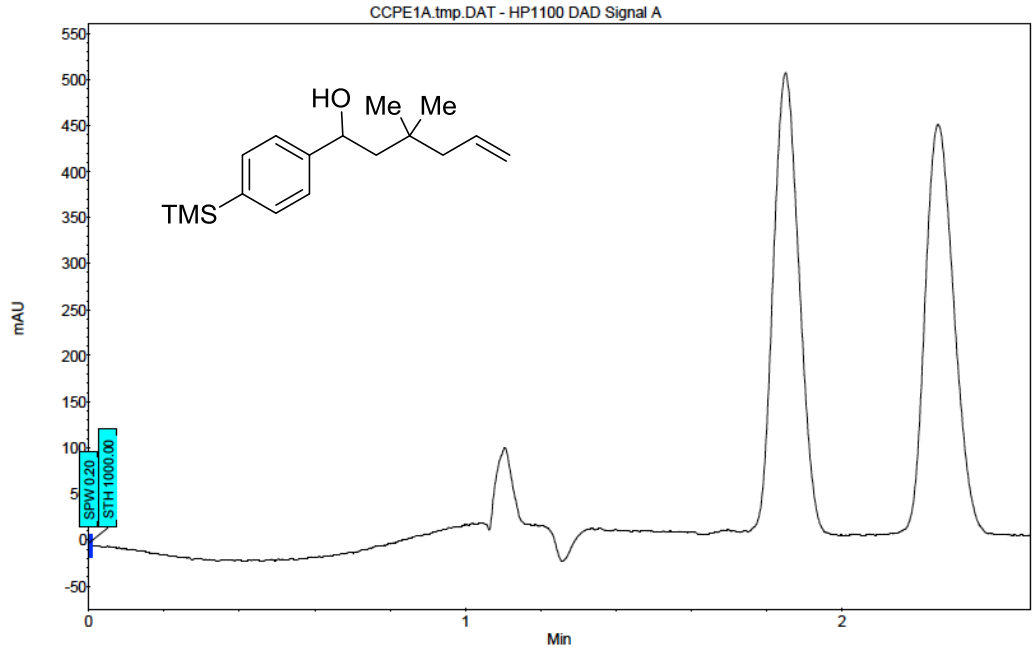


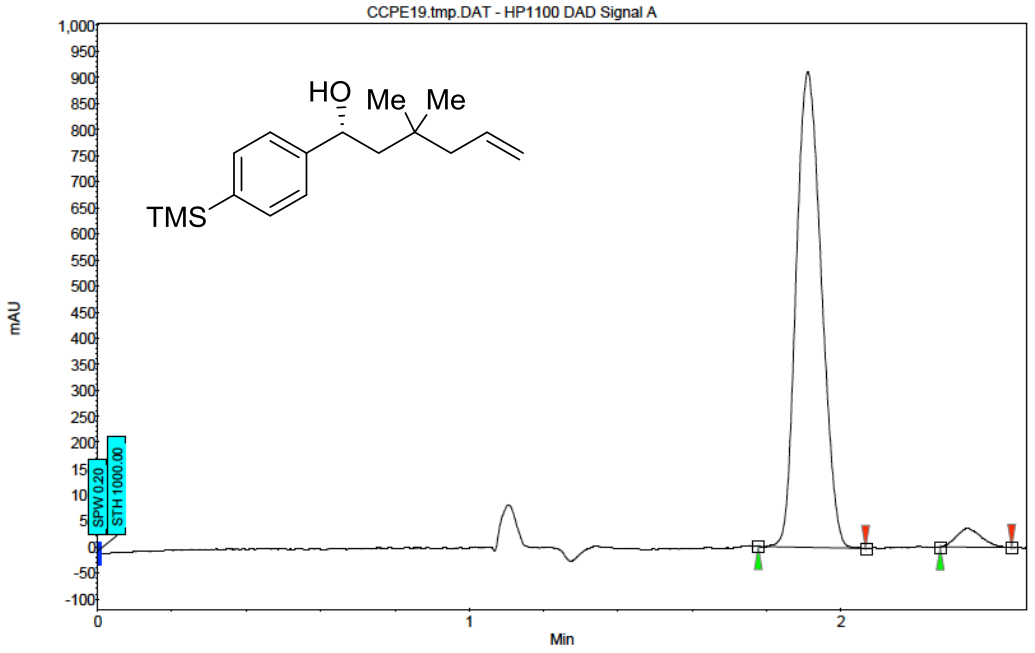
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.82	2.90	3.02	0.00	4.18	44.7	3.1	4.177
2	UNKNOWN	3.02	3.13	3.32	0.00	95.82	863.7	71.0	95.823
Total						100.00	908.4	74.1	100.000



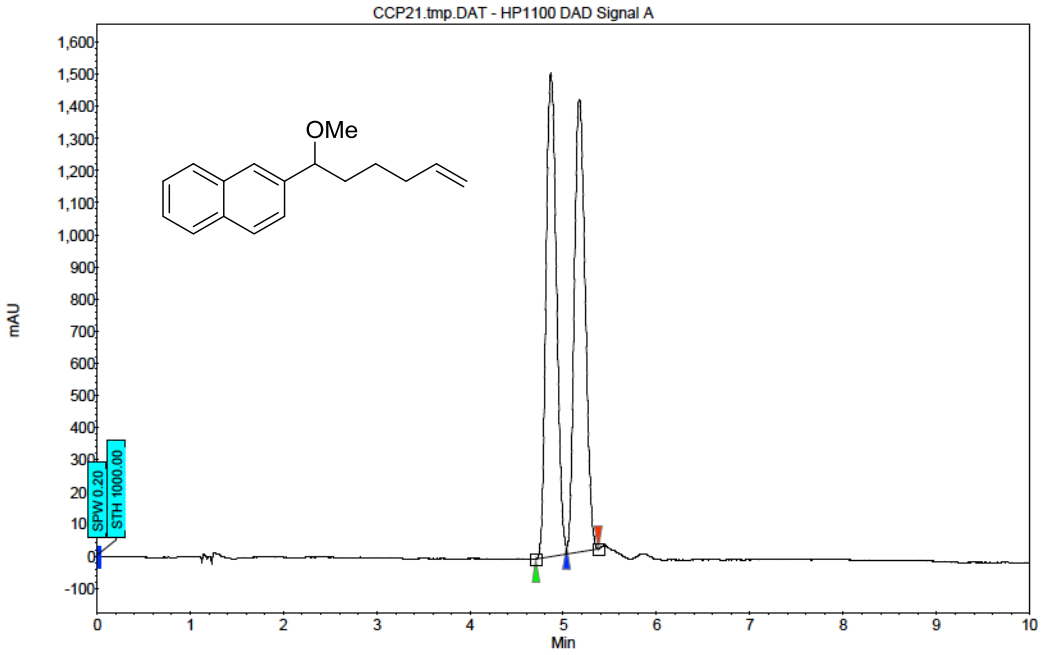


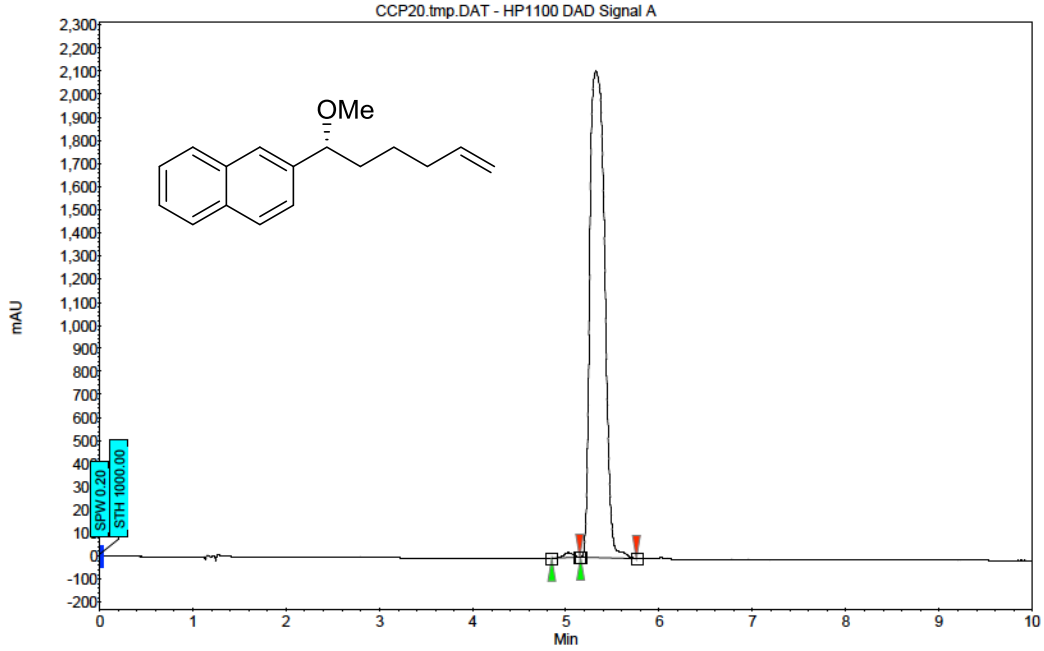
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8.98	9.43	9.99	0.00	94.74	284.7	121.7	94.739
2	UNKNOWN	9.99	10.31	10.75	0.00	5.26	15.5	6.8	5.261
Total						100.00	300.3	128.4	100.000



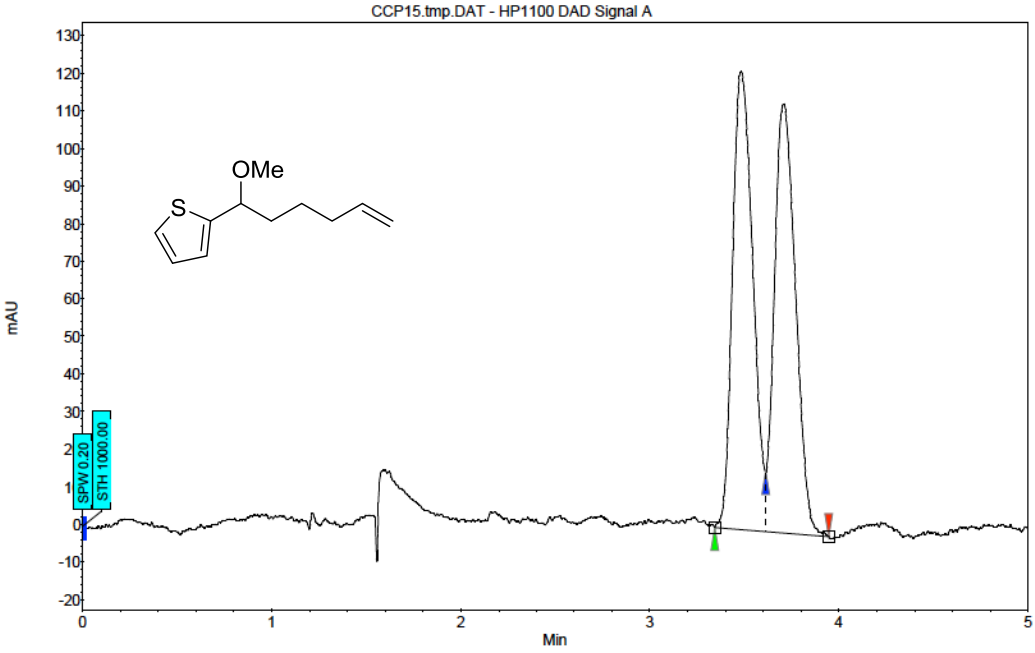


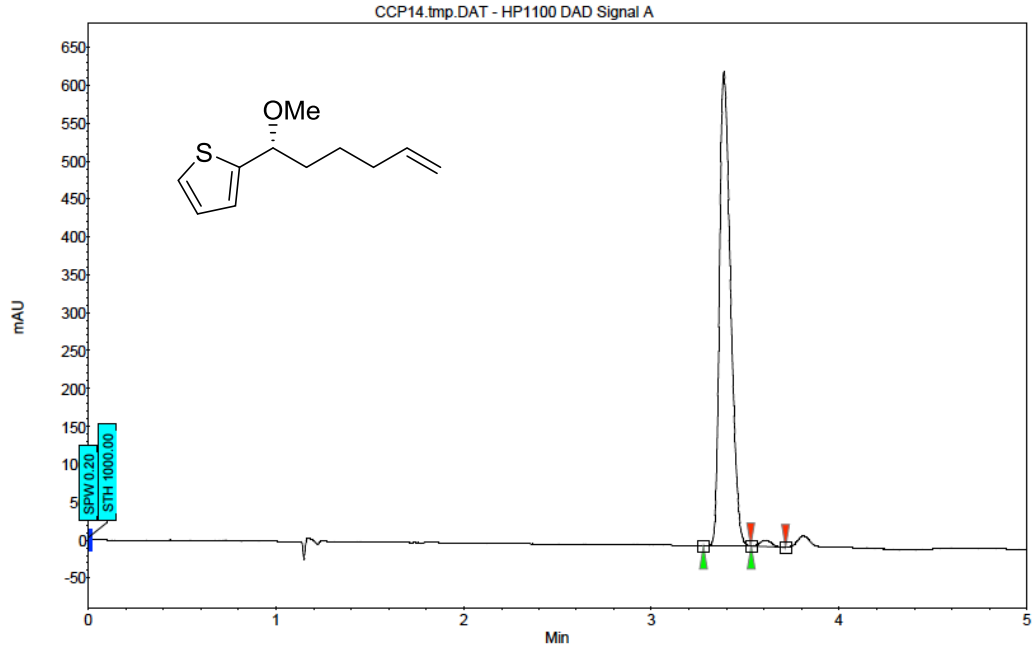
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.78	1.91	2.07	0.00	96.21	909.9	71.4	96.212
2	UNKNOWN	2.27	2.34	2.46	0.00	3.79	35.3	2.8	3.788
Total						100.00	945.1	74.2	100.000



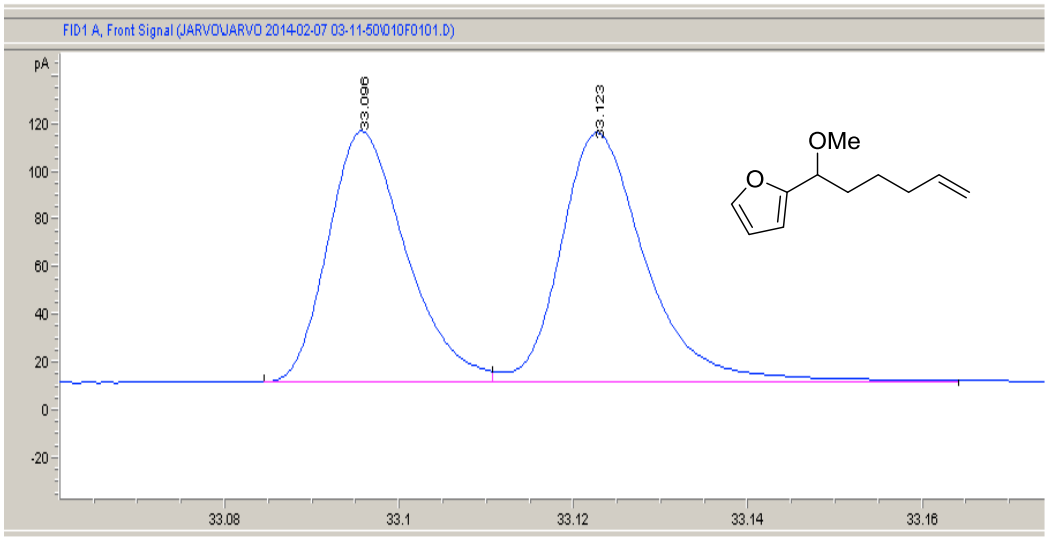


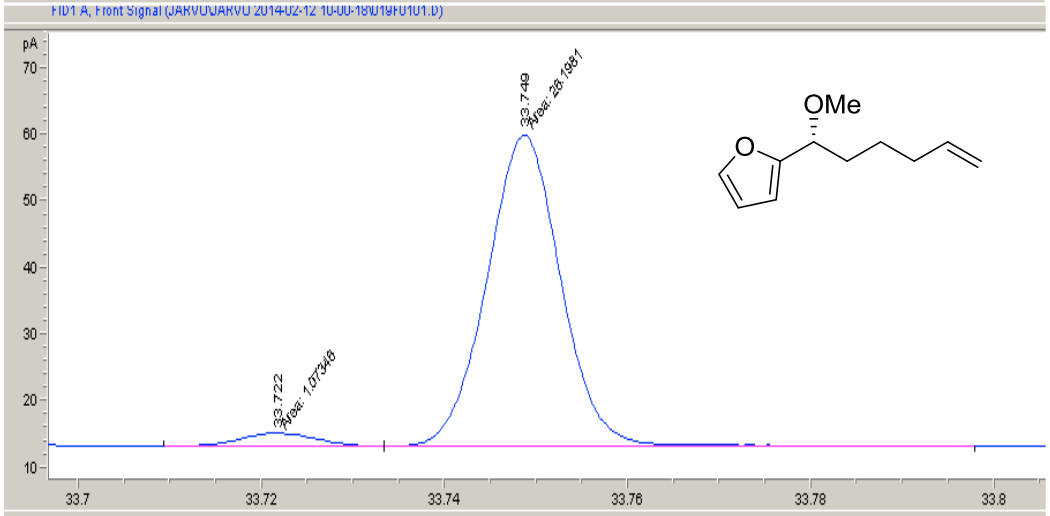
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.85	5.03	5.15	0.00	0.64	21.6	2.5	0.637
2	UNKNOWN	5.16	5.33	5.76	0.00	99.36	2105.2	384.1	99.363
Total						100.00	2126.8	386.5	100.000





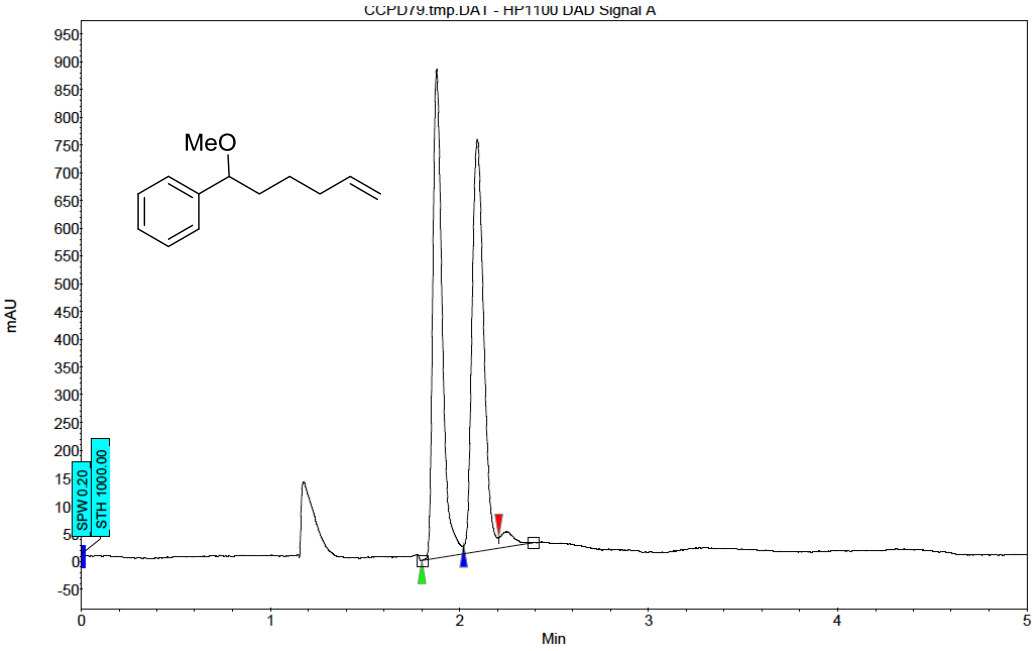
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.28	3.39	3.53	0.00	98.70	624.7	42.8	98.700
2	UNKNOWN	3.53	3.61	3.71	0.00	1.30	8.3	0.6	1.300
Total						100.00	633.0	43.4	100.000

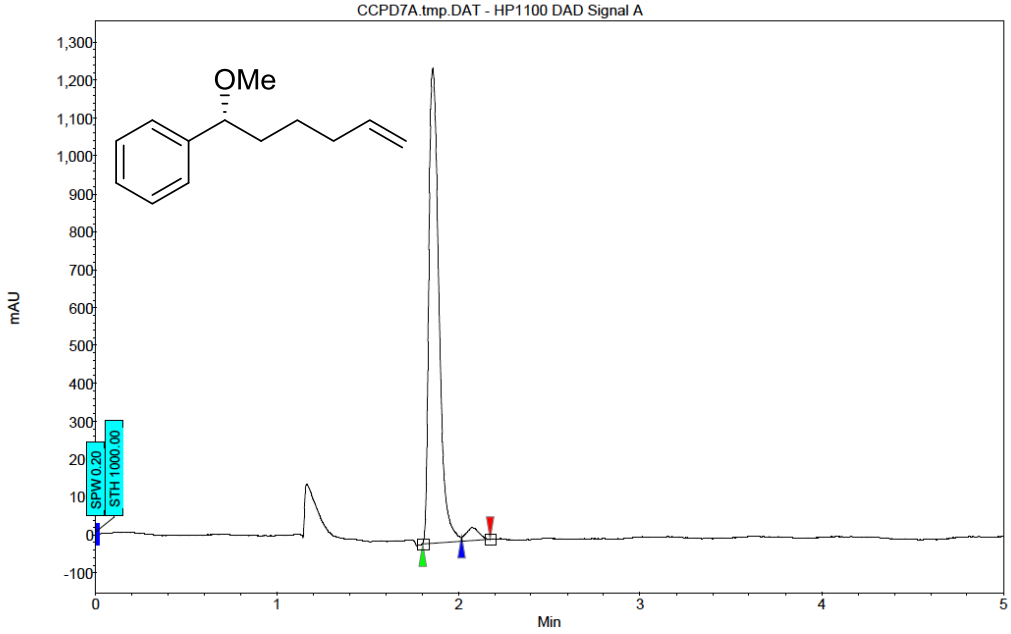




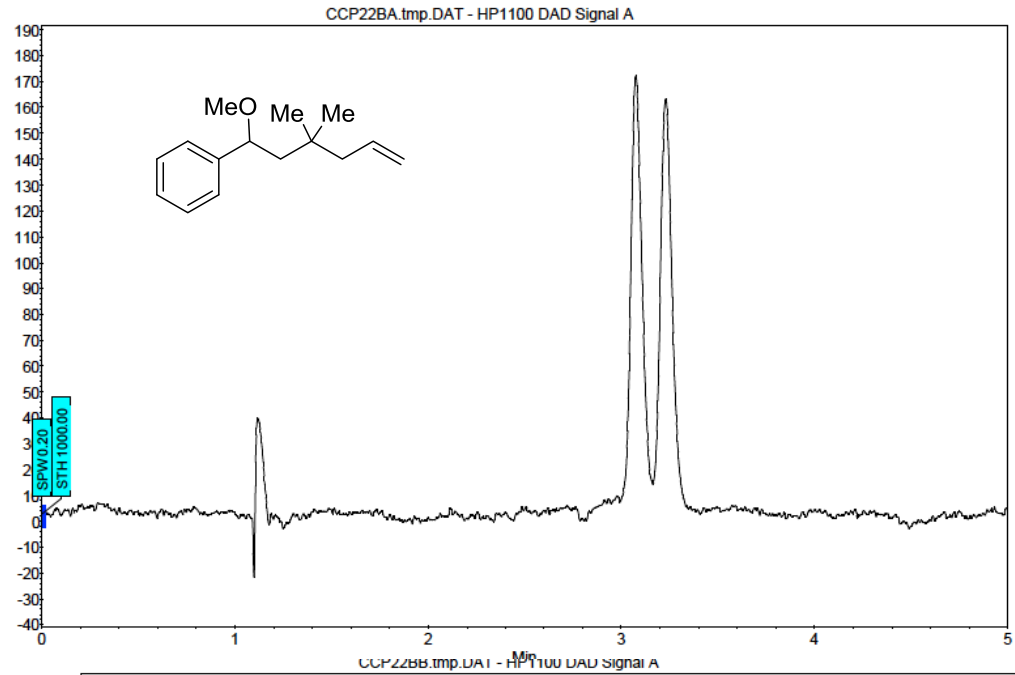
File Information	
GC-File	019F0101.D
File Path	C:\CHEM32\1\DATA\JARVO\JARVO 2014-02-12 10-00-18\
Date	12-Feb-14, 10:03:30
Sample	MOKACH3PROPRAC

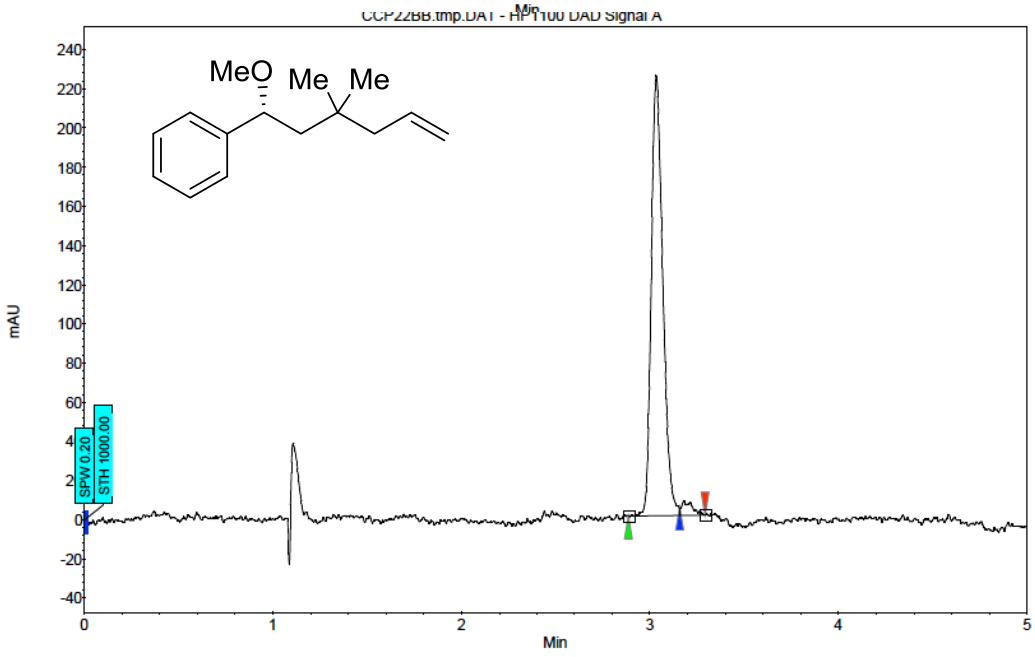
#	Time	Area	Height	Width	Area%	Symmetry
1	33.722	1.1	1.9	9.365E-3	3.936	1.325
2	33.749	26.2	47.1	9.2779E-3	96.064	1.414



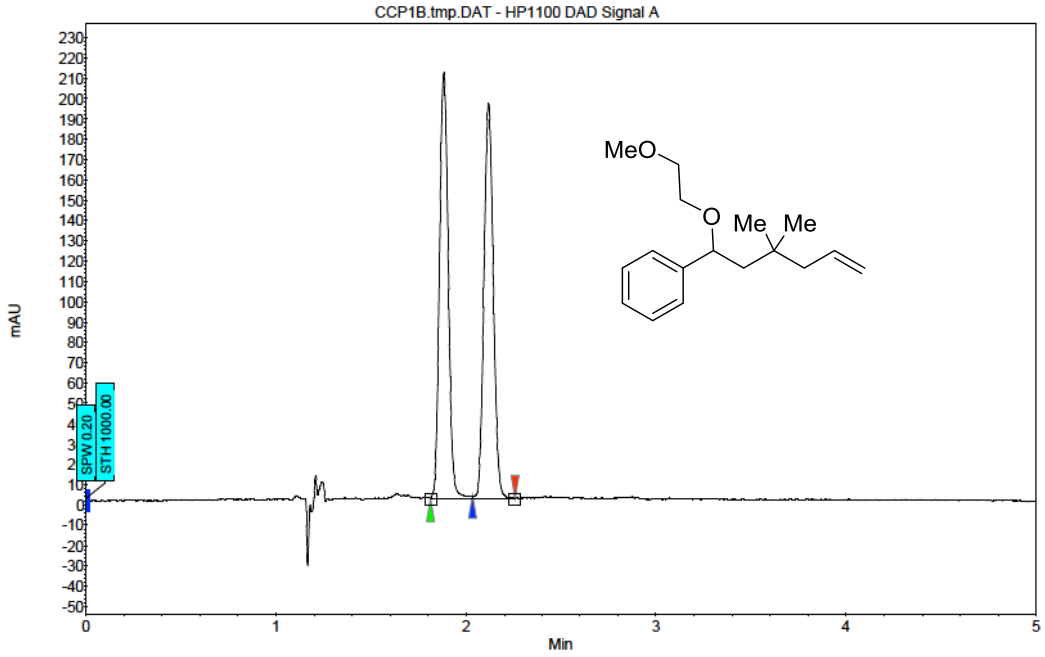


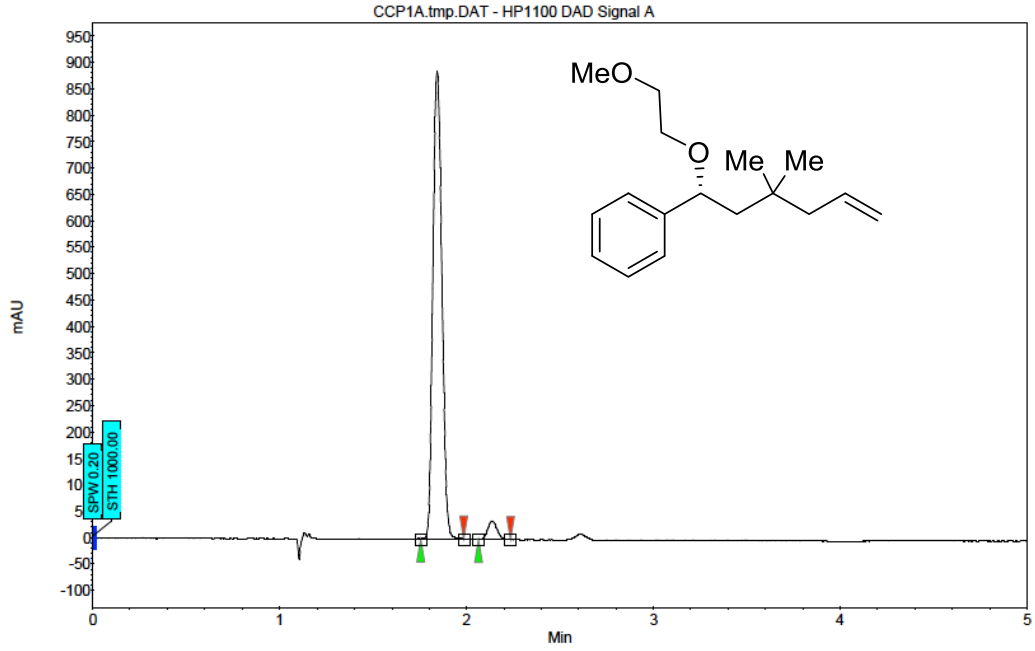
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.80	1.86	2.01	0.00	96.65	1252.1	80.4	96.654
2	UNKNOWN	2.01	2.07	2.17	0.00	3.35	34.3	2.8	3.346
Total						100.00	1286.4	83.2	100.000



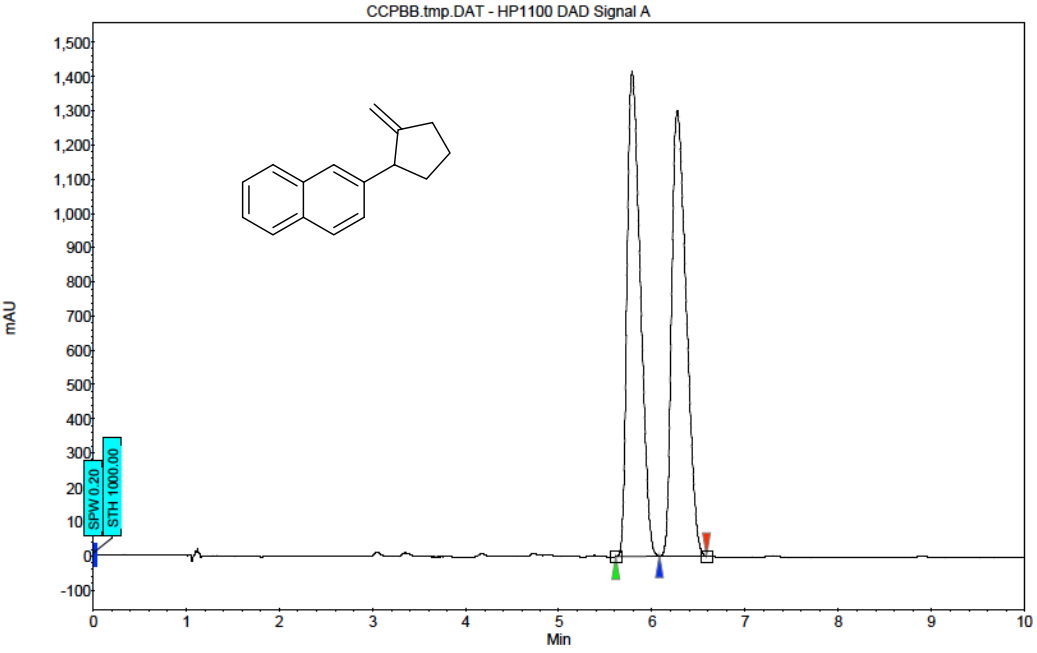


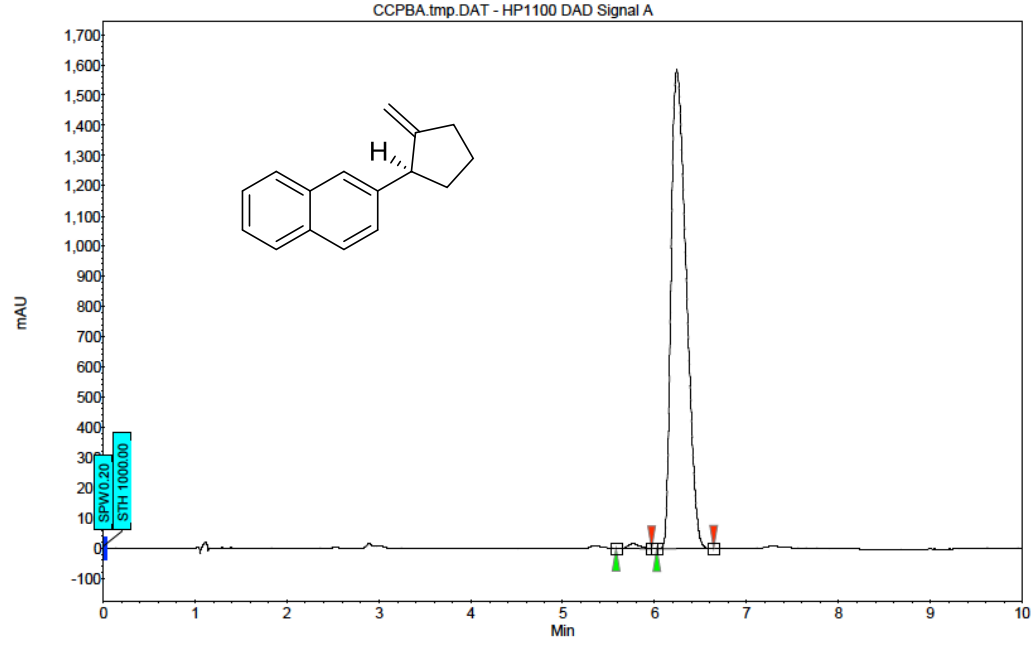
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.89	3.04	3.16	0.00	97.07	224.4	15.9	97.073
2	UNKNOWN	3.16	3.18	3.29	0.00	2.93	7.3	0.5	2.927
Total						100.00	231.8	16.3	100.000



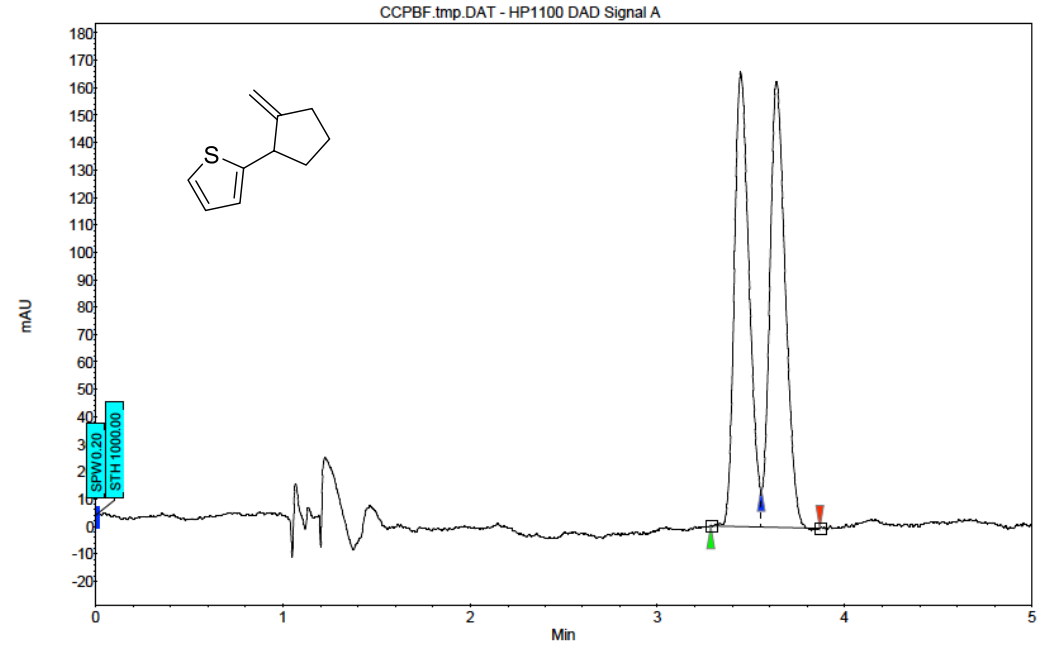


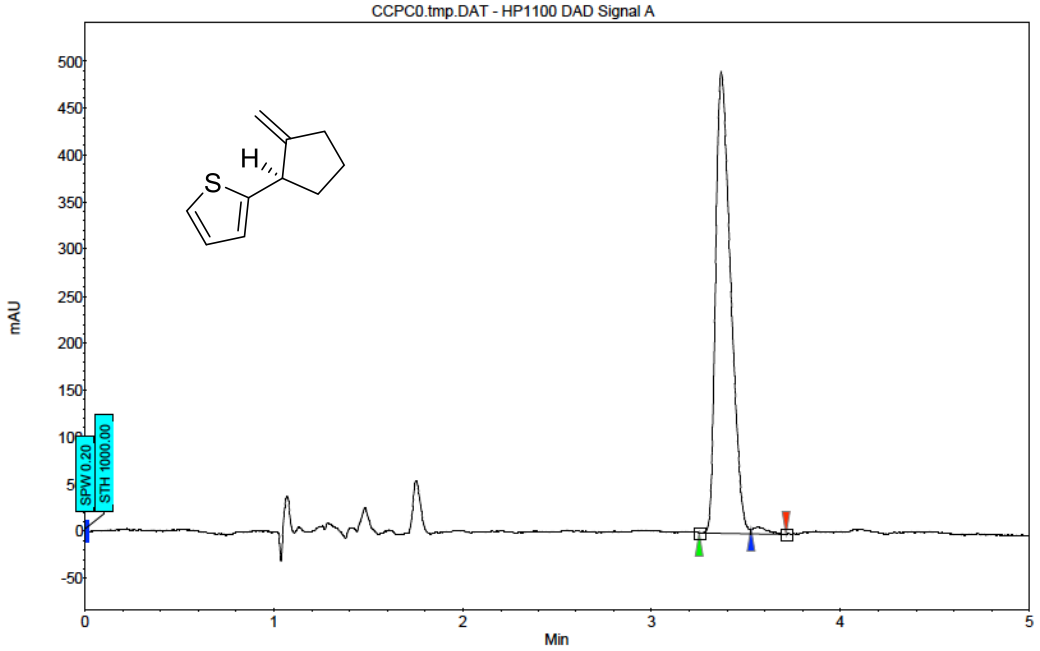
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.76	1.84	1.98	0.00	96.07	884.4	49.2	96.068
2	UNKNOWN	2.06	2.14	2.24	0.00	3.93	34.9	2.0	3.932
Total						100.00	919.3	51.2	100.000



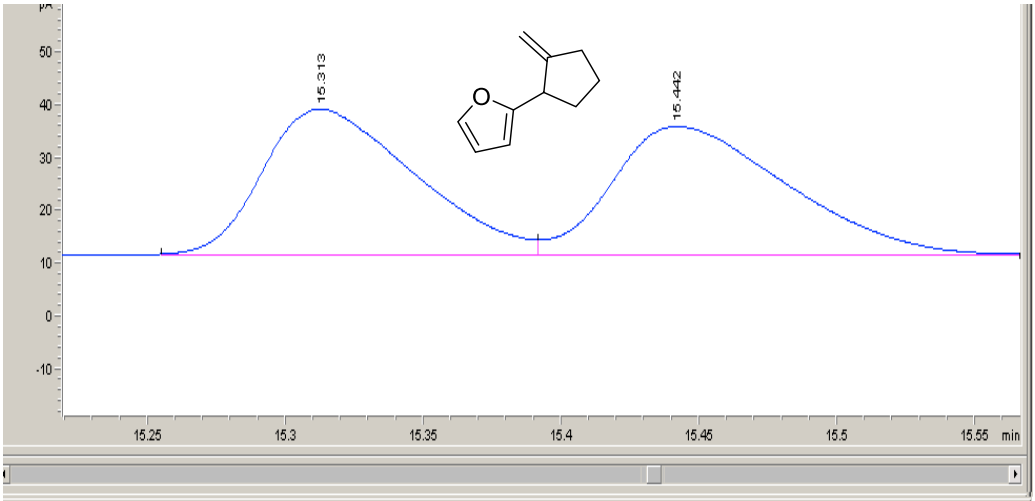


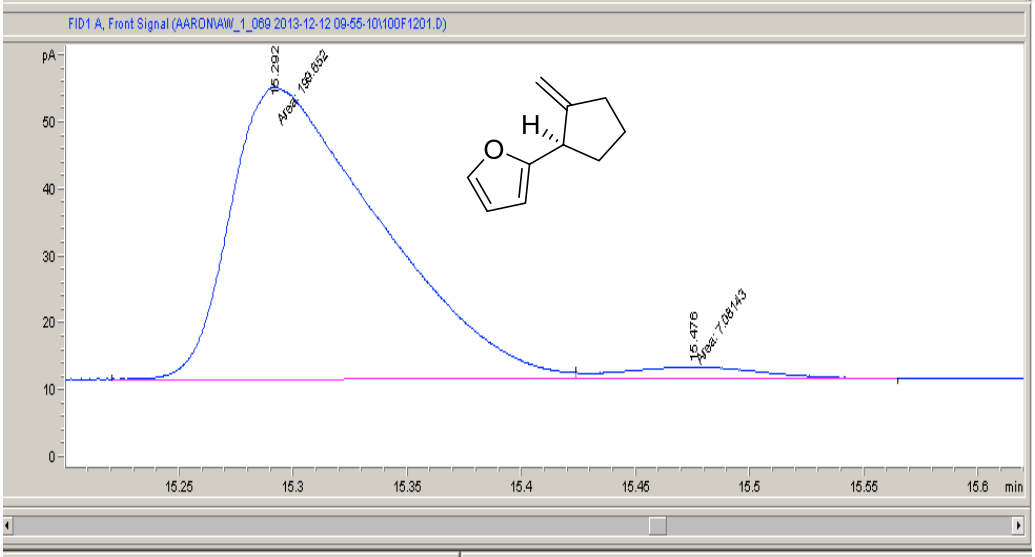
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.58	5.77	5.97	0.00	0.70	14.1	2.1	0.696
2	UNKNOWN	6.02	6.24	6.64	0.00	99.30	1585.0	301.4	99.304
Total						100.00	1599.1	303.5	100.000





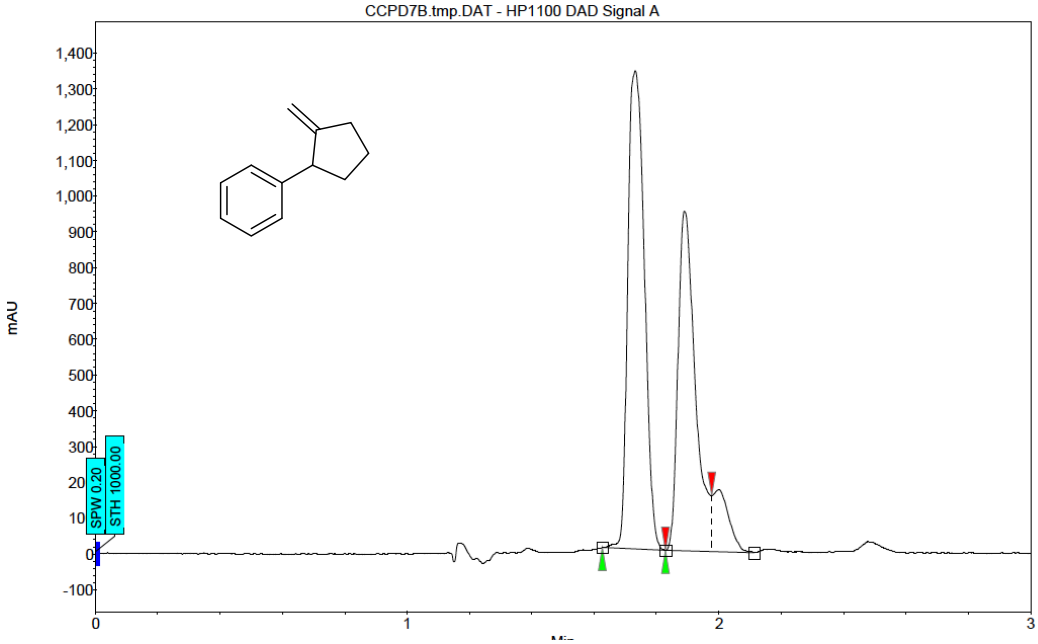
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.25	3.37	3.53	0.00	98.52	491.0	45.2	98.520
2	UNKNOWN	3.53	3.57	3.71	0.00	1.48	7.7	0.7	1.480
Total						100.00	498.7	45.9	100.000

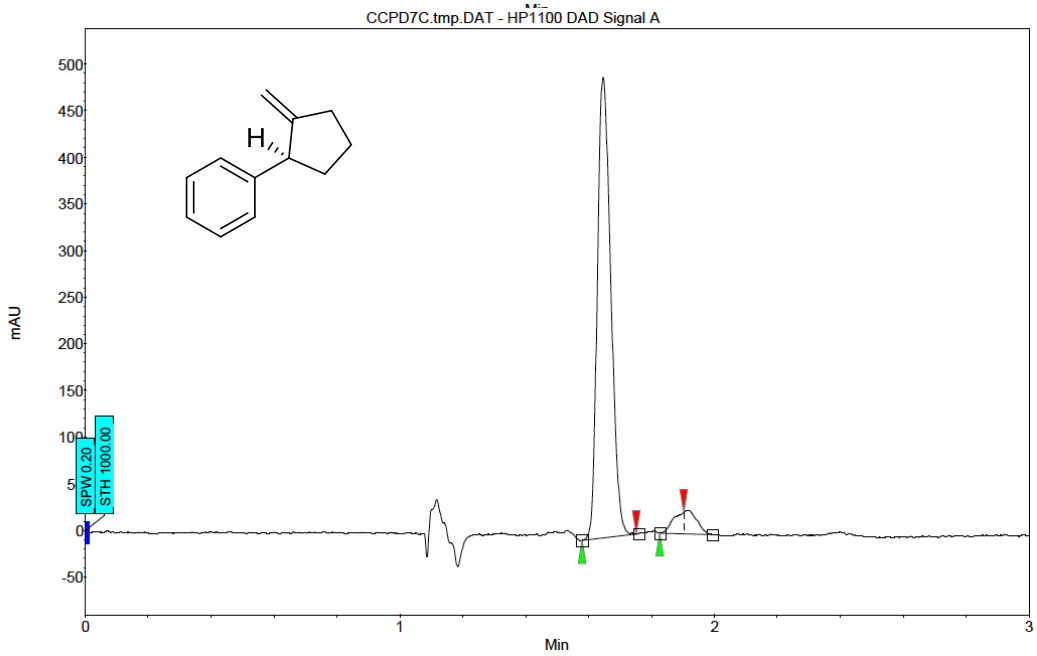




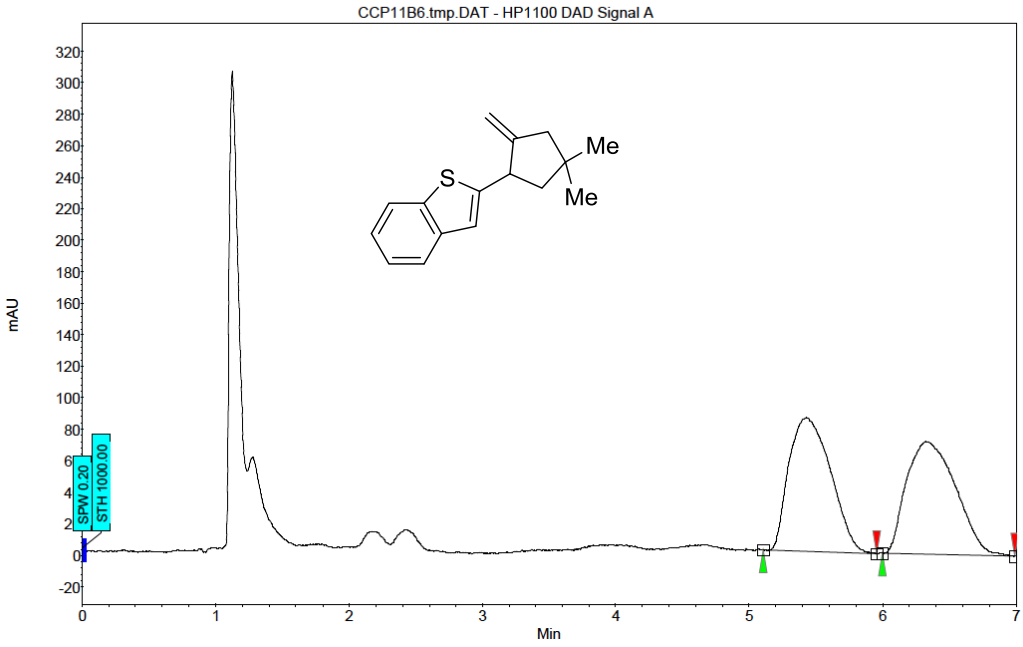
File Information	
GC-File	100F1201.D
File Path	C:\CHEM32\1\DATA\AARON\AW_1_069 2013-12-12 09-55-10\
Date	12-Dec-13, 12:53:49
Sample	MRH-VI-12

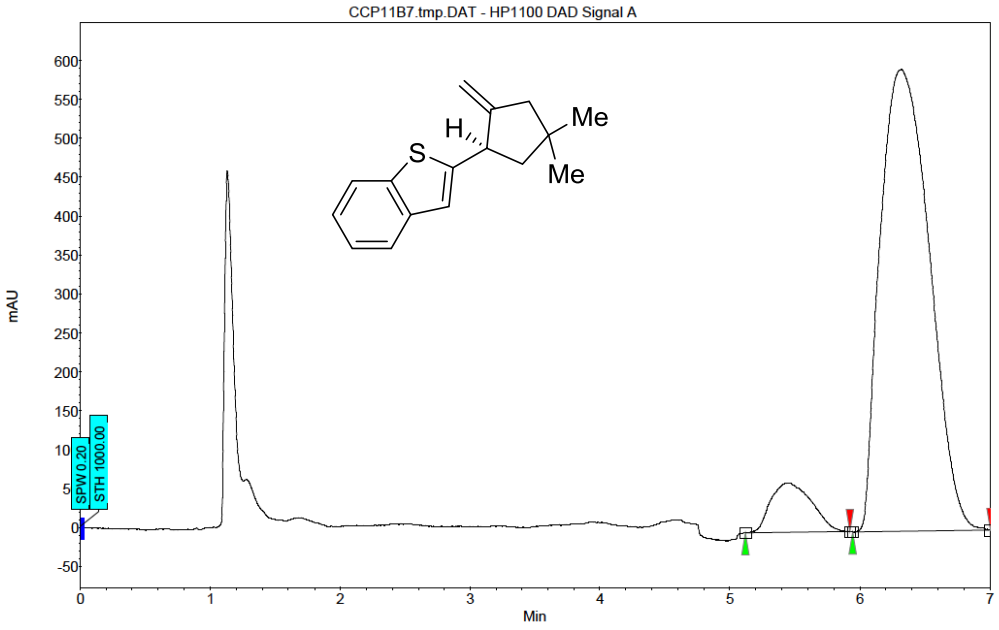
#	Time	Area	Height	Width	Area%	Symmetry
1	15.292	199.7	43.8	0.0759	96.575	0
2	15.476	7.1	1.7	0.071	3.425	1.204



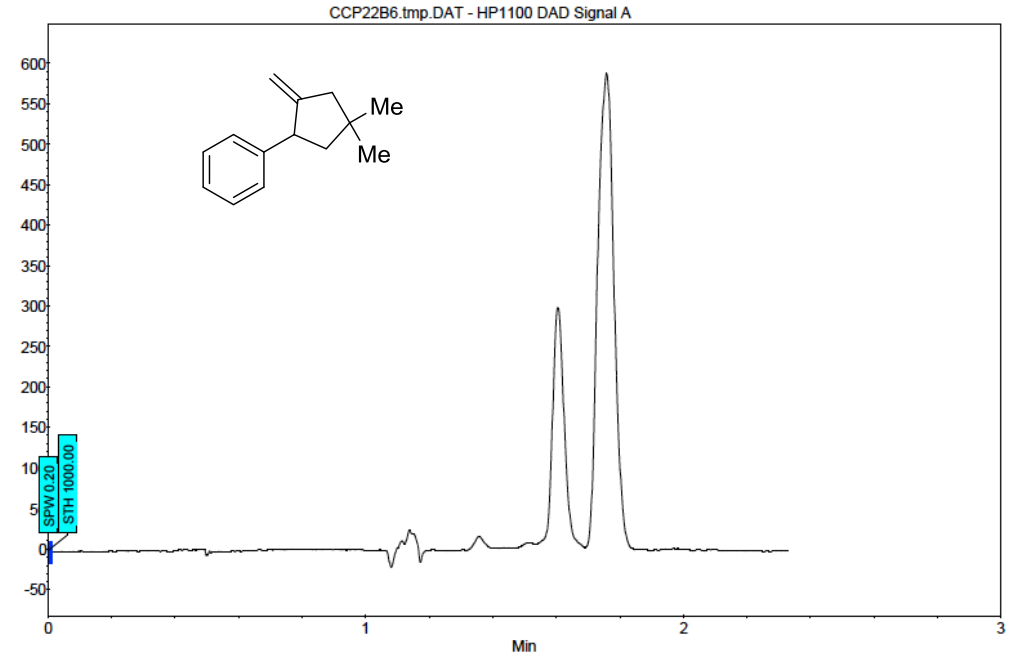


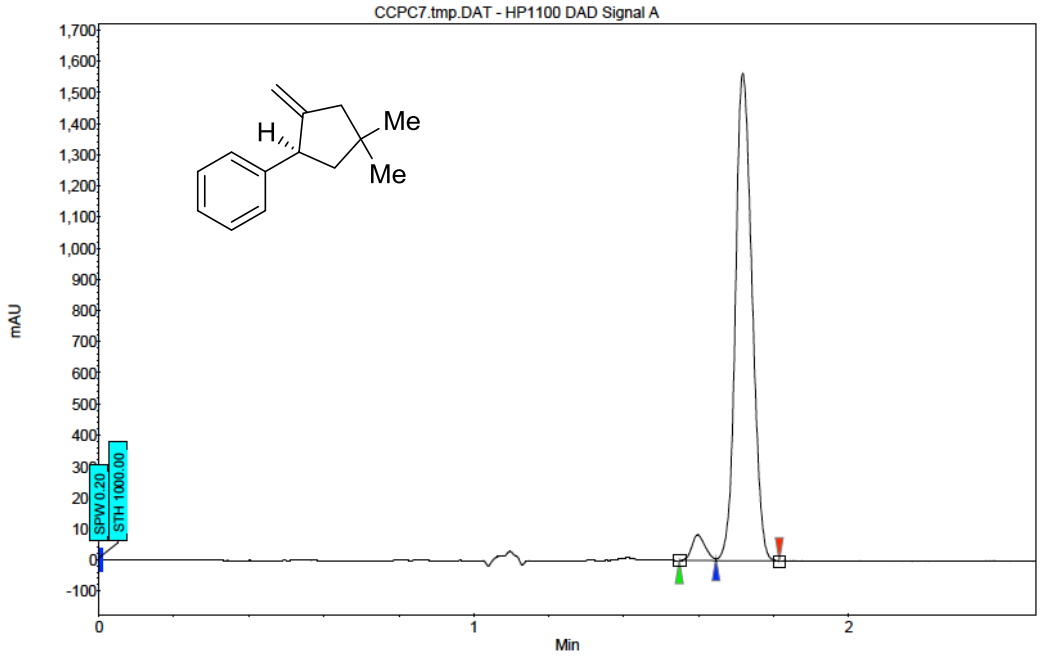
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.58	1.65	1.75	0.00	96.49	493.5	24.4	96.493
2	UNKNOWN	1.83	1.90	1.90	0.00	3.51	22.7	0.9	3.507
Total						100.00	516.1	25.3	100.000



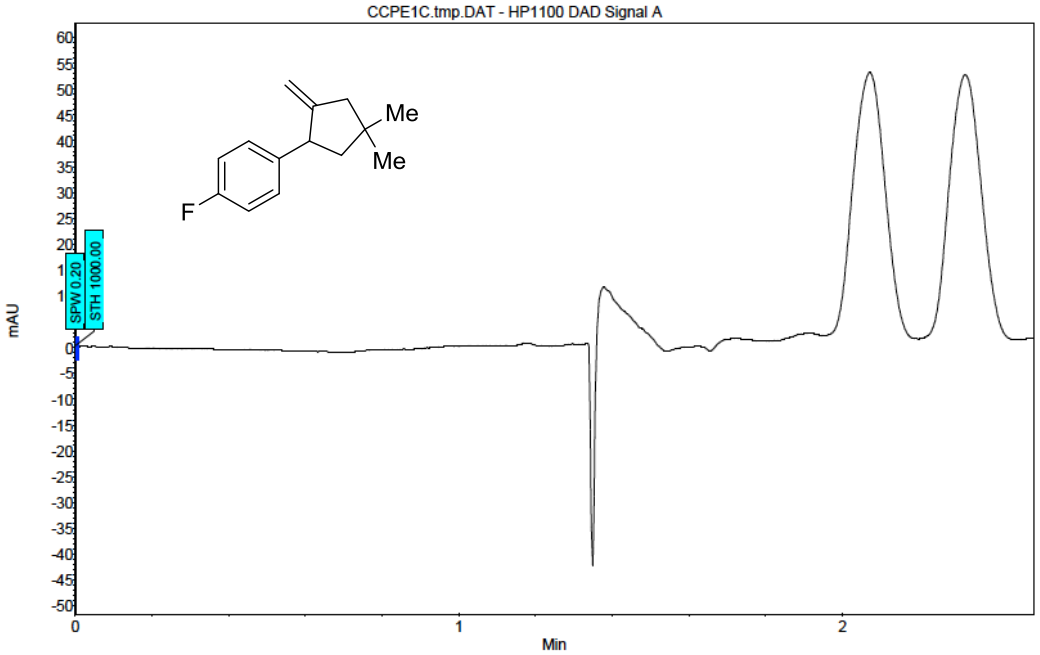


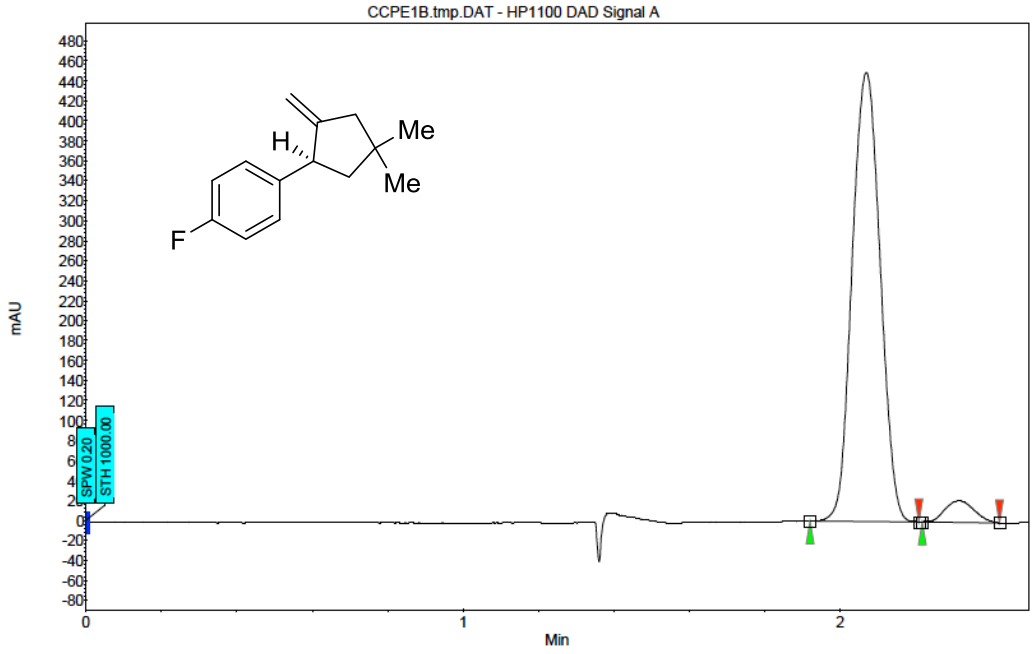
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.12	5.45	5.92	0.00	8.18	63.7	23.2	8.181
2	UNKNOWN	5.94	6.32	7.00	0.00	91.82	593.0	260.4	91.819
Total						100.00	656.7	283.6	100.000



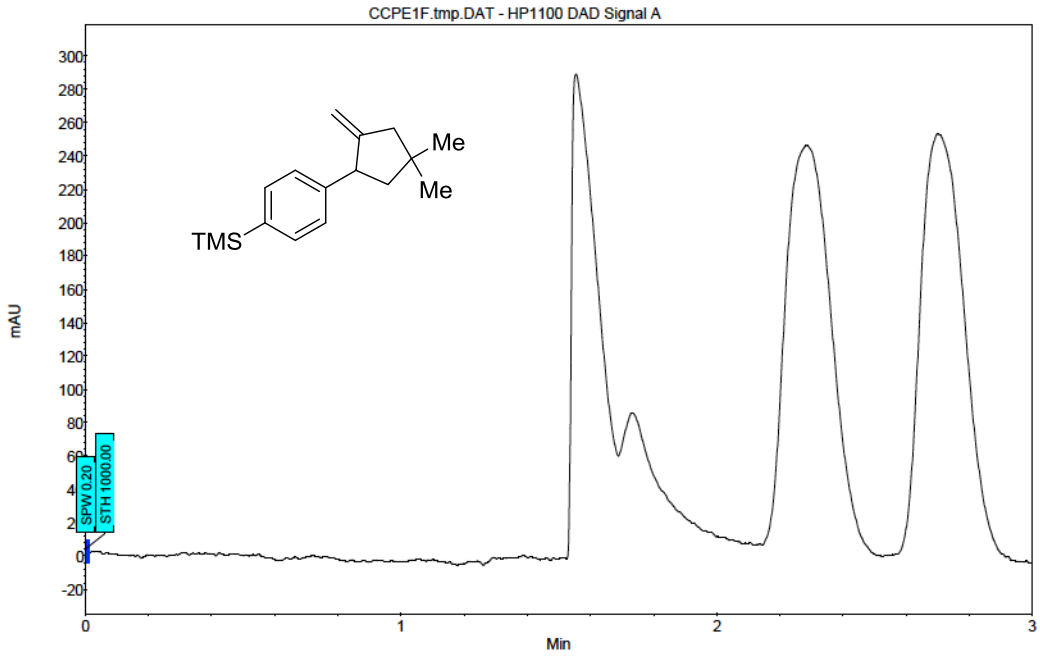


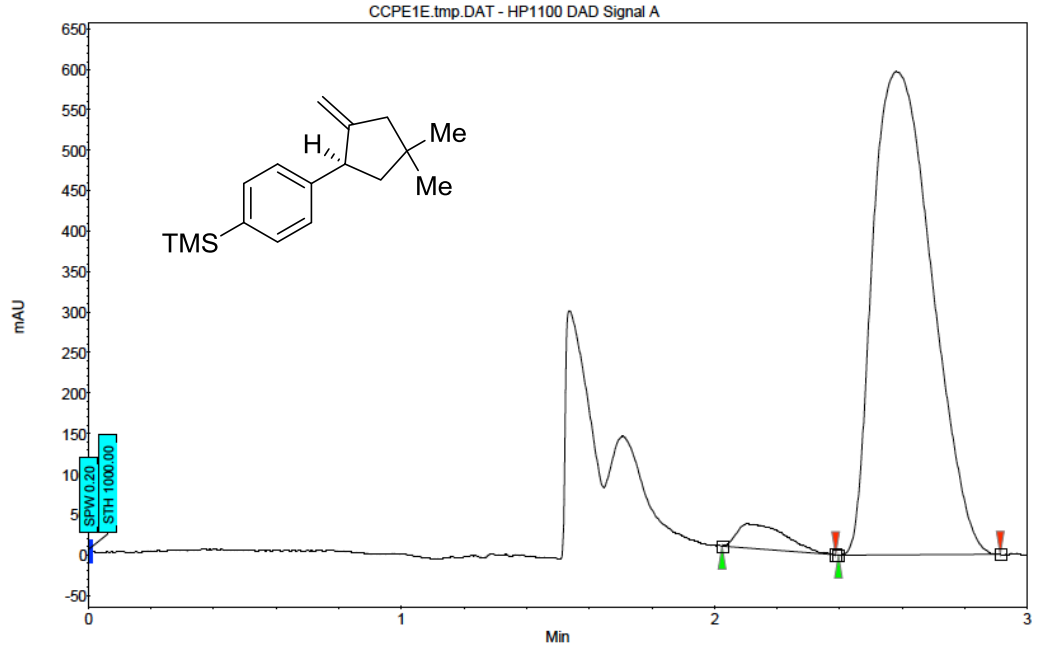
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.55	1.60	1.65	0.00	4.05	82.9	3.4	4.054
2	UNKNOWN	1.65	1.72	1.82	0.00	95.95	1563.6	80.0	95.946
Total						100.00	1646.4	83.4	100.000



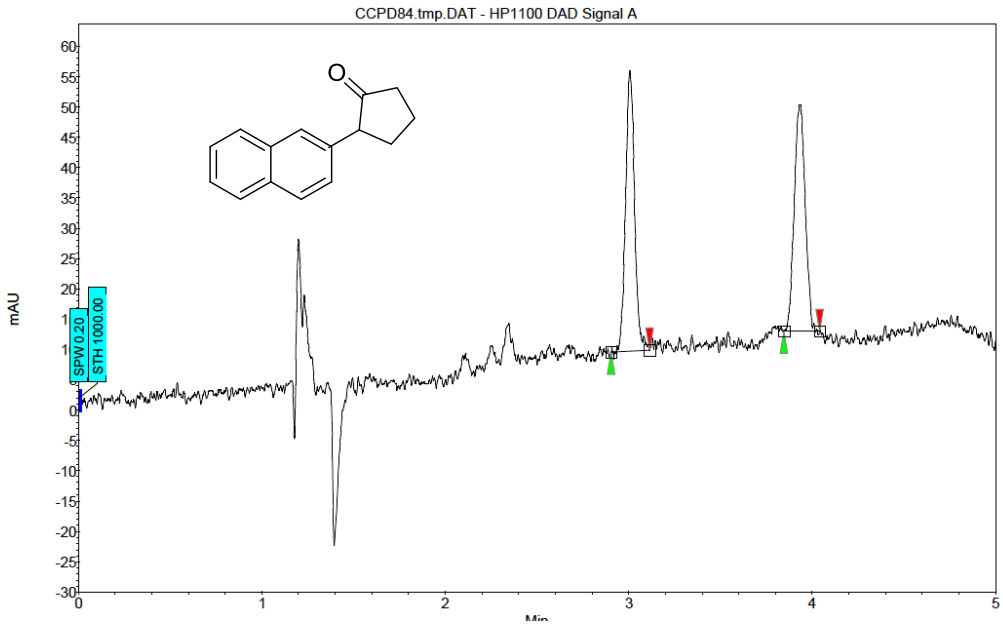


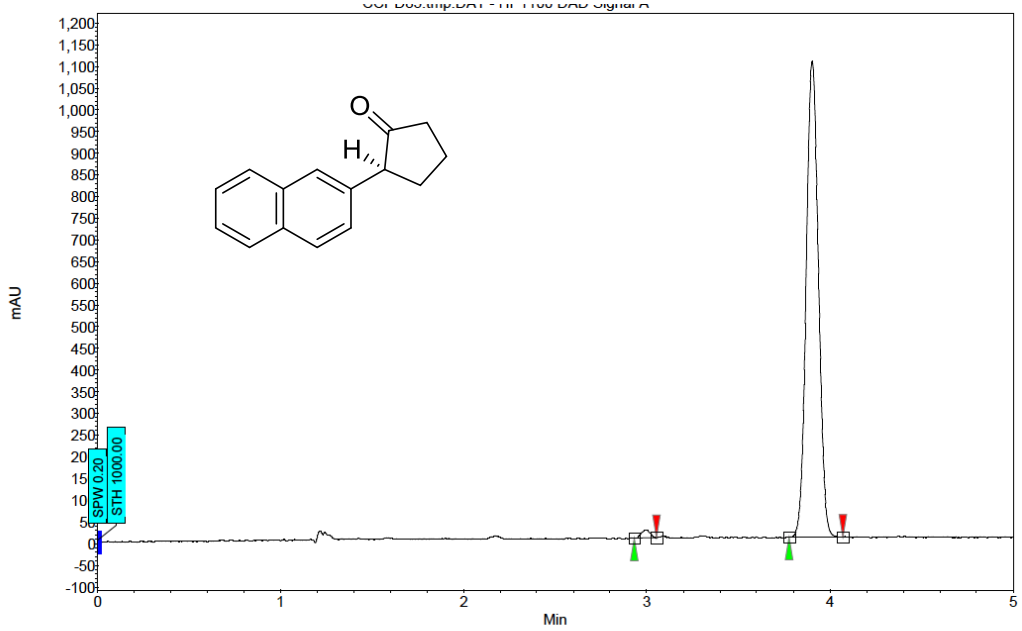
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.92	2.07	2.21	0.00	95.21	449.5	38.6	95.213
2	UNKNOWN	2.22	2.31	2.42	0.00	4.79	21.7	1.9	4.787
Total						100.00	471.2	40.6	100.000



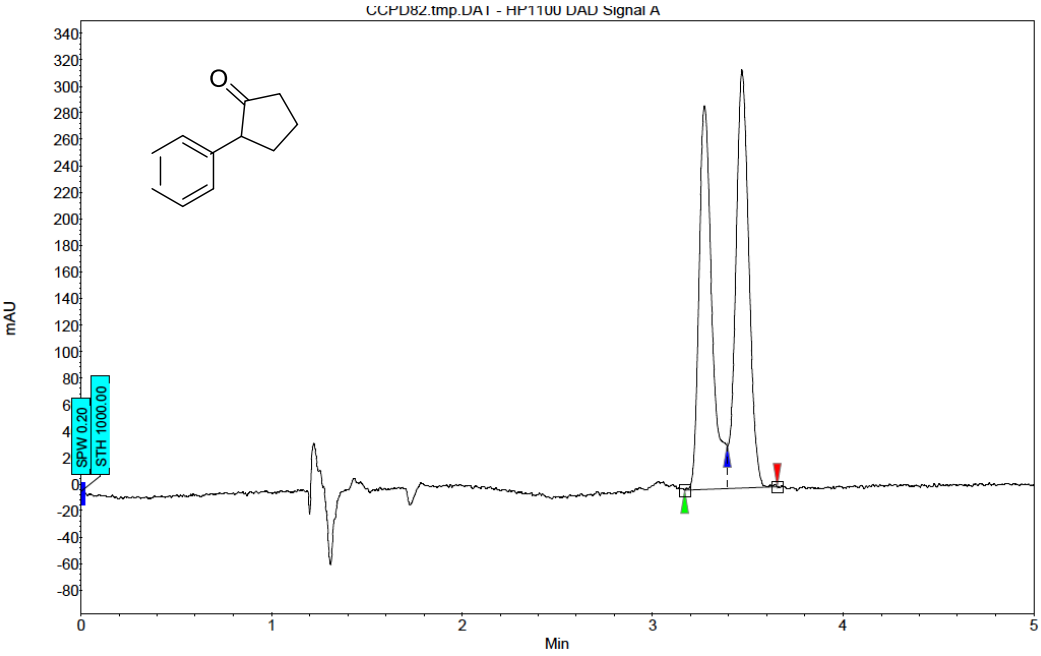


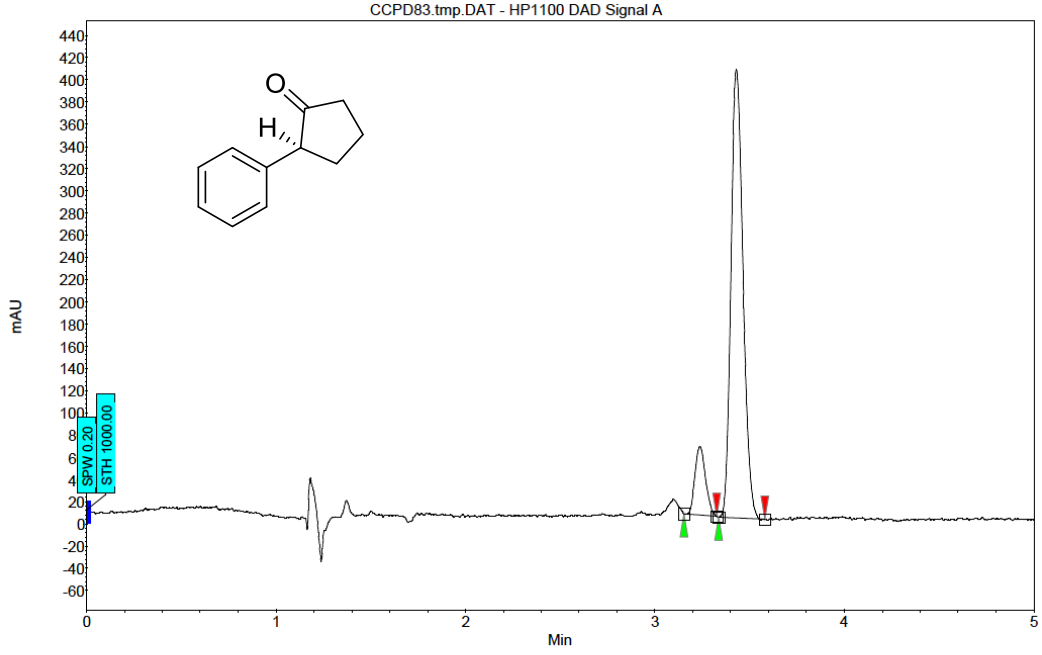
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.03	2.11	2.39	0.00	4.02	30.1	5.5	4.015
2	UNKNOWN	2.40	2.58	2.91	0.00	95.98	597.4	130.8	95.985
Total						100.00	627.5	136.2	100.000



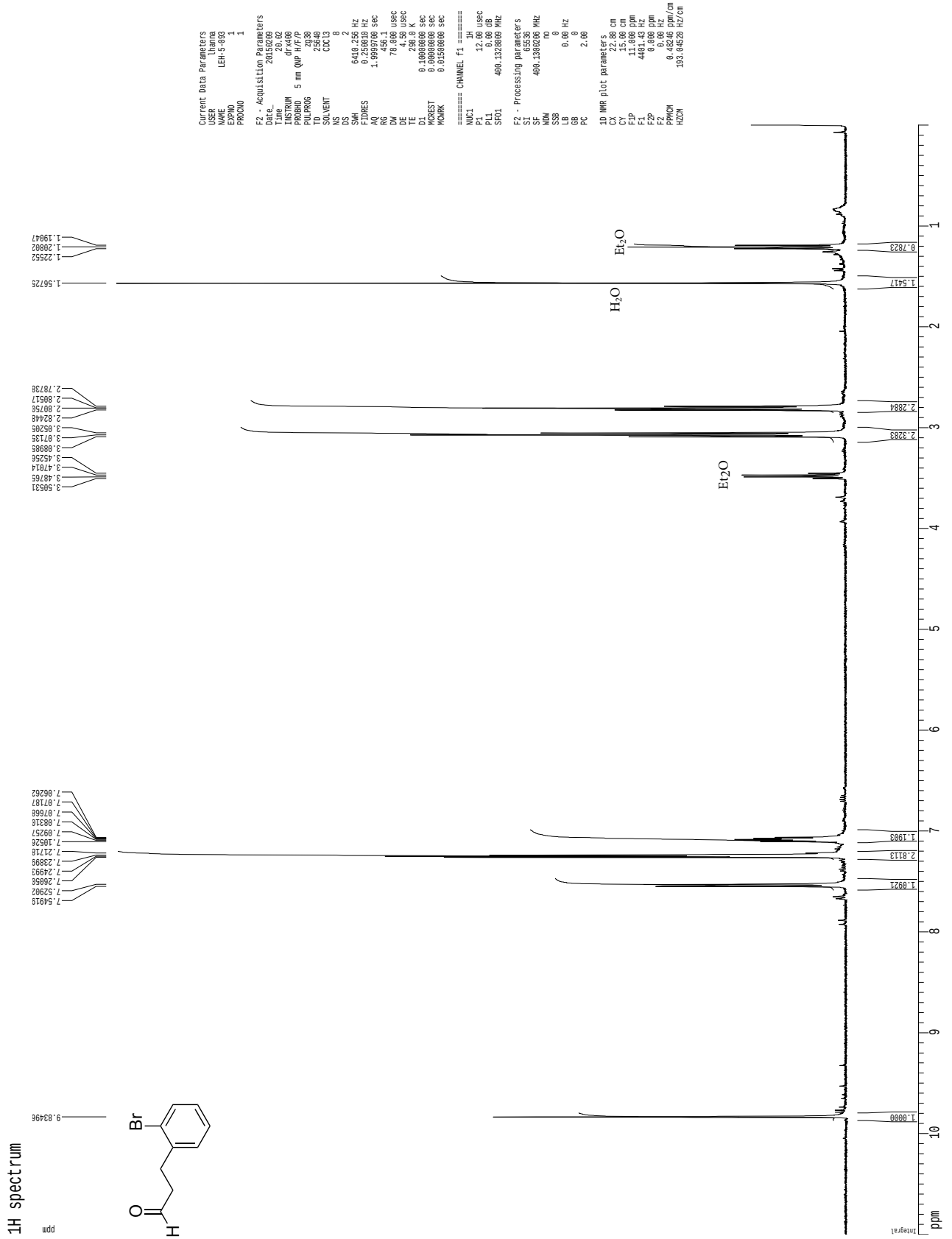


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	2.93	2.99	3.05	0.00	1.14	18.0	1.0	1.141
1	UNKNOWN	3.78	3.91	4.07	0.00	98.86	1096.7	85.6	98.859
Total						100.00	1114.8	86.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.15	3.24	3.33	0.00	11.78	61.5	4.0	11.780
2	UNKNOWN	3.34	3.43	3.58	0.00	88.22	403.6	29.7	88.220
Total						100.00	465.2	33.7	100.000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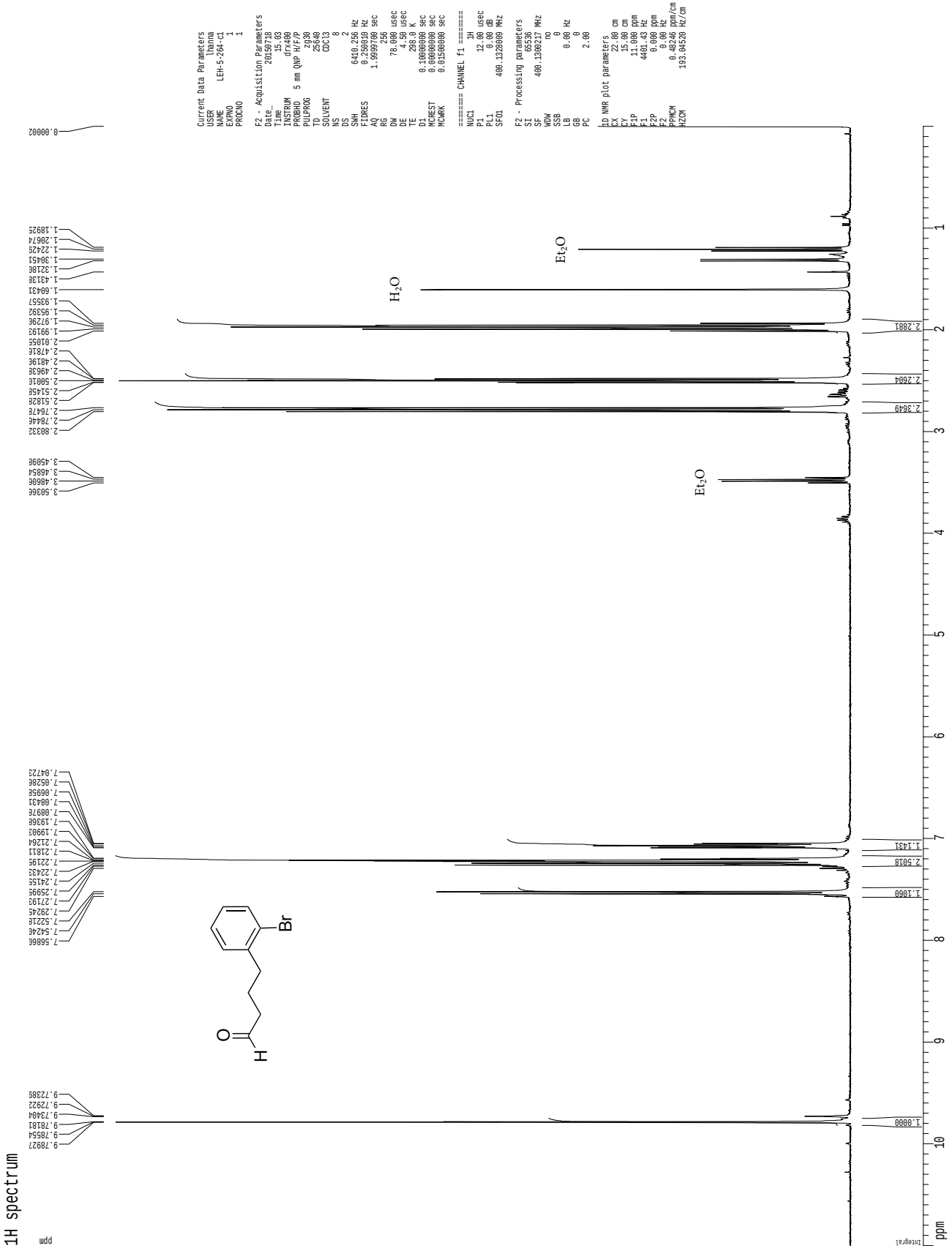
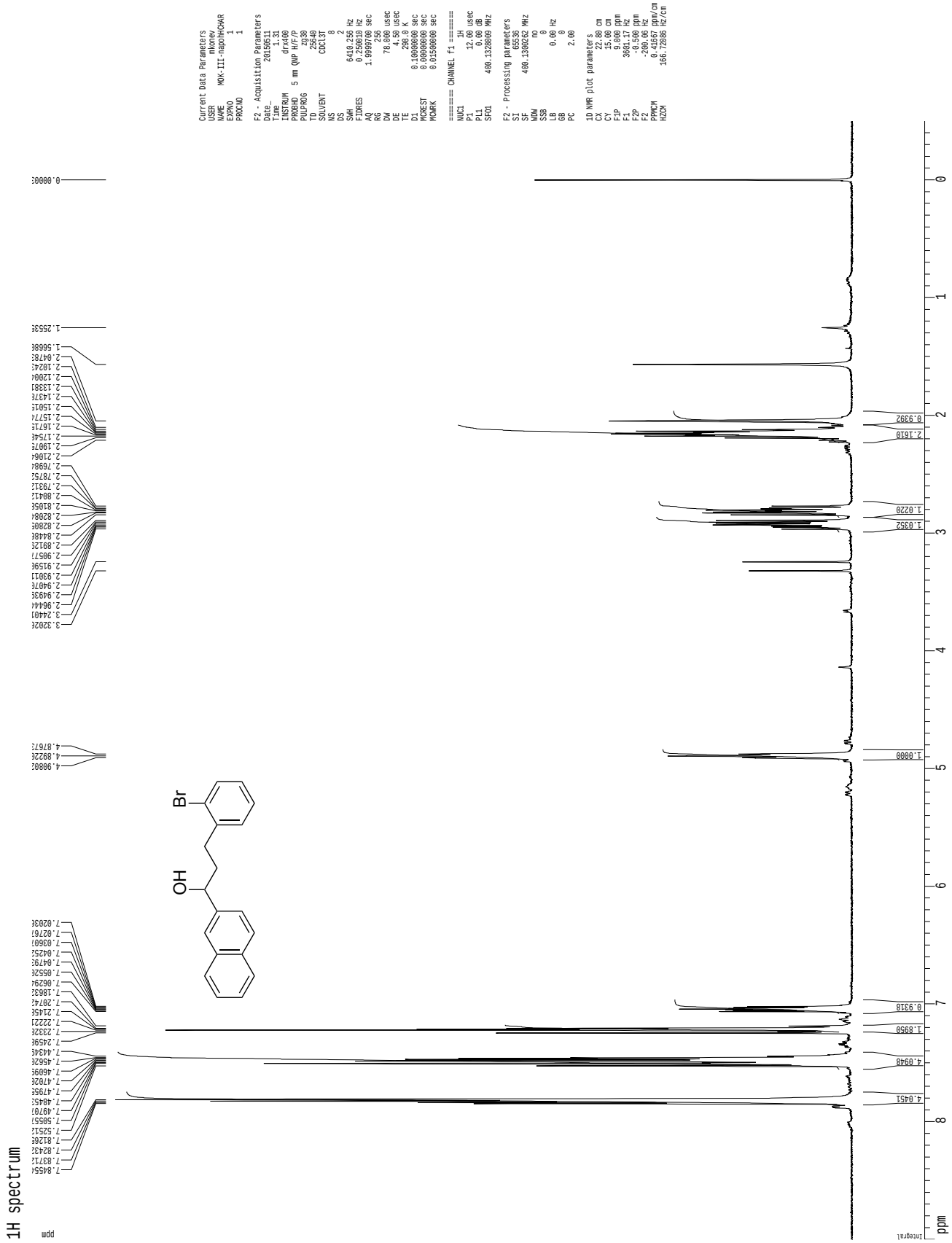
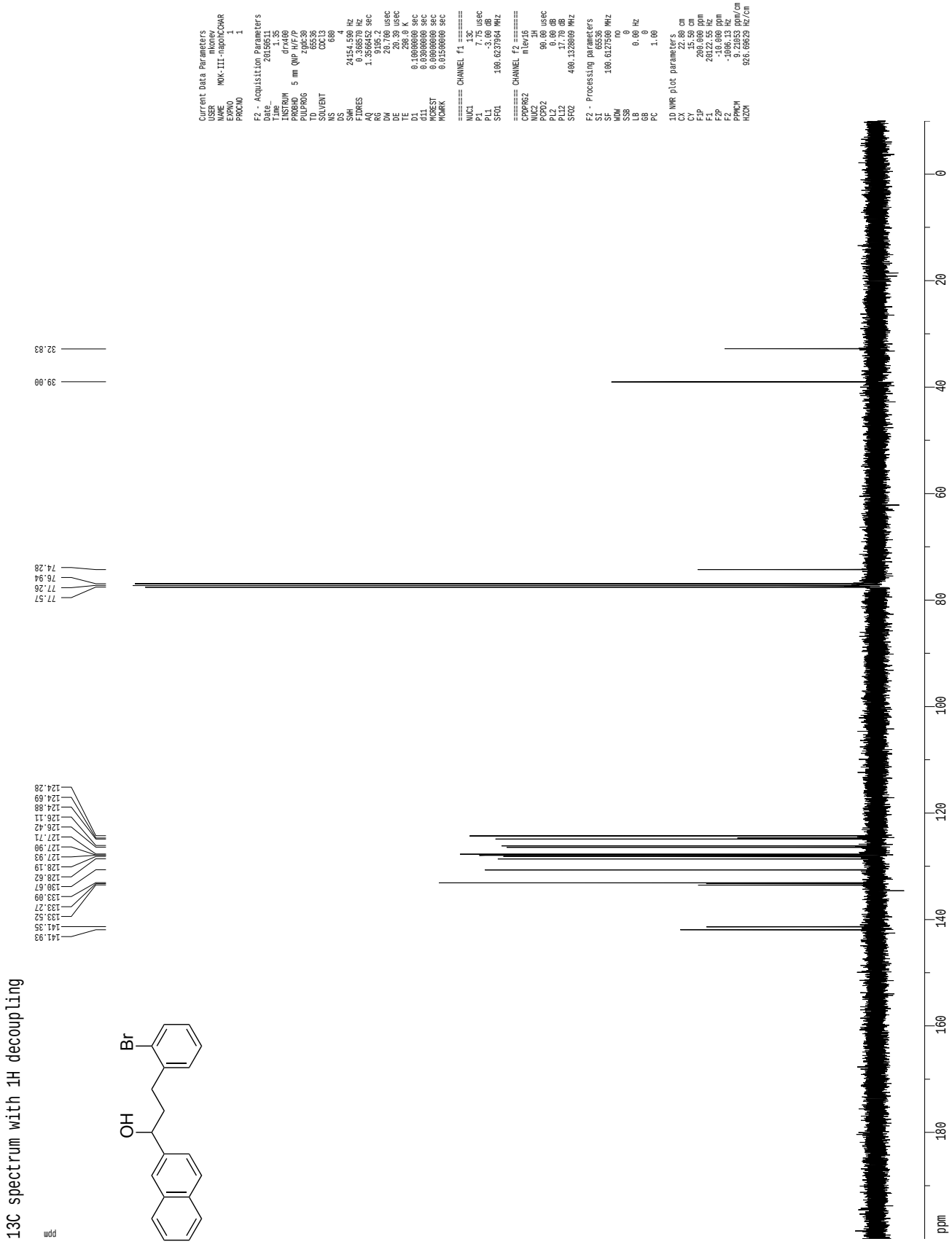
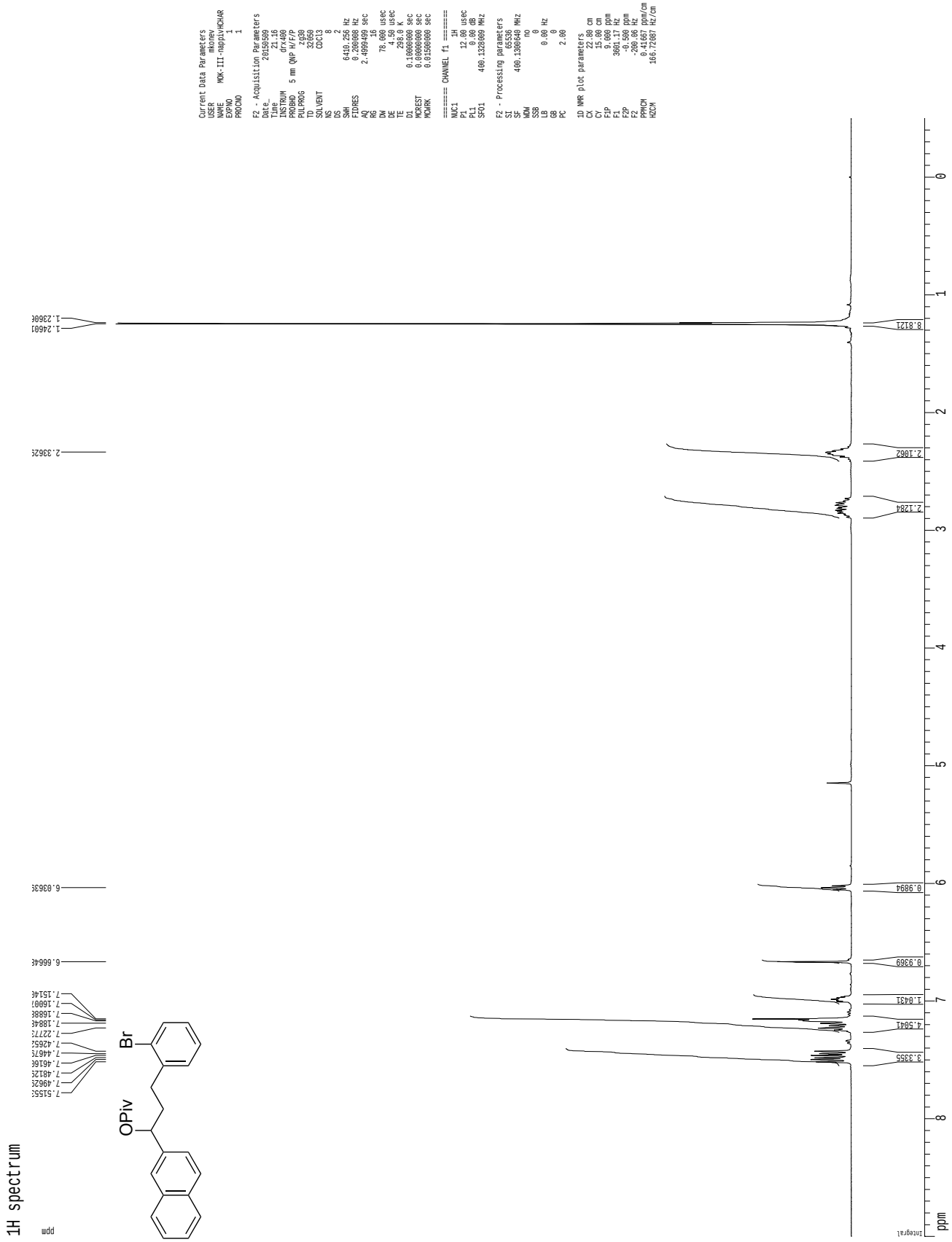
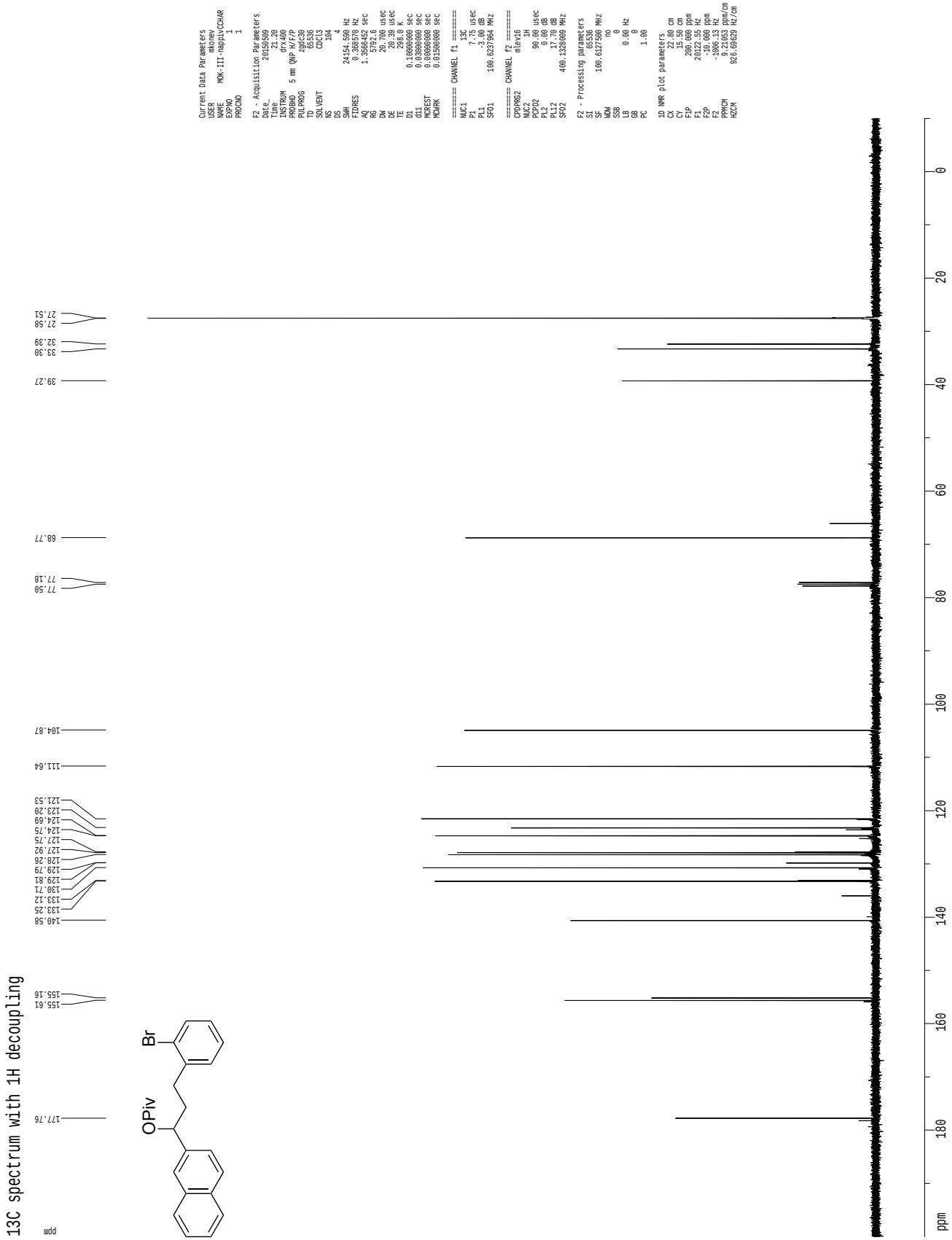
Current Data Parameters
USER thanma
NAME LEH-5-093
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150209
Time 20.02
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 456.1
DW 78.000 usec
DE 4.50 usec
TE 298.3 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300206 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 11.000 ppm
F1 4401.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.48246 ppm/cm
HZCM 193.04520 Hz/cm
Et2O
H2O
Et2O
Br
O
H
ppm
Integral

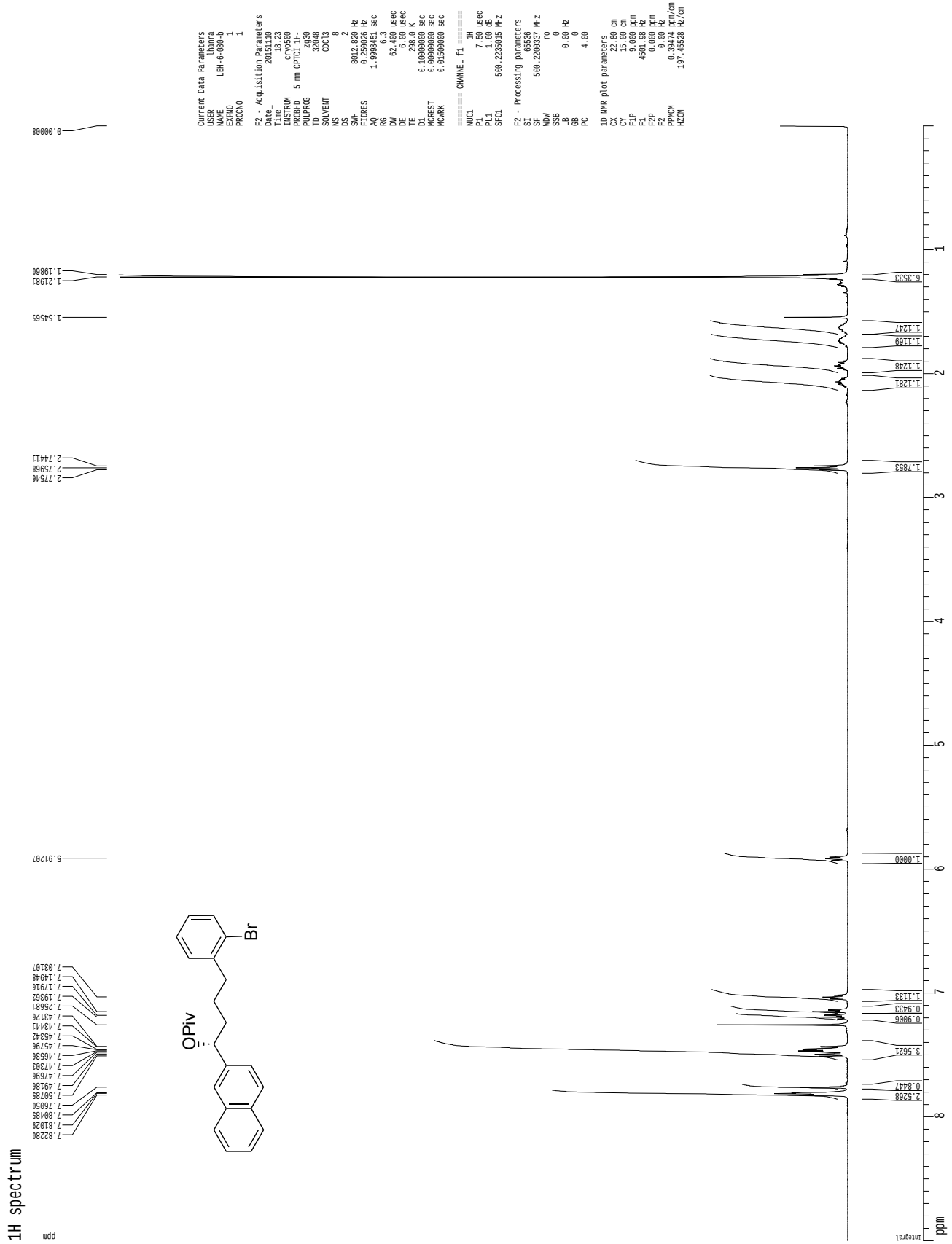

1H spectrum
Current Data Parameters
USER Thoma
NAME LEH-5-264-c1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150718
Time 15.03
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250093 Hz
AQ 1.9999700 sec
RG 256
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300227 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40240 ppm/cm
HZCM 193.04520 Hz/cm
Et2O
H2O
Et2O
Br
O
H
Integral
ppm

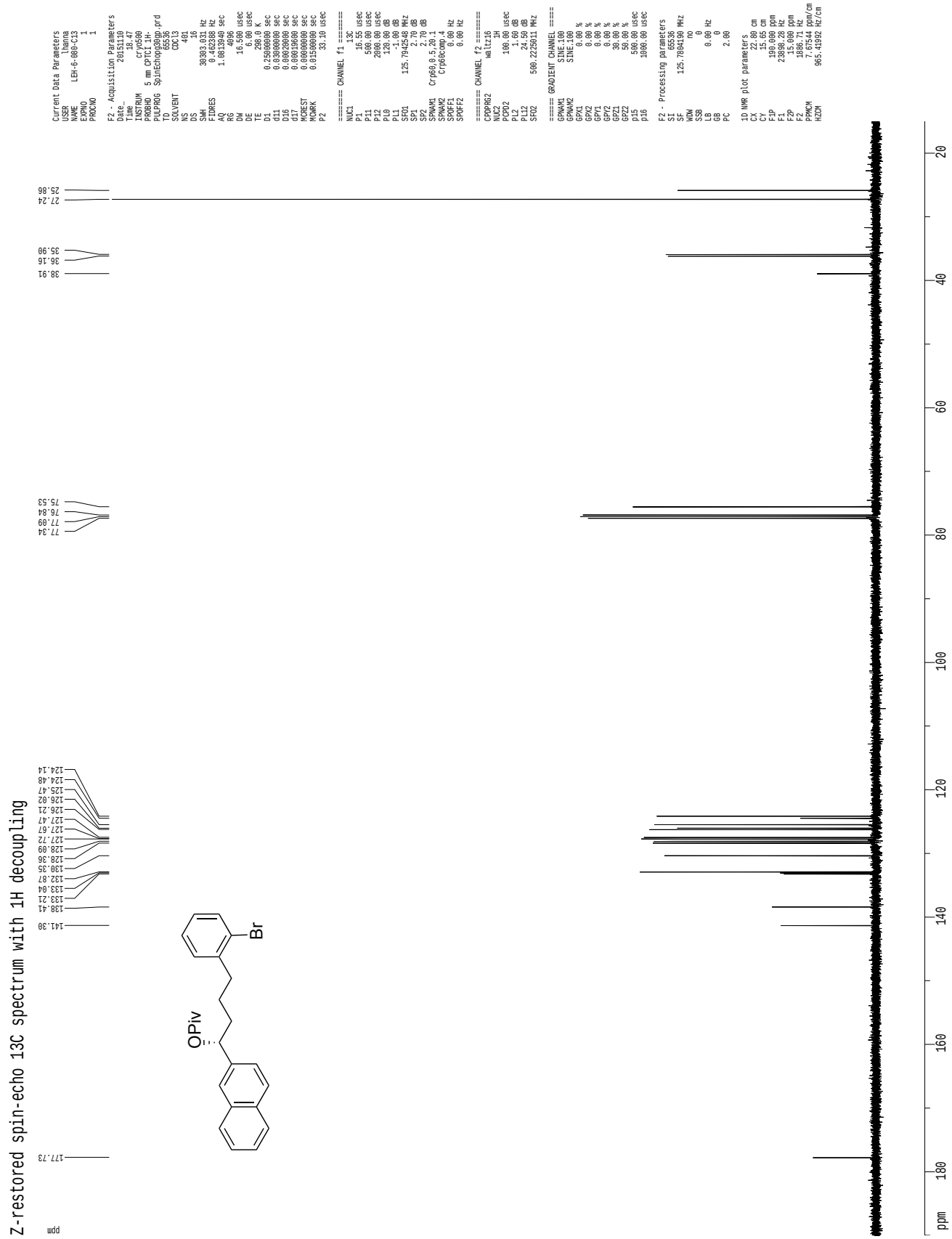

1H spectrum
Br
OH
ppm

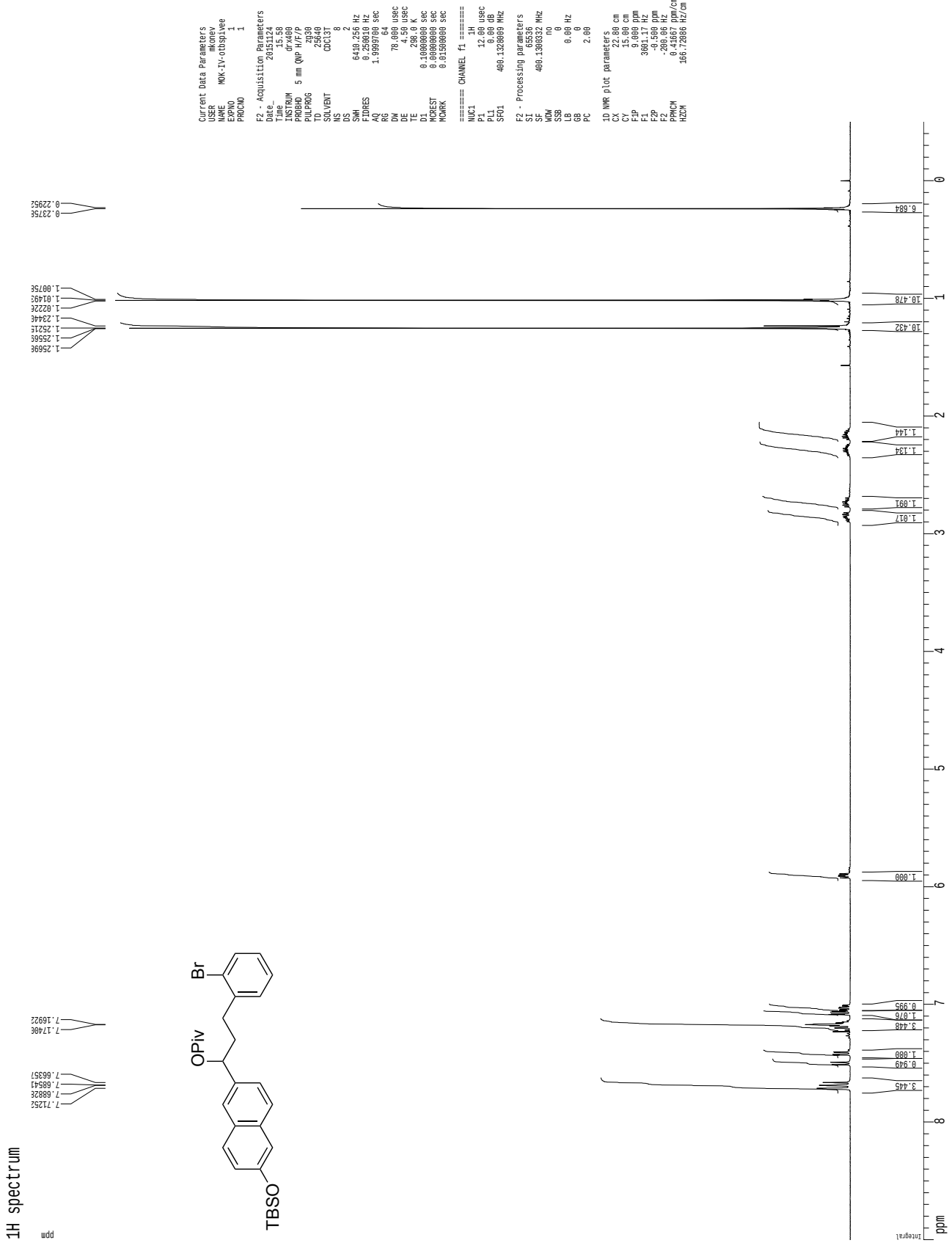

13C spectrum with 1H decoupling
Br
OH
ppm
Current Data Parameters
USER mkoney
NAME MOK-III-napothCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150511
Time 1.35
INSTRUM dru400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 680
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 91952
DW 20.700 usec
DE 20.30 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
====== CHANNEL f2 ======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
32.83
39.00
74.28
76.94
77.26
77.57
124.28
124.69
124.88
126.11
126.42
127.71
127.90
127.93
128.19
128.62
130.67
133.09
133.27
133.52
141.35
141.93
0
20
40
60
80
100
120
140
160
180
ppm

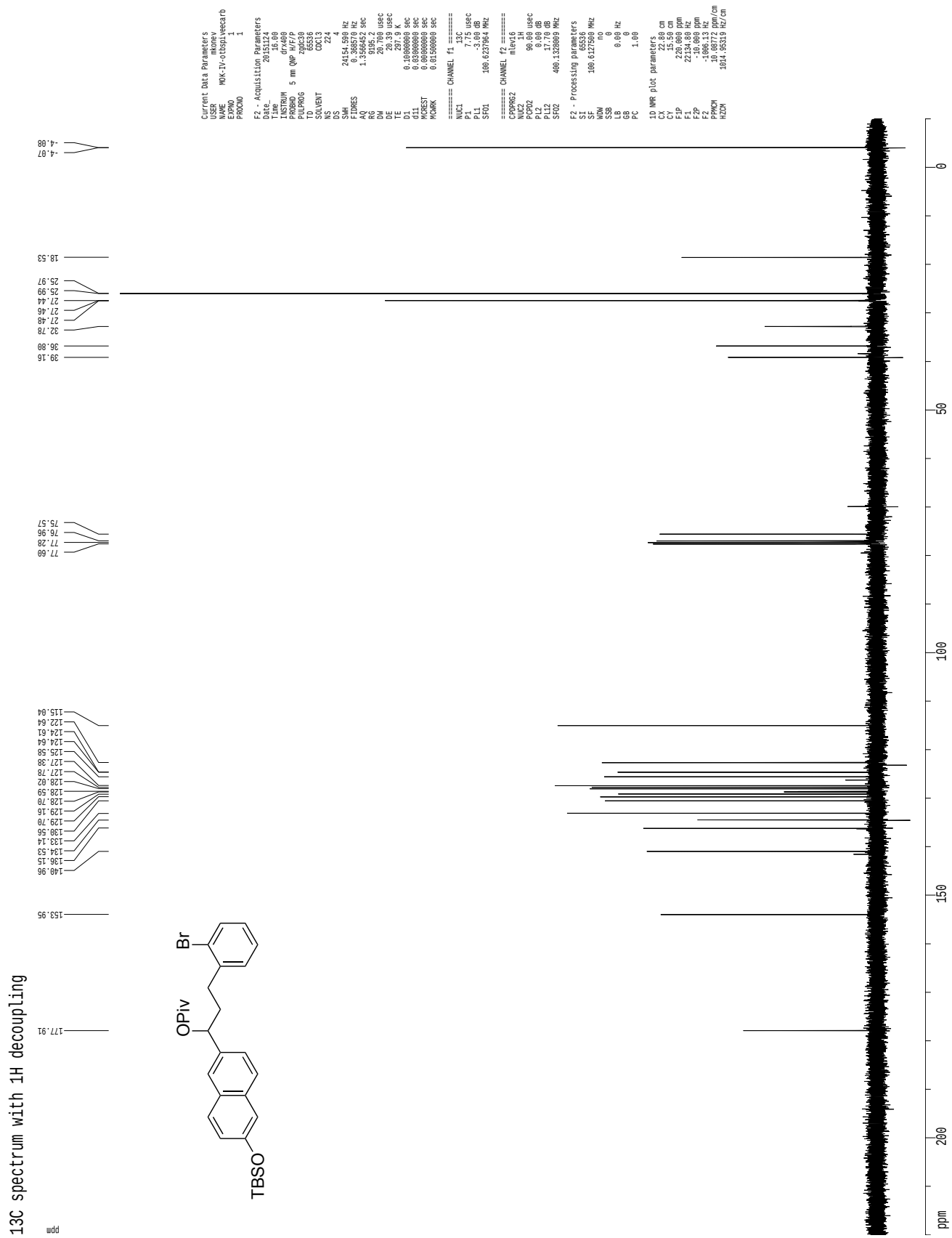

1H spectrum
ppm
OPiv
Br
Current Data Parameters
USER mkoney
NAME MOK-III-napiv4CHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 22.15
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32050
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.200008 Hz
AQ 2.4999499 sec
RG 16
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300040 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72087 Hz/cm
1.23606
1.24601
2.33626
6.03639
6.66646
7.15446
7.16001
7.16886
7.18846
7.22772
7.42652
7.44679
7.46166
7.48126
7.49626
7.51555
8.8121
7.1062
7.1284
0.9894
0.9369
1.0431
4.5041
3.3355
Integral
ppm


13C spectrum with 1H decoupling
ppm
Br
OPiv
177.76
155.61
155.16
140.58
133.25
133.13
130.71
129.81
129.79
128.26
127.92
127.75
124.75
124.69
123.20
121.53
111.64
104.87
77.50
77.18
68.77
39.27
33.30
32.39
27.58
27.51
Current Data Parameters
USER mkoney
NAME MOK-III-naptylCCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 22.20
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 104
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 5792.6
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.72653 ppm/cm
HZCM 926.68629 Hz/cm
0
20
40
60
80
100
120
140
160
180
ppm


1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-6-080-b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
2015i110
Time 18.23
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200337 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
0.00000
1.19866
1.21981
1.54565
2.74411
2.75966
2.77546
5.91207
7.03107
7.14948
7.17916
7.19362
7.25681
7.43126
7.43441
7.45342
7.45796
7.46536
7.47303
7.47696
7.49186
7.50785
7.76056
7.80485
7.81025
7.82286
6.35335
1.12477
1.11697
1.12487
1.12817
1.78537
1.00000
1.11337
0.94337
0.99067
3.56217
0.84477
7.52687
Integral
ppm
OPiv
Br


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
OPiv
Br
177.73
141.30
138.41
133.21
133.04
132.87
130.35
128.36
128.09
127.72
127.67
127.47
126.21
126.02
125.47
124.48
124.14
77.34
77.09
76.84
75.53
38.91
36.16
35.90
27.24
25.86
Current Data Parameters
USER Thanna
NAME LEH-6-080-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151110
Time 10.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 401
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 190.000 ppm
F2P 23898.28 Hz
F1 15.000 ppm
F2 1886.71 Hz
PPMCM 7.67544 ppm/cm
HZCM 965.41992 Hz/cm
20
40
60
80
100
120
140
160
180
ppm


1H spectrum
ppm
integral
Br
OPiv
TBSO
Current Data Parameters
USER mkonev
NAME MOK-IV-oiDsplvee
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151124
Time 15.58
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 64
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300032 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 200.05 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72806 Hz/cm
7.71252
7.68826
7.68541
7.66357
7.17406
7.16922
0.23756
0.22952
1.00756
1.01493
1.02226
1.23444
1.25215
1.25569
1.25698
6.684
10.478
10.432
1.144
1.134
1.091
1.017
1.000
0.995
1.076
3.448
1.080
0.949
3.445

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Current Data Parameters
USER      mkonev
NAME      MOK-IV-otbspivevecarb
EXPNO             1
PROCNO            1

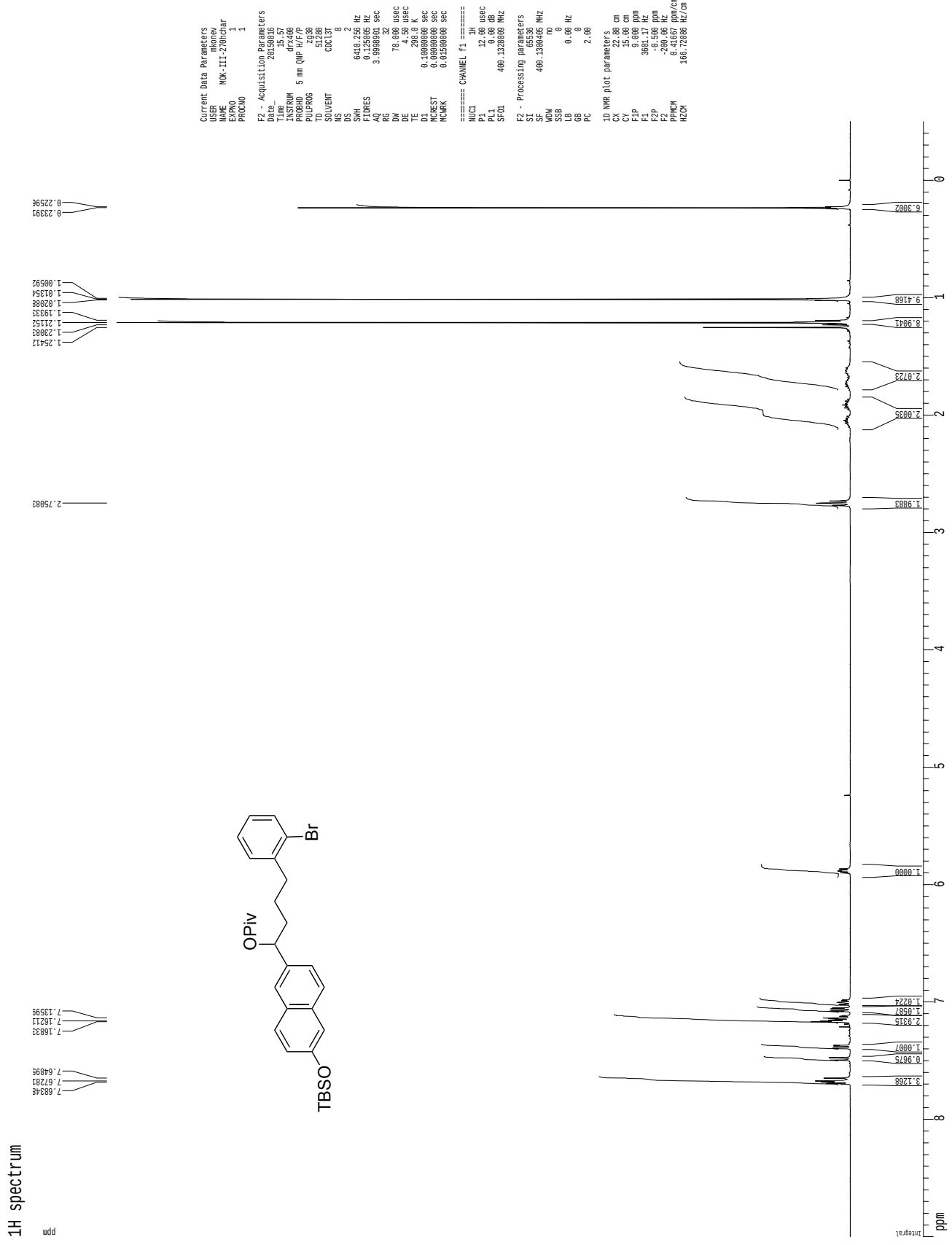
F2 - Acquisition Parameters
Date_      20151214
Time              16.60
INSTRUM         drx400
PROBHD  5 mm QNP H/F/P
PULPROG         zgpdc30
TD                65536
SOLVENT           CDCl3
NS                  224
DS                    4
SWH           24154.590 Hz
FIDRES        0.368570 Hz
AQ            1.3566452 sec
RG                9195.2
DW               20.700 usec
DE                6.50 usec
TE                297.9 K
D1            0.10000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

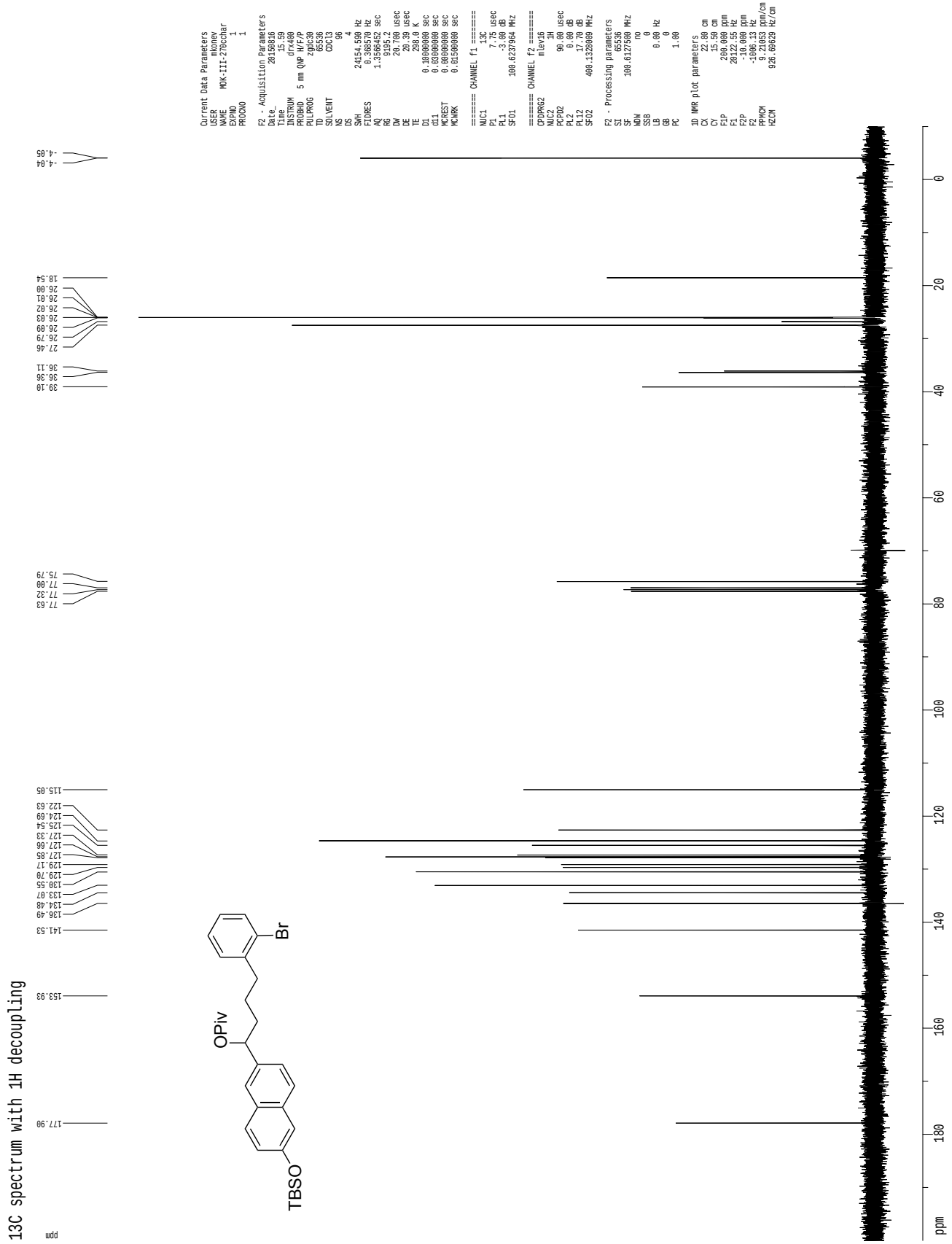
======= CHANNEL f1 =======
NUC1               13C
P1                7.75 usec
PL1               -3.00 dB
SFO1       100.6237964 MHz

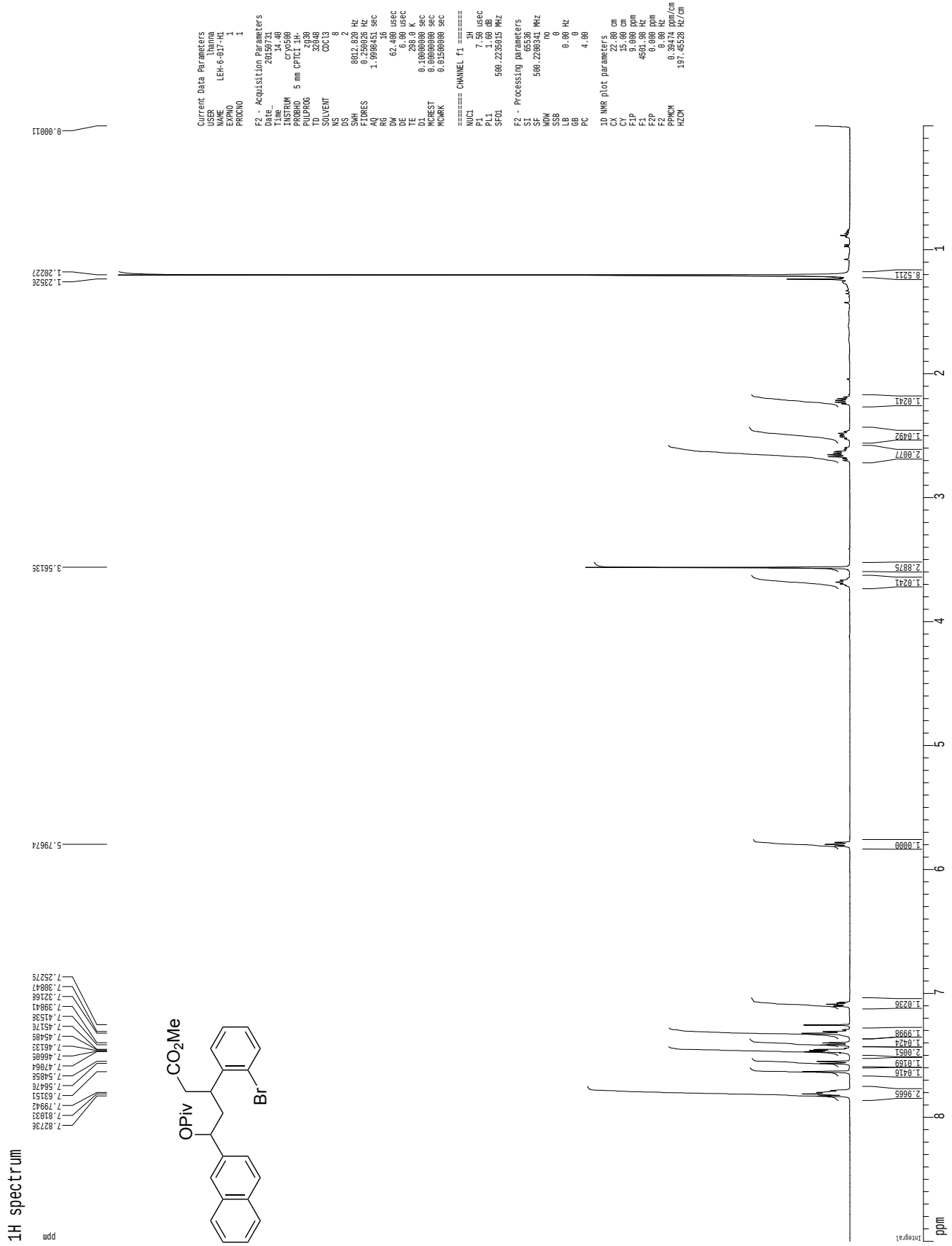
======= CHANNEL f2 =======
CPDPRG2         mlev16
NUC2               1H
PCPD2            90.00 usec
PL2               0.00 dB
PL12             17.70 dB
SFO2       400.1320009 MHz

F2 - Processing parameters
SI                65536
SF         100.6127680 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

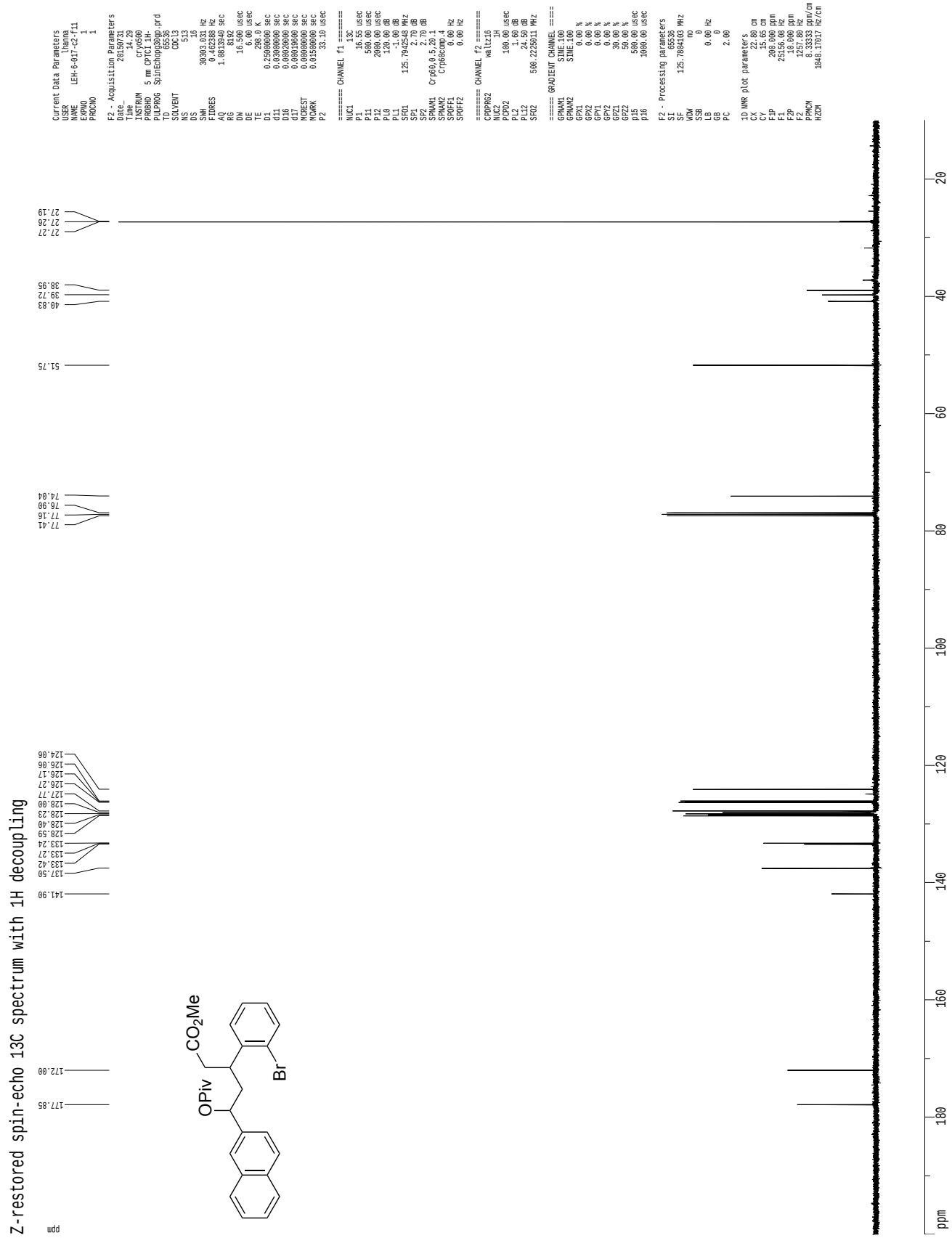
1D NMR plot parameters
CX               22.80 cm
CY               15.50 cm
F1P             220.000 ppm
F1            22134.80 Hz
F2P            -10.000 ppm
F2           -1006.13 Hz
PPMCM          10.00772 ppm/cm
HZCM           1014.95319 Hz/cm
```

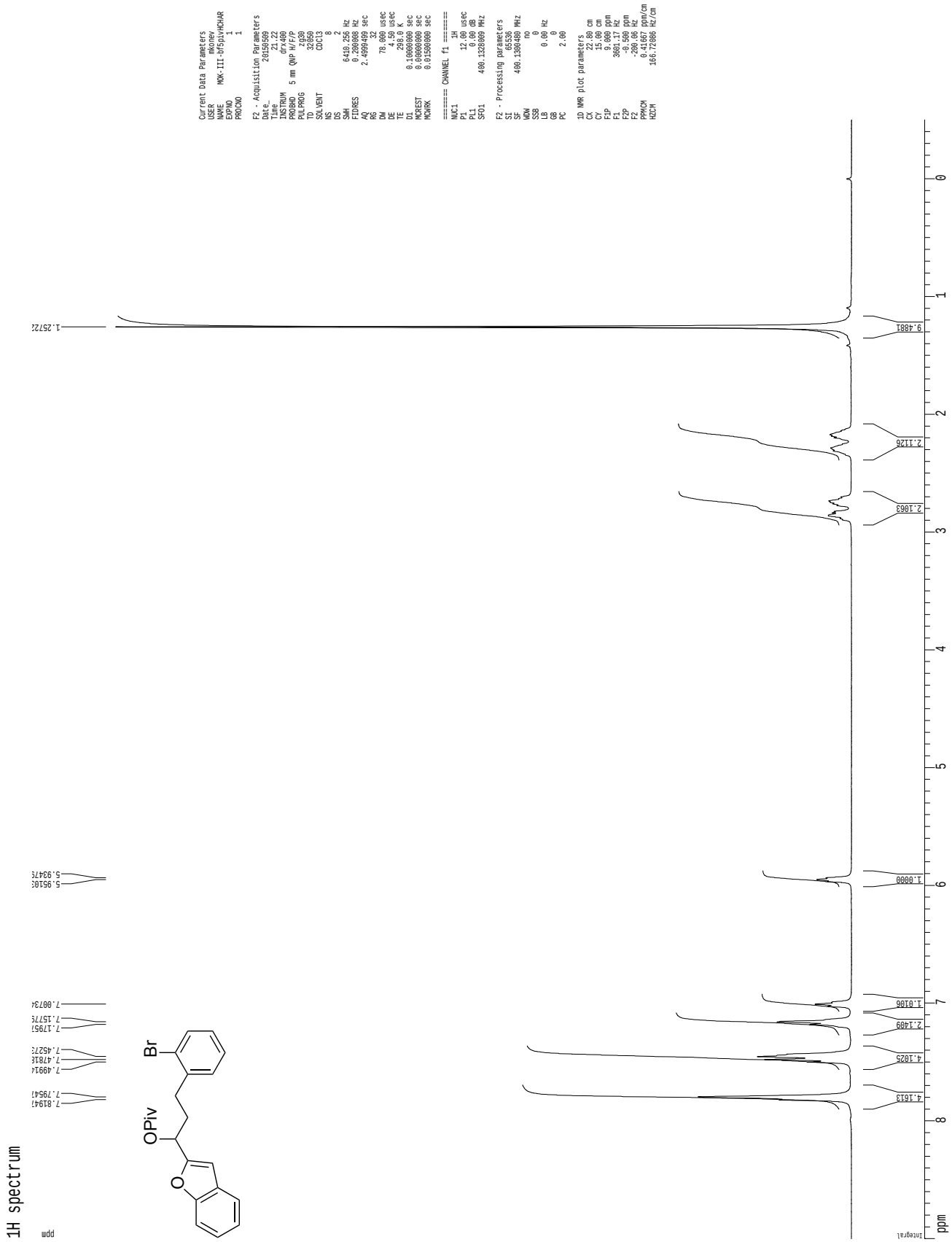

1H spectrum
Current Data Parameters
USER mvgray
NAME MOK-III-270mhchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150816
Time 15.57
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 51280
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.125095 Hz
AQ 3.9980895 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300405 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -200.00 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72080 Hz/cm
OPiv
Br
TBSO
ppm
Integral


13C spectrum with 1H decoupling
ppm
-4.04
-4.05
18.54
26.00
26.01
26.02
26.03
26.02
26.09
26.79
27.46
36.11
36.36
39.10
75.79
77.00
77.32
77.63
115.05
122.63
124.69
125.54
127.33
127.66
127.85
129.17
129.70
130.55
133.07
134.48
136.49
141.53
153.93
177.90
OPiv
Br
TBSO
Current Data Parameters
USER mhoren
NAME MOK-III-270char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150816
Time 15.59
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 6912
DW 20.700 usec
DE 20.30 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlevi6
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328000 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -4.110 ppm
F2 -1106.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
ppm
Integral
OPiv
CO2Me
Br

Z-restored spin-echo 13C spectrum with 1H decoupling



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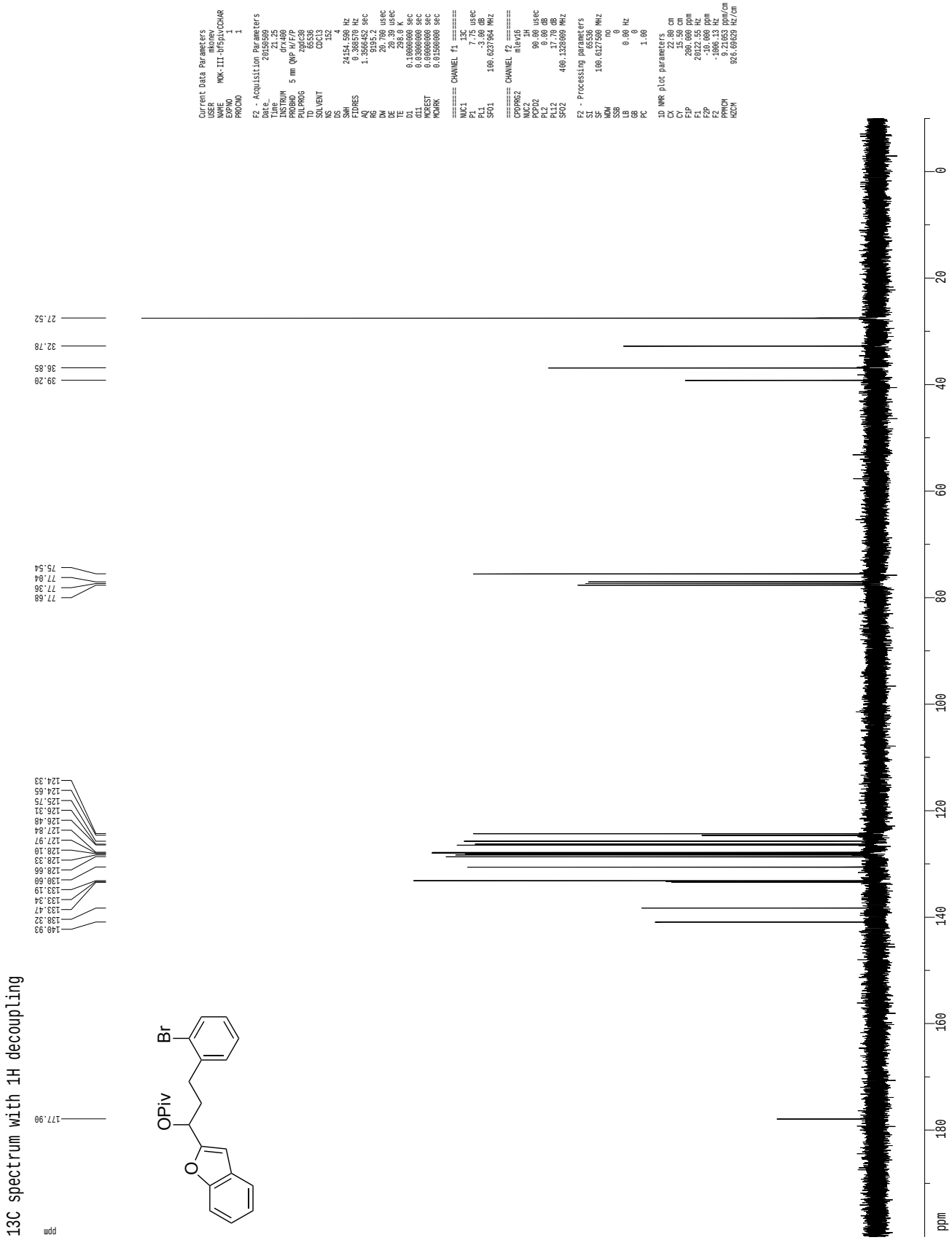
Current Data Parameters
USER      mkoney
NAME      MOK-III-bf5pivHCHAR
EXPNO         1
PROCNO        1

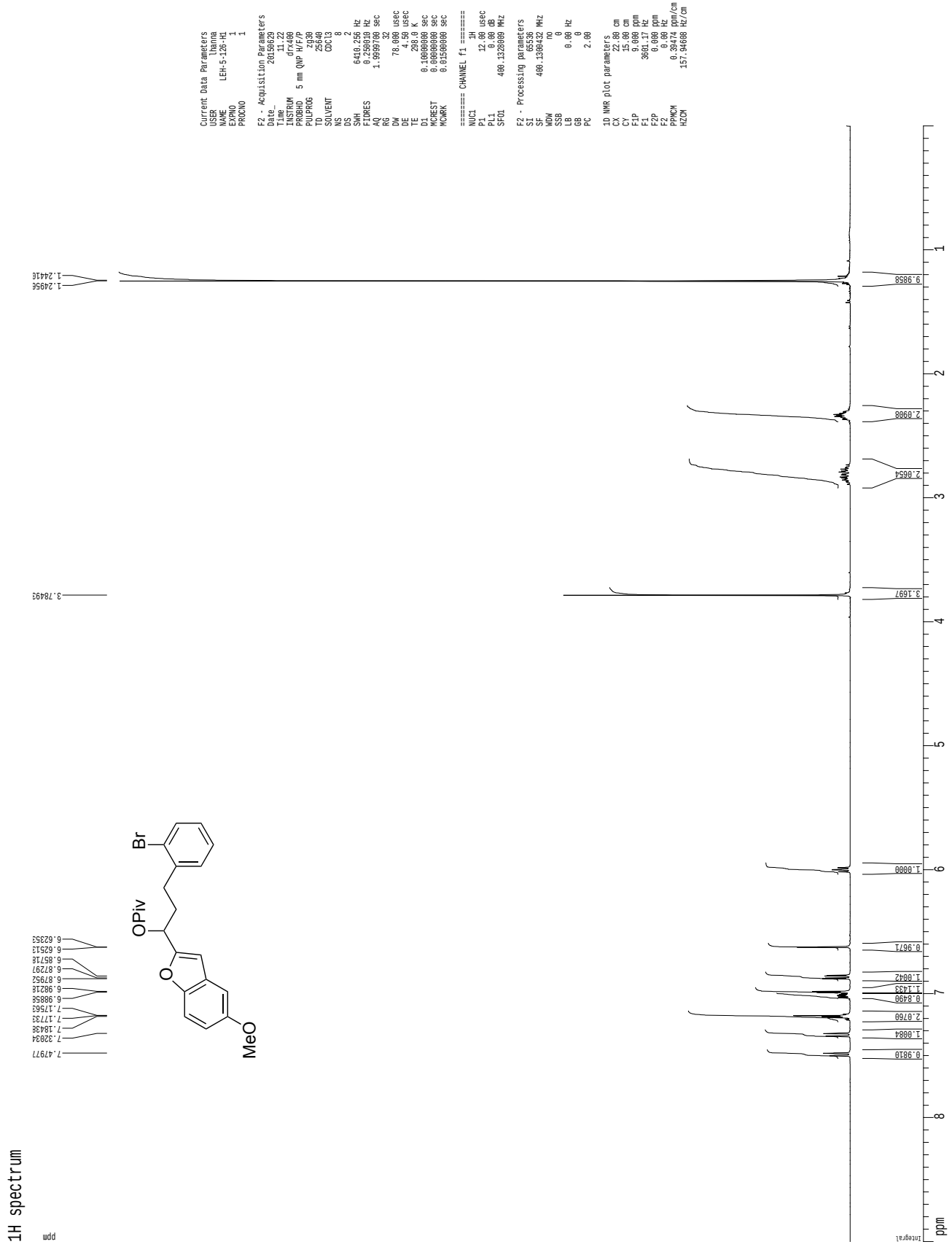
F2 - Acquisition Parameters
Date_      20150909
Time       21.22
INSTRUM      dfx400
PROBHD  5 mm QNP H/F/P
PULPROG      zg30
TD           32050
SOLVENT      CDCl3
NS               8
DS               2
SWH         6410.256 Hz
FIDRES      0.200008 Hz
AQ          2.4999499 sec
RG              32
DW           78.000 usec
DE            4.50 usec
TE           298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1             1H
P1           12.00 usec
PL1           0.00 dB
SFO1     400.1320009 MHz

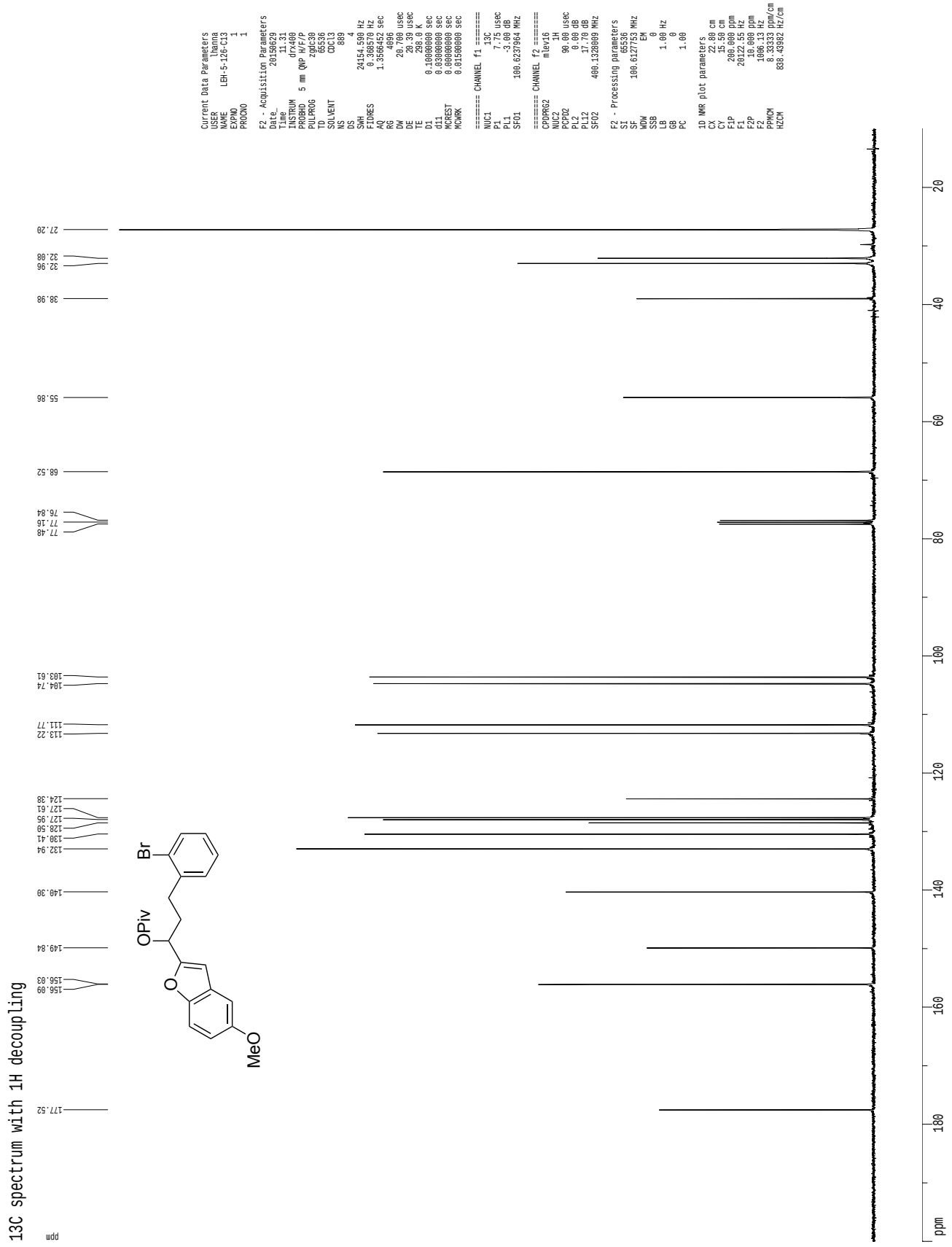
F2 - Processing parameters
SI            65536
SF       400.1300480 MHz
WDW              no
SSB               0
LB             0.00 Hz
GB                0
PC             2.00

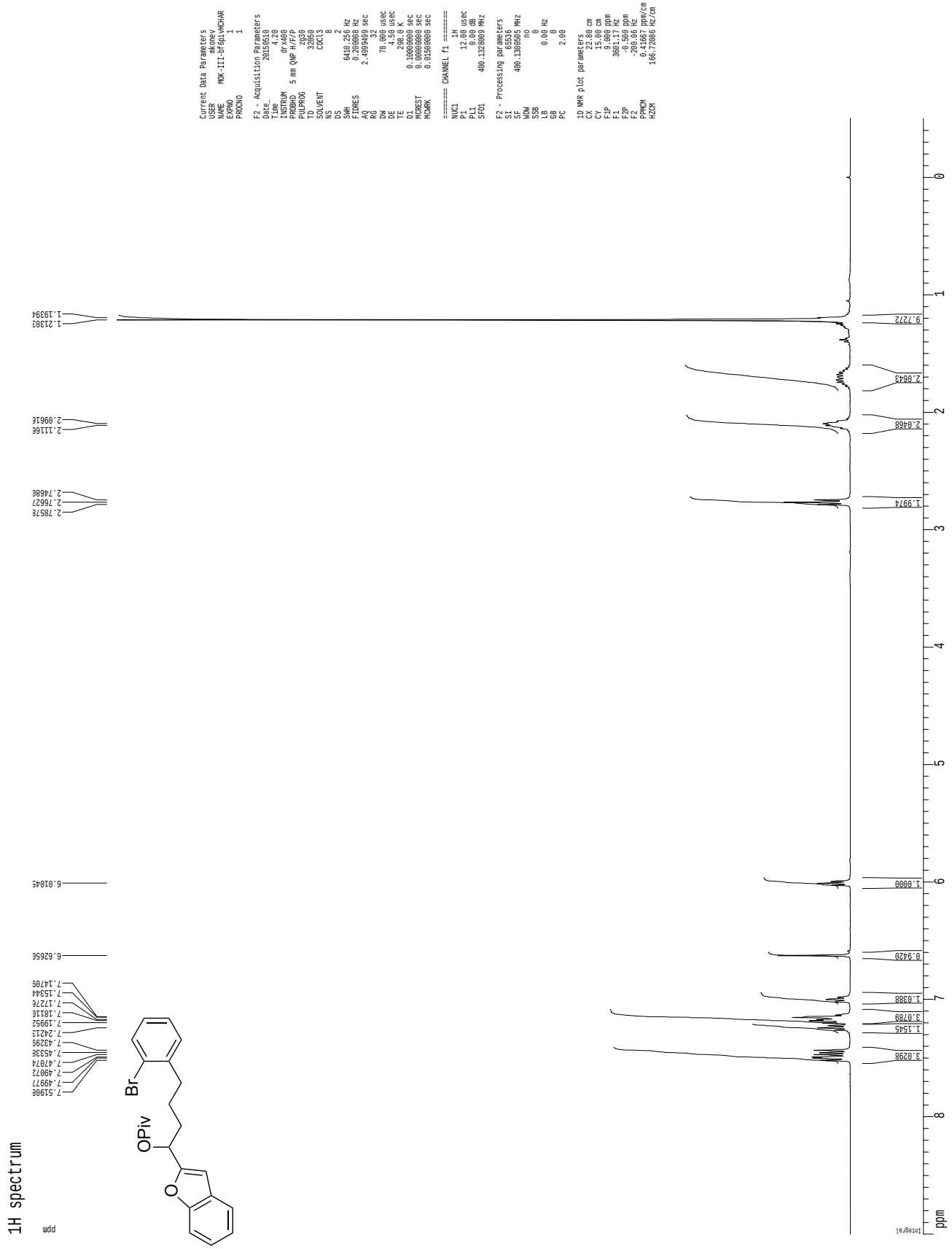
1D NMR plot parameters
CX           22.80 cm
CY           15.00 cm
F1P           9.0000 ppm
F1         3601.17 Hz
F2P          -0.500 ppm
F2         -200.06 Hz
PPMCM         0.41667 ppm/cm
HZCM        166.72086 Hz/cm
```

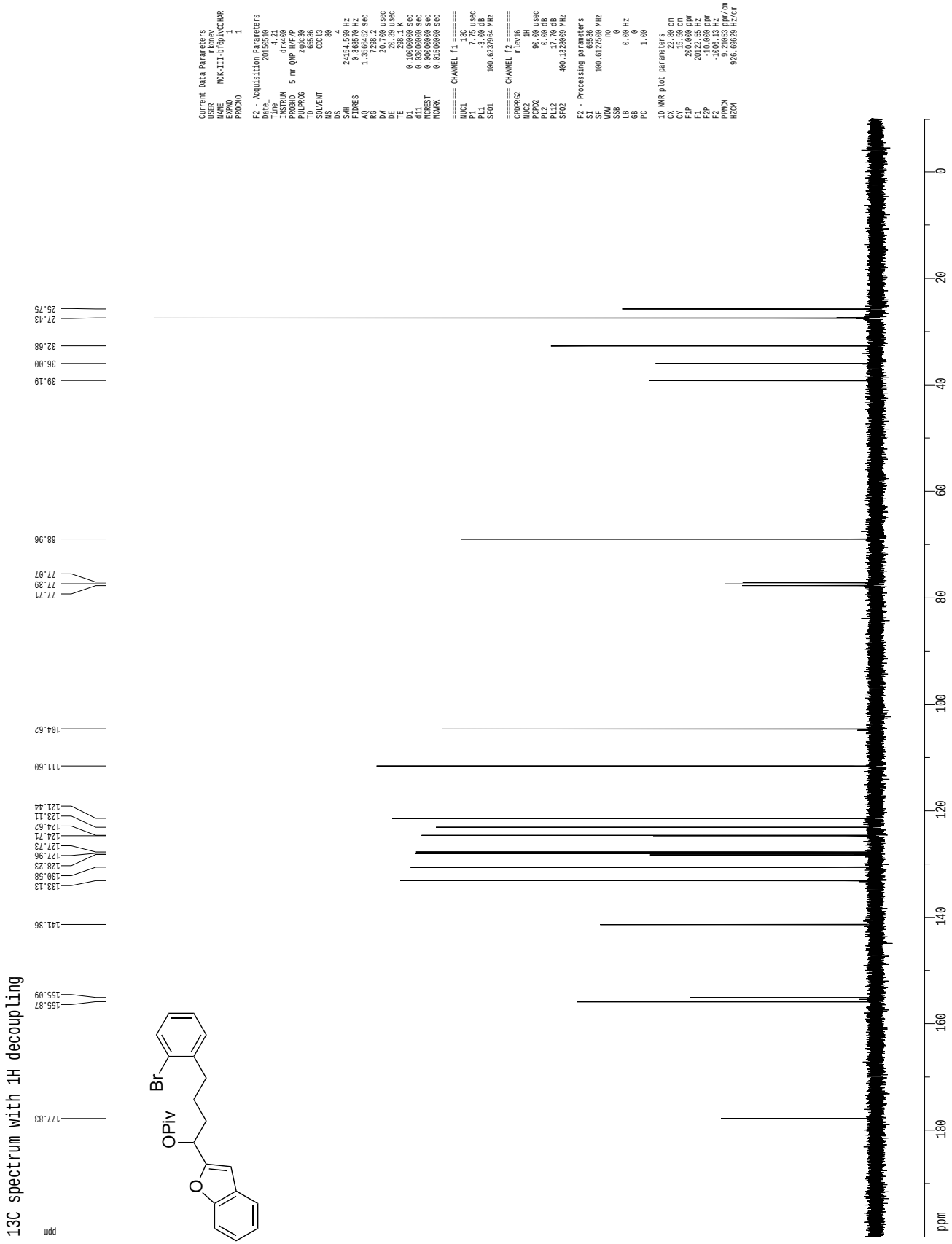

13C spectrum with 1H decoupling
ppm
Br
OPiv
O
177.90
140.93
138.92
133.47
133.34
133.19
130.60
128.66
128.33
128.10
127.97
127.84
126.48
126.31
125.75
124.65
124.33
77.68
77.36
77.04
75.54
39.20
36.85
32.78
27.52
Current Data Parameters
USER mkoney
NAME MOK-III-bf5pivCCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150909
Time 22.25
INSTRUM dfrx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 152
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 0.72653 ppm/cm
HZCM 926.68629 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
Current Data Parameters
USER Ilyana
NAME LEH-5-126-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150629
Time 11.22
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250163 Hz
AQ 1.9999799 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 16555
SF 400.1300432 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94608 Hz/cm
1.24416
1.24956
3.78493
7.47977
7.32034
7.18438
7.17733
7.17563
6.98856
6.98218
6.87952
6.87297
6.85718
6.62513
6.62353
9.9858
2.0908
2.0654
3.1697
1.0000
0.9671
1.0042
1.1433
0.8490
2.0760
1.0084
0.9810
Br
OPiv
O
MeO
ppm
Integral

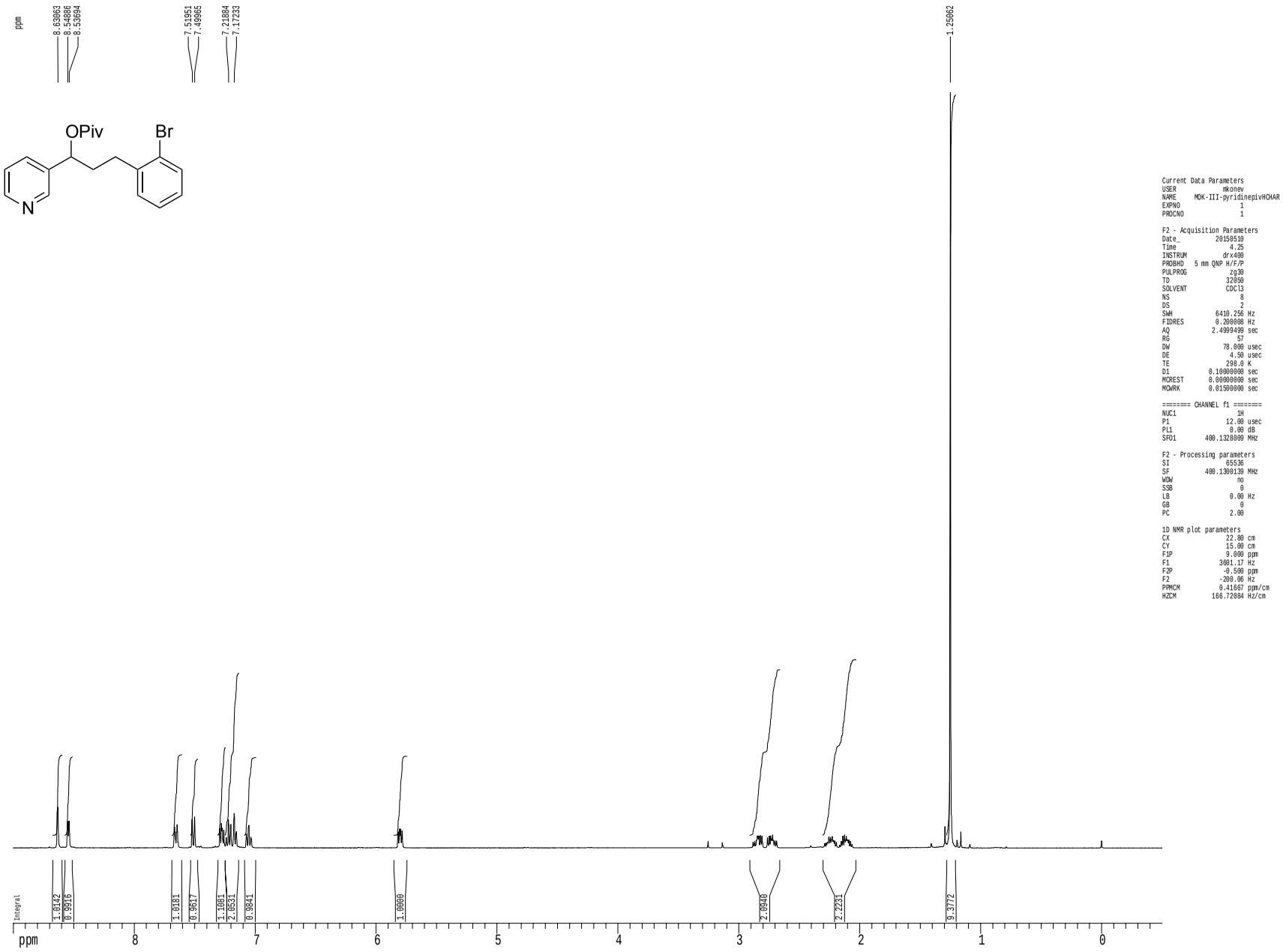
13C spectrum with 1H decoupling




1H spectrum
Br
OPiv
O
ppm
Integral
Current Data Parameters
USER mkoney
NAME MOX-III-bf6pivMeCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150610
Time 17.43
INSTRUM dr400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32050
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.200080 Hz
AQ 2.4999499 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300005 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72066 Hz/cm
1.19394
1.21302
2.09616
2.11166
2.74686
2.76627
2.78578
6.01045
6.62656
7.14708
7.15344
7.17276
7.18116
7.19952
7.24213
7.42298
7.45338
7.47074
7.49072
7.49977
7.51908
9.7272
2.0643
2.0468
1.9974
1.0000
0.9420
1.0388
3.0789
1.1545
3.0298


13C spectrum with 1H decoupling
Current Data Parameters
USER mkonev
NAME MOK-III-bf6pivOCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150510
Time 17.21
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 80
DS 4
SWH 24154.589 Hz
FIDRES 0.368578 Hz
AQ 1.3566452 sec
RG 7208.2
DW 20.700 usec
DE 20.30 usec
TE 298.1 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328809 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 5.21053 ppm/cm
HZCM 926.60629 Hz/cm
25.75
27.43
32.68
36.00
39.19
68.96
77.07
77.39
77.71
104.62
111.60
121.44
123.11
124.62
124.71
127.73
127.96
128.23
130.58
133.13
141.36
155.09
155.87
177.83
ppm
0
20
40
60
80
100
120
140
160
180
Br
OPiv
O

1H spectrum

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Current Data Parameters
USER              mkonev
NAME      MDK-III-pyridinepivHCHAR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150510
Time                4.25
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 32050
SOLVENT             CDCl3
NS                     8
DS                     2
SWH             6410.256 Hz
FIDRES           0.200008 Hz
AQ              2.4999499 sec
RG                    57
DW                78.000 usec
DE                 4.50 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======= CHANNEL f1 =======
NUC1                  1H
P1                 12.00 usec
PL1                 0.00 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300139 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.00 cm
CY                 15.00 cm
F1P                9.000 ppm
F1               3601.17 Hz
F2P               -0.500 ppm
F2               -200.06 Hz
PPMCM             0.41667 ppm/cm
HZCM            166.72084 Hz/cm
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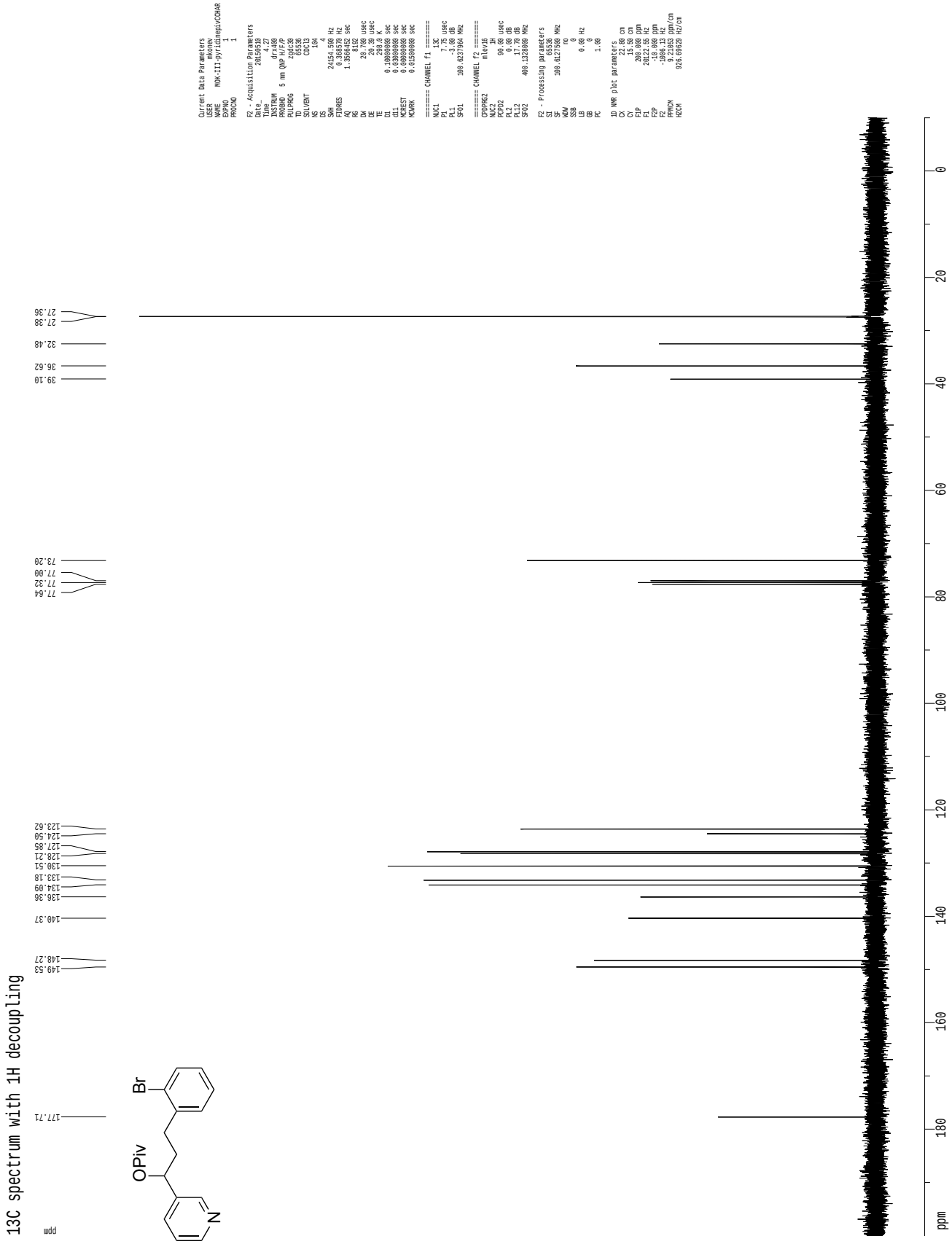
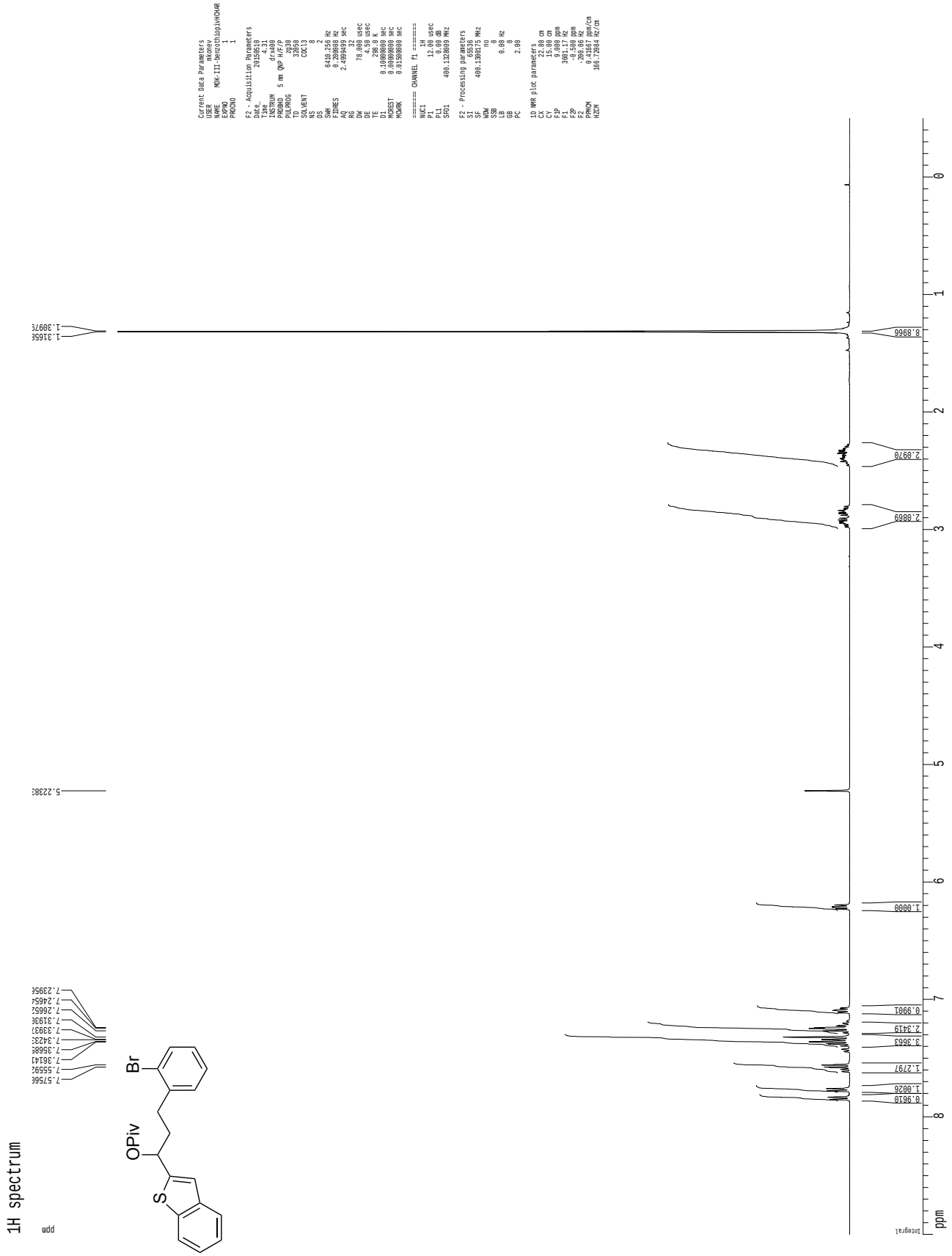
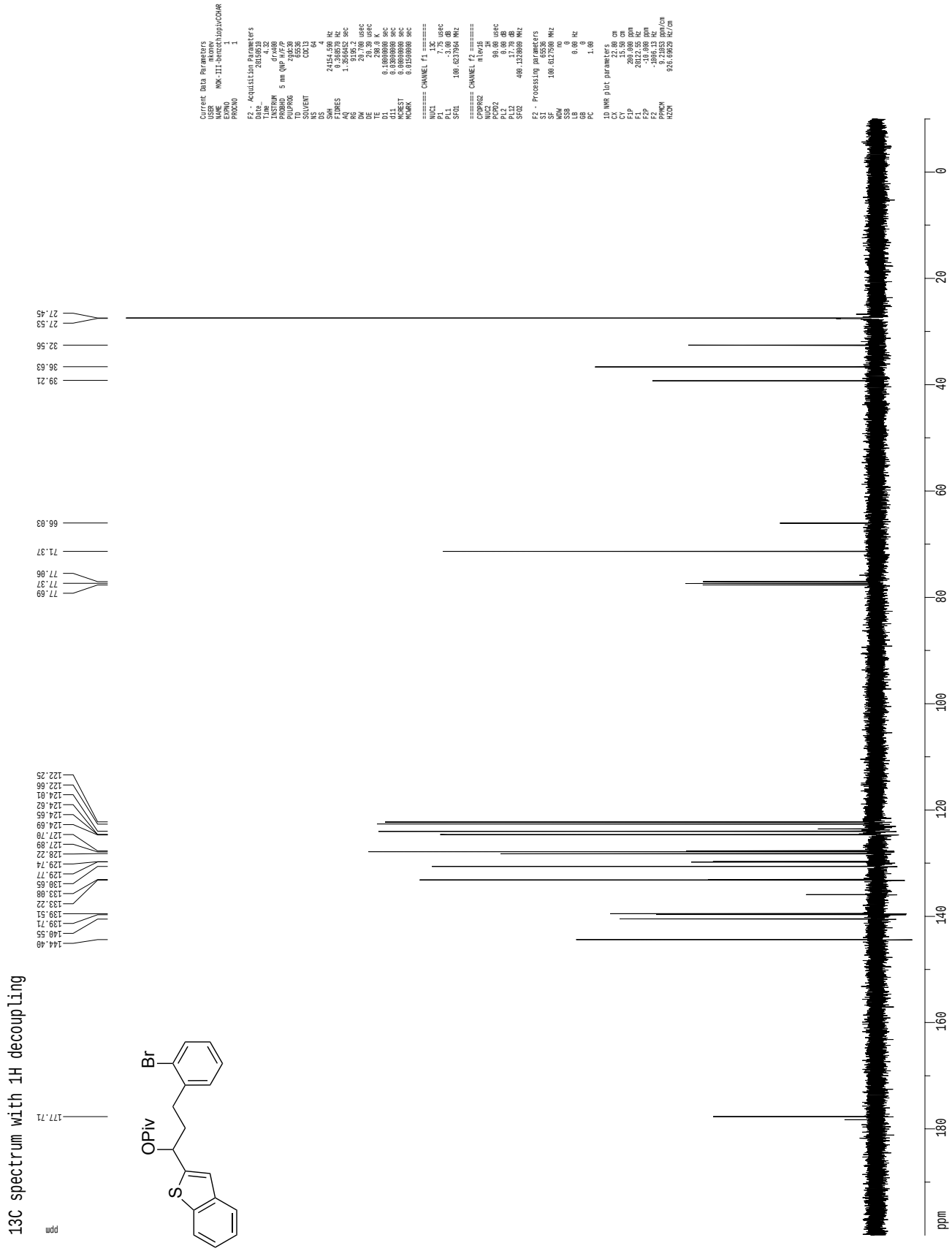
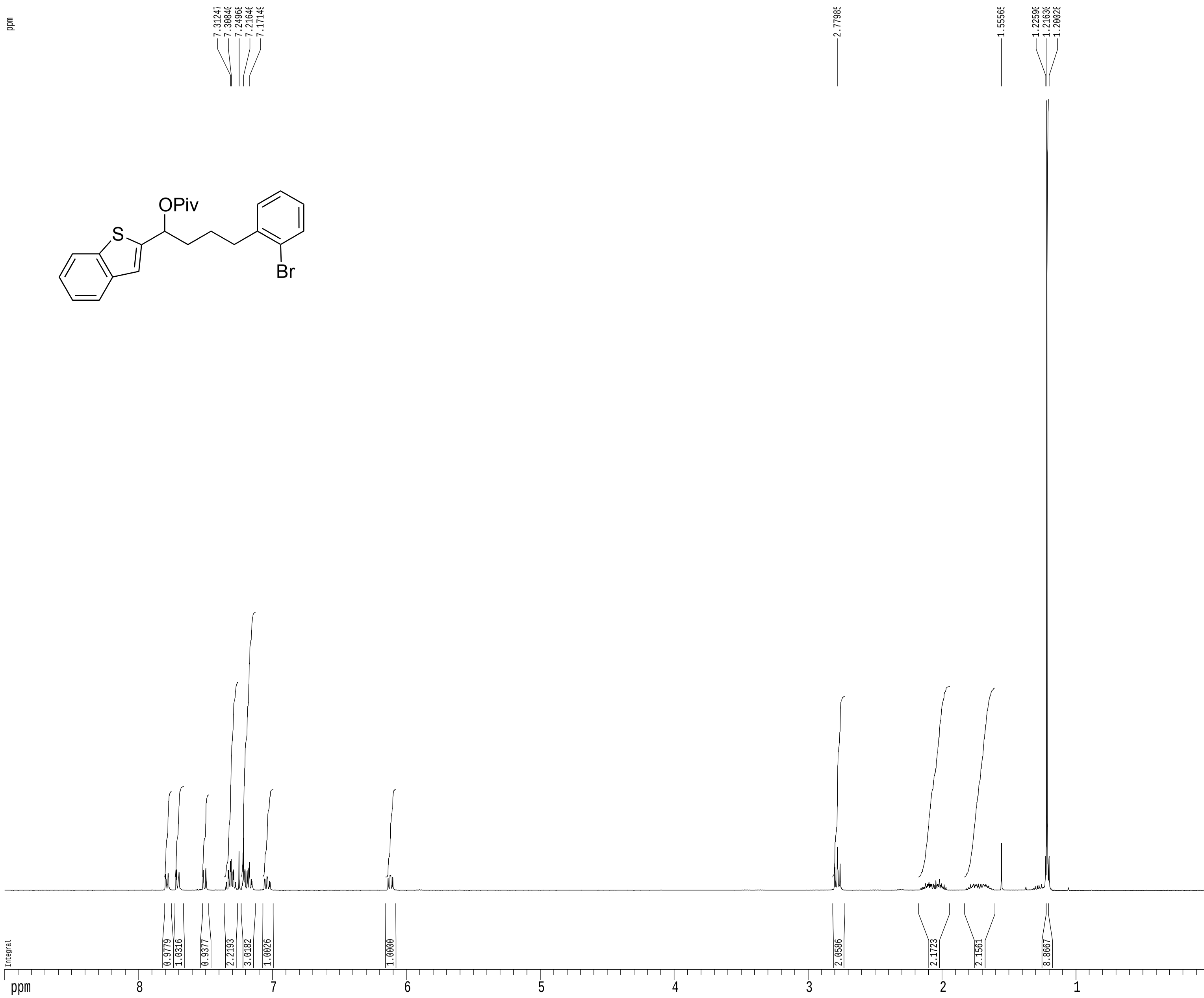

13C spectrum with 1H decoupling
ppm
Br
OPiv
N
177.71
149.53
148.27
140.37
136.36
134.09
133.18
130.51
128.21
127.85
124.50
123.62
77.64
77.32
77.00
73.20
39.10
36.62
32.48
27.38
27.36
Current Data Parameters
USER mkohlev
NAME MOK-III-pyridinepivCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150618
Time 4.27
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 104
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566432 sec
RG 18.02
DW 20.700 usec
DE 20.30 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328000 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21453 ppm/cm
HZCM 926.69629 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
Br
OPiv
S
ppm
integral
Current Data Parameters
USER mkomrev
NAME MOK-III-benzothiophVHCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150610
Time 4.31
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32859
SOLVENT CDCl3
NS 2
DS 2
SWH 6410.256 Hz
FIDRES 0.293038 Hz
AQ 2.4896640 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300115 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.12 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72884 Hz/cm
7.57566
7.55592
7.36141
7.35668
7.34236
7.33931
7.31936
7.26652
7.24654
7.23956
5.22383
1.31656
1.30976
8.8966
2.0970
2.0869
1.0000
0.9901
2.3419
3.3663
1.2797
1.0026
0.9610


13C spectrum with 1H decoupling
ppm
Br
OPiv
S
177.71
144.40
140.55
139.71
139.51
133.22
133.08
130.65
129.77
129.74
128.22
127.89
127.70
124.69
124.65
124.62
124.01
122.66
122.25
77.69
77.37
77.06
71.37
66.03
39.21
36.63
32.56
27.53
27.45
0
20
40
60
80
100
120
140
160
180
ppm

1H spectrum

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Current Data Parameters
USER              lhanna
NAME      LEH-6-025-c2-f32
EXPNO                  1
PROCNO                 1

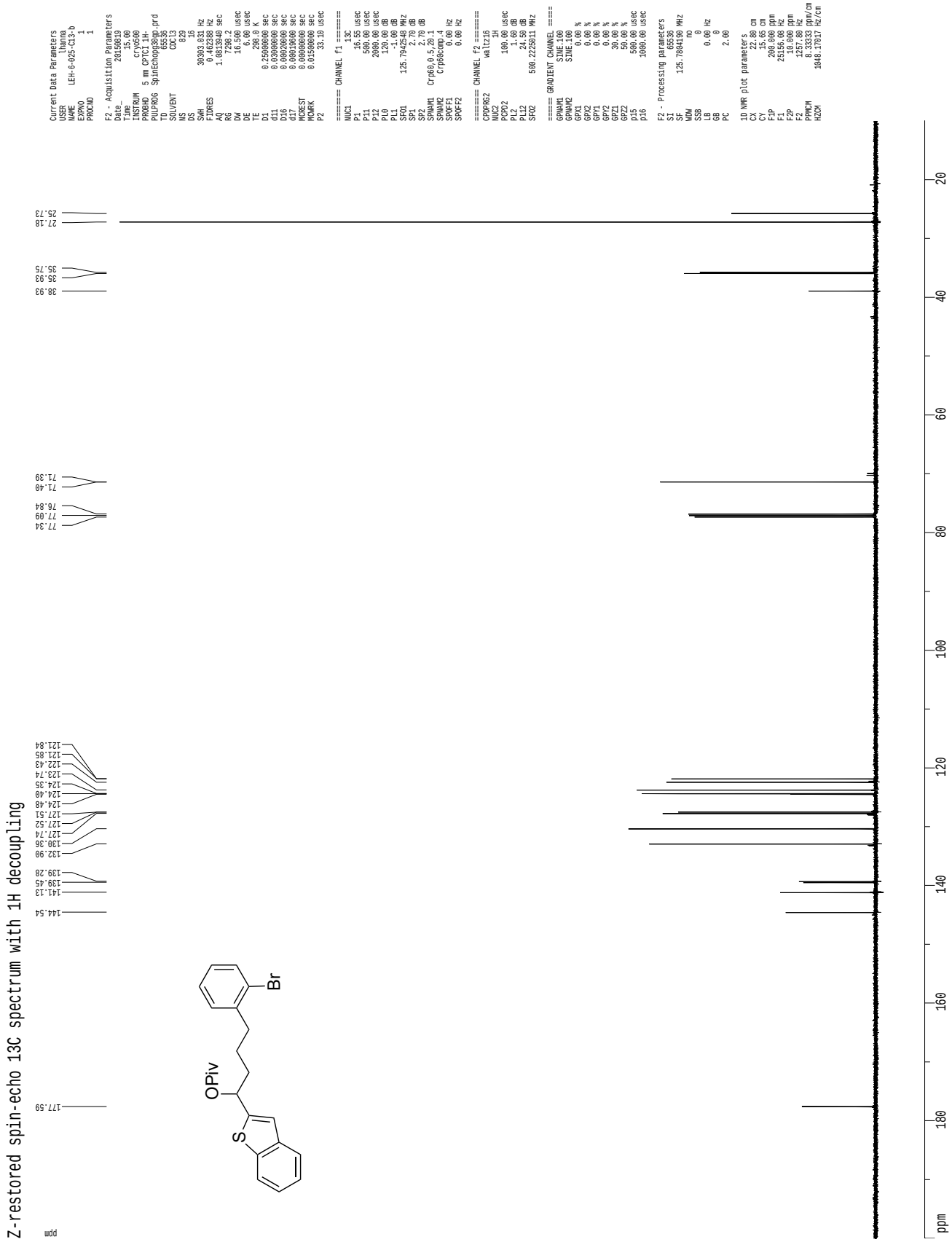
F2 - Acquisition Parameters
Date_           20150818
Time               20.25
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 25640
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6410.256 Hz
FIDRES          0.250010 Hz
AQ             1.9999700 sec
RG                   256
DW                78.000 usec
DE                  4.50 usec
TE                 298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                 0.00 dB
SFO1         400.1328009 MHz

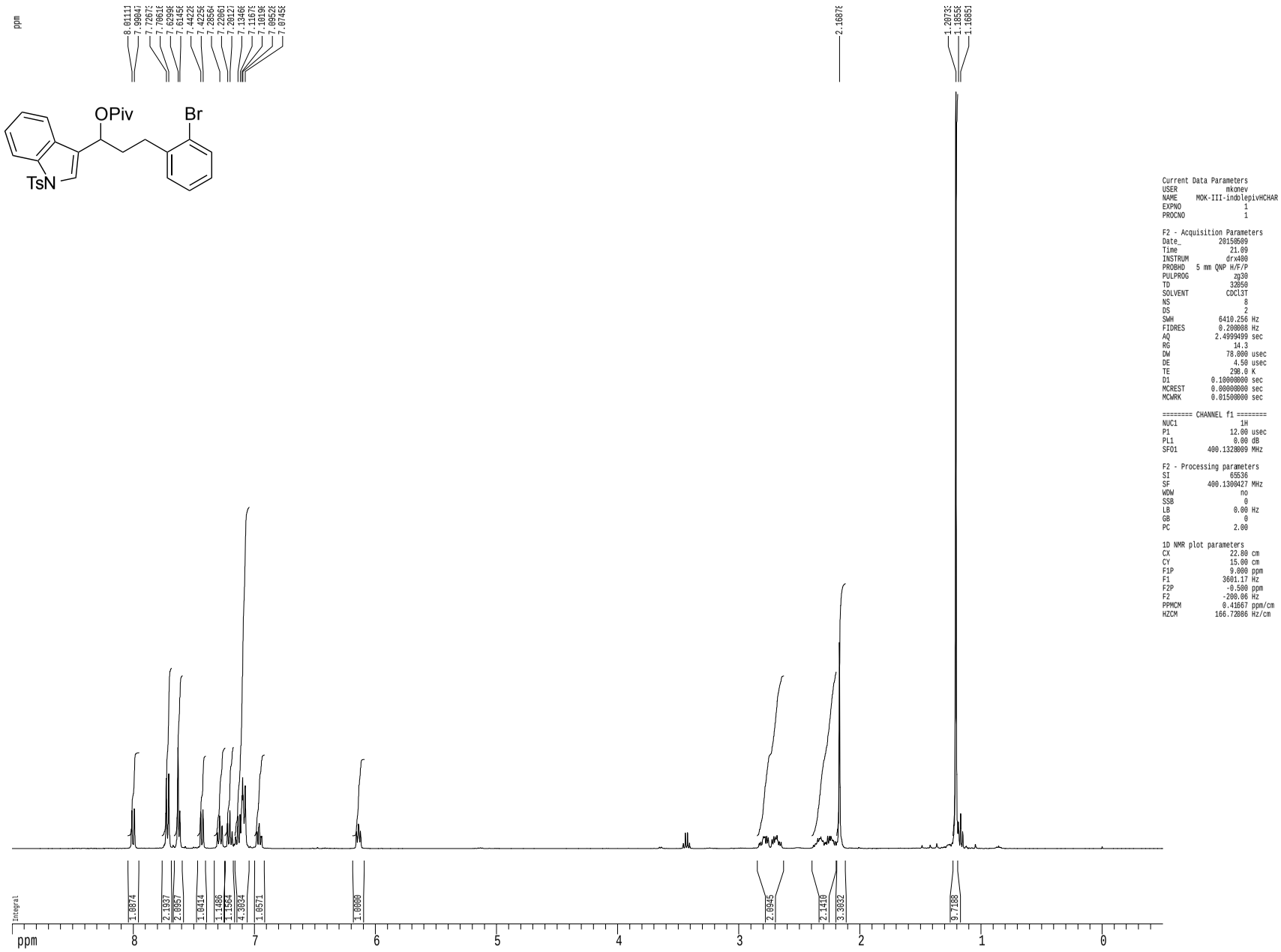
F2 - Processing parameters
SI                 65536
SF           400.1300256 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P                9.000 ppm
F1               3601.17 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.39474 ppm/cm
HZCM           157.94608 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



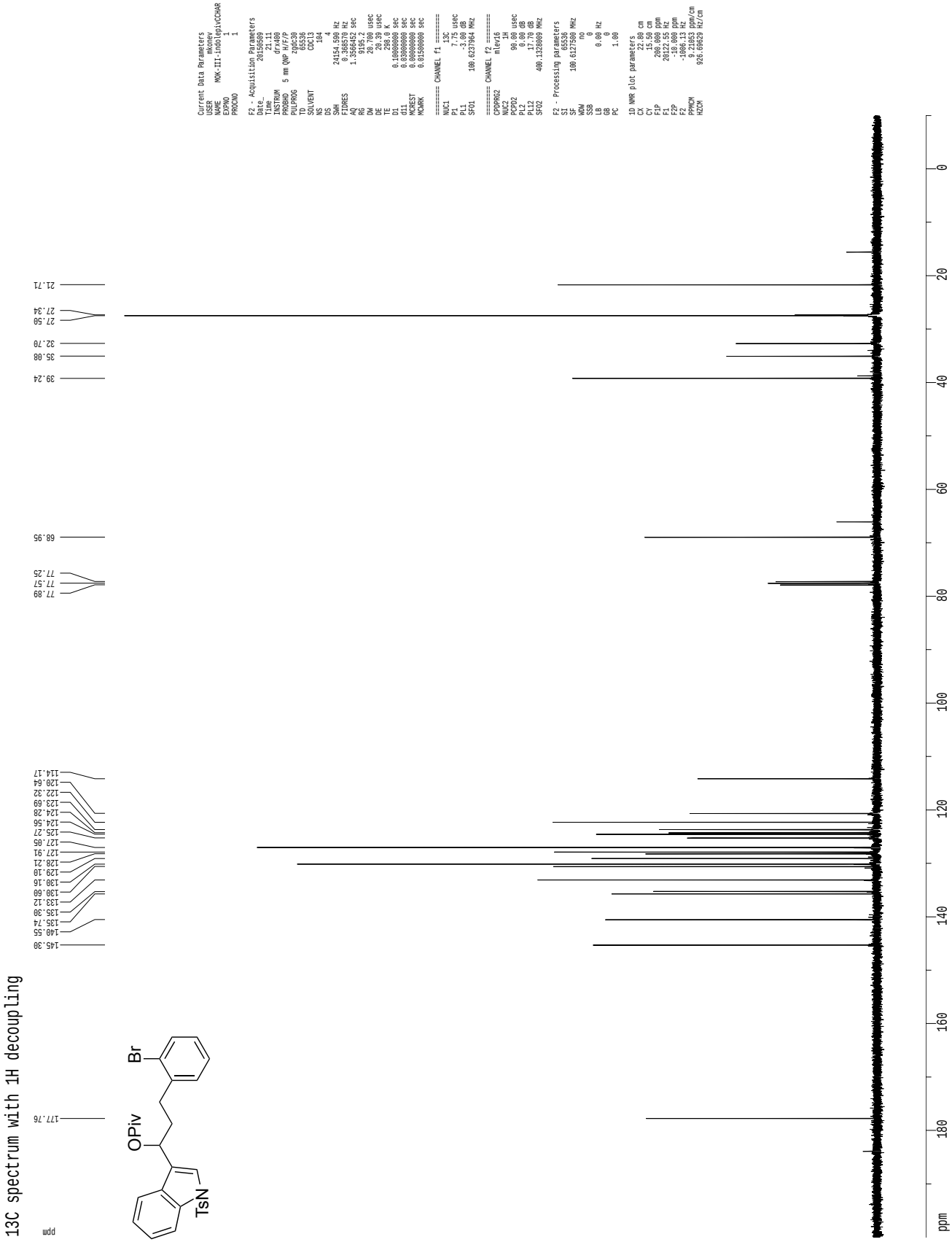

```
Current Data Parameters
USER            mkonev
NAME      MOK-III-indolepivHCHAR
EXPNO                1
PROCNO               1

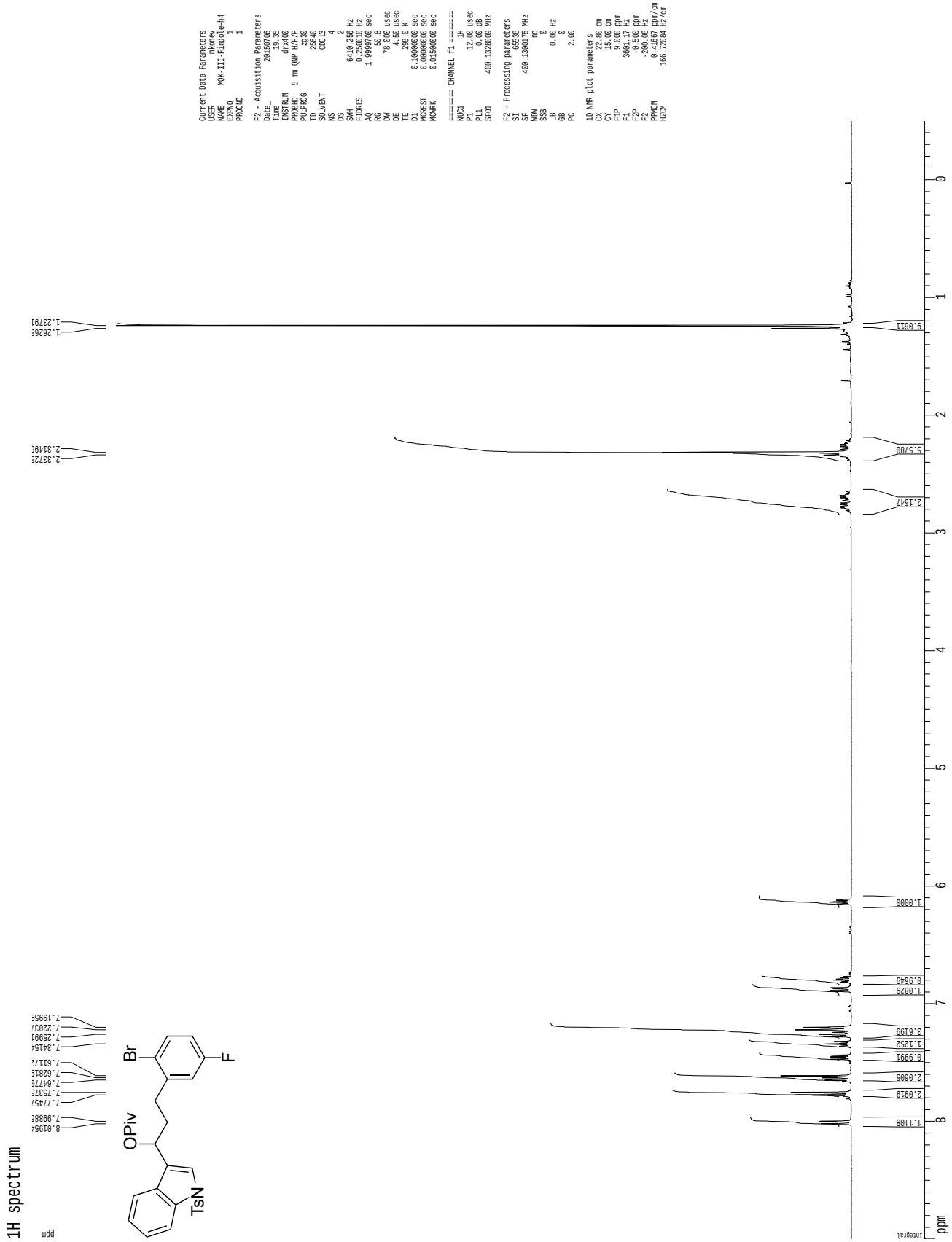
F2 - Acquisition Parameters
Date_         20150509
Time              21.09
INSTRUM         drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zg30
TD              32050
SOLVENT         CDCl3T
NS                  8
DS                  2
SWH           6410.256 Hz
FIDRES        0.200008 Hz
AQ            2.4999499 sec
RG                14.3
DW               78.000 usec
DE                4.50 usec
TE               298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1                12.00 usec
PL1                0.00 dB
SFO1       400.1328089 MHz

F2 - Processing parameters
SI                65536
SF          400.1300427 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2             -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM         166.72086 Hz/cm
```


13C spectrum with 1H decoupling
ppm
Br
OPiv
TsN
177.76
145.30
140.55
135.74
135.30
133.12
130.69
130.16
129.10
128.21
127.91
127.65
125.27
124.56
124.28
123.69
122.32
120.64
114.17
77.89
77.57
77.25
68.95
39.24
35.08
32.70
27.50
27.34
21.71
Current Data Parameters
USER mkonev
NAME MOX-III-indolepivCCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 21.11
INSTRUM drx400
PROBHD 5 mm QNP H/F/C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 104
DS 4
SWH 24154.589 Hz
FIDRES 0.368578 Hz
AQ 1.3564452 sec
RG 9195.2
DW 20.700 usec
DE 20.30 usec
TE 298.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.555 Hz
F2P -100.000 ppm
F2 -10060.113 Hz
PPMCM 0.21053 ppm/cm
HZCM 926.69829 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
ppm
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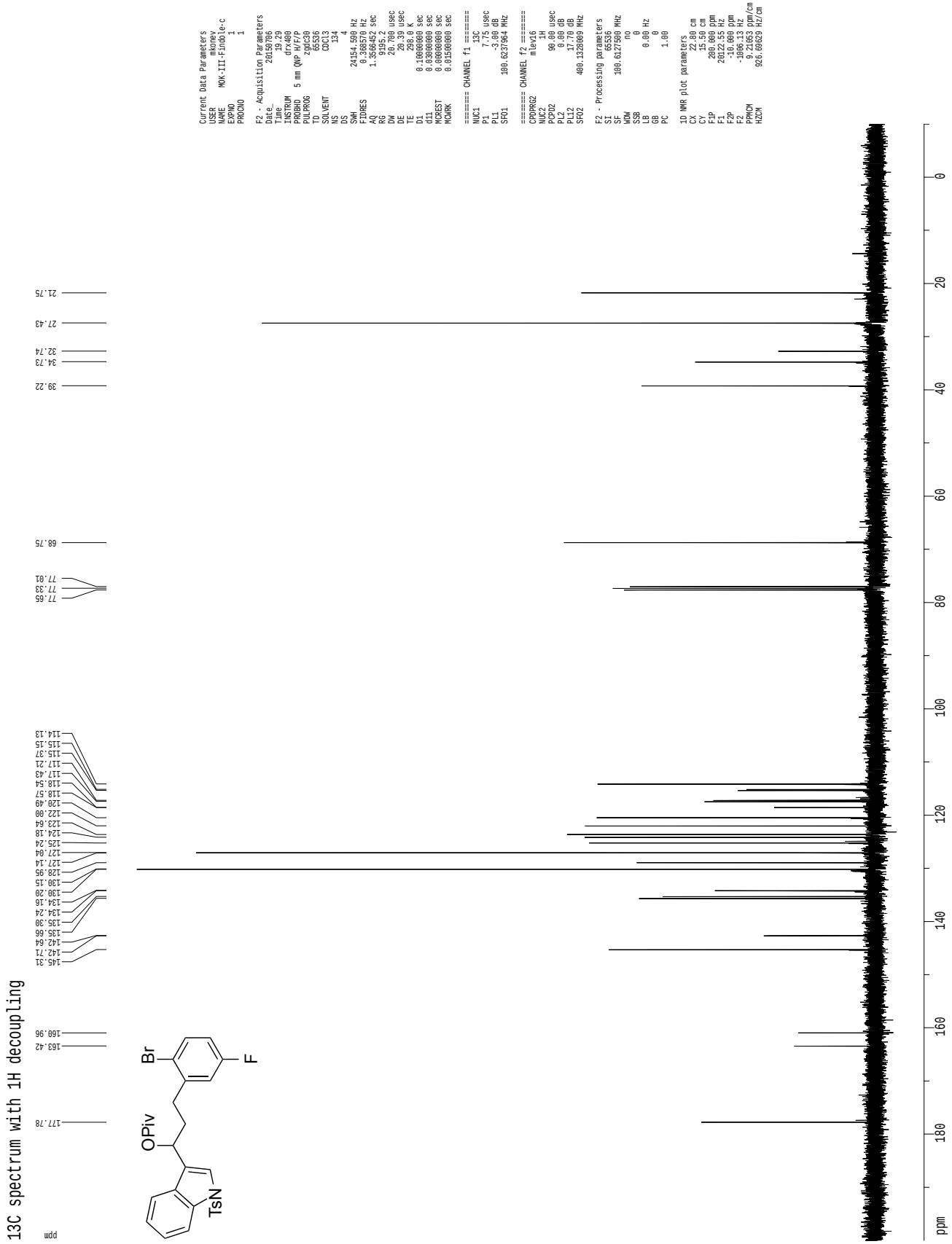
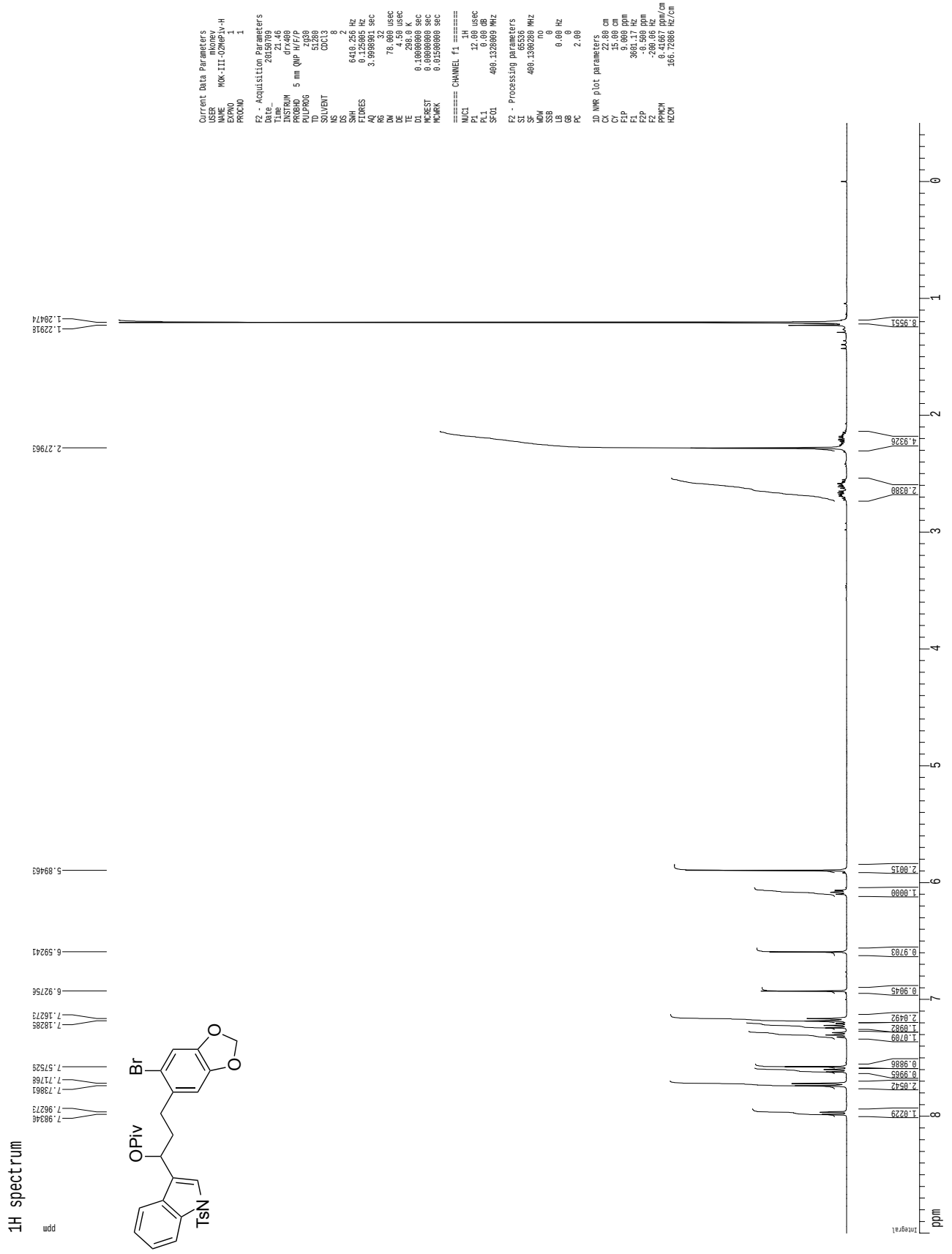
7.99868
7.77451
7.75376
7.64776
7.62815
7.61172
7.34154
7.25991
7.22031
7.19956
OPiv
Br
F
TsN
Current Data Parameters
USER mkoney
NAME MOK-III-Findole-h4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150706
Time 13.35
INSTRUM dir400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 4
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 50.8
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
1.23791
1.26266
2.31496
2.33725
9.0611
5.5780
2.1547
1.0000
0.9649
1.0829
3.6199
1.1252
0.9991
2.0605
2.0919
1.1108
Integral
ppm


13C spectrum with 1H decoupling
Current Data Parameters
USER mkoney
NAME MOK-III-Fin0le-c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150706
Time 19.29
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 134
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 17.70 dB
PL12 400.1328809 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 27.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
21.75
27.43
32.74
34.73
39.22
68.75
77.01
77.33
77.65
114.13
115.15
115.37
117.21
117.43
118.54
118.57
120.49
122.00
123.64
124.18
125.24
127.04
127.14
128.95
130.15
130.20
134.16
134.24
135.30
135.66
142.64
142.71
145.31
160.96
163.42
177.78
Br
F
OPiv
TsN
ppm
0
20
40
60
80
100
120
140
160
180



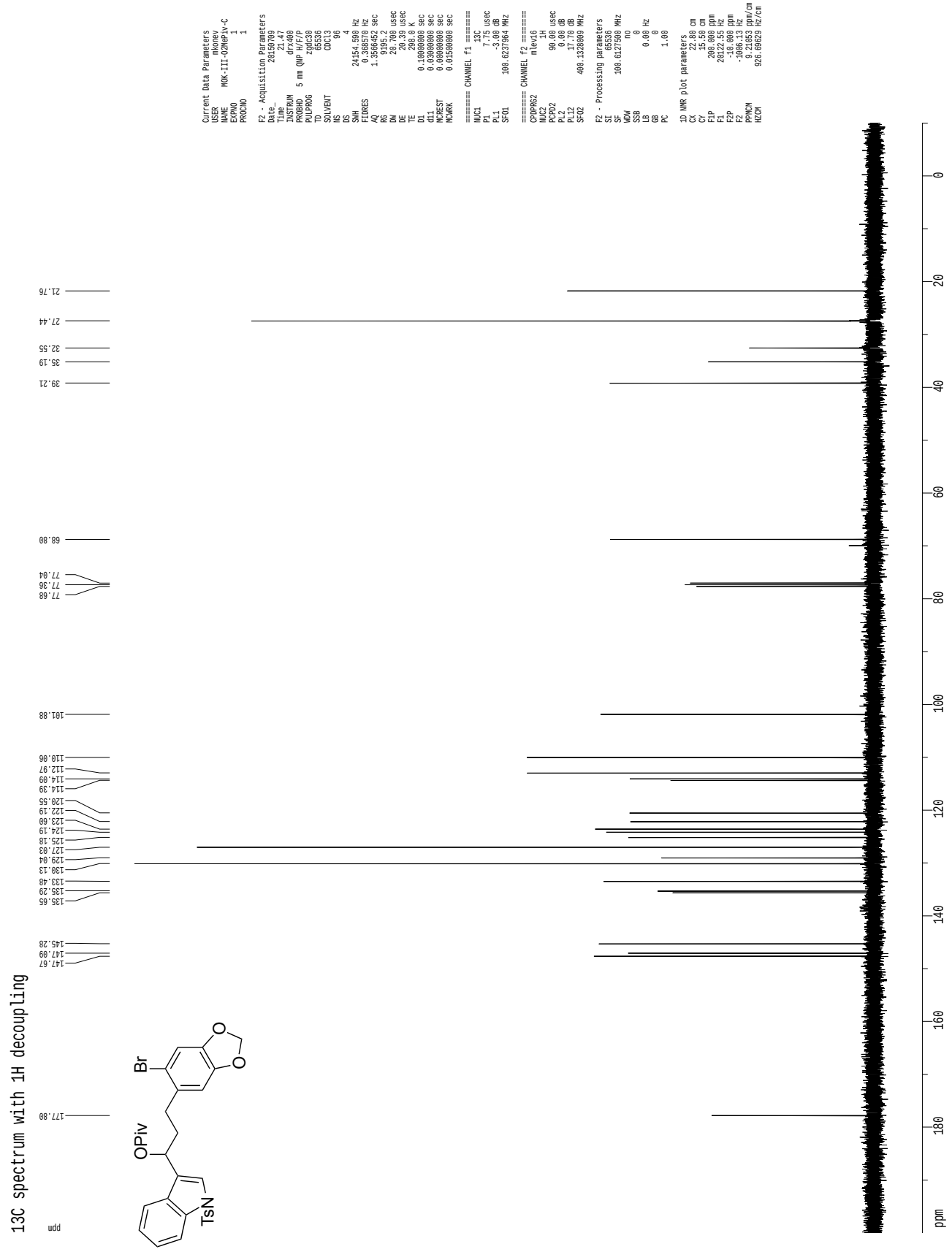

```
Current Data Parameters
USER      mkonev
NAME      MOK-III-02MeP1v-H
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_      20150709
Time_            21.46
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zg30
TD              51200
SOLVENT         CDCl3
NS                  8
DS                  0
SWH          6410.256 Hz
FIDRES       0.125085 Hz
AQ           3.9998901 sec
RG                 32
DW              78.000 usec
DE               4.50 usec
TE              298.0 K
D1           0.10000000 sec
MCREST       0.09000000 sec
MCWRK        0.01500000 sec

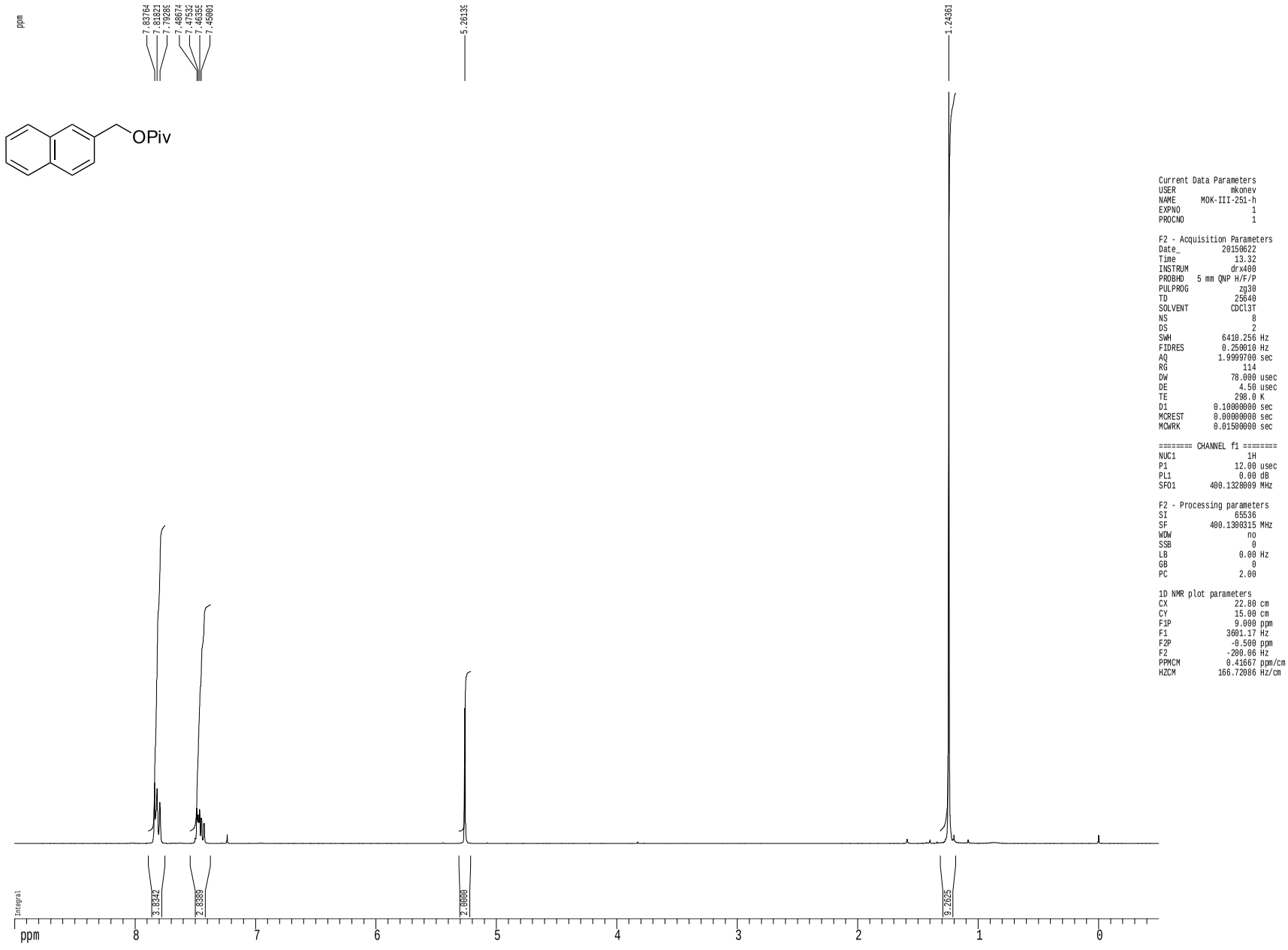
======= CHANNEL f1 =======
NUC1               1H
P1              12.00 usec
PL1              0.00 dB
SFO1        400.1320009 MHz

F2 - Processing parameters
SI              65536
SF          400.1300200 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              2.00

1D NMR plot parameters
CX             22.80 cm
CY             15.00 cm
F1P             9.000 ppm
F1           3601.17 Hz
F2P            -0.500 ppm
F2           -200.046 Hz
PPMCM         0.41667 ppm/cm
HZCM        166.72086 Hz/cm
```


13C spectrum with 1H decoupling
Current Data Parameters
USER mkonev
NAME MOK-III-026ePiv-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150709
Time 21.47
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.09000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SF01 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SF02 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1906.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
177.80
147.67
147.09
145.28
135.65
135.29
133.48
130.13
129.04
127.93
125.18
124.19
123.60
122.19
120.55
114.39
114.09
112.97
110.06
101.88
68.80
77.68
77.36
77.04
39.21
35.19
32.55
27.44
21.76
ppm
0
20
40
60
80
100
120
140
160
180
OPiv
Br
TsN
O
O

1H spectrum



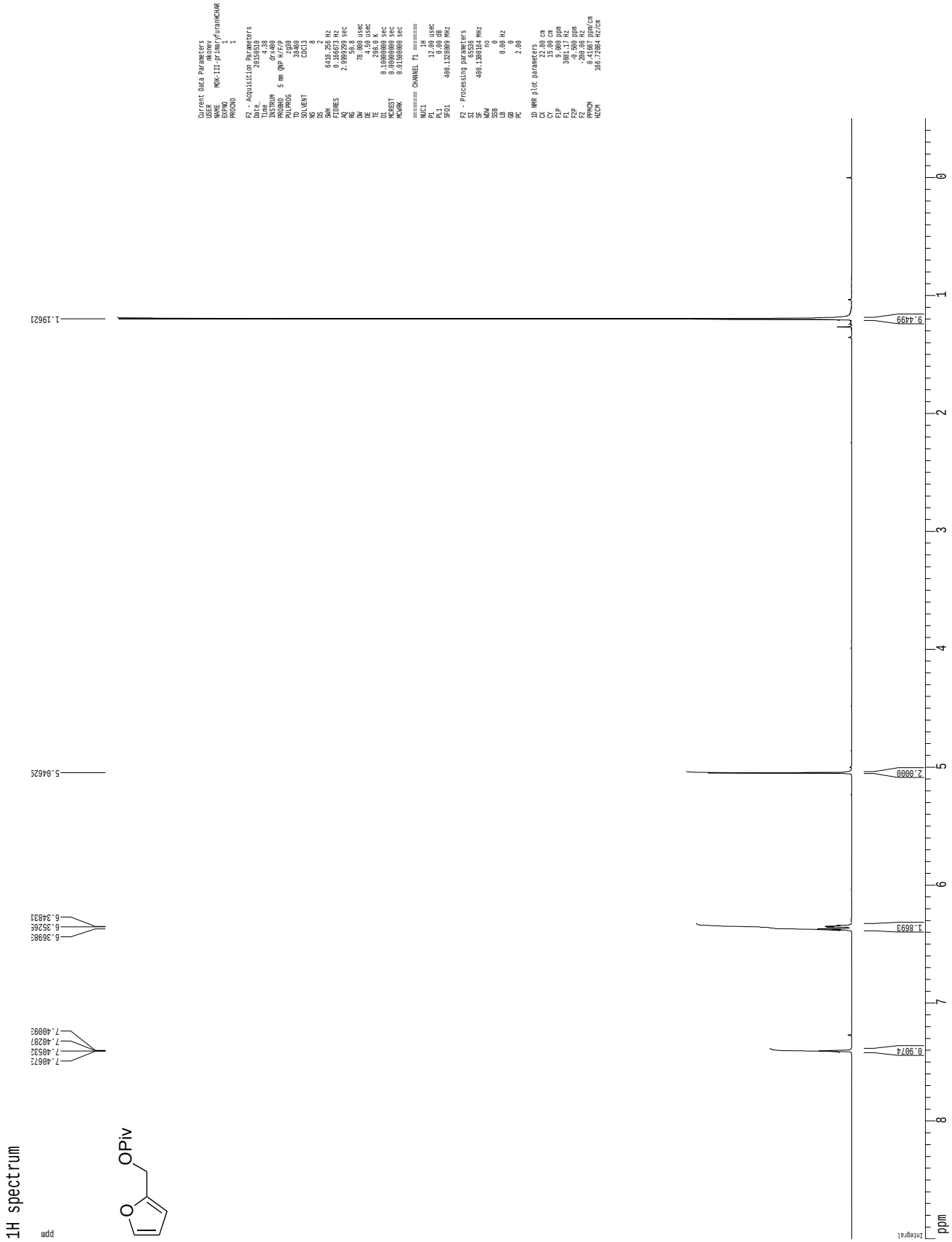
Current Data Parameters
USER mkonev
NAME MOK-III-251-h
EXPNO 1
PROCNO 1

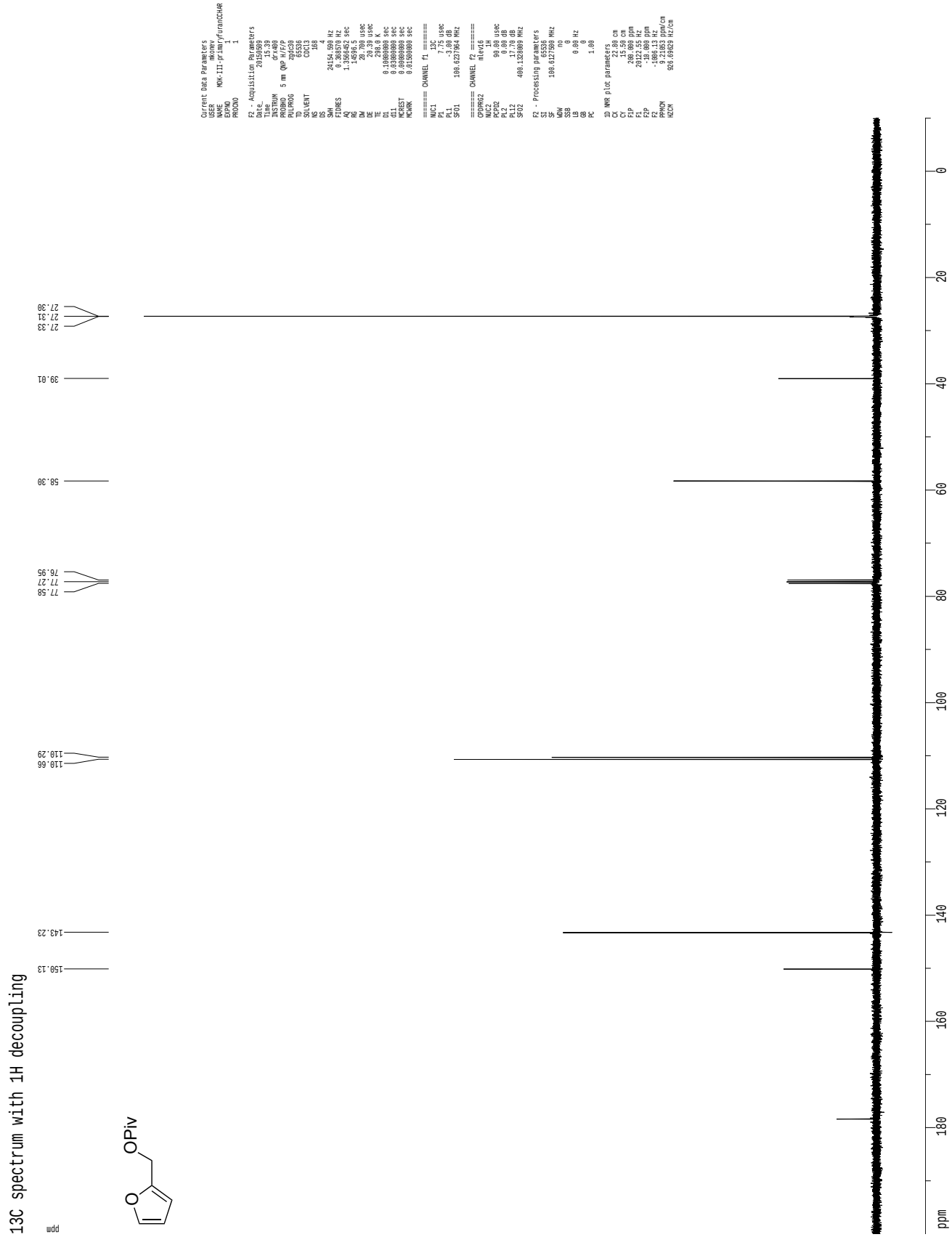
F2 - Acquisition Parameters
Date_ 20150622
Time 13.32
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 114
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

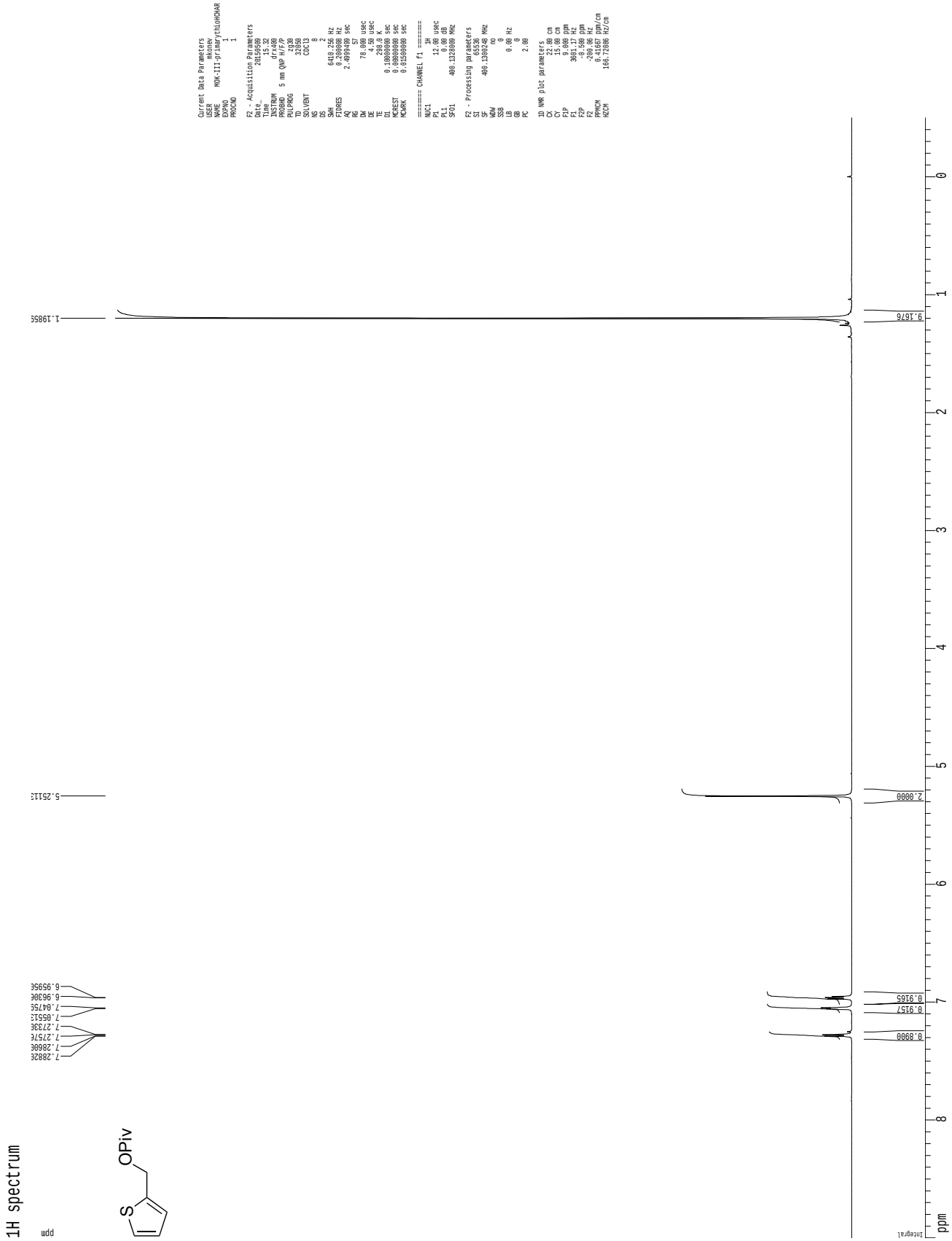
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300315 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72086 Hz/cm


1H spectrum
OPiv
O
ppm
7.4067
7.4053
7.4028
7.4009
6.3698
6.3526
6.3483
5.0462
1.1962
9.4499
2.0000
1.8693
0.9074
Integral
ppm


13C spectrum with 1H decoupling
ppm
OPiv
O
150.13
143.23
110.66
110.29
77.58
77.27
76.95
58.30
39.01
27.38
27.31
27.33
Current Data Parameters
USER mkonev
NAME MOK-III-primaryfuranOCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 13.53
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 168
DS 4
SWH 241.546 Hz
FIDRES 0.368553 Hz
AQ 1.3565452 sec
RG 14596.5
DW 20.700 usec
DE 6.50 usec
TE 298.1 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237984 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 15.50 cm
F1P 200.000 ppm
F2P -10.000 ppm
PPMCM 1.85431413 ppm/cm
HZCM 926.69829 Hz/cm
0
20
40
60
80
100
120
140
160
180
ppm


1H spectrum
OPiv
S
ppm
Integral
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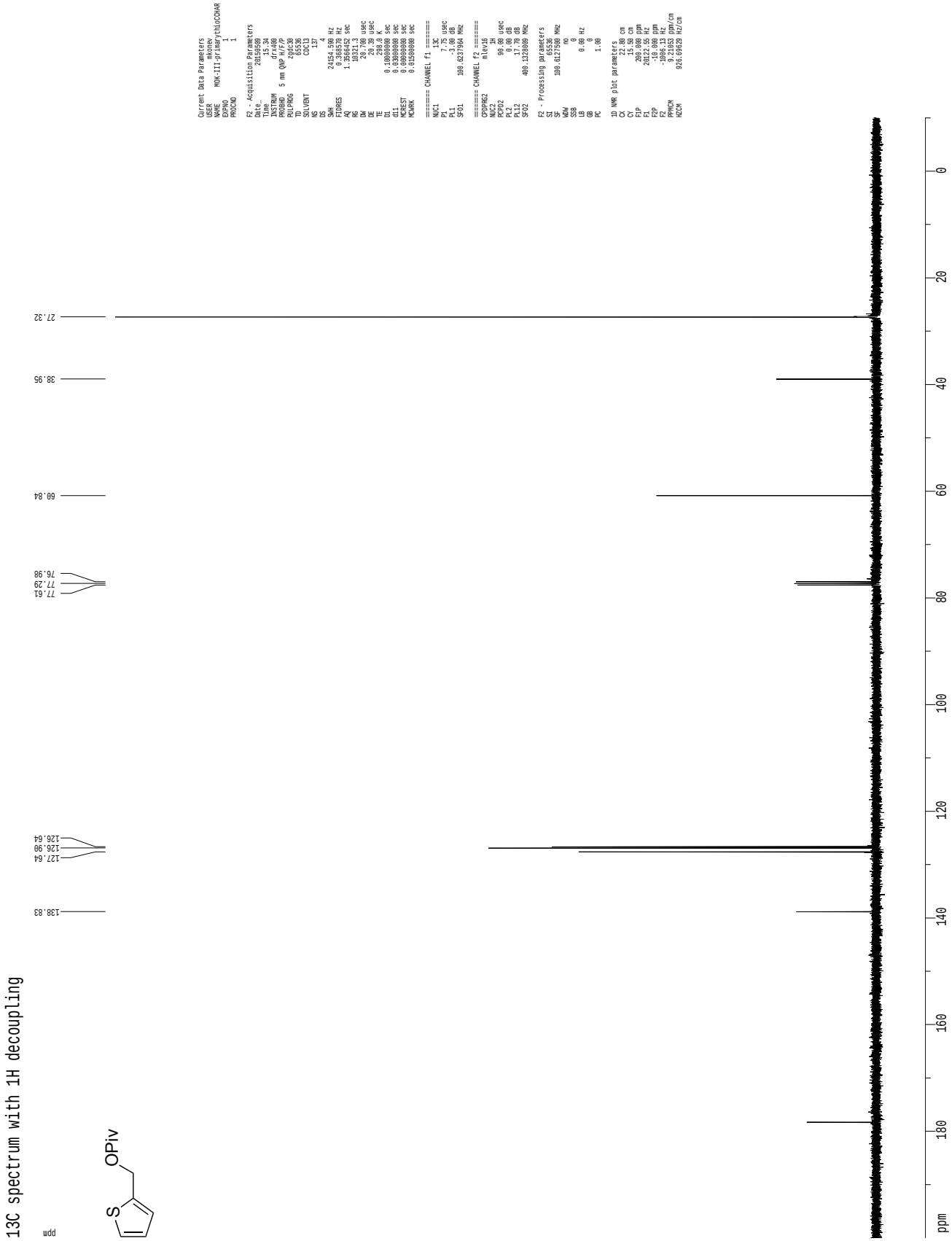
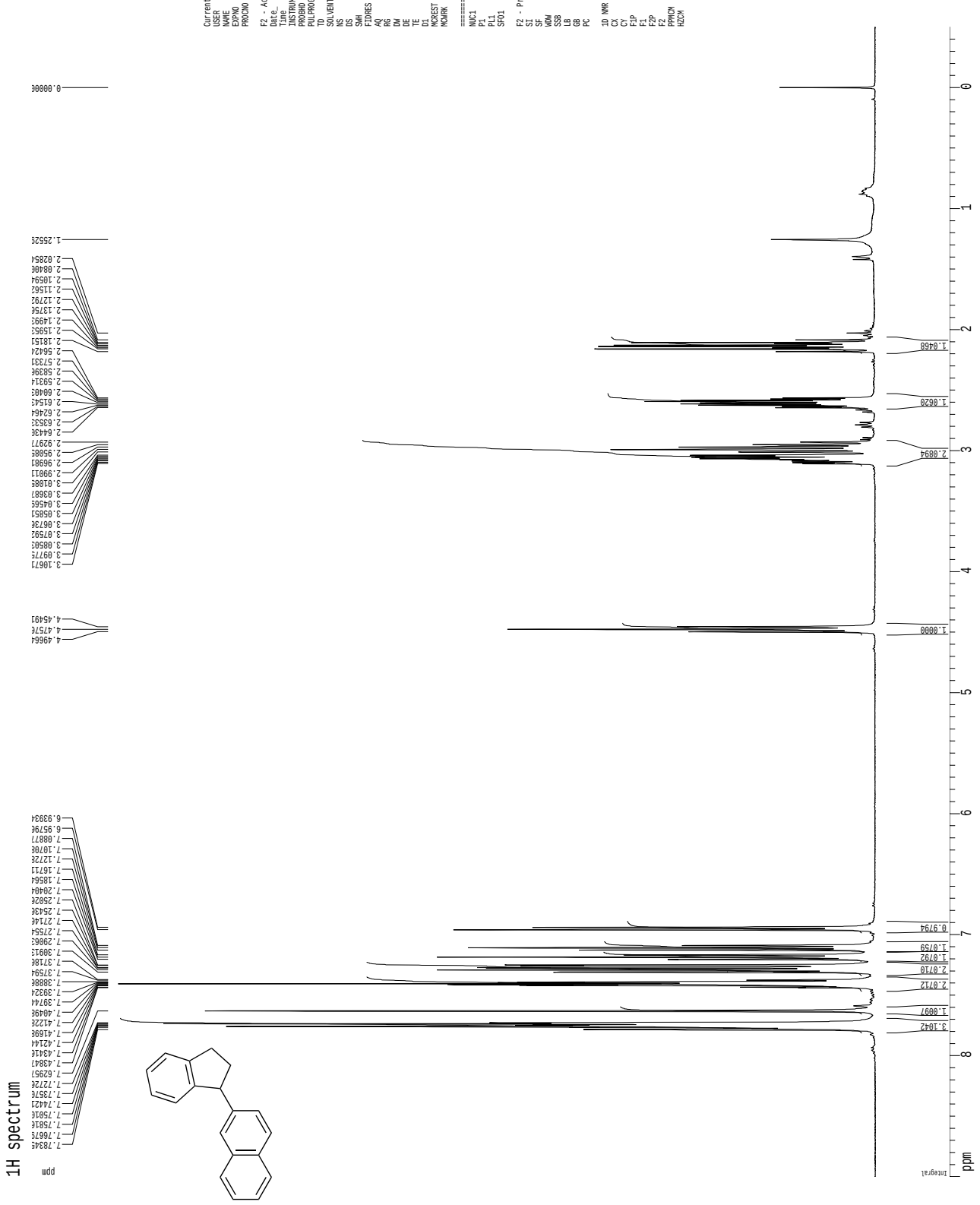
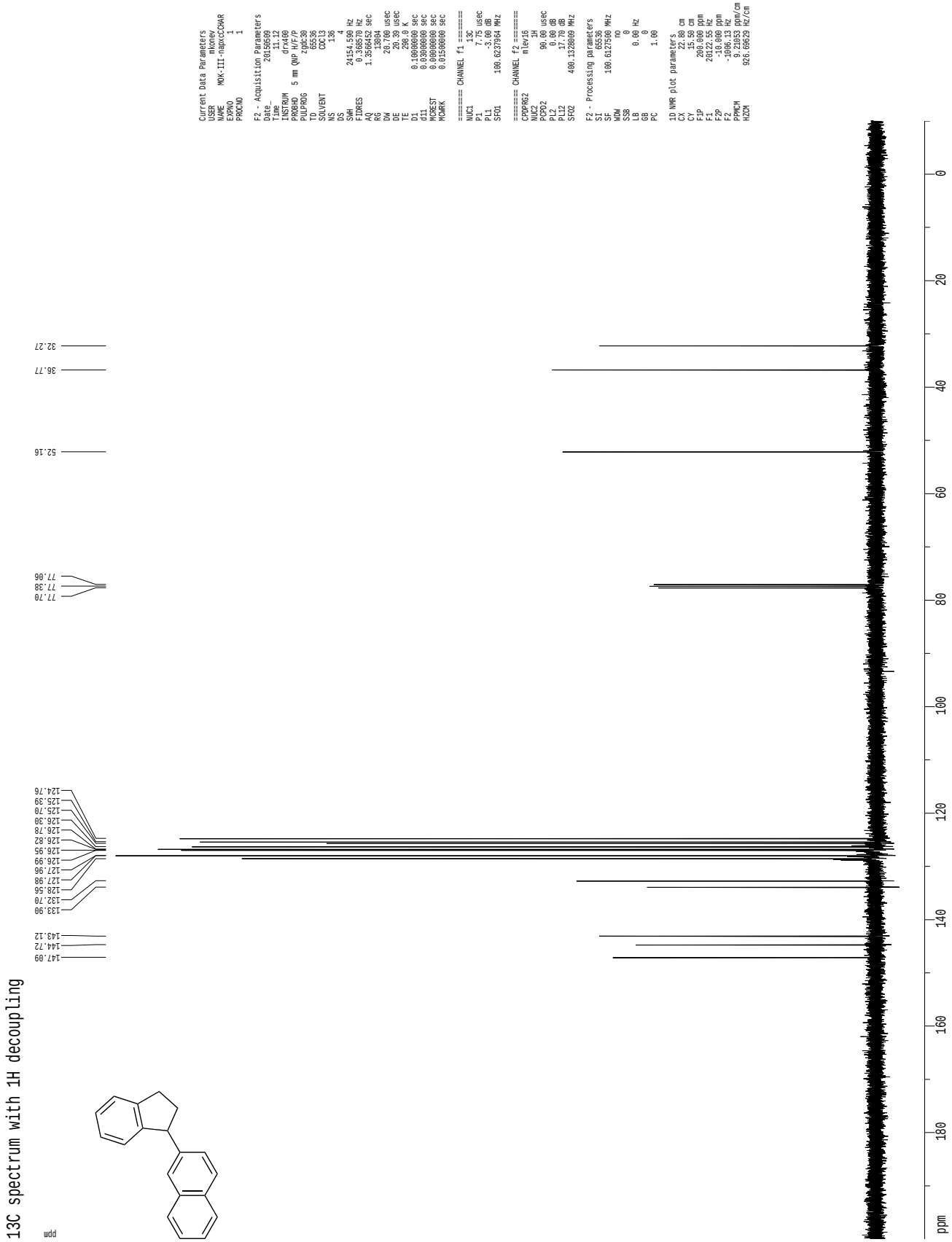
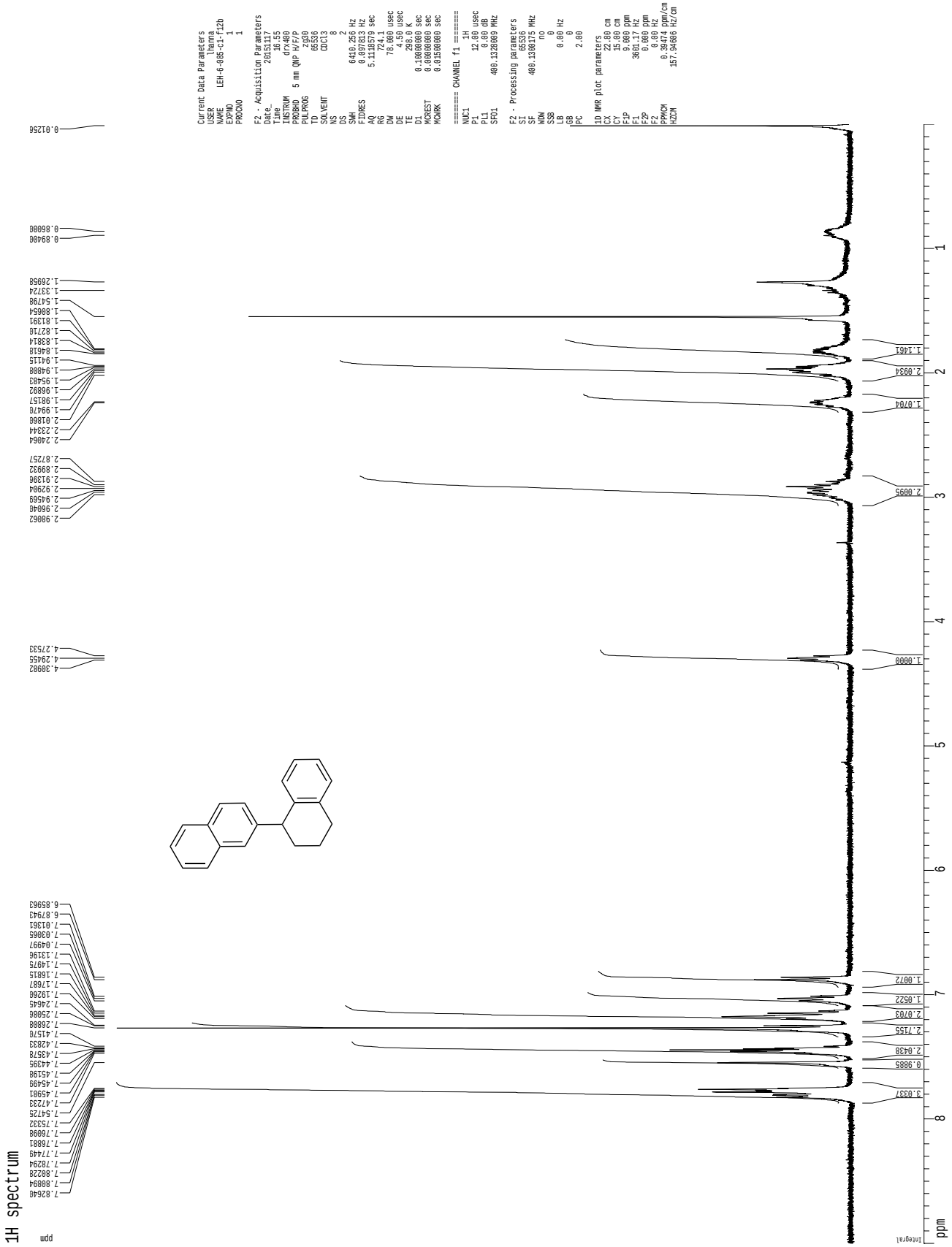
5.2513
6.9596
6.9630
7.0475
7.0551
7.2733
7.2757
7.2806
7.2882
9.1676
2.0000
0.9165
0.9157
0.8900

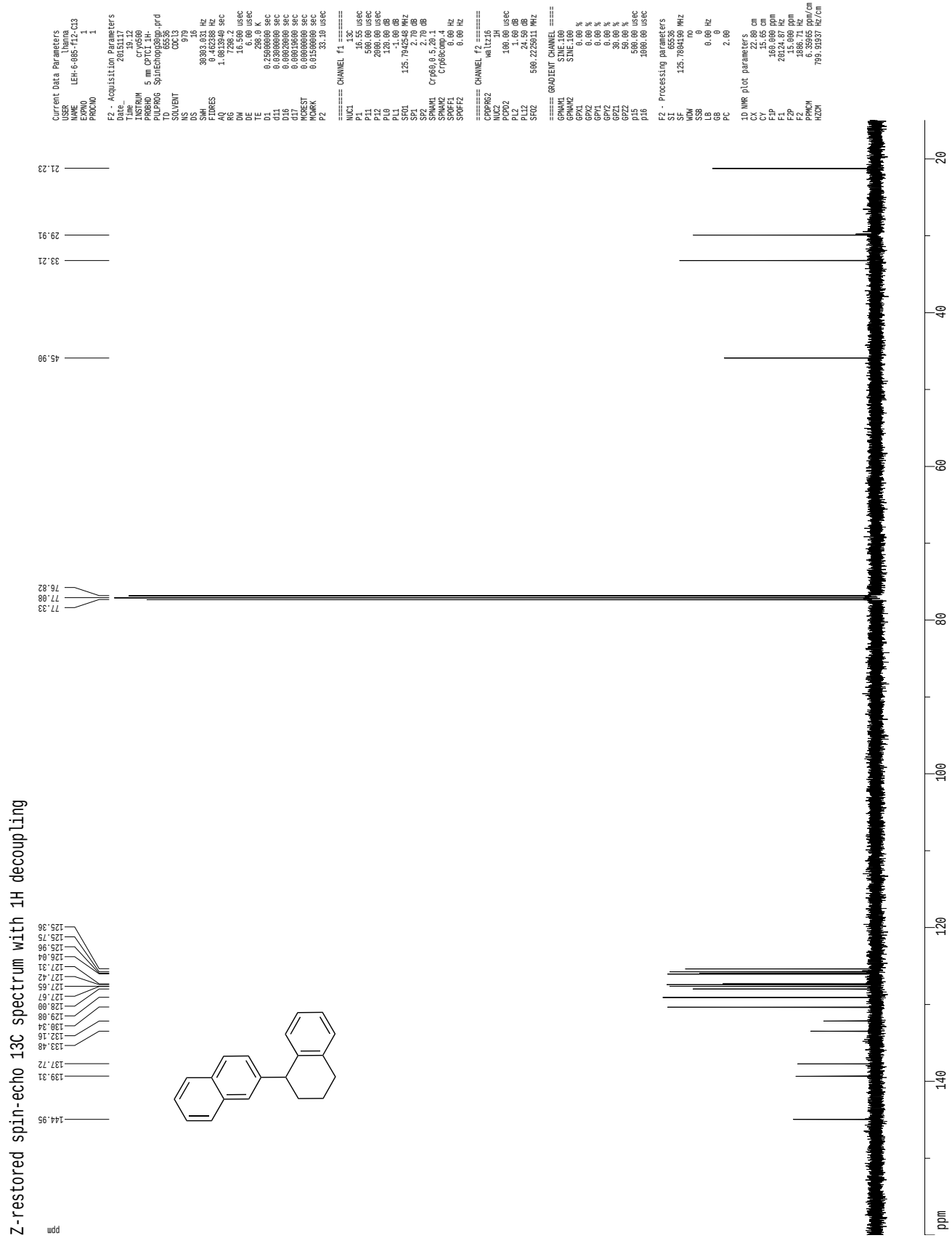

13C spectrum with 1H decoupling
ppm
OPiv
S
Current Data Parameters
USER mkonev
NAME MOK-III-primarythioCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150609
Time 15:34
INSTRUM dpx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 112
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3568452 sec
RG 181.1
DW 20.700 usec
DE 20.30 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 12.00 cm
CY 15.35 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -4066.13 Hz
PPMCM 9.21043 ppm/cm
HZCM 926.69629 Hz/cm
138.83
127.64
126.90
126.64
77.61
77.29
76.98
60.84
38.95
27.32
ppm
0
20
40
60
80
100
120
140
160
180

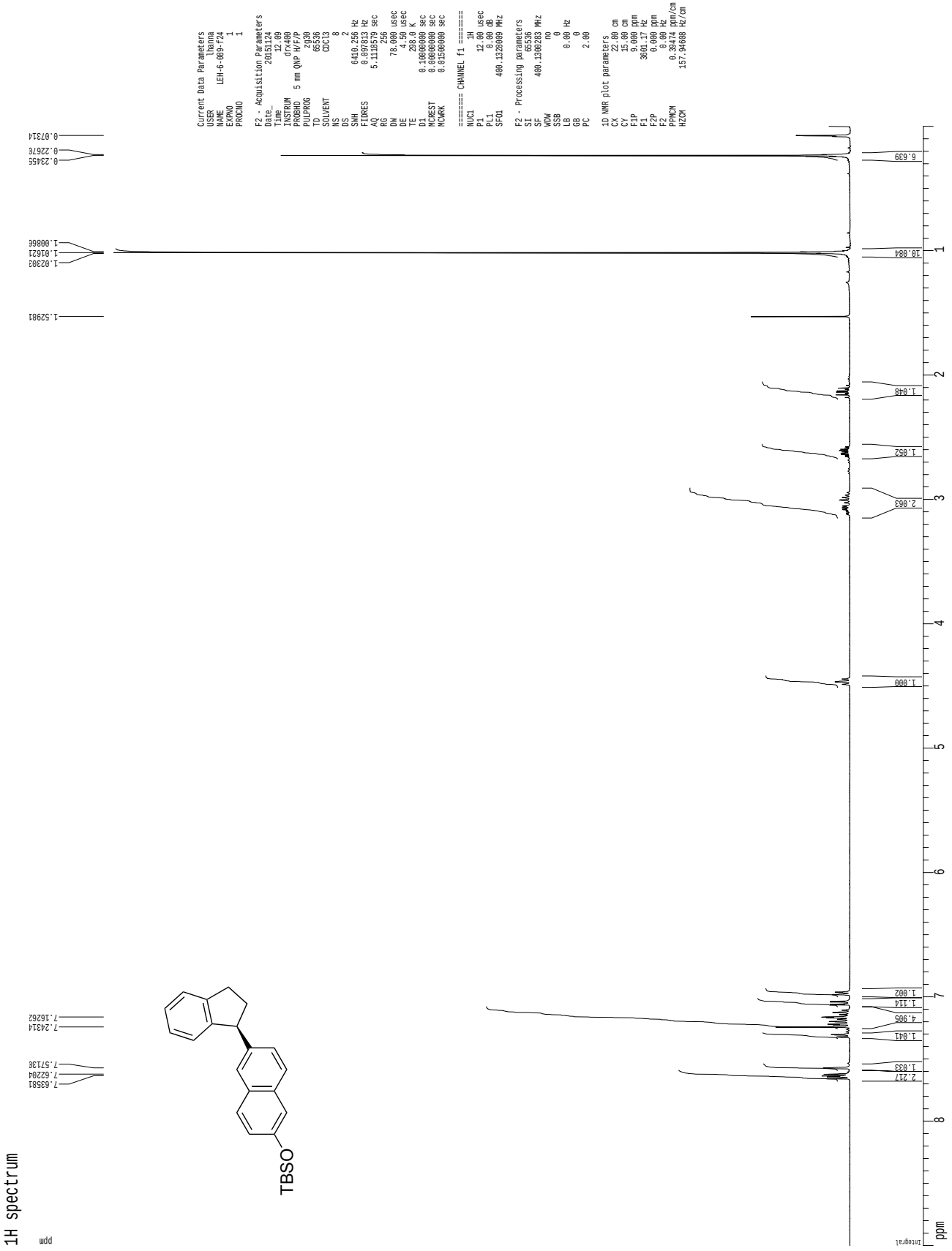

1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-III-naptoCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150609
Time 11.10
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.200008 Hz
AQ 2.4999499 sec
RG 57
DW 78.0000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300733 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.590 ppm
F2 -236.05 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72087 Hz/cm
ppm
Integral
1.0000
0.9794
1.0092
1.0759
1.0792
2.0710
2.0712
1.0097
3.1042
1.0468
1.0620
2.0894

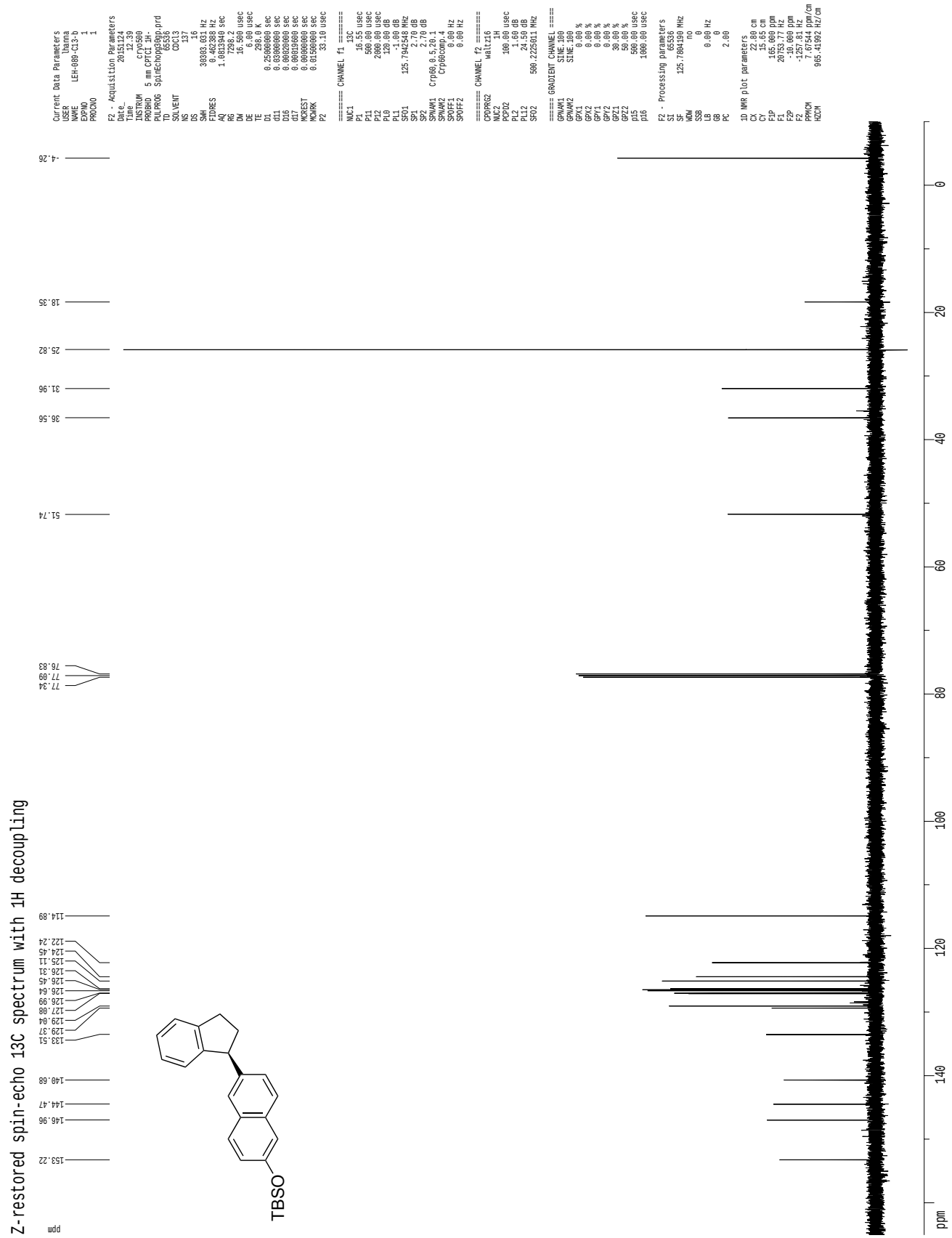

13C spectrum with 1H decoupling
Current Data Parameters
USER mkoney
NAME MOK-III-napyCCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 11.12
INSTRUM dirx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 136
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 15084
DW 20.700 usec
DE 20.30 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
32.27
36.77
52.16
77.06
77.38
77.70
124.76
125.39
125.70
126.30
126.78
126.82
126.95
126.99
127.96
127.98
128.56
132.70
133.90
143.12
144.72
147.09
ppm
0
20
40
60
80
100
120
140
160
180

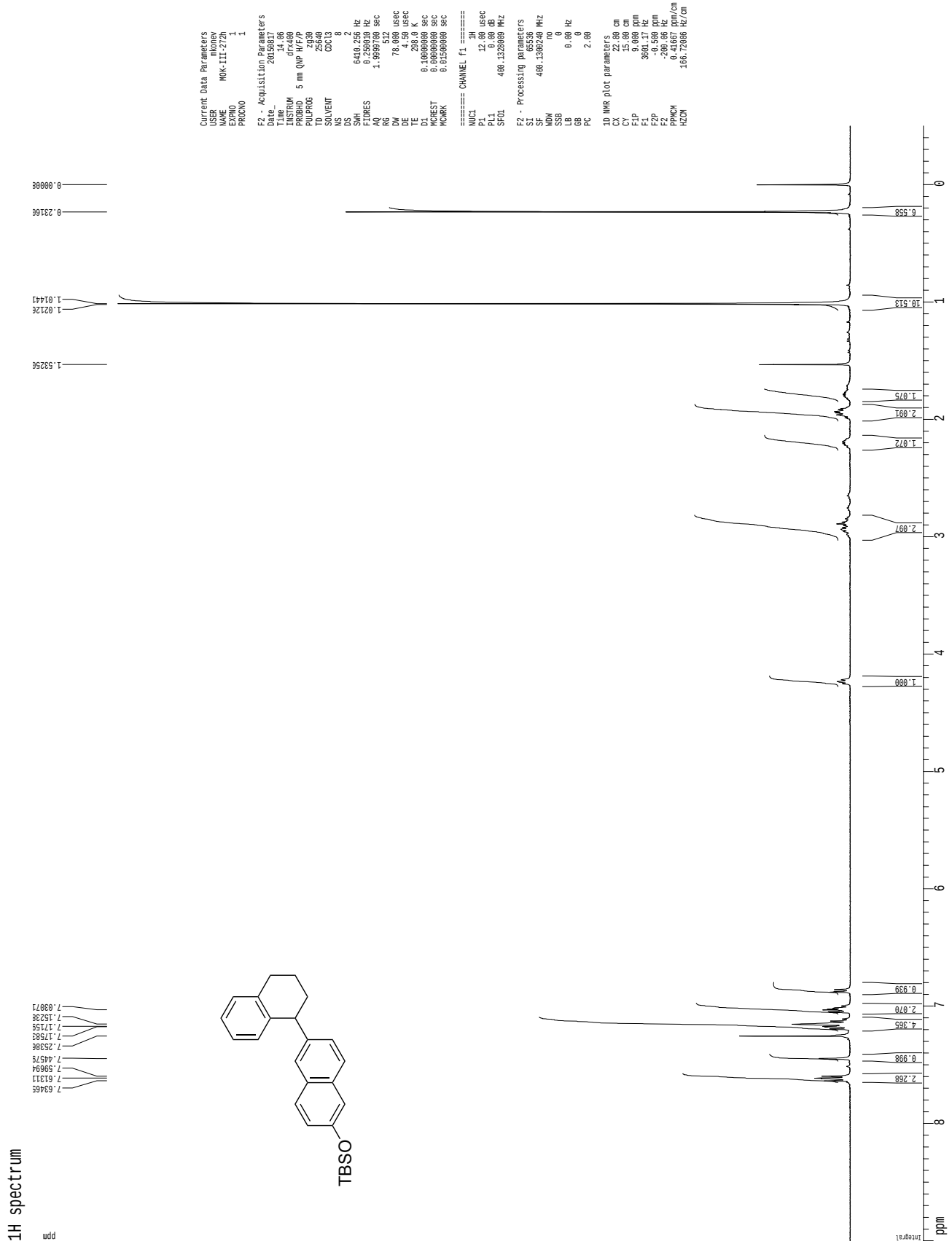

1H spectrum
Current Data Parameters
USER Obama
NAME LEH-6-085-c1-f12b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 16.55
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 724.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94806 Hz/cm
0.01256
0.89408
0.86088
1.26958
1.33724
1.54798
1.80654
1.81391
1.82718
1.83814
1.84618
1.94115
1.94808
1.95483
1.96892
1.98157
1.99470
2.01866
2.23344
2.24064
2.87257
2.89932
2.91396
2.92904
2.94569
2.96040
2.98062
4.27533
4.29455
4.30982
6.85963
6.87943
7.01361
7.03065
7.04997
7.13196
7.14975
7.16815
7.17687
7.19266
7.24645
7.25086
7.26808
7.41570
7.42833
7.43578
7.44395
7.45198
7.45499
7.46981
7.47233
7.54725
7.75332
7.76098
7.76881
7.77449
7.78294
7.80228
7.80894
7.82640
1.1461
2.0934
1.0704
2.0095
1.0000
1.0072
1.0522
2.0703
2.7155
2.0438
0.9885
3.0337
Integral
ppm
1
2
3
4
5
6
7
8
ppm

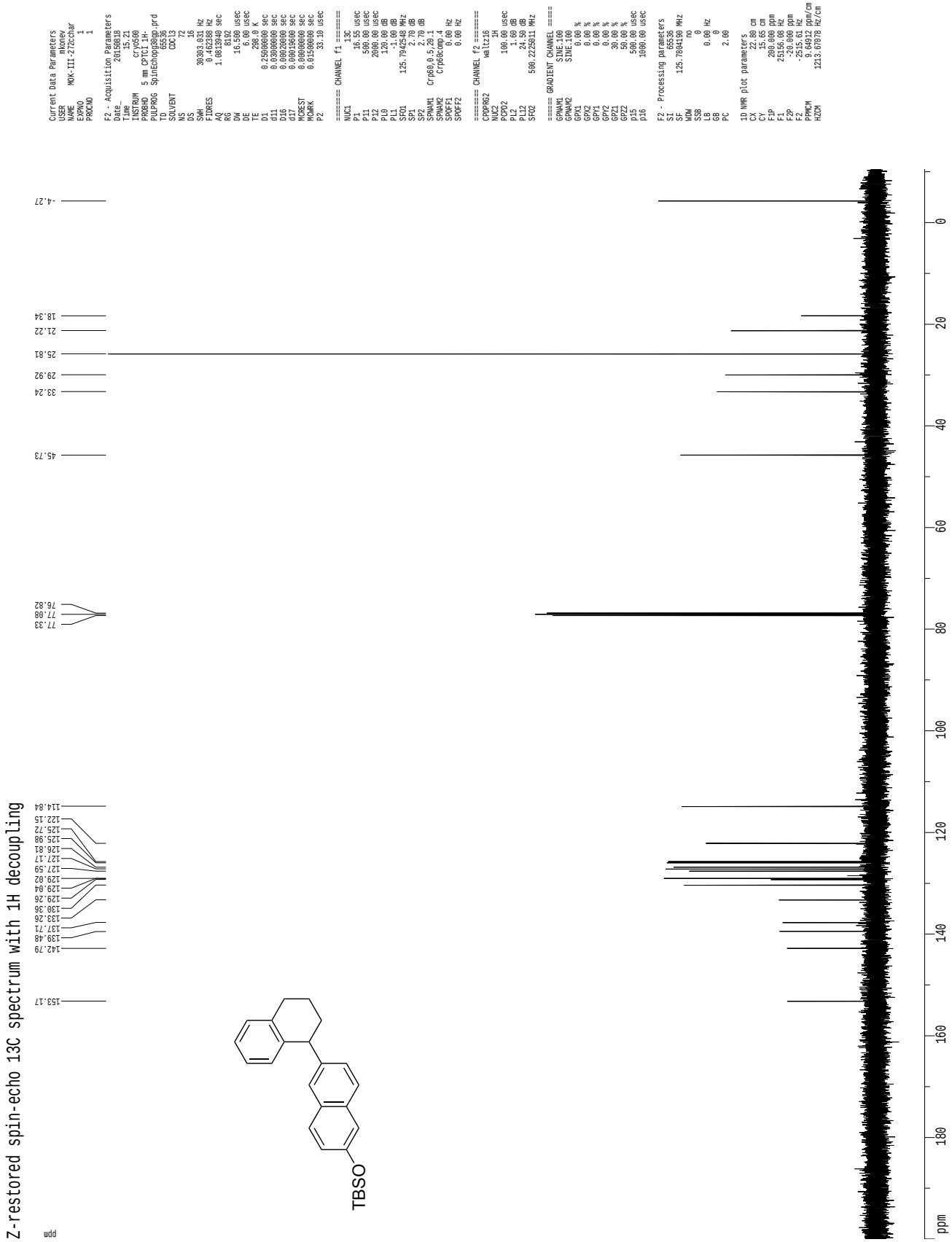
Z-restored spin-echo 13C spectrum with 1H decoupling

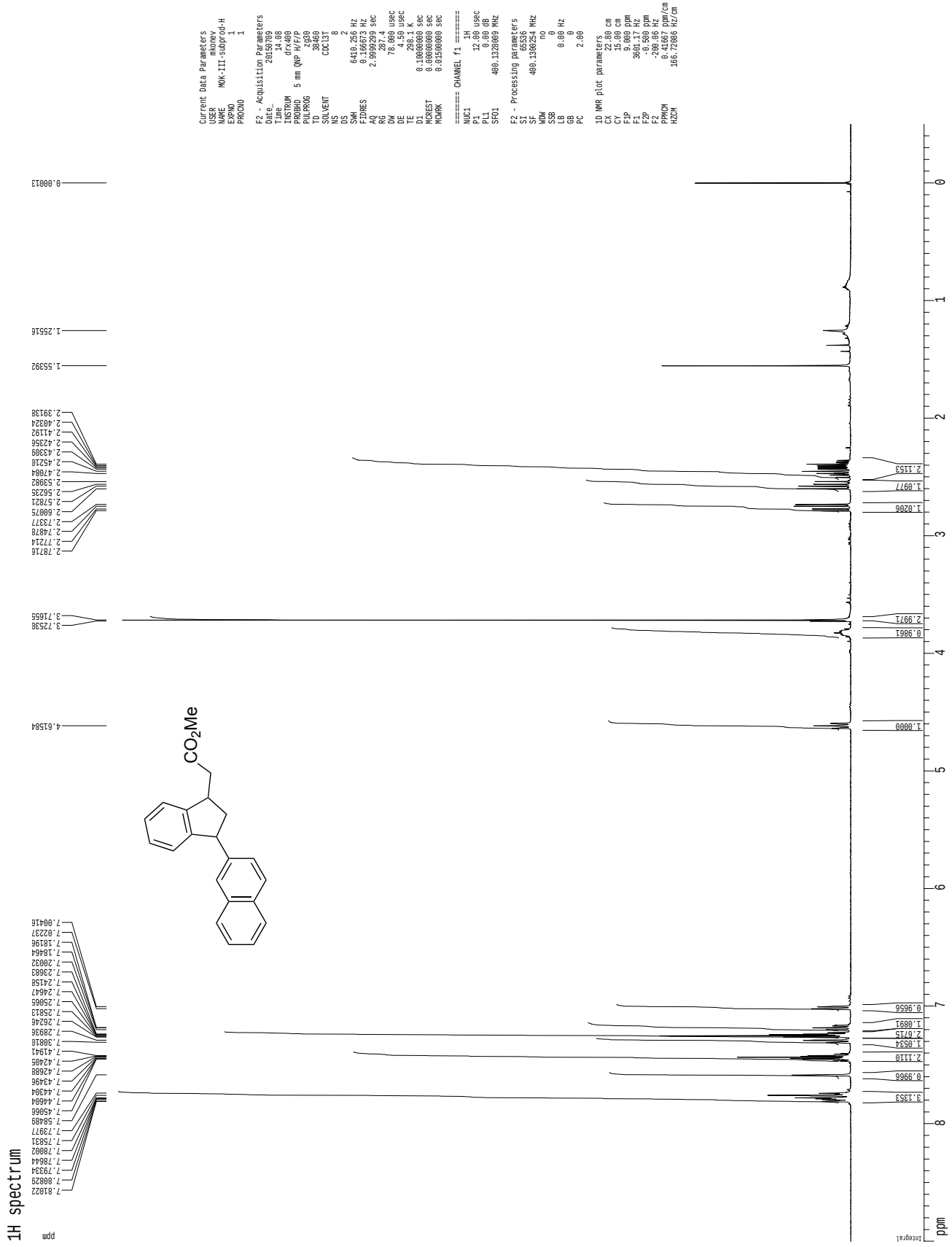


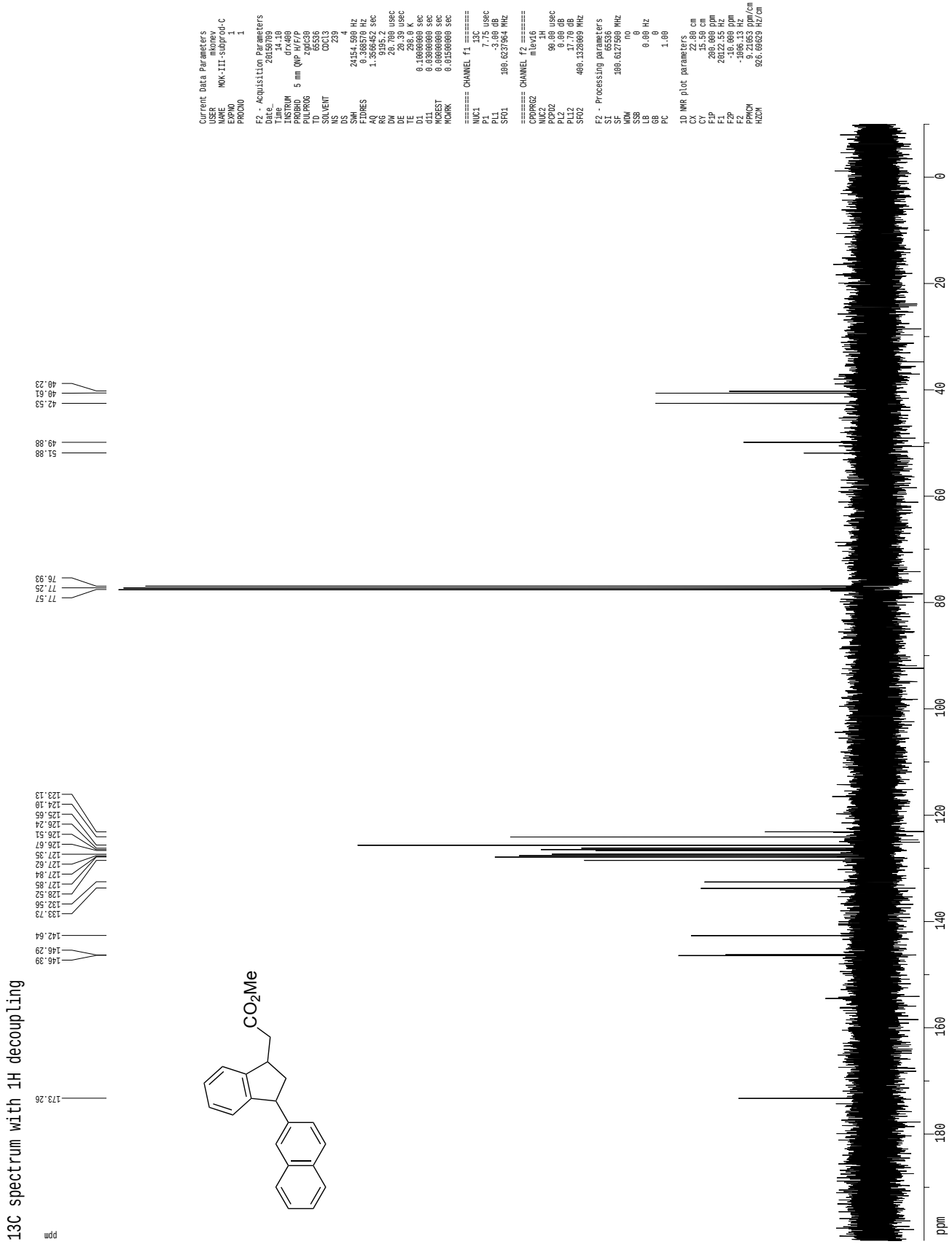

1H spectrum
TBSO
ppm
Integral
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters

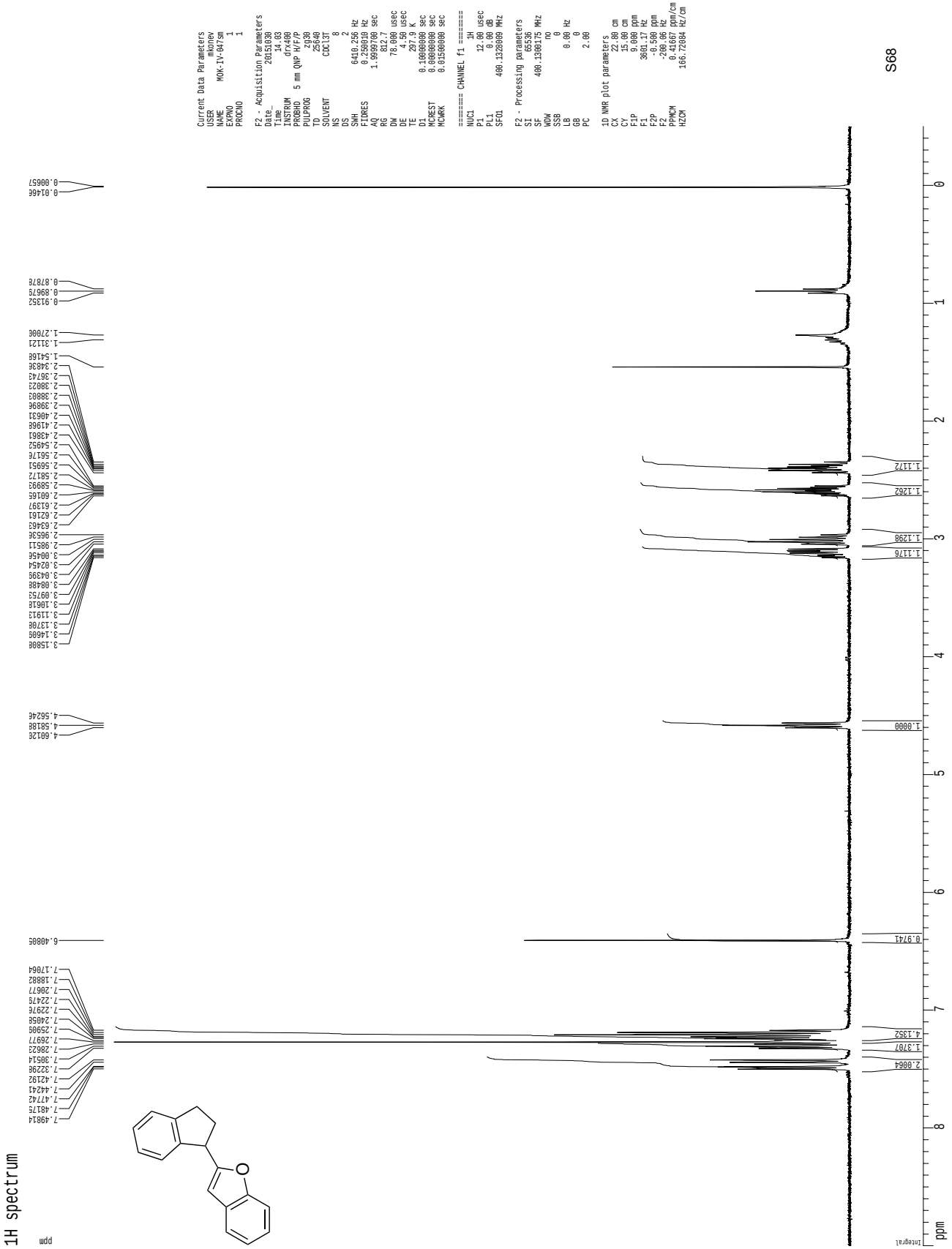

Z-restored spin-echo 13C spectrum with 1H decoupling
TBSO
ppm
Current Data Parameters
USER Dhanna
NAME LEH-089-C13-b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151124
Time 12.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 137
DS 16
SWH 38303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 600.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp80,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 165.000 ppm
F1 20753.77 Hz
F2P -10.000 ppm
F2 -1257.81 Hz
PPMCM 7.67544 ppm/cm
HZCM 965.41992 Hz/cm
-4.26
18.35
25.82
31.96
36.56
51.74
76.83
77.09
77.34
114.89
122.24
124.45
125.11
126.31
126.45
126.64
126.99
127.08
129.04
129.37
133.51
140.68
144.47
146.96
153.22
0
20
40
60
80
100
120
140
ppm


1H spectrum
TBSO
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
0.00000
0.23166
1.01441
1.02126
1.53256
7.03071
7.15238
7.17159
7.17583
7.25386
7.44579
7.59694
7.61311
7.63465
6.558
10.513
1.075
2.091
1.072
2.097
1.000
0.939
2.070
4.365
0.998
2.268
ppm
Integral

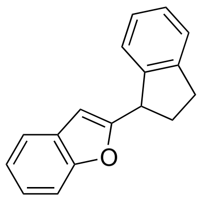

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
TBSO
Current Data Parameters
USER mkonev
NAME MOK-III-272cChar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150818
Time 15.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 72
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.08139940 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 200.000 ppm
F1 25156.08 Hz
F2P -20.000 ppm
F2 -2515.61 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67078 Hz/cm
-4.27
18.34
21.22
25.81
29.92
33.24
45.73
76.82
77.08
77.33
114.84
122.15
125.72
125.98
126.81
127.17
127.59
129.02
129.04
129.26
130.36
133.26
137.71
139.48
142.79
153.17
0
20
40
60
80
100
120
140
160
180
ppm

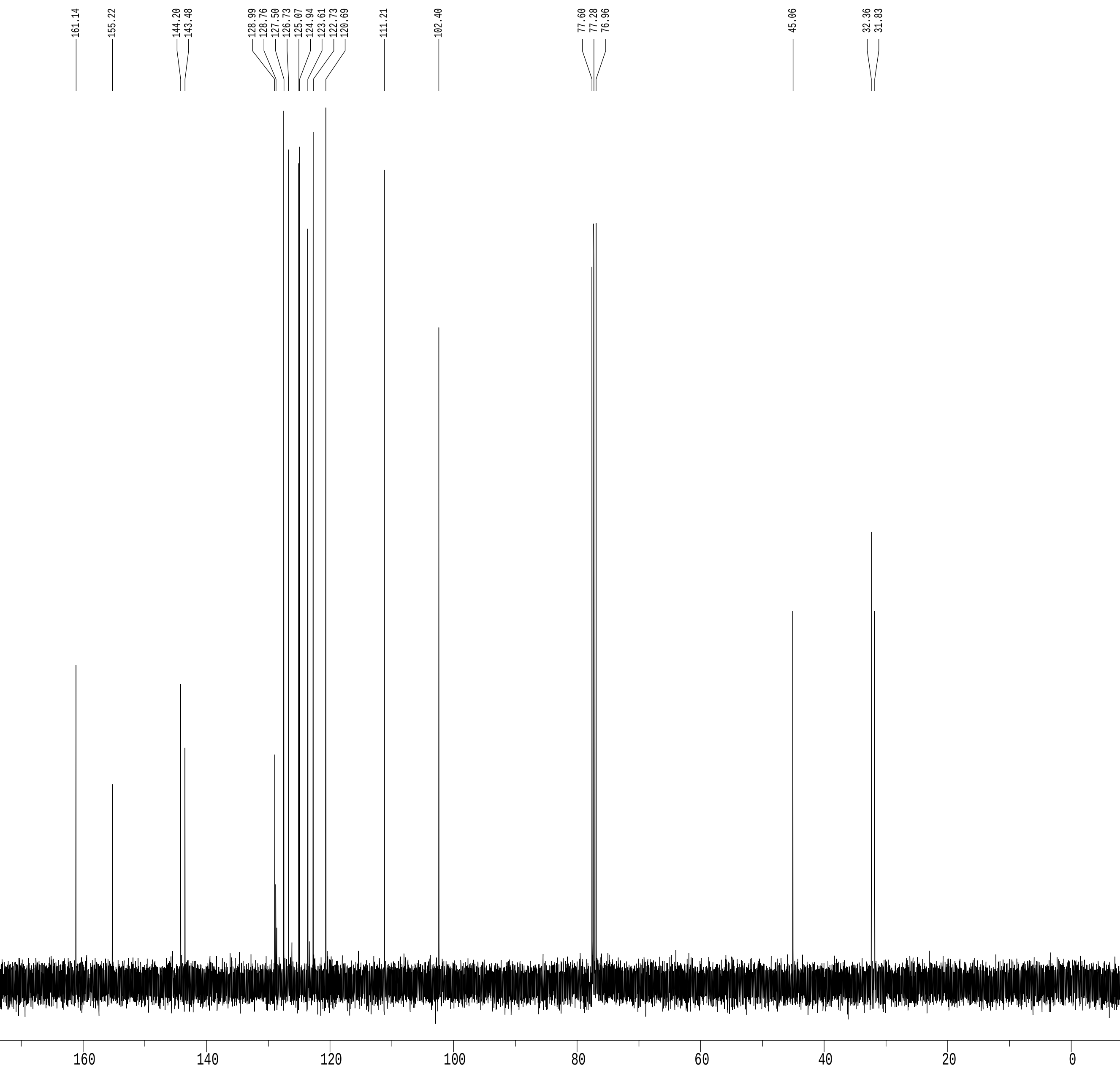

1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-III-subprod-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150709
Time 14.08
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 38460
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.166673 Hz
AQ 2.9999239 sec
RG 287.4
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.09000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300254 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -200.86 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72086 Hz/cm
0.00013
1.25516
1.55392
2.39138
2.40324
2.41192
2.42356
2.43309
2.45210
2.47084
2.53982
2.56235
2.57821
2.60075
2.73377
2.74878
2.77214
2.78716
3.72536
3.74655
4.61584
7.00416
7.02237
7.18196
7.18464
7.20032
7.23683
7.24158
7.24647
7.25065
7.25813
7.26246
7.28936
7.30816
7.41941
7.42405
7.42688
7.43496
7.44304
7.44604
7.45066
7.58489
7.73977
7.75831
7.78002
7.78644
7.79334
7.80829
7.81022
2.1153
1.0977
1.0206
2.9971
0.9861
1.0000
0.9656
1.0891
2.0715
1.0534
2.1110
0.9966
3.1353
integral
ppm
CO2Me


13C spectrum with 1H decoupling
ppm
173.26
146.39
146.29
142.64
133.73
132.56
128.52
127.85
127.84
127.62
127.35
126.67
126.51
126.24
125.65
124.10
123.13
77.57
77.25
76.93
51.88
49.88
42.53
40.61
40.23
CO2Me
Current Data Parameters
USER mkonev
NAME MOX-III-subprod-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150709
Time 14.10
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 234
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 17.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 15.50 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
S68
ppm
Integral
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
0.00657
0.01466
0.87878
0.89675
0.91352
1.27006
1.31121
1.54168
1.11772
1.12762
1.12988
1.11776
1.00000
0.97741
4.13527
1.37077
2.00644

13C spectrum with 1H decoupling



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Current Data Parameters
USER                 mkonev
NAME     MOK-III-benzofuranxcCCHAR
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150509
Time                  11.20
INSTRUM              drx400
PROBHD    5 mm QNP H/F/P
PULPROG              zgdc30
TD                    65536
SOLVENT               CDCl3
NS                      208
DS                        4
SWH            24154.590 Hz
FIDRES          0.368570 Hz
AQ             1.3566452 sec
RG                   9195.2
DW                   20.700 usec
DE                    20.39 usec
TE                    298.0 K
D1            0.10000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     7.75 usec
PL1                   -3.00 dB
SFO1          100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2              mlev16
NUC2                     1H
PCPD2                 90.00 usec
PL2                    0.00 dB
PL12                  17.70 dB
SFO2          400.1328009 MHz

F2 - Processing parameters
SI                    65536
SF            100.6127500 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    22.80 cm
CY                    15.50 cm
F1P                 200.000 ppm
F1                 20122.55 Hz
F2P                 -10.000 ppm
F2                 -1006.13 Hz
PPMCM               9.21053 ppm/cm
HZCM              926.69629 Hz/cm
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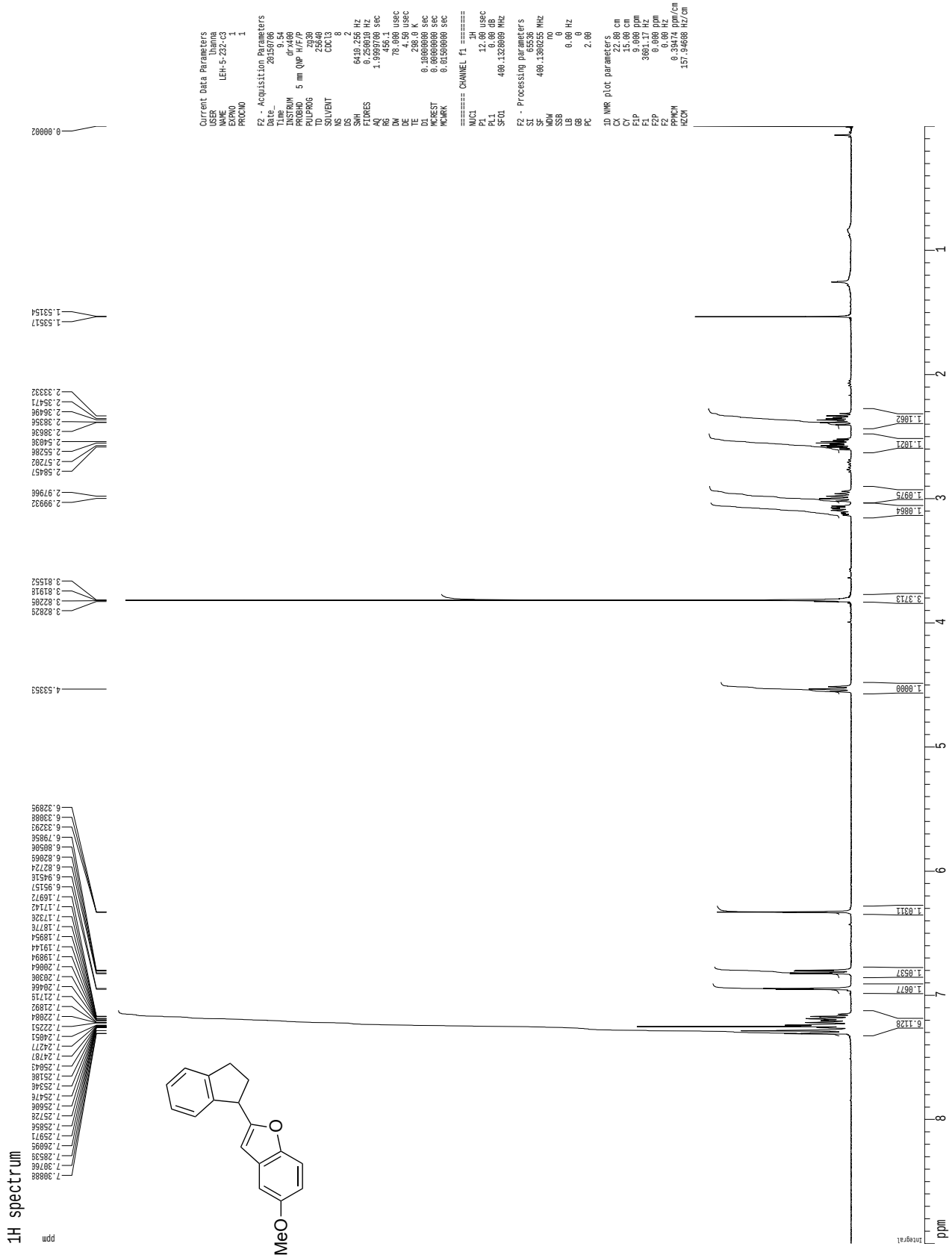
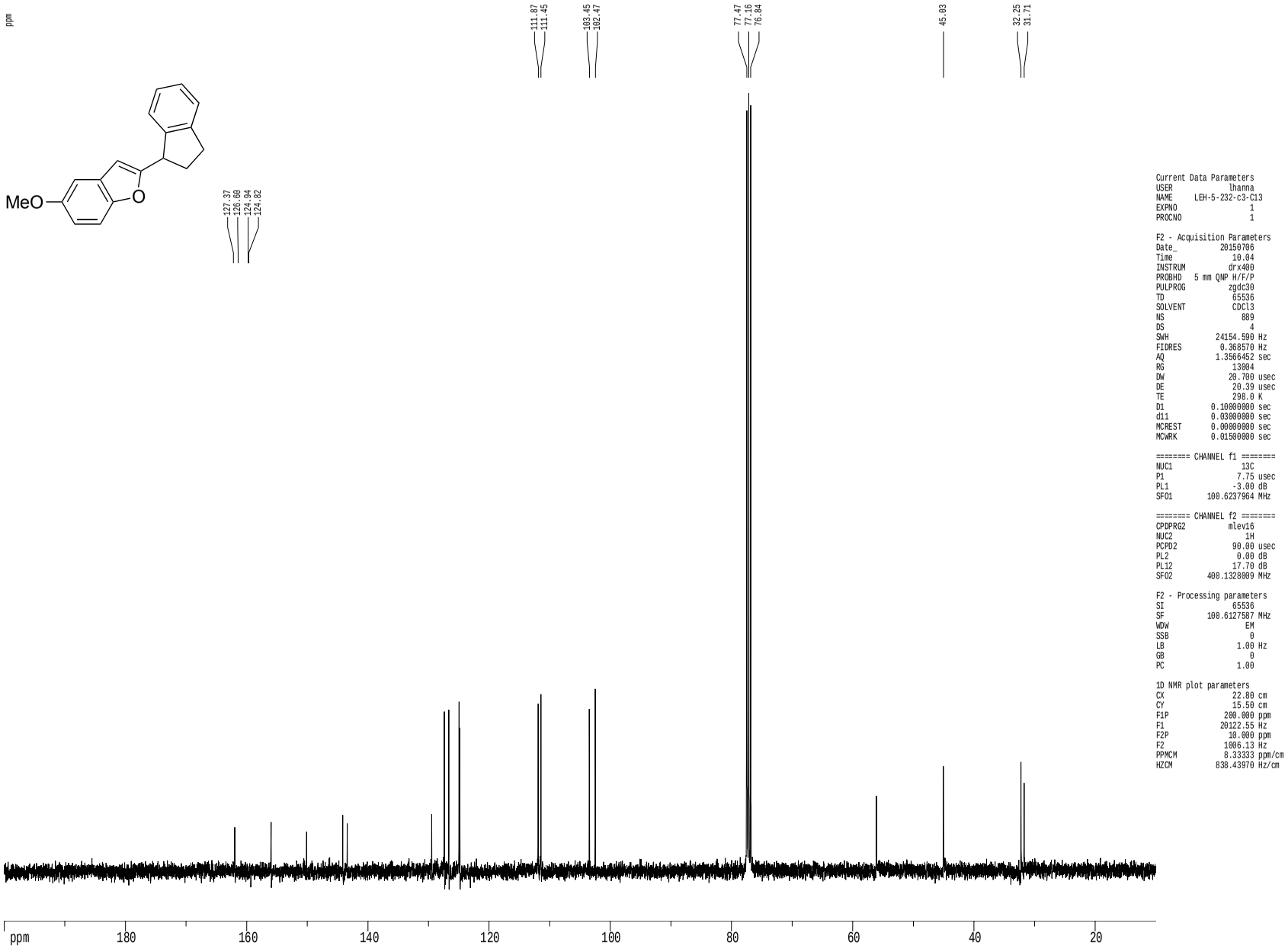

1H spectrum
MeO
ppm

13C spectrum with 1H decoupling



Current Data Parameters
USER lhanna
NAME LEH-5-232-c3-C13
EXPNO 1
PROCNO 1

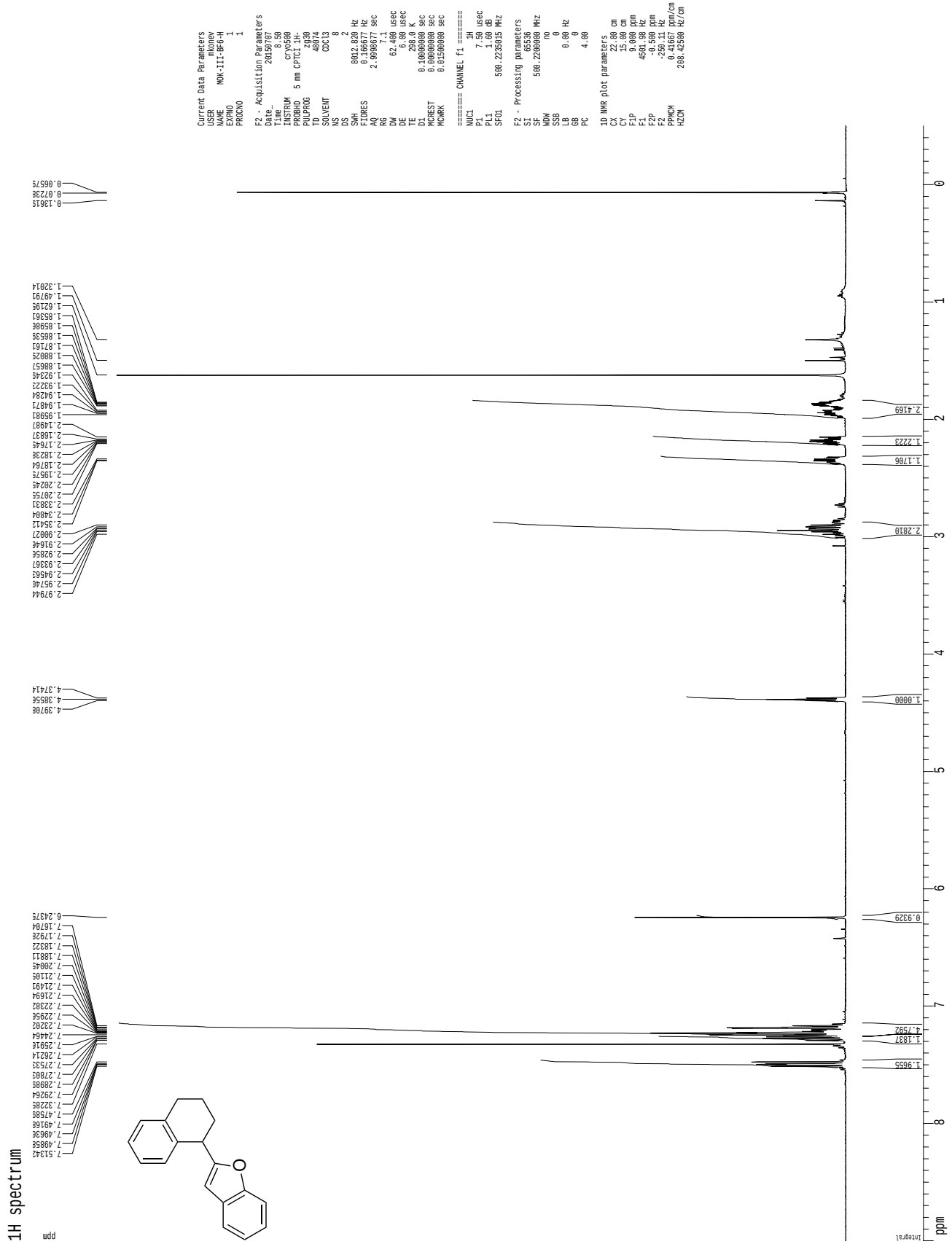
F2 - Acquisition Parameters
Date_ 20150706
Time 10.04
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 889
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 13004
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

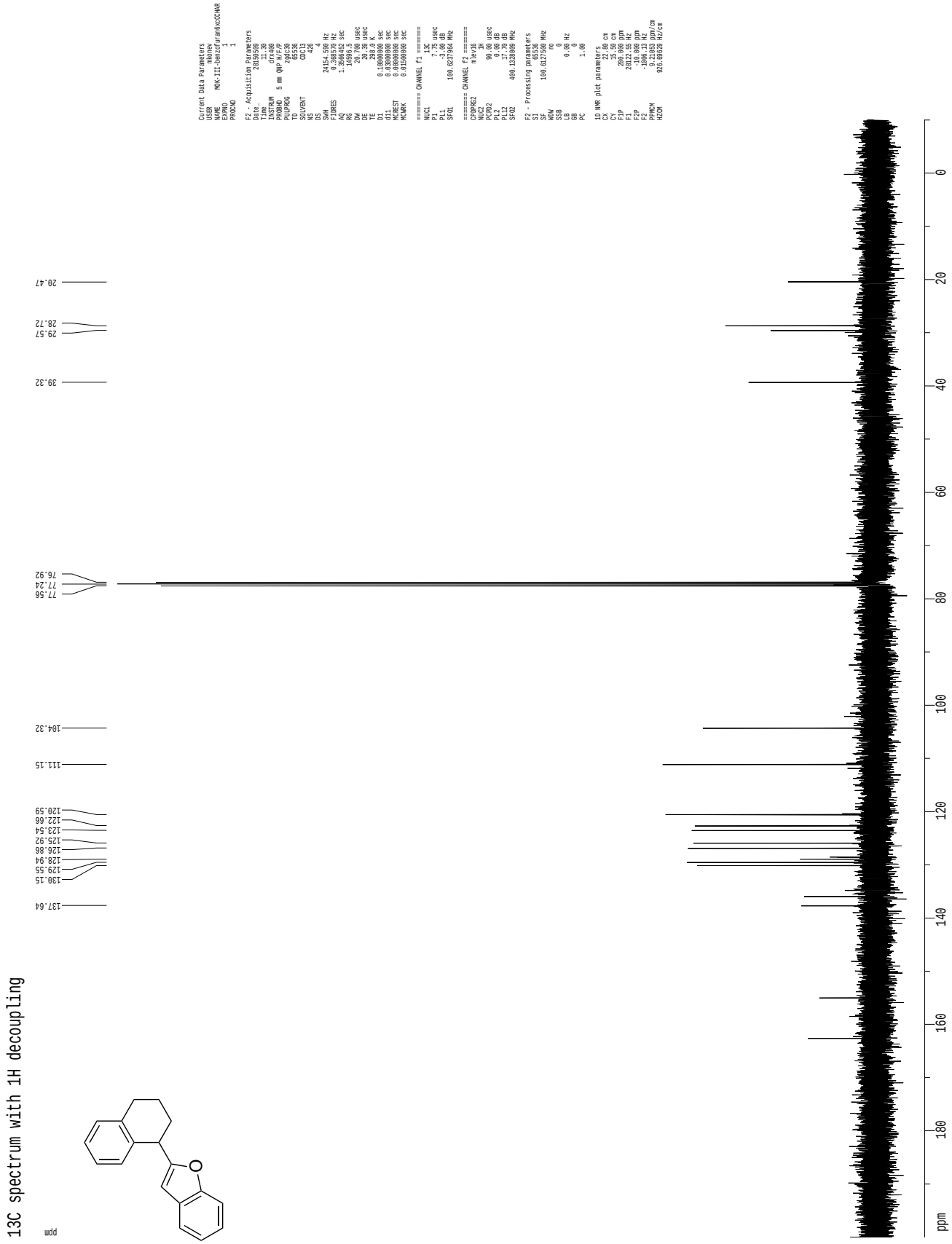
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

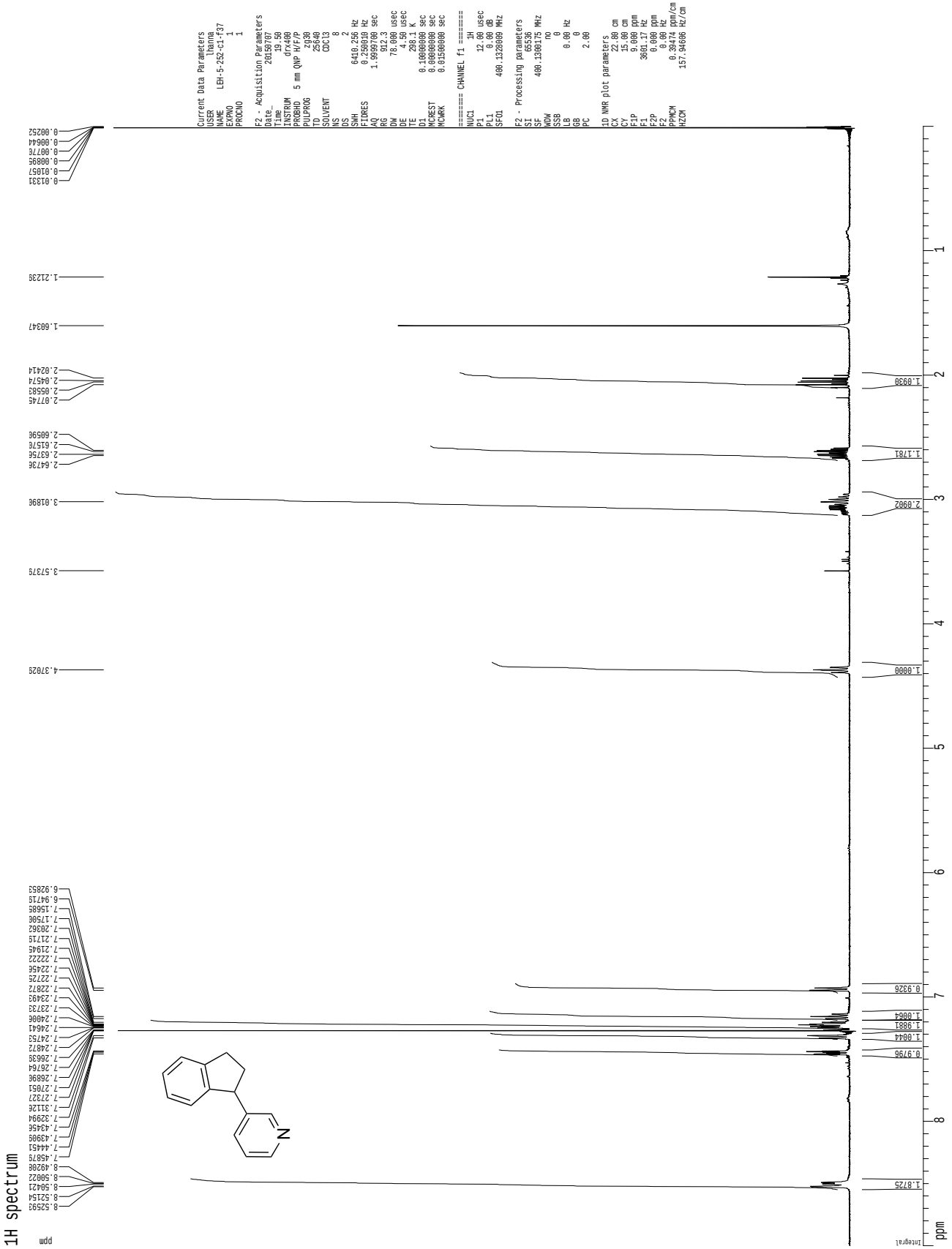
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127587 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

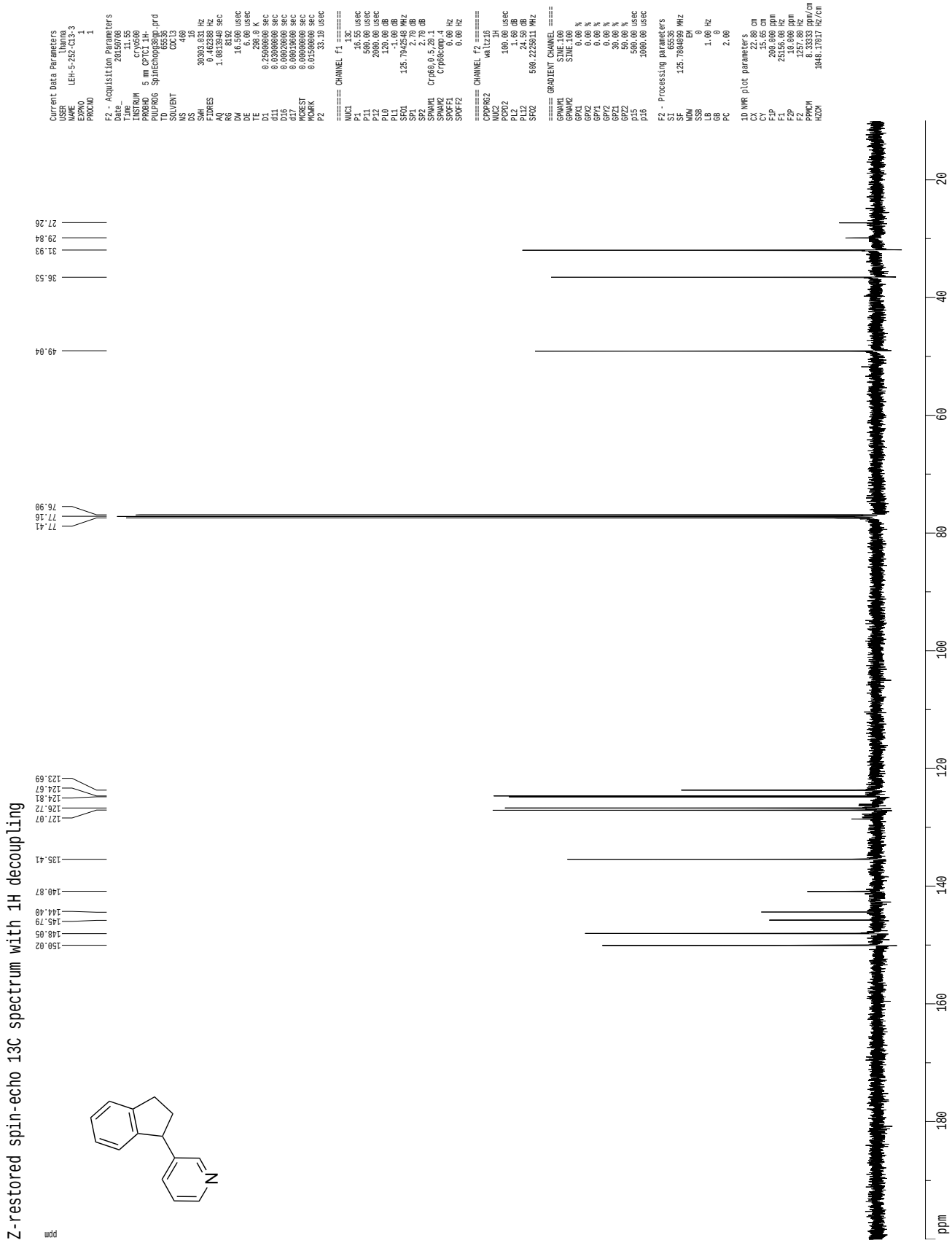
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 10.000 ppm
F2 1006.13 Hz
PPMCM 8.33333 ppm/cm
HZCM 838.43970 Hz/cm

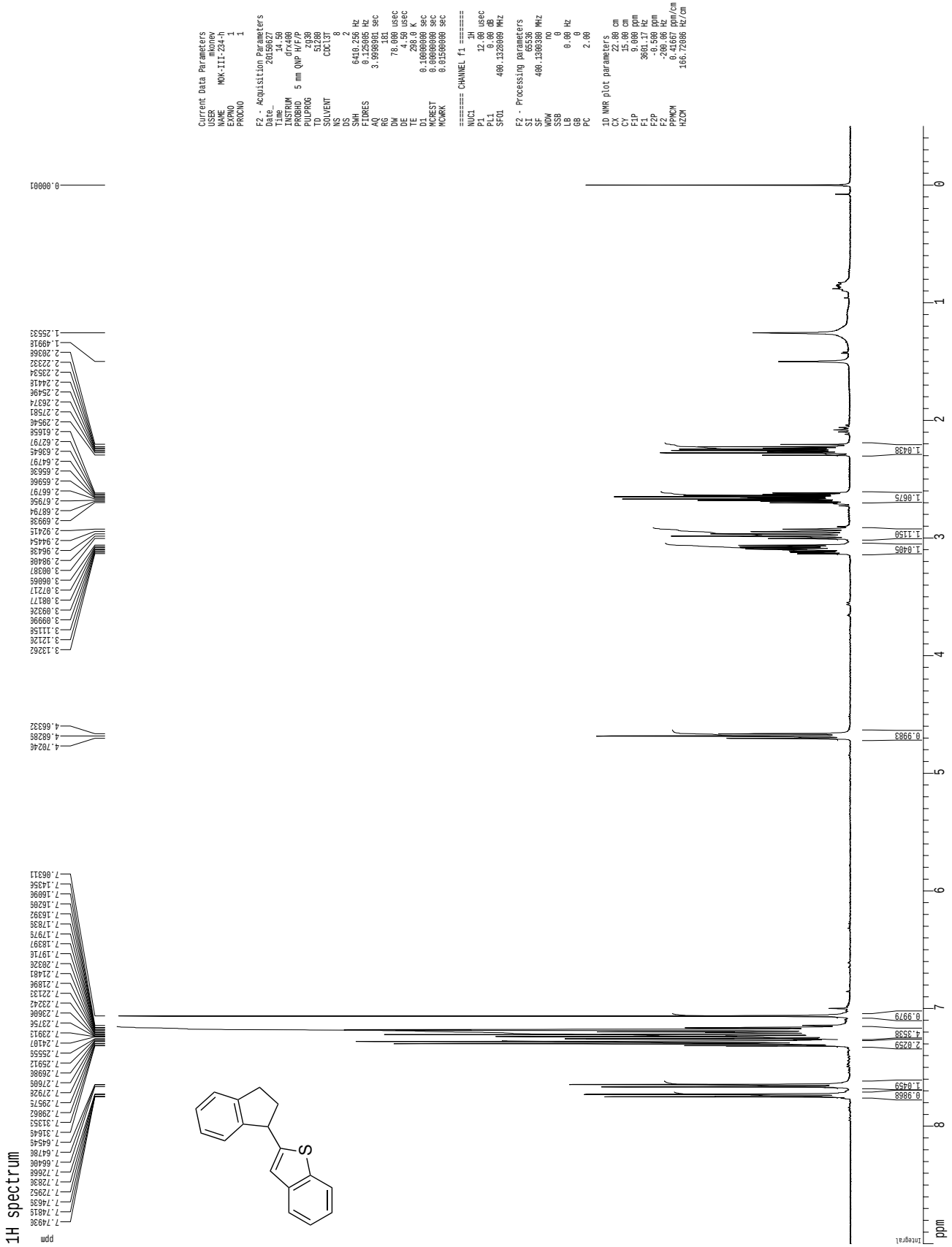

1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-III-BF6-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150707
Time 8.50
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 48074
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.166677 Hz
AQ 2.9998677 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P -0.696 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42500 Hz/cm
0.06575
0.07238
0.13615
1.32014
1.49791
1.62195
1.85361
1.86986
1.86535
1.87161
1.88025
1.88657
1.92345
1.93223
1.94284
1.94871
1.95981
2.14987
2.16837
2.17645
2.18238
2.18764
2.19575
2.20245
2.20756
2.33831
2.34804
2.35412
2.90027
2.91646
2.92856
2.93367
2.94563
2.95746
2.97944
4.37408
4.38556
4.37414
6.24375
7.16704
7.17928
7.18322
7.18811
7.20046
7.21106
7.21491
7.21694
7.22382
7.22956
7.23202
7.24464
7.25916
7.26214
7.27533
7.27883
7.28985
7.29264
7.32285
7.47588
7.49168
7.49636
7.49858
7.51342
2.41691
1.22231
1.17061
2.28101
1.00000
0.93291
4.75921
1.18371
1.96551
integral
ppm
0
1
2
3
4
5
6
7
8
ppm

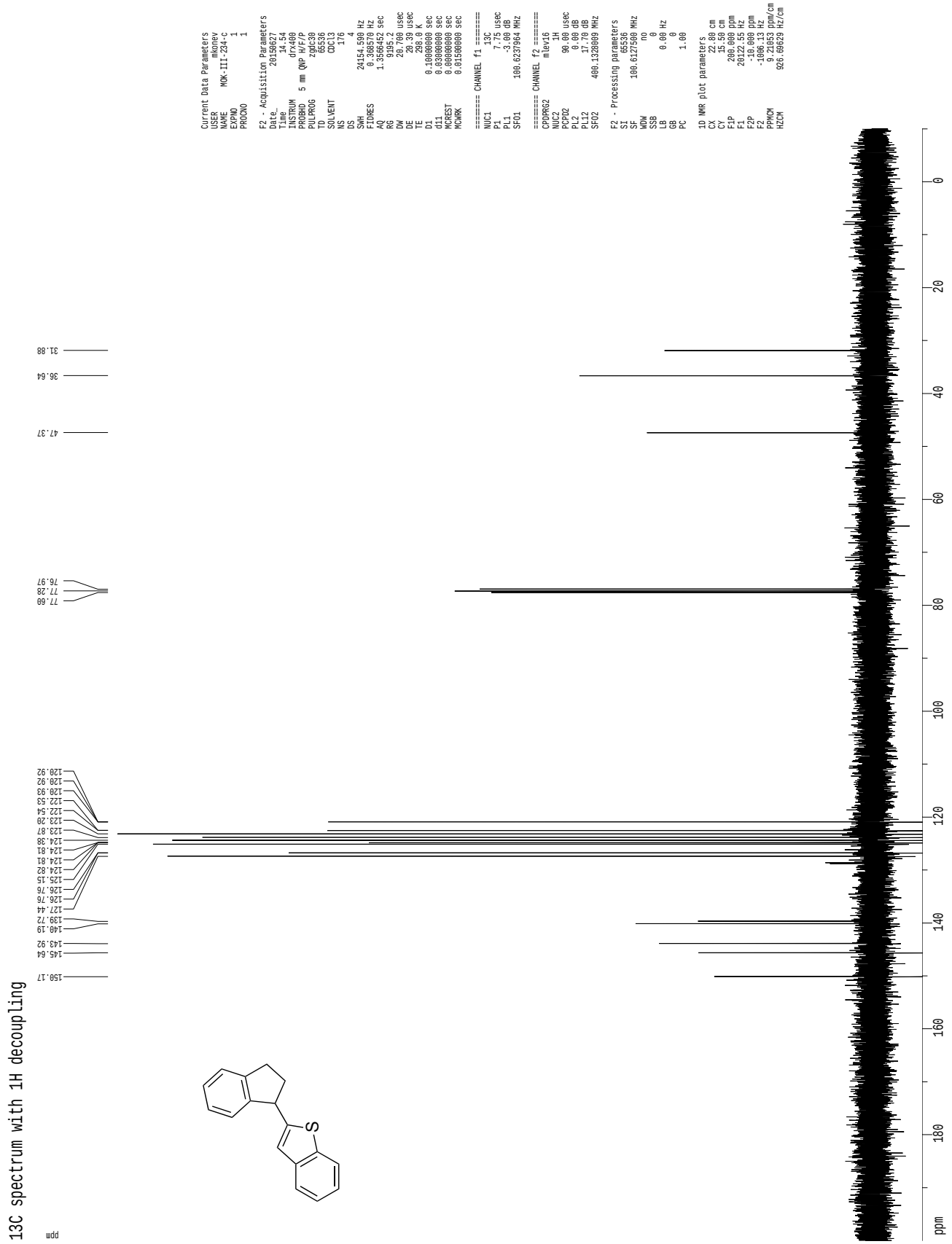

13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER mkoniev
NAME MOK-III-benzofuran6ocCCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20135599
Time 11.36
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 426
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566462 sec
RG 145949.5
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUCL 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237994 MHz
======== CHANNEL f2 ========
CPDPRG2 mlevi6
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320089 MHz
F2 - Processing parameters
SI 65536
SF 100.6127560 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1986.11 Hz
PPMCM 0.21355 ppm/cm
HZCM 926.68828 Hz/cm
20.47
28.72
29.57
39.32
76.92
77.24
77.56
104.32
111.15
120.59
122.66
123.54
125.92
126.86
128.94
129.55
130.15
137.64
0
20
40
60
80
100
120
140
160
180
ppm

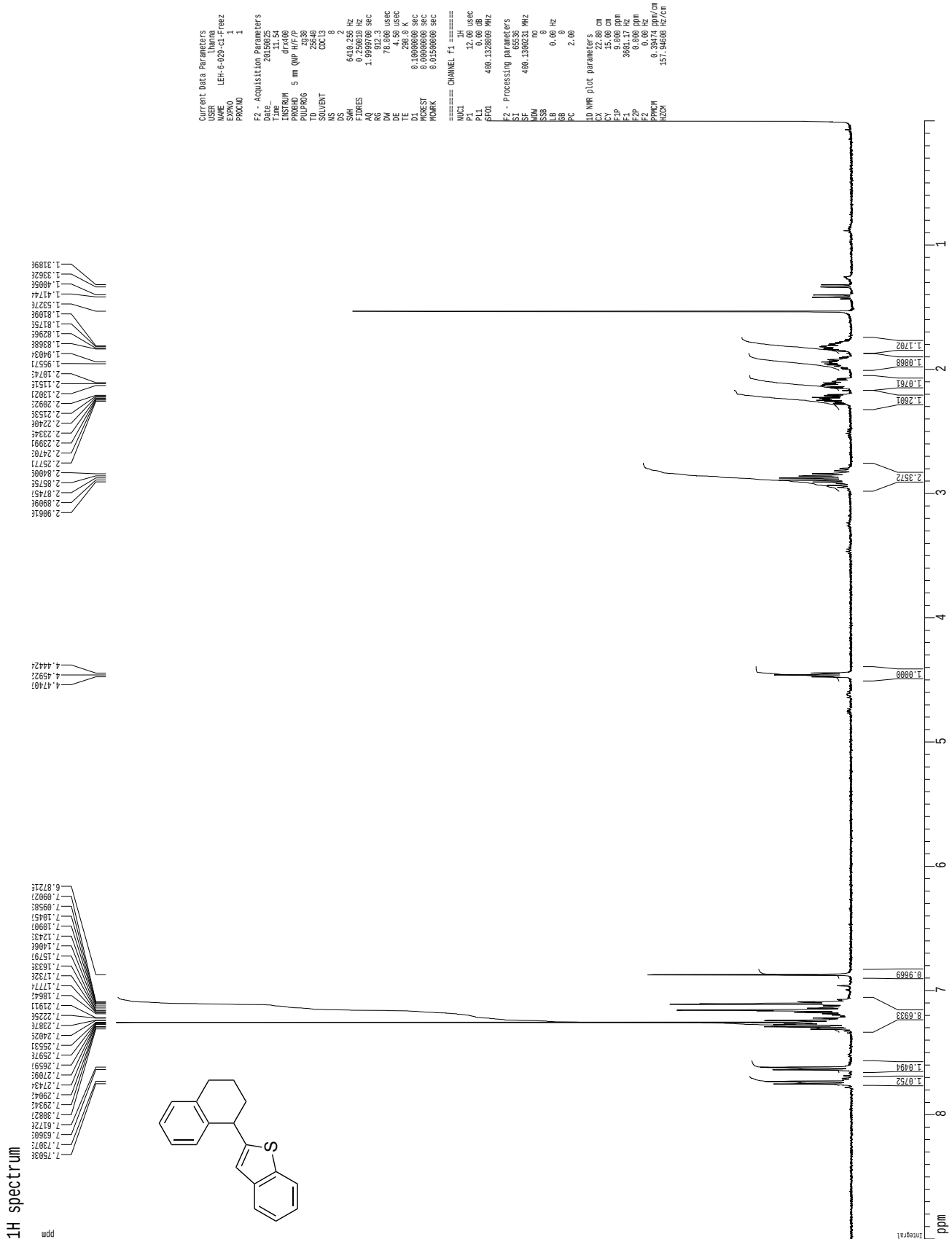

1H spectrum
Current Data Parameters
USER Thesis
NAME LEH-5-252-c1-f37
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150707
Time 19.50
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250098 Hz
AQ 1.9961088 sec
RG 912.3
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
0.00252
0.00644
0.00776
0.00896
0.01057
0.01331
1.21239
1.60347
2.02414
2.04574
2.05583
2.07745
2.60596
2.61576
2.63756
2.64736
3.01896
3.57379
4.37029
6.92853
6.94718
7.15685
7.17506
7.20362
7.21718
7.21946
7.22222
7.22456
7.22726
7.22872
7.23493
7.23733
7.24006
7.24641
7.24753
7.24872
7.26639
7.26764
7.26896
7.27051
7.27327
7.31126
7.32994
7.49456
7.49905
7.44451
7.45875
8.49268
8.50022
8.50421
8.52154
8.52593
1.0930
1.1781
2.0902
1.0000
0.9326
1.0064
1.9881
1.0044
0.9796
1.8725
Integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

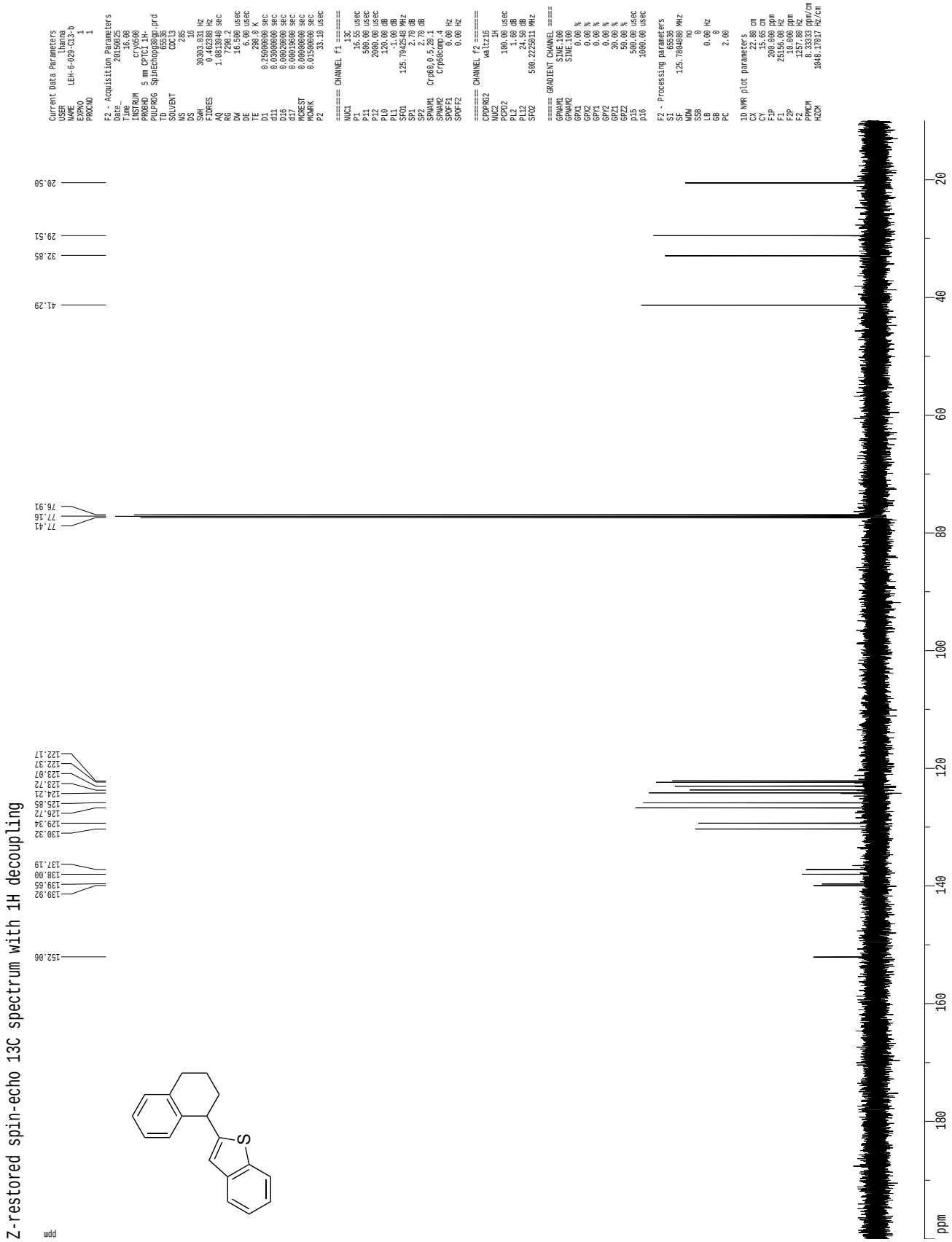


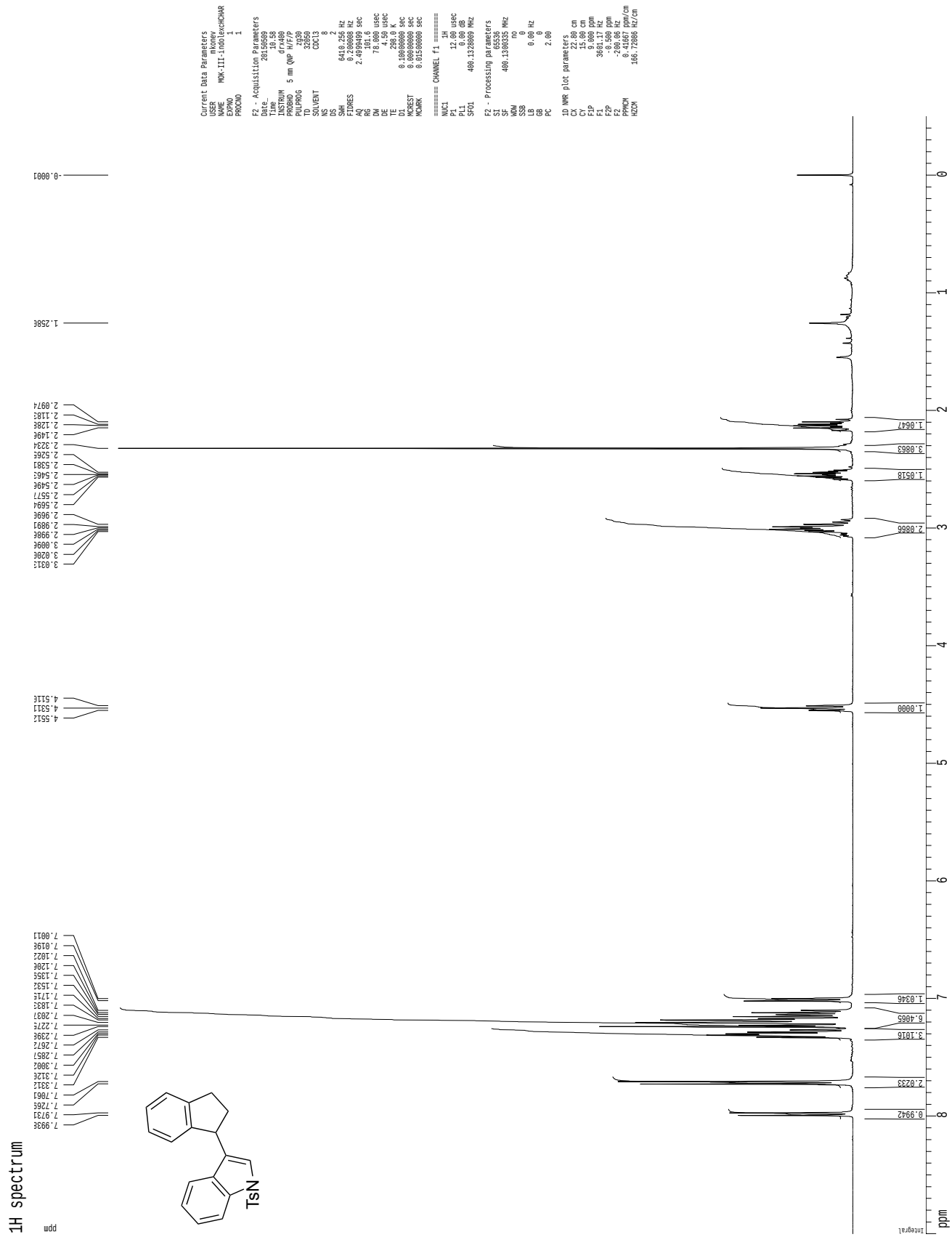

1H spectrum
Current Data Parameters
USER mcgrady
NAME MCK-III-234-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150627
Time 14.50
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 51200
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.125095 Hz
AQ 3.9998601 sec
RG 181
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300338 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -208.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72080 Hz/cm
0.00001
1.25532
1.49918
2.20386
2.22322
2.23534
2.24418
2.25496
2.26374
2.27583
2.29546
2.61658
2.62797
2.63645
2.64797
2.65636
2.65966
2.66797
2.67956
2.68794
2.69938
2.92415
2.94454
2.96438
2.98408
3.00387
3.06065
3.07217
3.08177
3.09326
3.09996
3.11158
3.12126
3.13262
4.66532
4.68285
4.70246
7.06311
7.14356
7.16096
7.16296
7.16392
7.17835
7.17975
7.18397
7.19716
7.20328
7.21483
7.21896
7.22123
7.23242
7.23606
7.23756
7.23912
7.24107
7.25556
7.25912
7.26906
7.27605
7.27928
7.29575
7.29862
7.31353
7.31645
7.64545
7.64786
7.66406
7.72668
7.72952
7.72836
7.74539
7.74813
7.74936
ppm
1.0438
1.0675
1.1150
1.0405
0.9983
0.9979
4.3538
2.0259
1.0459
0.9868
Integral
ppm
0
1
2
3
4
5
6
7
8
S

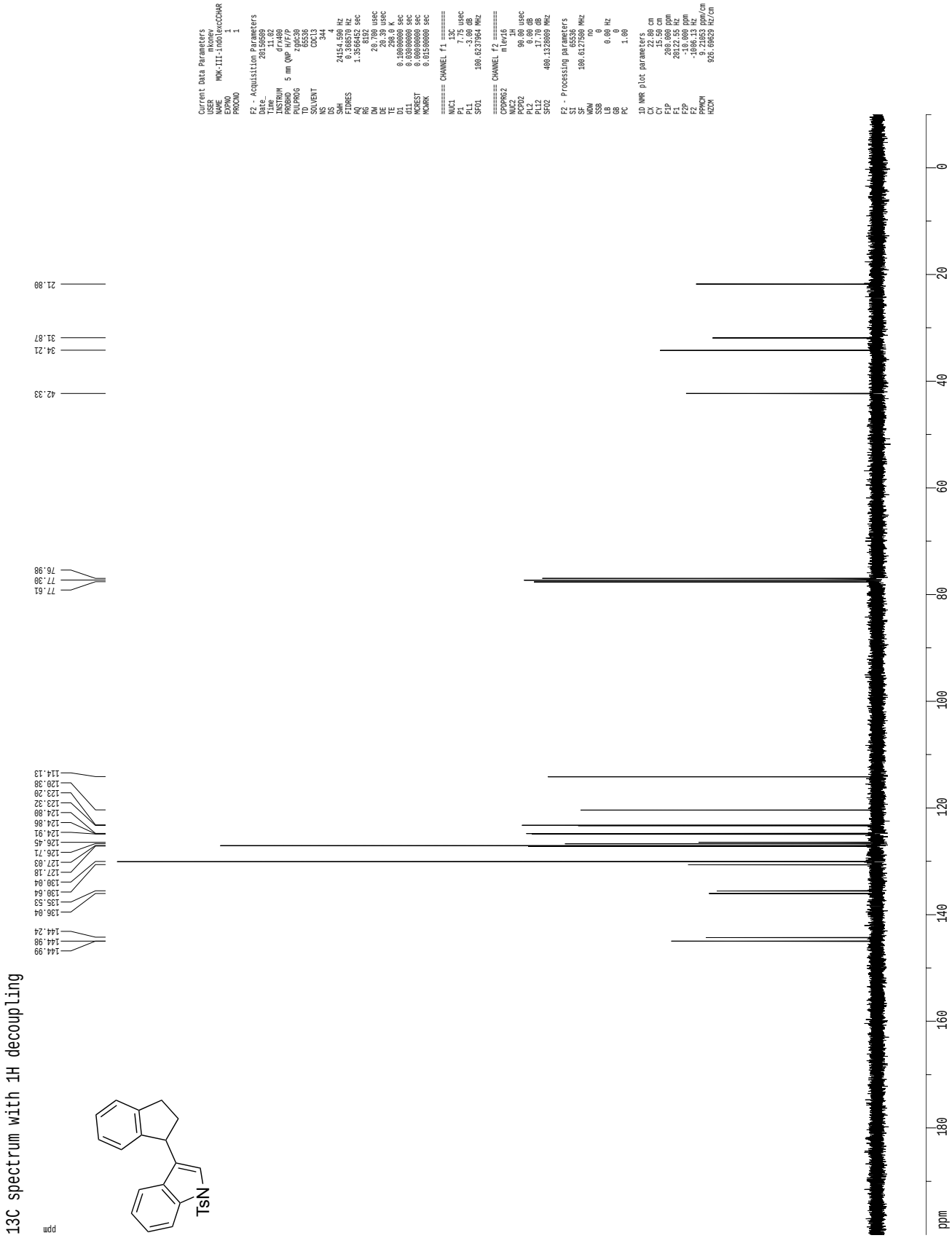

13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER mkonev
NAME MOK-III-234-c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150627
Time 14.54
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.35566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.76 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127560 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
31.88
36.64
47.37
76.97
77.28
77.60
120.92
120.92
120.93
122.53
122.54
123.20
123.87
124.38
124.81
124.81
124.82
126.15
126.76
126.76
127.44
139.72
140.19
143.92
145.64
150.17
S
0
20
40
60
80
100
120
140
160
180
ppm

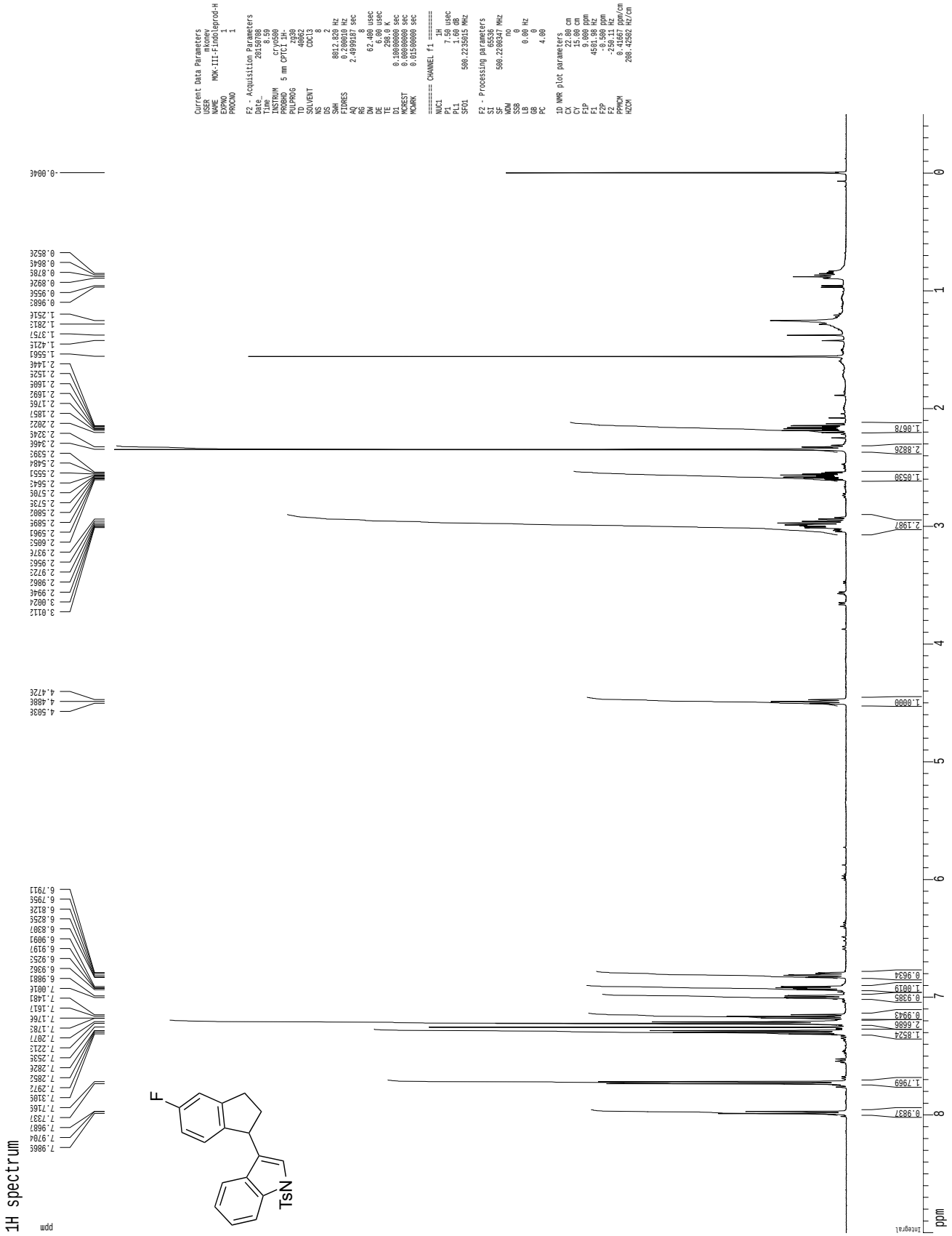

1H spectrum
Current Data Parameters
USER lbanna
NAME LEH-6-029-c1-Freez
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
TIME 11.54
INSTRUM dfrq400
PROBHD 5 mm QNP H/C/D
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 512.3
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300231 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94608 Hz/cm
1.1702
1.0868
1.0761
1.2601
2.3572
1.0000
0.9669
8.6933
1.0494
1.0752
Integral
ppm
S

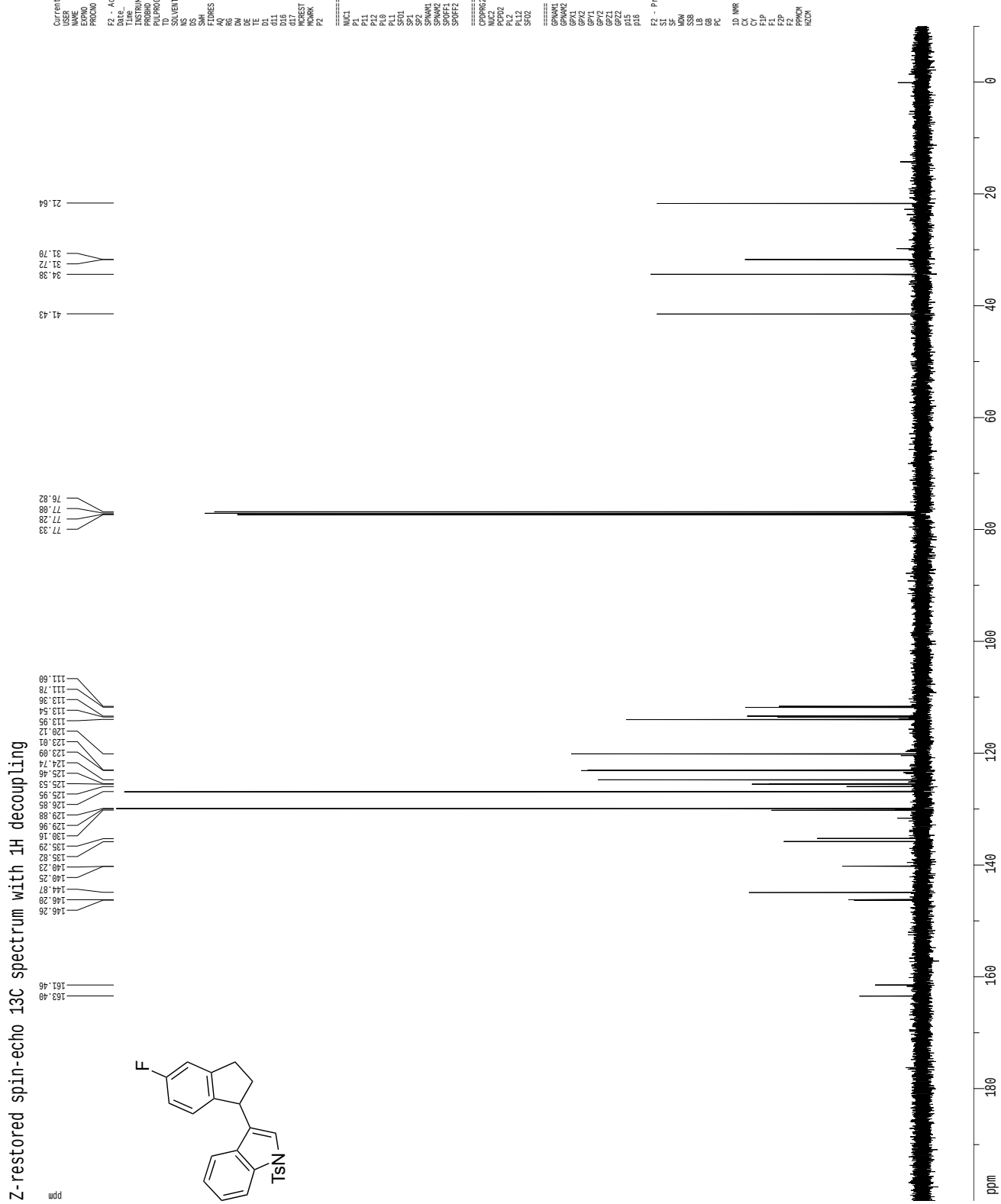
Z-restored spin-echo 13C spectrum with 1H decoupling

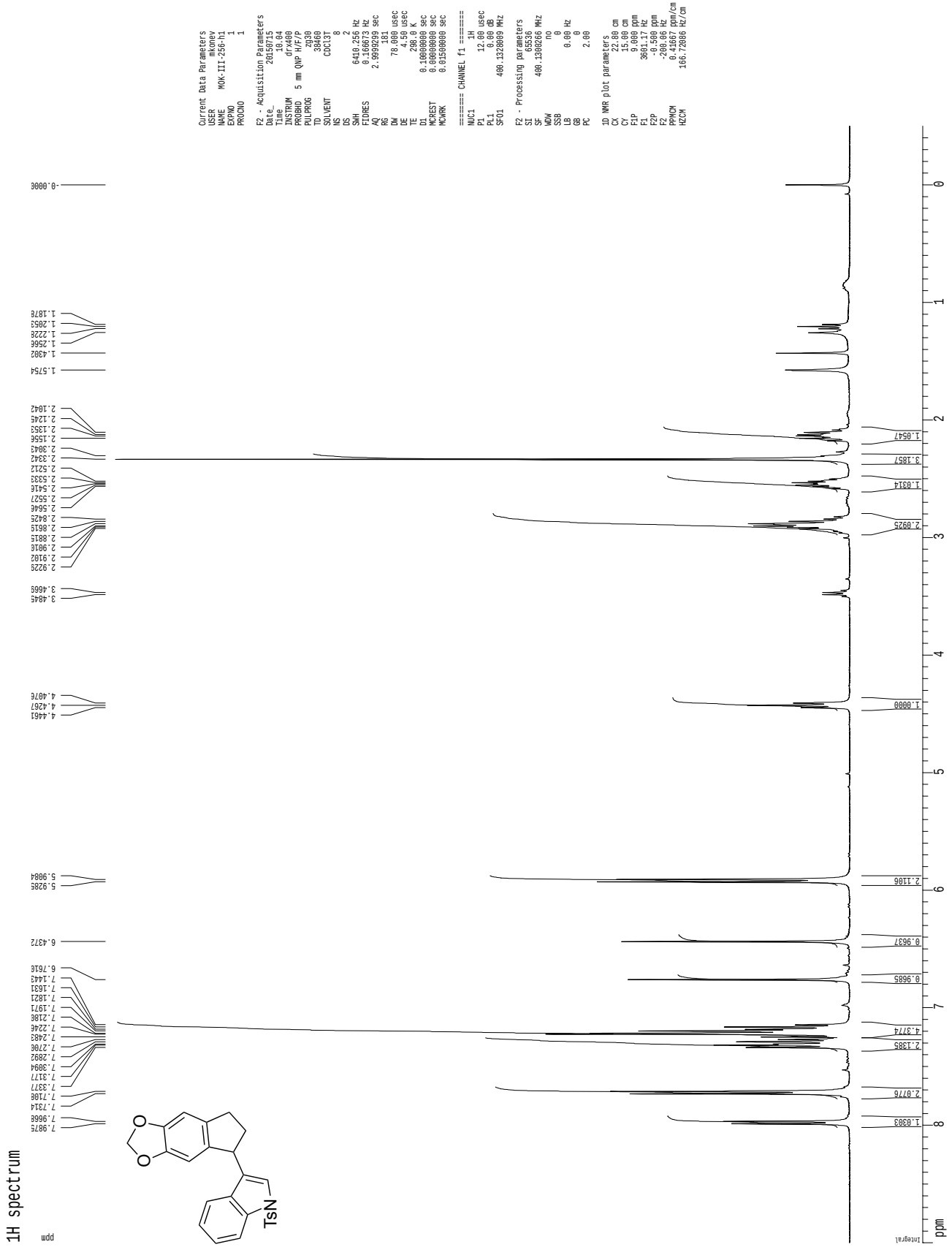



1H spectrum
Current Data Parameters
USER mkonev
NAME MOX-III-indolexchAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 10.58
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.195648 Hz
AQ 2.5559040 sec
RG 181.5
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300335 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 0.000 ppm
F1 3001.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72086 Hz/cm
TsN
ppm
Integral


13C spectrum with 1H decoupling
TsN
ppm
Current Data Parameters
USER mkonev
NAME MOX-III-indolexcCHAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150509
Time 11.02
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 344
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3564416 sec
RG 8192
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237984 MHz
======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D WNR plot parameters
CX 22.00 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
21.80
31.87
34.21
42.33
76.98
77.30
77.61
114.13
120.38
123.20
123.32
124.80
124.86
124.91
126.45
126.71
127.03
127.18
130.04
130.64
135.53
136.04
144.24
144.98
144.99
ppm


1H spectrum
Current Data Parameters
USER mKonev
NAME MOK-III-Flindoleprod-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150708
Time 8.59
INSTRUM CryoDRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 40962
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.195628 Hz
AQ 2.4999107 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200347 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 27.80 cm
CY 15.000 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41657 ppm/cm
HZCM 208.42502 Hz/cm
F
TsN
ppm
Integral
1.0678
2.8826
1.0530
2.1987
1.0000
0.9634
1.0019
0.9385
0.9943
2.6686
1.8524
1.7969
0.9837


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
F
TsN
Current Data Parameters
USER mdw
NAME MOK-III-Findolegrod-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150708
Time 9.03
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchop130gp.prd
TD 65536
SOLVENT CDCl3
NS 672
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.08113940 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d12 0.00001600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
PL1 500.00 usec
PL2 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.79425548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.22229011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.78041190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.65 cm
F1P 209.000 ppm
F1 25155.88 Hz
F2P -10.000 ppm
F2 -1257.800 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.59378 Hz/cm
21.64
31.70
31.72
34.38
41.43
76.82
77.08
77.28
77.33
111.60
111.78
113.36
113.54
113.95
120.12
123.01
123.09
124.74
125.46
125.53
125.96
126.85
129.88
129.96
130.16
135.29
135.82
140.23
140.25
144.87
146.20
146.26
161.46
163.40
0
20
40
60
80
100
120
140
160
180
ppm


1H spectrum
ppm
TsN
Integral
Current Data Parameters
USER mtovey
NAME MOK-III-256-h1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150715
Time 10.04
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 38460
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.166573 Hz
AQ 2.9999819 sec
RG 181
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300266 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -209.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72800 Hz/cm
0.0000
1.5754
1.4302
1.2228
1.2053
1.1878
2.1042
2.1246
2.1353
2.1556
2.3043
2.3342
2.5212
2.5333
2.5416
2.5527
2.5646
2.8425
2.8619
2.8815
2.9016
2.9102
2.9229
3.4845
3.4669
4.4076
4.4267
4.4461
5.9084
5.9286
6.4372
6.7616
7.1443
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7.1821
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7.2706
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7.3377
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7.7314
7.9668
7.9875
1.0547
3.1857
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4.3774
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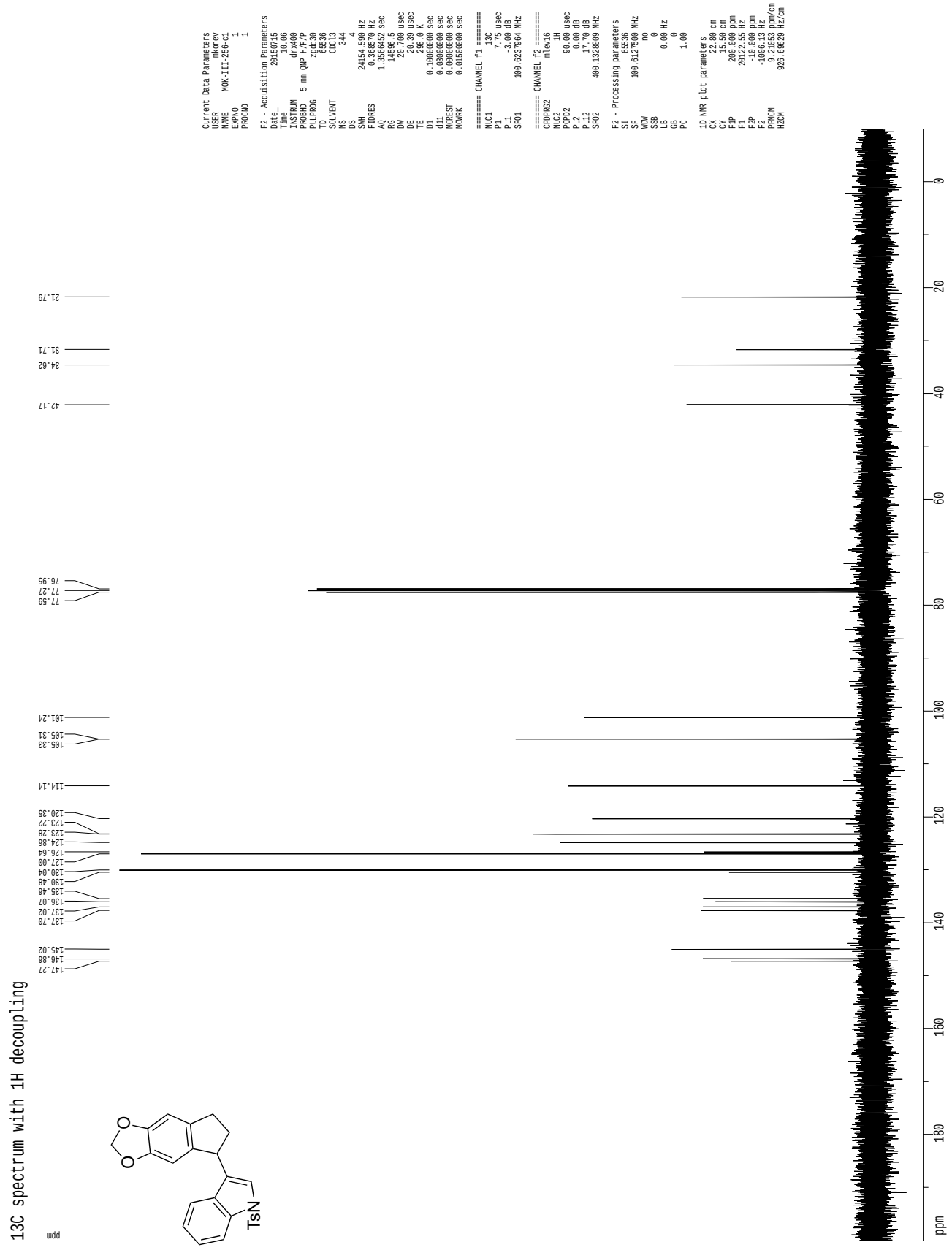
2.0776
1.0303


13C spectrum with 1H decoupling
Current Data Parameters
USER mkonev
NAME MOK-III-236-c1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150715
Time 18.06
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 344
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 14596.5
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237984 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127560 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.21053 ppm/cm
HZCM 926.69629 Hz/cm
21.79
31.71
34.62
42.17
76.95
77.27
77.59
101.24
105.31
105.33
114.14
120.35
123.22
123.28
124.86
126.64
127.00
130.04
130.48
135.46
136.07
137.02
137.70
145.02
146.86
147.27
TsN
ppm
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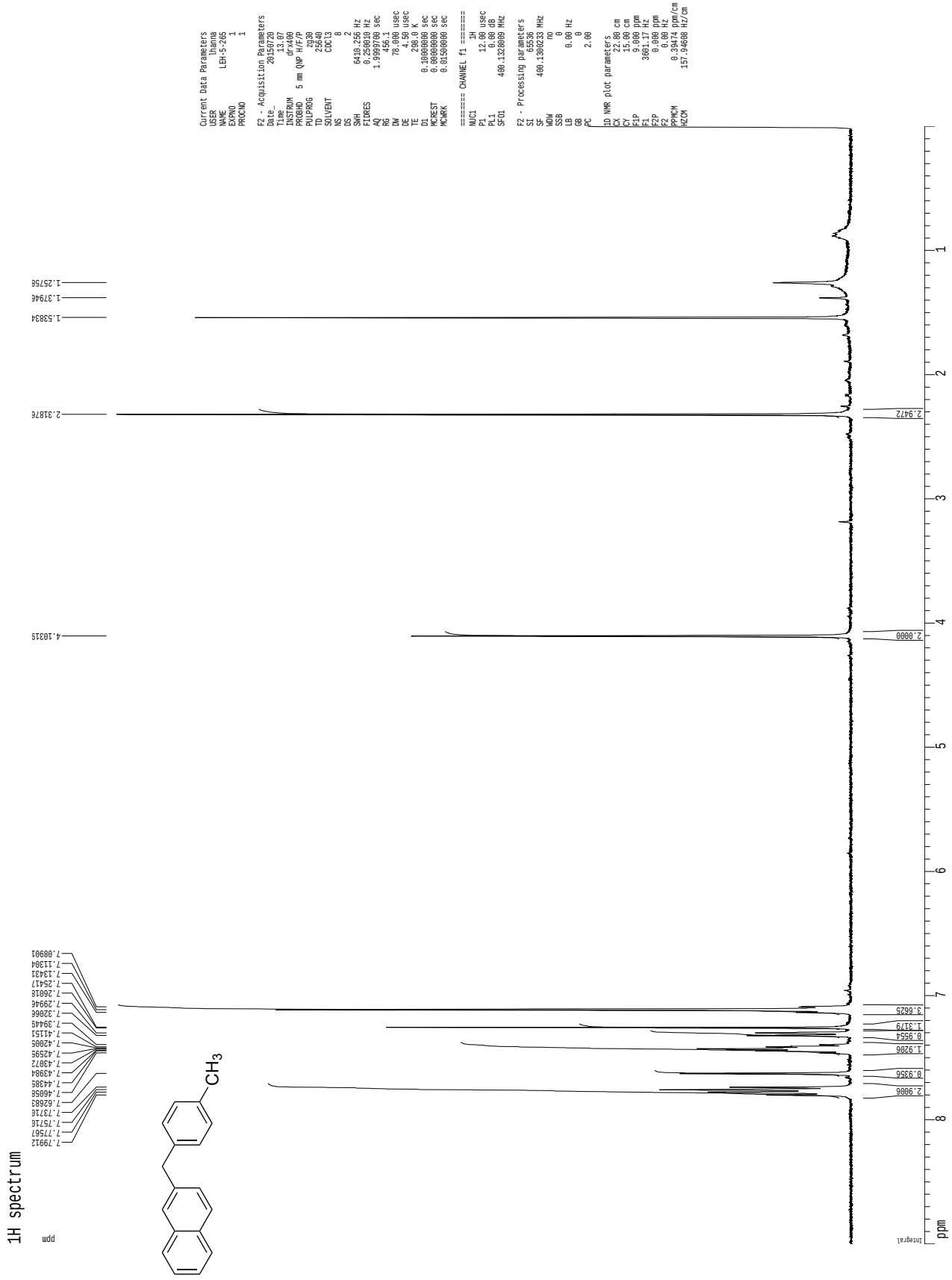
20
40
60
80
100
120
140
160
180


1H spectrum
ppm
Integral
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USER lhanna
NAME LEH-5-265
EXPNO 1
PROCNO 1
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Date_ 20150720
Time 13.07
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.255 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 456.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300233 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.0000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94698 Hz/cm
1.25758
1.37946
1.53834
2.31876
4.10319
7.79912
7.77567
7.75714
7.73714
7.62683
7.46058
7.44385
7.43984
7.43072
7.42596
7.42006
7.41151
7.39449
7.32066
7.29946
7.26018
7.25417
7.13431
7.11384
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2.0000
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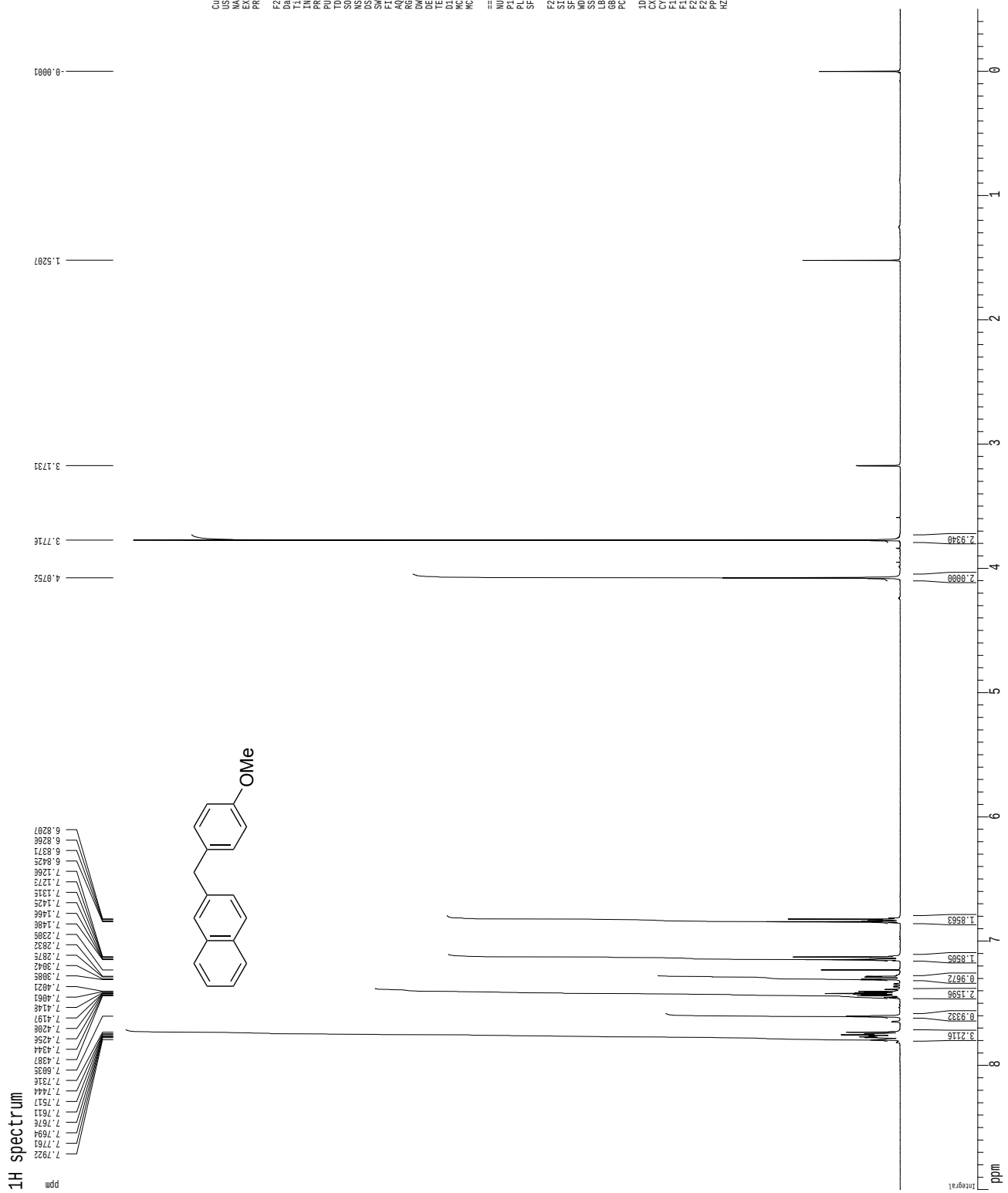
0.9554
1.9206
0.9356
2.9006
CH3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ppm
Integral
OMe
Current Data Parameters
USER mydemo
NAME MOK-III-253
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 12.33
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32050
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.200000 Hz
AQ 2.4999490 sec
RG 256
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300332 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72080 Hz/cm
7.7922
7.7761
7.7694
7.7676
7.7611
7.7512
7.7444
7.7316
7.6035
7.4387
7.4344
7.4256
7.4208
7.4197
7.4148
7.4061
7.4021
7.3985
7.3042
7.2875
7.2832
7.2309
7.1486
7.1466
7.1425
7.1315
7.1273
7.1266
6.8473
6.8371
6.8266
6.8207
0.0001
1.5207
3.1731
3.7716
4.0752
2.9340
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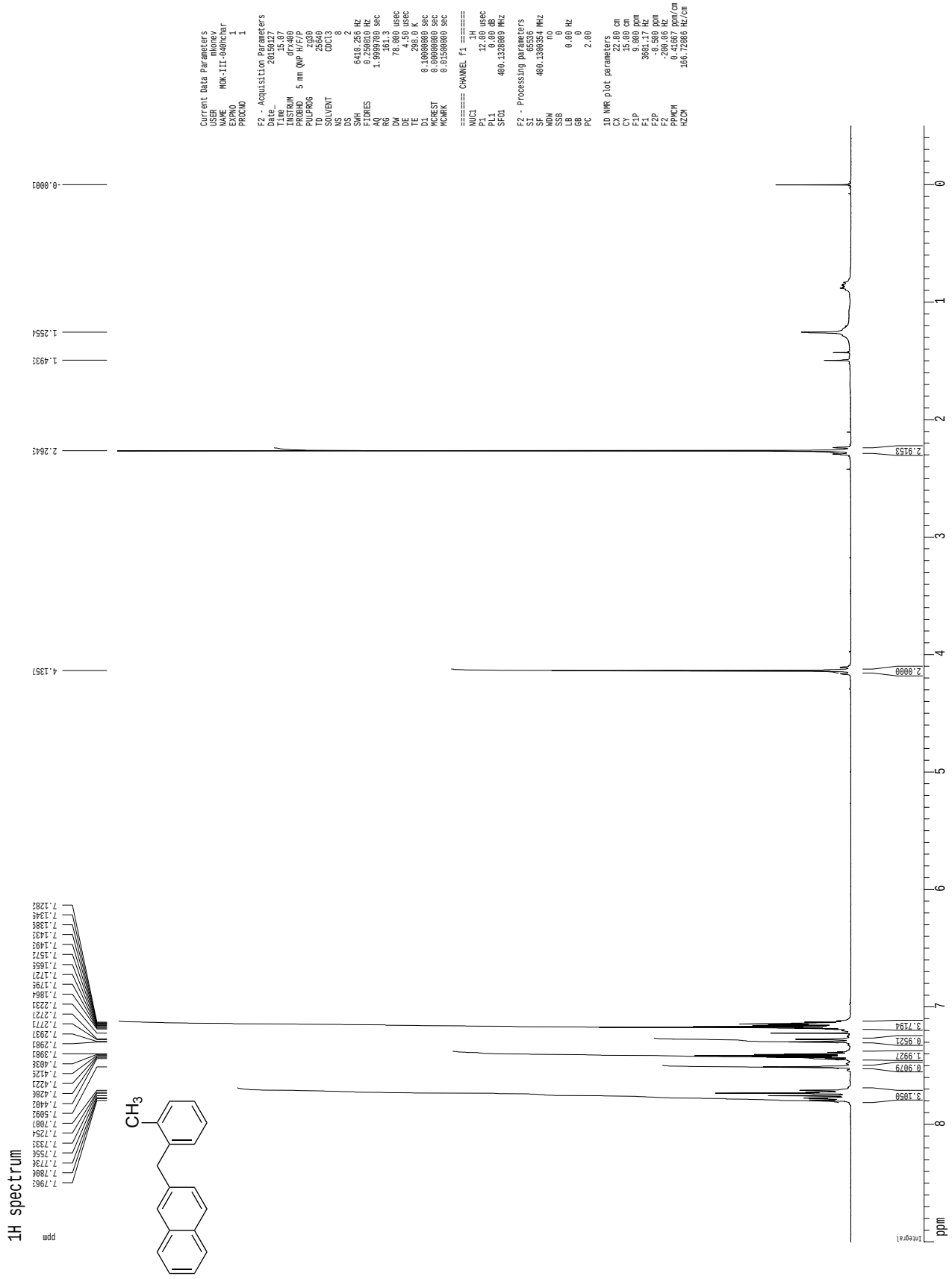
1.8563
1.8505
0.9672
2.1596
0.9332
3.2116


1H spectrum
ppm
integral
CH3
Current Data Parameters
USER mkoney
NAME MOK-III-040hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150127
Time 15.07
INSTRUM dfx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999708 sec
RG 161.3
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300354 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 0.000 ppm
F1 3681.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72086 Hz/cm
0.0001
1.2554
1.4933
2.2643
4.1357
7.1282
7.1346
7.1386
7.1433
7.1492
7.1572
7.1656
7.1721
7.1796
7.1864
7.2281
7.2727
7.2773
7.2937
7.2983
7.3983
7.4038
7.4125
7.4221
7.4286
7.4402
7.5092
7.7087
7.7254
7.7333
7.7556
7.7736
7.7806
7.7963
2.9153
2.0000
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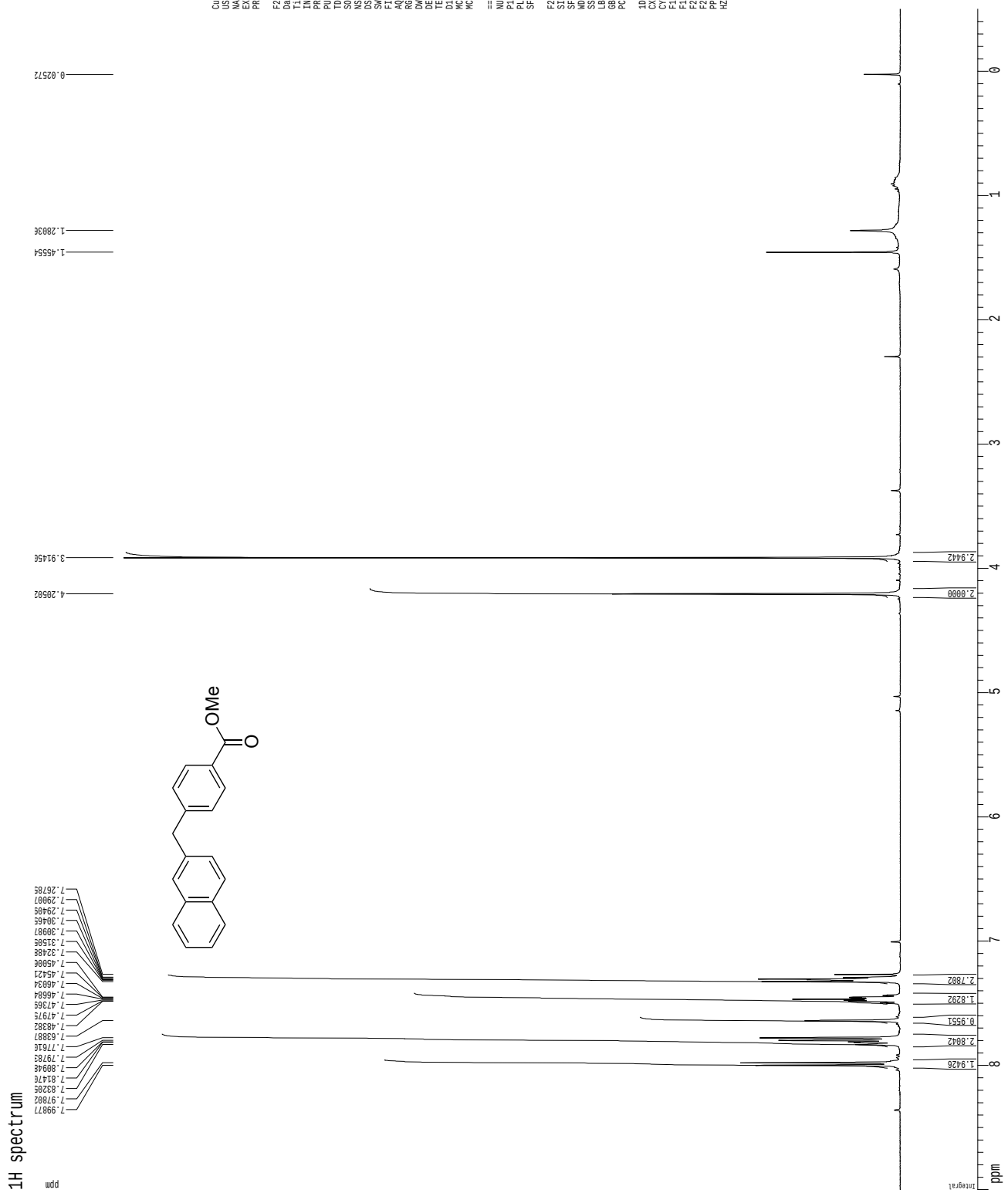
0.9521
1.9927
0.9079
3.1050


1H spectrum
Current Data Parameters
USER mvinay
NAME MOK-III-150
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150302
Time 18.56
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250078 Hz
AQ 1.9961088 sec
RG 203.2
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0.00 Hz
LB 0
GB 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -280.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
0.02572
1.28036
1.45554
3.91456
4.20502
7.99877
7.98202
7.83206
7.81476
7.80948
7.79783
7.77616
7.63887
7.48382
7.47975
7.47365
7.46684
7.46034
7.45421
7.46006
7.32488
7.31506
7.30987
7.30465
7.29405
7.29007
7.26785
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O
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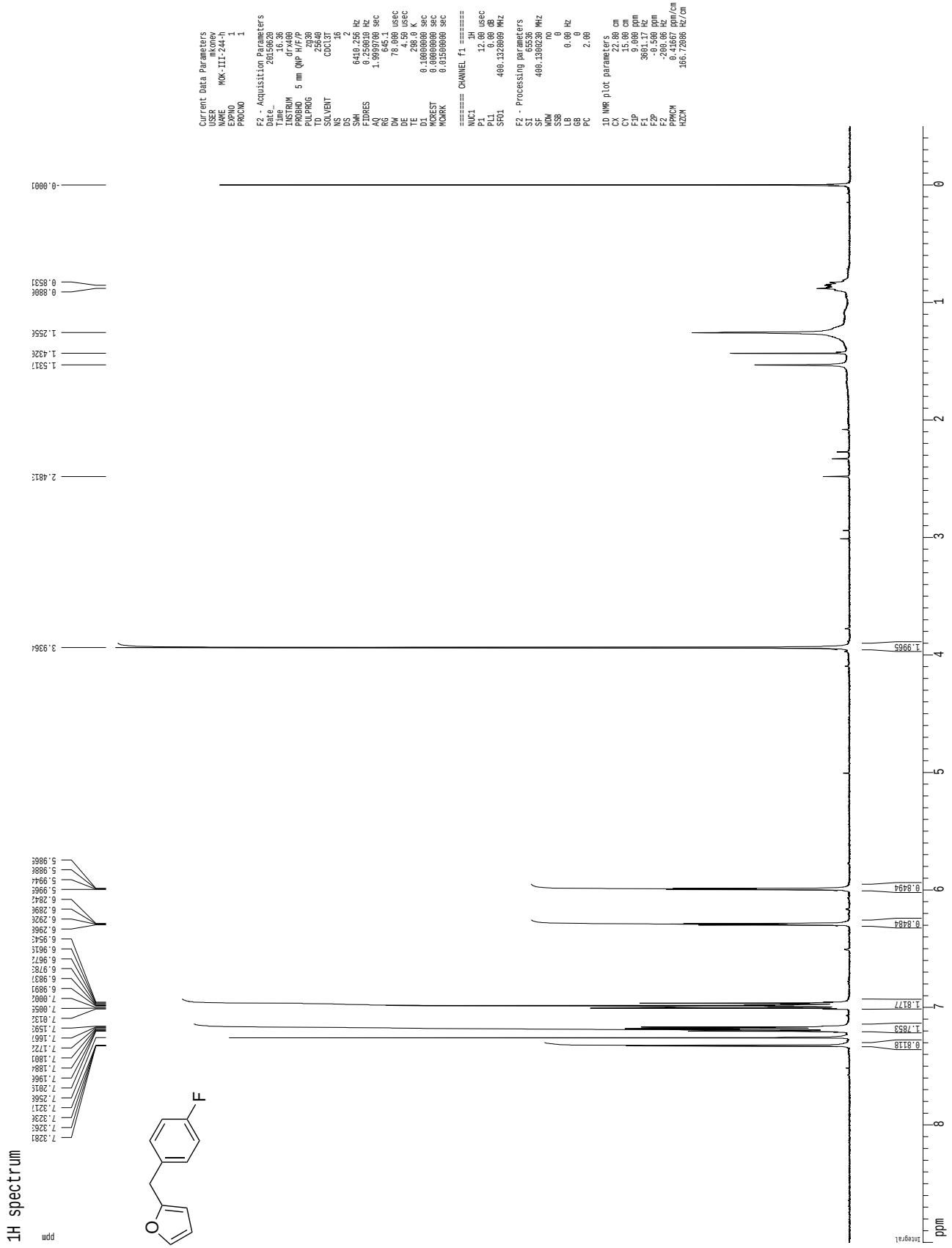
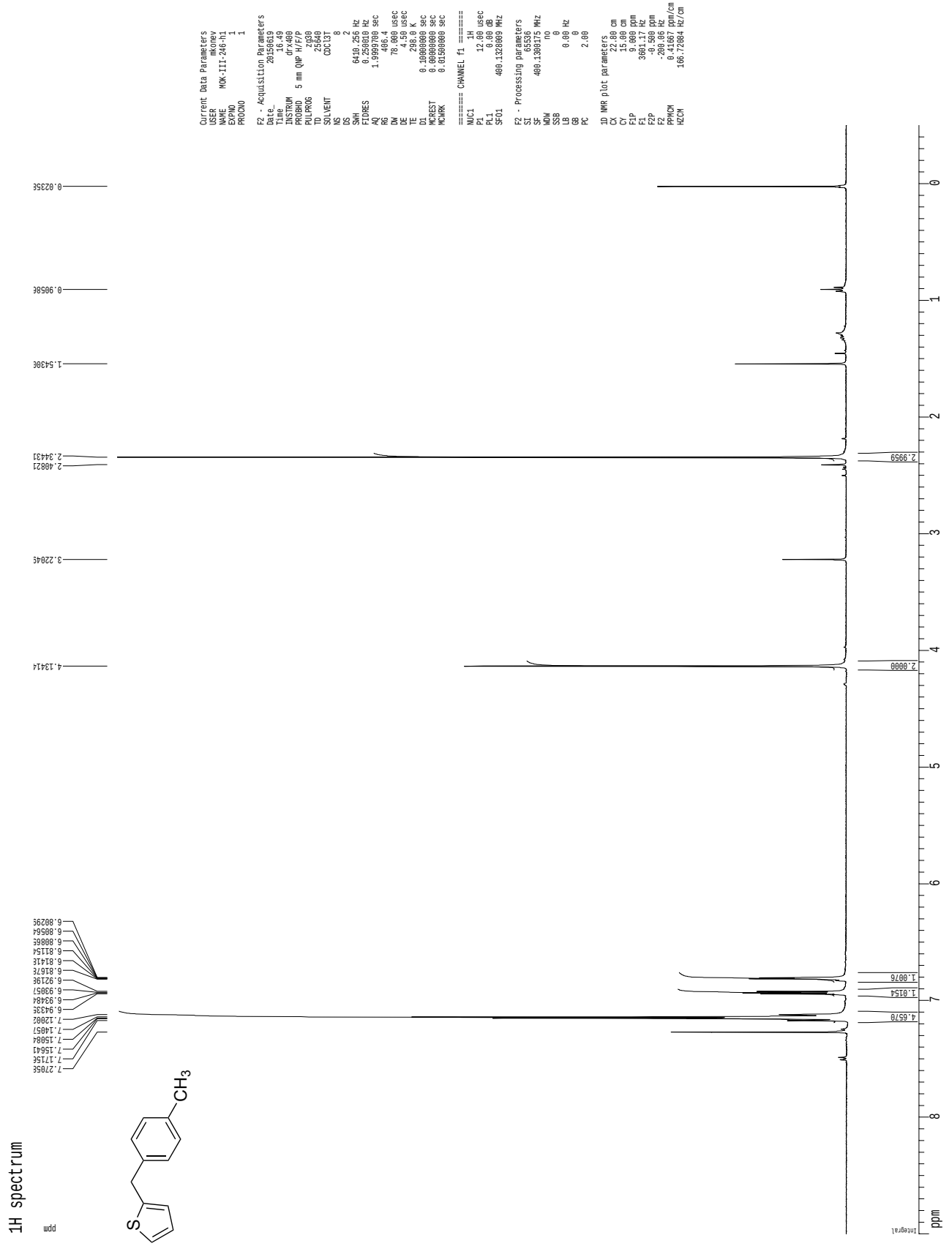
2.0000
2.7802
1.8292
0.9551
2.8042
1.9426
Integral
ppm


1H spectrum
Current Data Parameters
USER mkuney
NAME MOK-III-244-h
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150620
Time 16.36
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999709 sec
RG 456.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72308 Hz/cm
0.0001
0.8531
0.8806
1.2556
1.4326
1.5317
2.4813
3.9364
1.9965
0.8494
0.8484
1.8177
1.7853
0.8118
7.3281
7.3263
7.3236
7.3217
7.2566
7.2015
7.1966
7.1884
7.1801
7.1721
7.1661
7.1596
7.0152
7.0056
7.0008
6.9931
6.9783
6.9672
6.9615
6.9543
6.2961
6.2926
6.2896
6.2842
5.9969
5.9944
5.9886
5.9866
F
O
ppm
integral


1H spectrum
ppm
Integral
Current Data Parameters
USER mkonev
NAME MOK-III-246-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150619
Time 16.49
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999709 sec
RG 406.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.000 cm
F1P 9.000 ppm
F1 3661.17 Hz
F2P -0.560 ppm
F2 -209.98 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
0.02356
0.90586
1.54306
2.34431
2.40821
3.22045
4.13414
6.80296
6.80564
6.80866
6.81154
6.81414
6.81676
6.92196
6.93057
6.93484
6.94335
7.12002
7.14057
7.15084
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7.17156
7.27056
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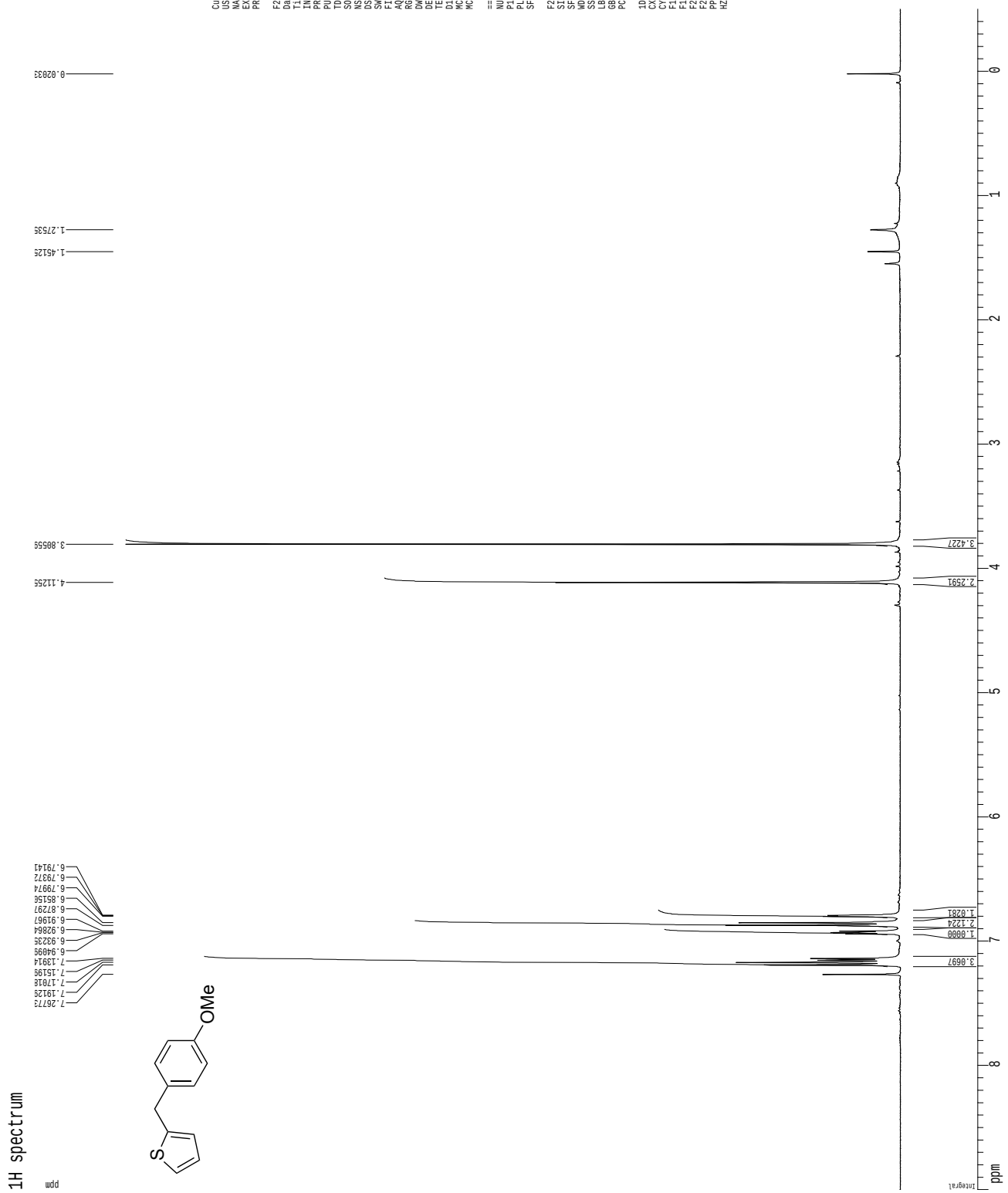
1.00760
1.01540
4.65700
CH3
S


1H spectrum
Current Data Parameters
USER mvlaev
NAME MOK-II-159pure
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150304
Time 21.51
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250098 Hz
AQ 1.9961088 sec
RG 287.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0.00 Hz
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -208.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
0.02033
1.45126
1.27536
3.80556
4.11256
7.26773
7.19126
7.17018
7.15198
7.13914
6.94099
6.92336
6.92864
6.91967
6.87297
6.85156
6.79974
6.79372
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2.2591
1.0281
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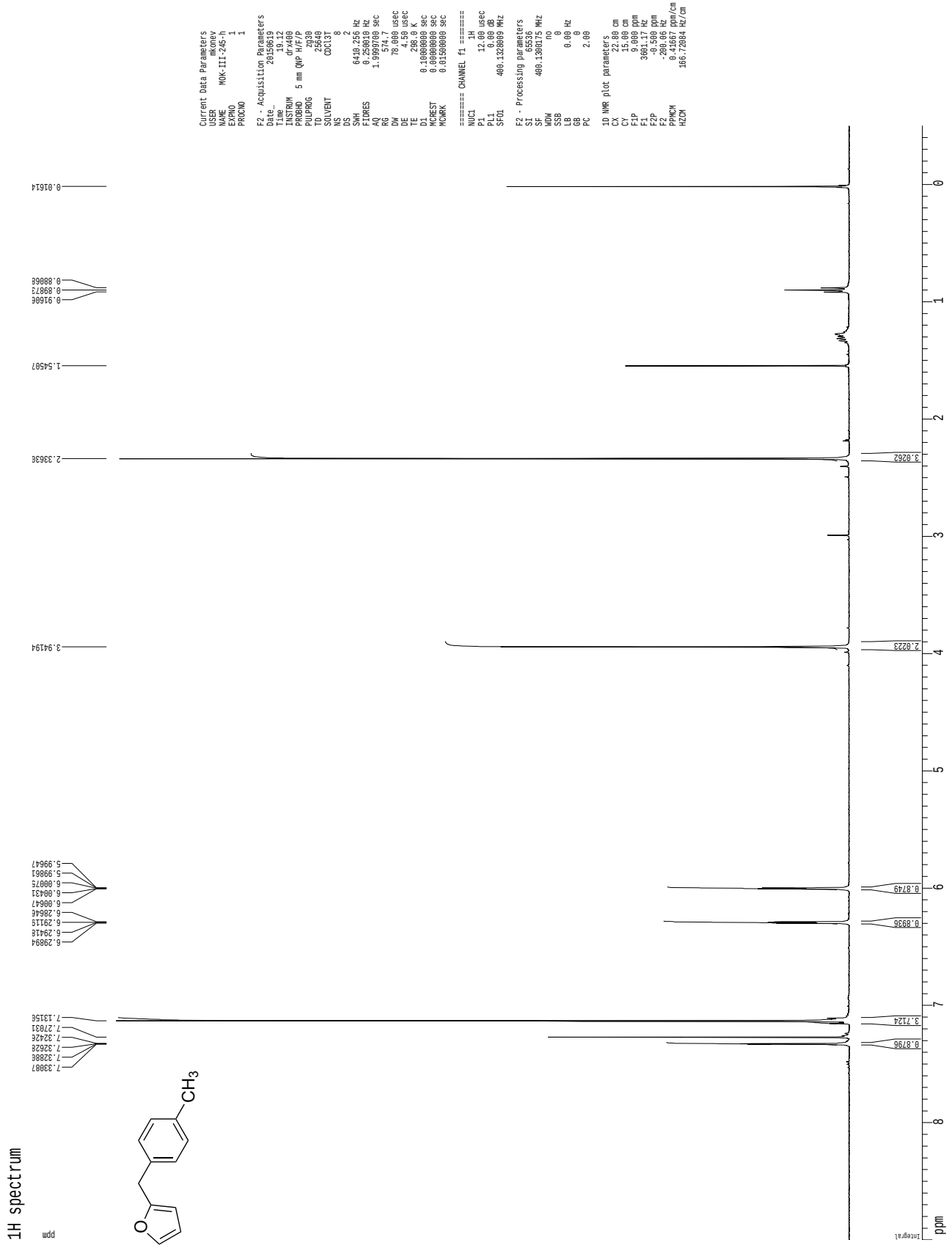
3.0697
OMe
S
integral
ppm
ppm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Current Data Parameters
USER mkonev
NAME MOK-III-245-h
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150619
Time 19.12
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999707 sec
RG 574.7
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72884 Hz/cm
0.01614
0.88068
0.89873
0.91606
1.54507
2.33636
3.94194
5.99647
5.99861
6.00075
6.00431
6.00647
6.28646
6.29119
6.29418
6.29894
7.13156
7.27031
7.32426
7.32628
7.32886
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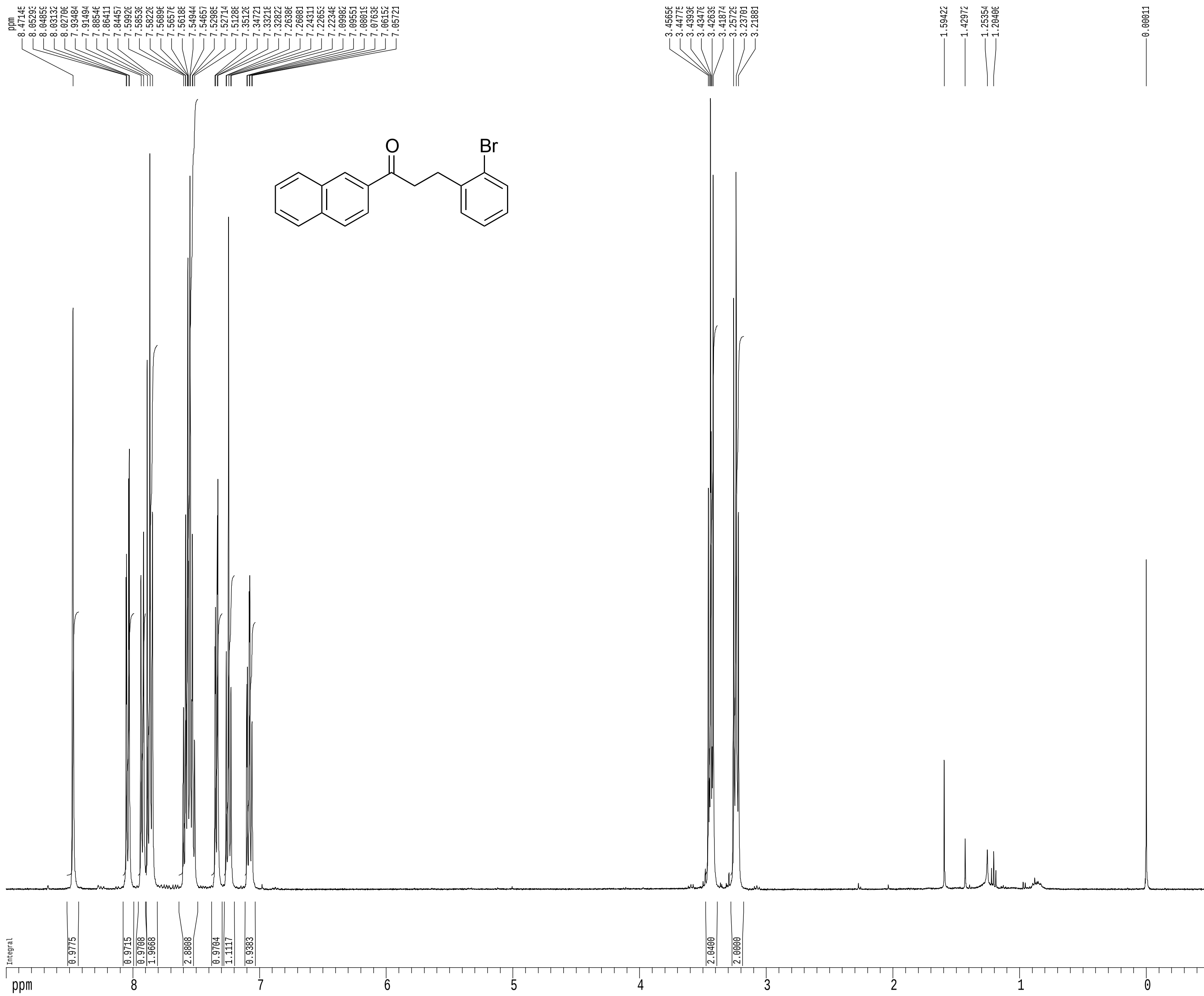
3.7124
0.8796
ppm
Integral

1H spectrum

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Current Data Parameters
USER              mkonev
NAME      MOK-III-105hchar
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150126
Time               14.23
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 38460
SOLVENT            CDCl3T
NS                     8
DS                     2
SWH            6410.256 Hz
FIDRES         0.166673 Hz
AQ            2.9999299 sec
RG                   181
DW                78.000 usec
DE                  4.50 usec
TE                 298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                 0.00 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300273 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P                9.000 ppm
F1               3601.17 Hz
F2P               -0.500 ppm
F2               -200.06 Hz
PPMCM            0.41667 ppm/cm
HZCM           166.72086 Hz/cm
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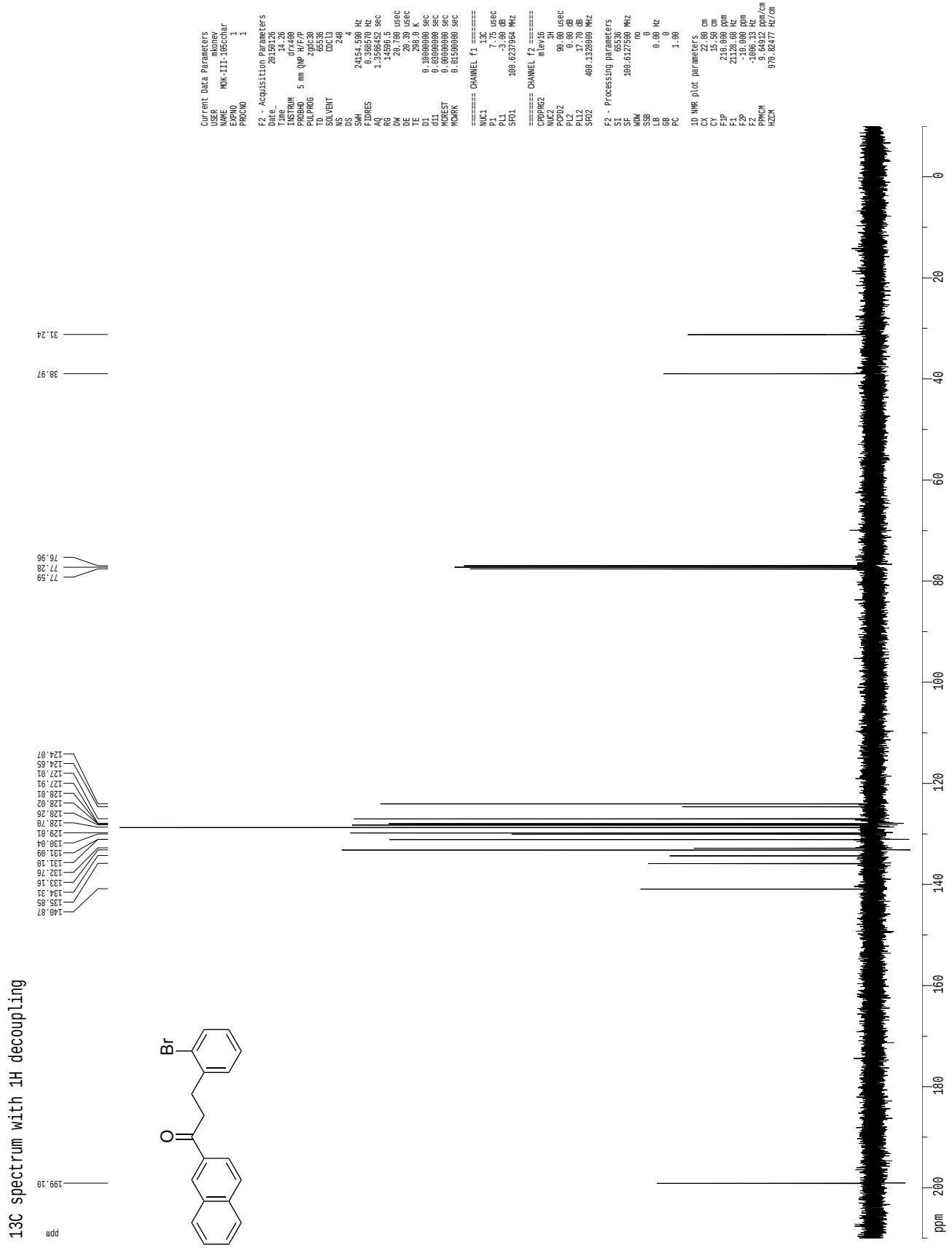
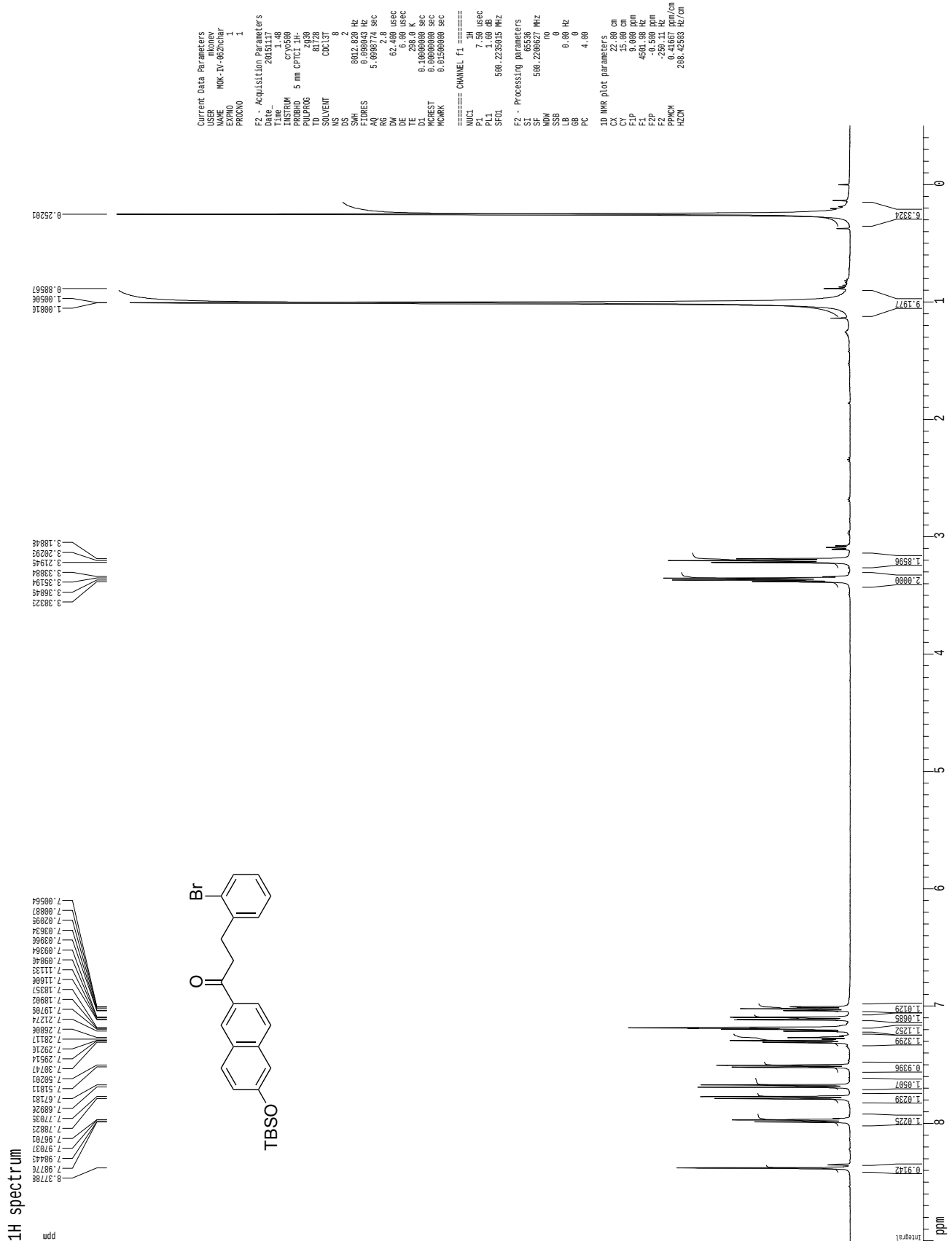

13C spectrum with 1H decoupling
Current Data Parameters
USER mMorrev
NAME MOK-III-165cnar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150126
Time 14.26
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 248
DS 4
SWH 24154.590 Hz
FIDRES 0.368579 Hz
AQ 1.3566452 sec
RG 14596
DW 20.700 usec
DE 20.38 usec
TE 298.8 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======= CHANNEL f2 =======
CPDPRG2 mlevi6
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127580 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 216.000 ppm
F1 21728.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm
199.10
149.87
135.85
134.31
133.16
132.76
131.10
131.09
130.04
129.81
128.70
128.26
128.02
128.01
127.91
127.01
124.65
124.07
77.59
77.28
76.96
38.97
31.24
Br
O
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm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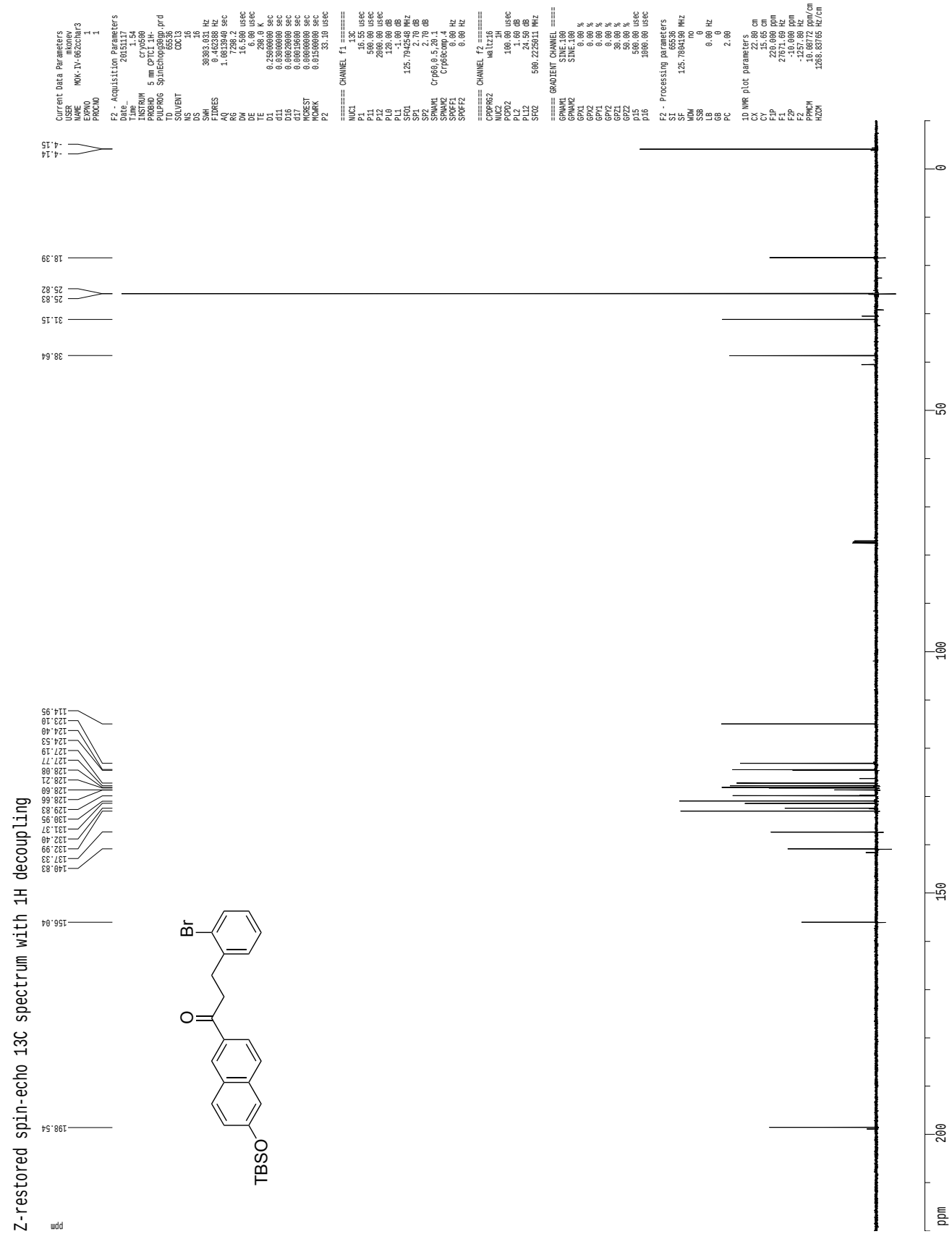
Br
O
TBSO
Current Data Parameters
USER mkonev
NAME MOK-IV-062hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 1.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 2.8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200627 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.460 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm
0.25201
0.88567
1.00016
1.00506
3.38323
3.36849
3.35194
3.33884
3.27945
3.20293
3.18848
7.00564
7.00887
7.02695
7.03634
7.03966
7.09364
7.09846
7.11133
7.11606
7.18357
7.18902
7.19705
7.21274
7.26806
7.28117
7.29216
7.29514
7.30147
7.50201
7.51811
7.67181
7.68926
7.77035
7.78823
7.96701
7.97037
7.98443
7.98776
8.37786
6.3324
9.1977
1.8596
2.0000
1.0129
1.0685
1.1252
1.3299
0.9396
1.0507
1.0239
1.0225
0.9142
Integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling

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Current Data Parameters
USER      mkonev
NAME      MOK-IV-062cher3
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_      20151117
Time           1.54
INSTRUM      cryo500
PROBHD  5 mm CPTCI 1H-
PULPROG  SpinEchoPg30gp.prd
TD          65536
SOLVENT       CDCl3
NS              16
DS               4
SWH        30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG              7208.2
DW             16.500 usec
DE              6.00 usec
TE            298.0 K
D1         2.0000000 sec
D11        0.0300000 sec
D16        0.0002000 sec
d17        0.0001900 sec
MCREST     0.0000000 sec
MCWRK      0.0150000 sec
P2            33.10 usec

======== CHANNEL f1 ========
NUC1             13C
P1             16.55 usec
P11           500.00 usec
P12          2000.00 usec
PL0           120.00 dB
PL1            -1.00 dB
SFO1     125.7942548 MHz
SP1            2.70 dB
SP2            2.70 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1         0.00 Hz
SPOFF2         0.00 Hz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2             1H
PCPD2       100.00 usec
PL2            1.60 dB
PL12          24.50 dB
SFO2     500.2229911 MHz

===== GRADIENT CHANNEL =====
GPNAM1       SINE.100
GPNAM2       SINE.100
GPX1          0.00 %
GPX2          0.00 %
GPY1          0.00 %
GPY2          0.00 %
GPZ1         30.00 %
GPZ2         50.00 %
p15        5000.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI          65536
SF     125.7884190 MHz
WDW            no
SSB             0
LB           0.00 Hz
GB              0
PC           2.00

1D NMR plot parameters
CX          22.80 cm
CY          15.65 cm
F1P       220.000 ppm
F1      27671.69 Hz
F2P      -10.000 ppm
F2     -1257.80 Hz
PPMCM    10.08772 ppm/cm
HZCM   1268.83765 Hz/cm
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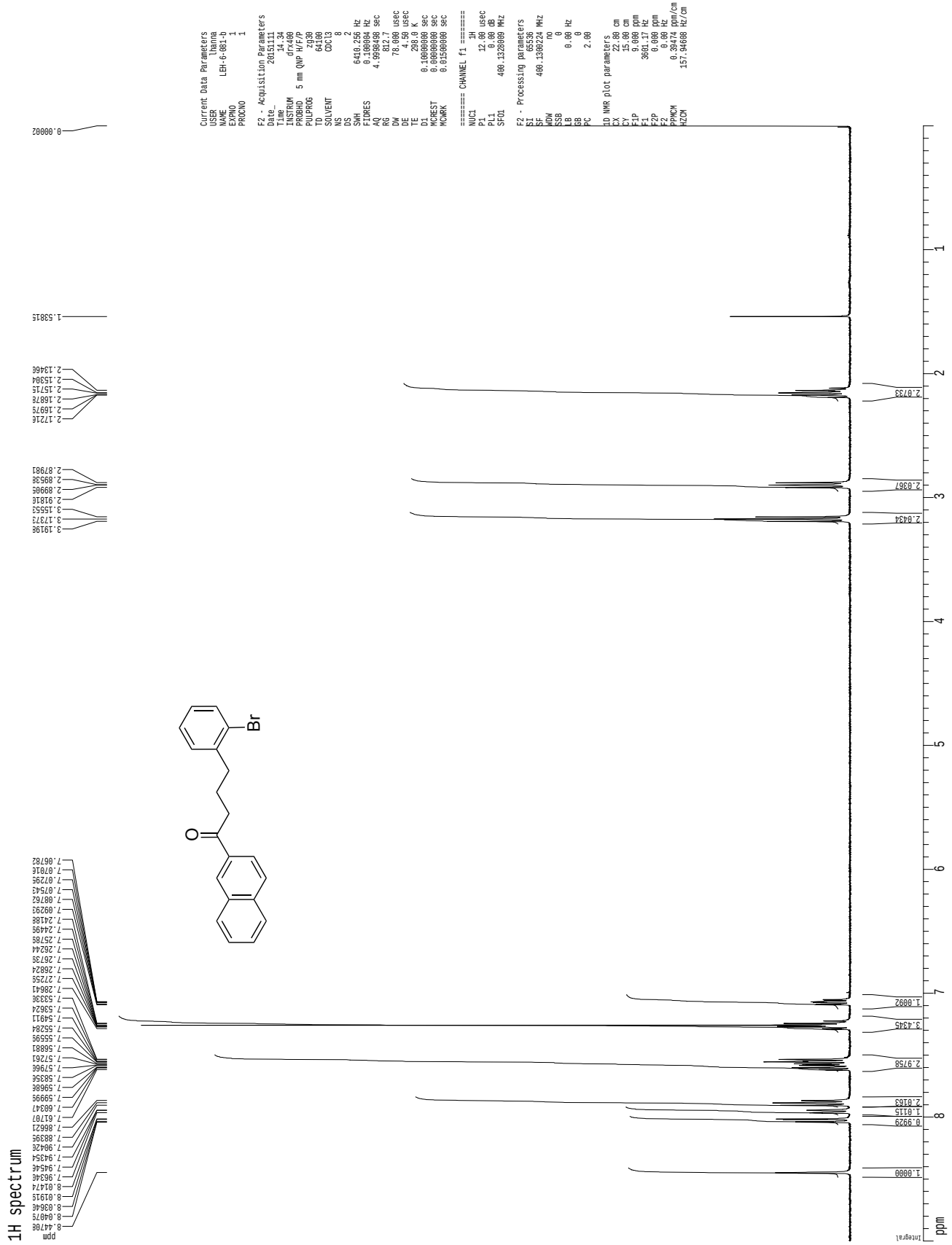
1H spectrum
Current Data Parameters
USER Jhyuna
NAME LEH-6-081-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151111
Time 14.34
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 64100
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.100096 Hz
AQ 4.9961549 sec
RG 812.7
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300224 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
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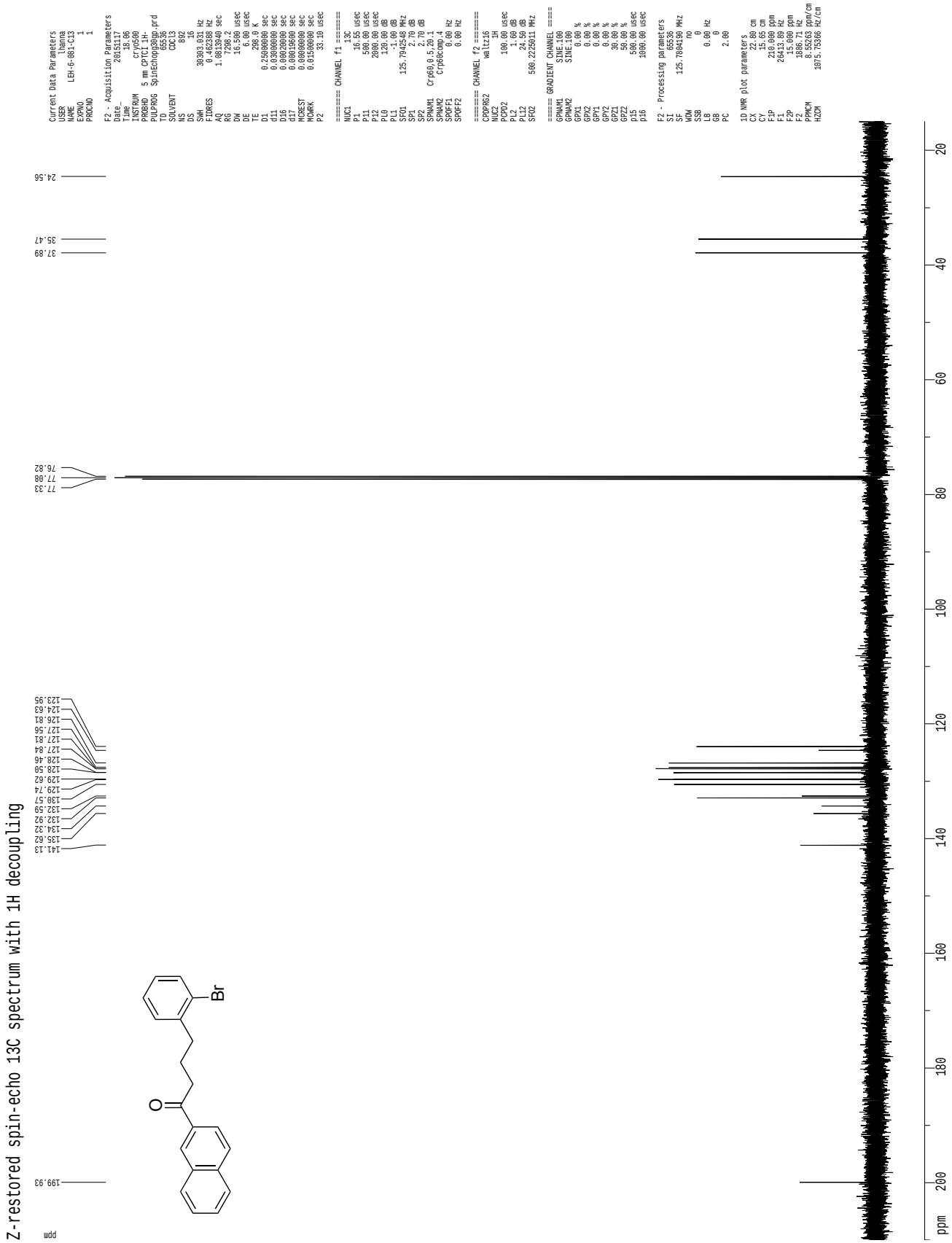
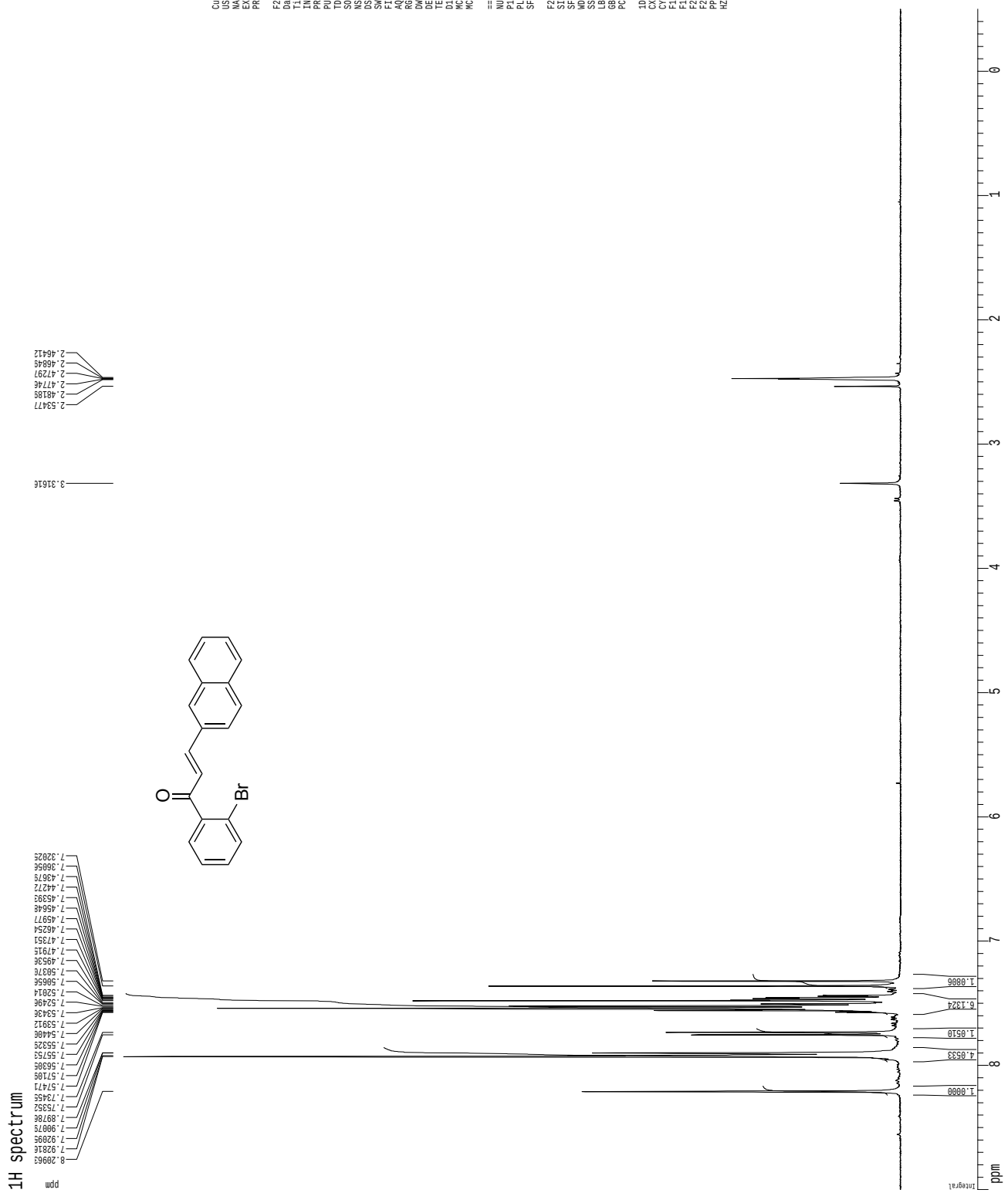
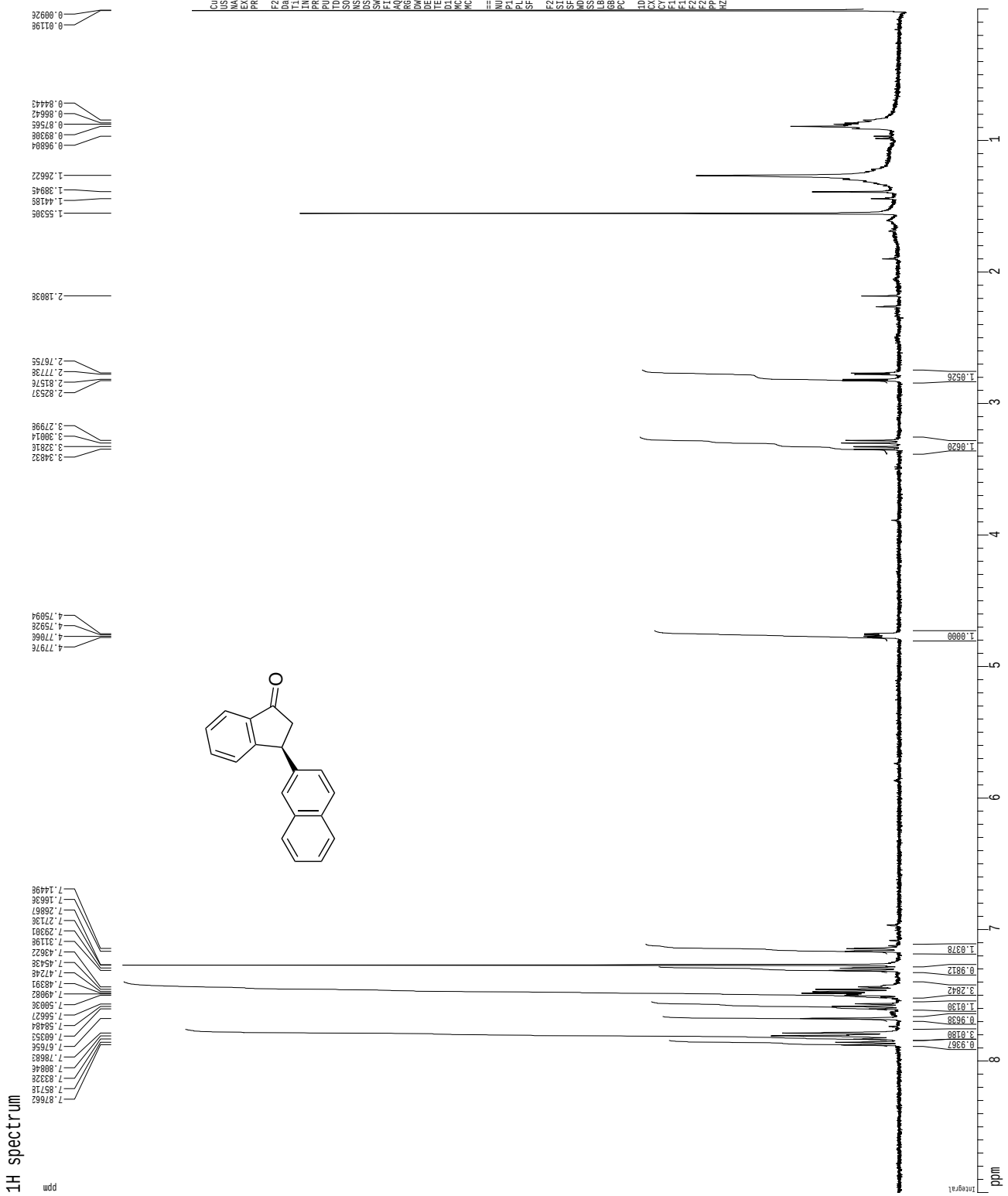
HZCM 157.94608 Hz/cm
Br
O
ppm
Integral

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER mkovary
NAME MOK-IV-chalcone
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151112
Time 17.24
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097753 Hz
AQ 5.112575 sec
RG 203.2
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -280.00 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72984 Hz/cm
O
Br
ppm
Integral
1.0000
4.0533
1.0510
6.1324
1.0806


1H spectrum
Current Data Parameters
USER Thesis
NAME LEH-6-93S-c1-f20
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150831
Time 13.56
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.250098 Hz
AQ 1.998080 sec
RG 1024
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
0.00926
0.01198
0.84443
0.86642
0.87565
0.89308
0.96804
1.26622
1.38945
1.44185
1.55306
2.18038
2.76755
2.77738
2.81576
2.82537
3.27998
3.30014
3.32816
3.94832
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4.76926
4.77066
4.77976
7.14498
7.16636
7.26867
7.27136
7.29301
7.31198
7.43622
7.45438
7.47248
7.48391
7.49982
7.50036
7.56627
7.58484
7.60355
7.67656
7.78683
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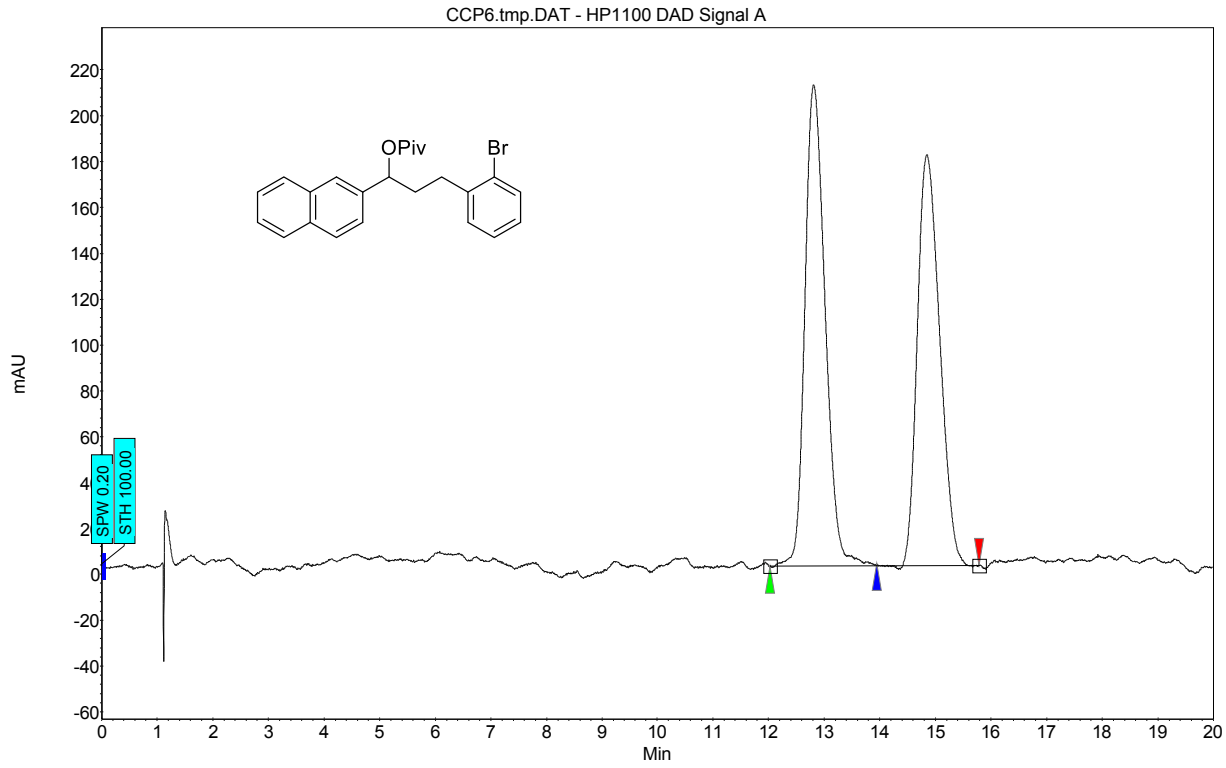
7.83326
7.85718
7.87662
1.0526
1.0620
1.0000
1.0378
0.9812
3.2842
1.0130
0.9538
3.0180
0.9367
Integral
ppm

Method Name:MOK-II-266rac
Run Name:LEH-5-RAC-Np-Piv-Br-22

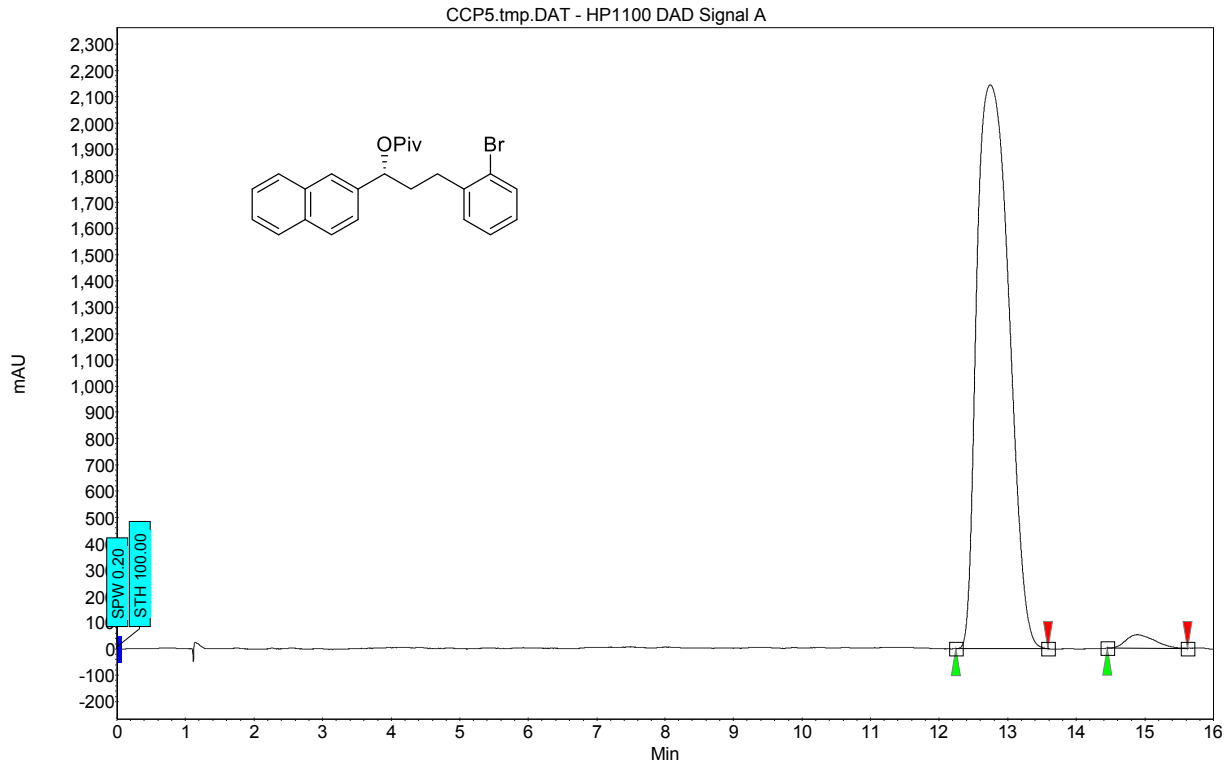
Date:5/10/2015
Time:9:53:59 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.02	12.81	13.95	0.00	51.51	209.7	88.4	51.507
2	UNKNOWN	13.95	14.86	15.79	0.00	48.49	179.2	83.2	48.493
Total						100.00	388.8	171.5	100.000

Method Name:MOK-II-266rac
Run Name:MOK-2-Np-Piv-Br-ee-22

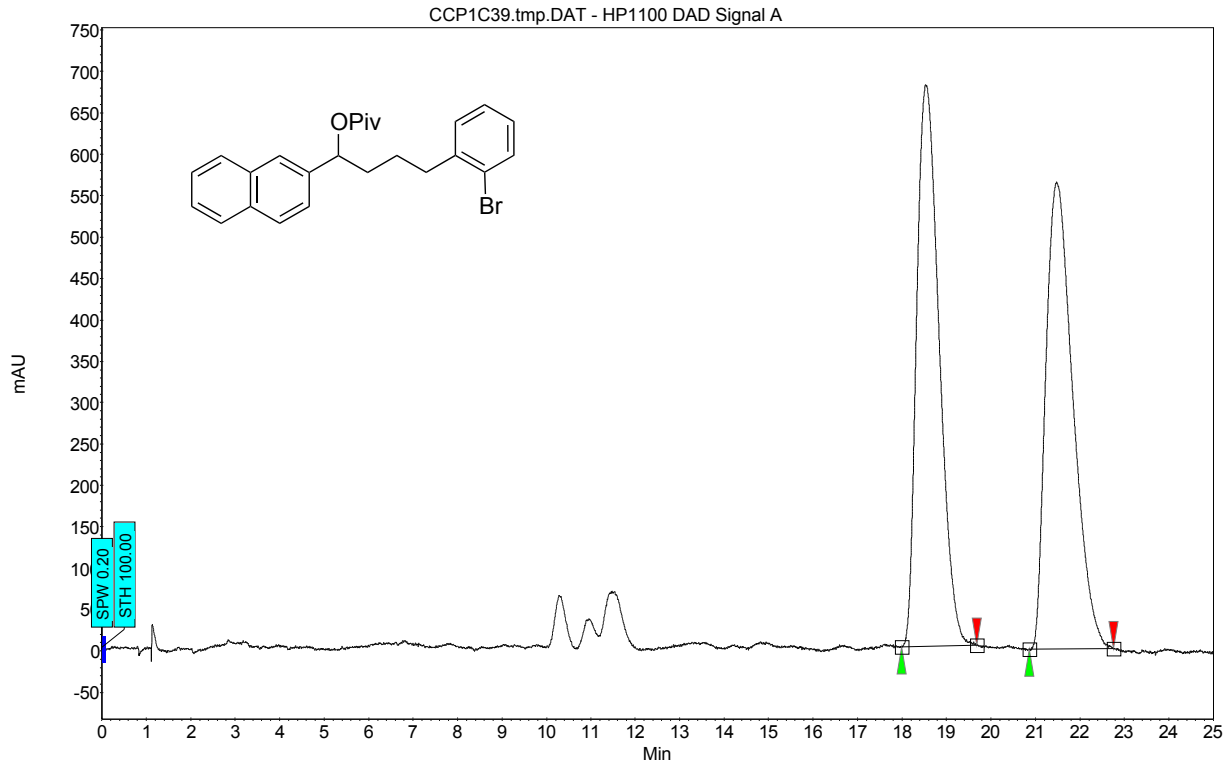
Date:5/10/2015
Time:9:43:48 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.25	12.74	13.59	0.00	97.90	2143.7	1166.3	97.901
2	UNKNOWN	14.45	14.90	15.62	0.00	2.10	51.7	25.0	2.099
Total						100.00	2195.5	1191.3	100.000

Method Name:MOK-II-266rac
Run Name:LEH-6-080-D3

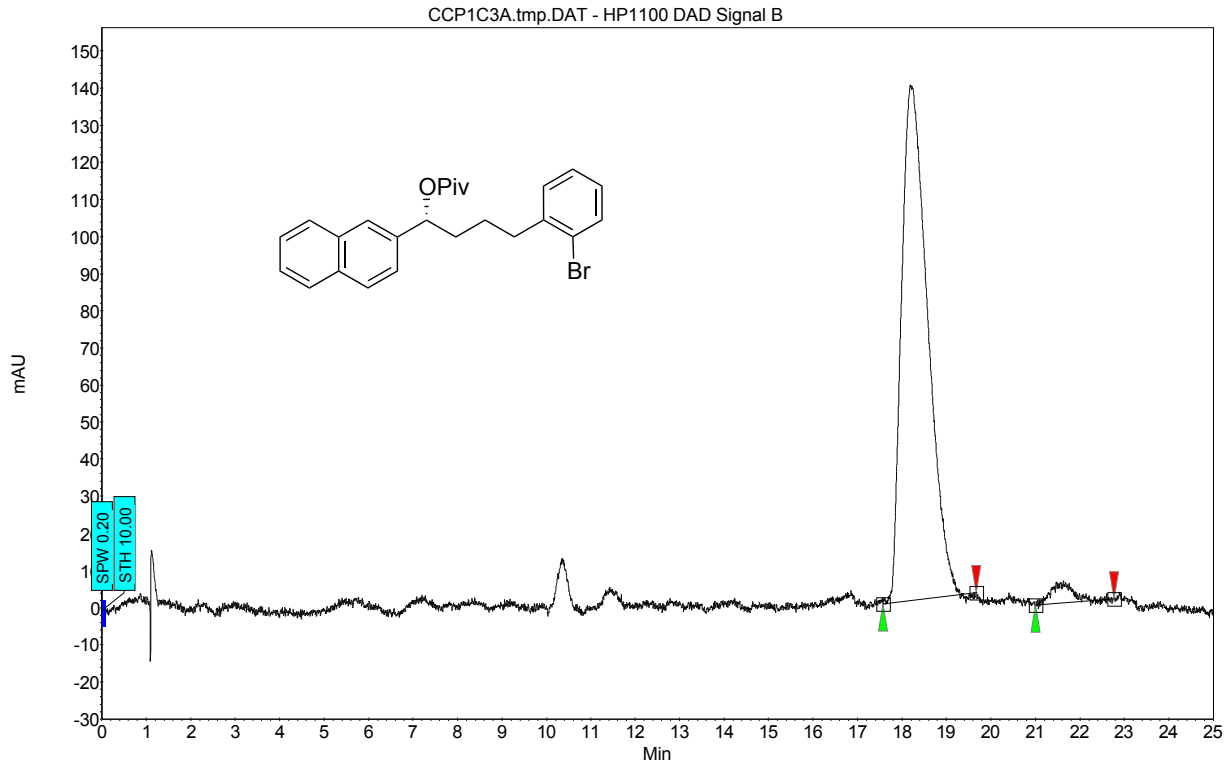
Date:11/24/2015
Time:7:09:09 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	17.99	18.53	19.68	0.00	49.85	678.1	395.5	49.846
2	UNKNOWN	20.86	21.48	22.75	0.00	50.15	564.1	397.9	50.154
Total						100.00	1242.2	793.3	100.000

Method Name:MOK-II-266rac
Run Name:LEH-5-084-A3

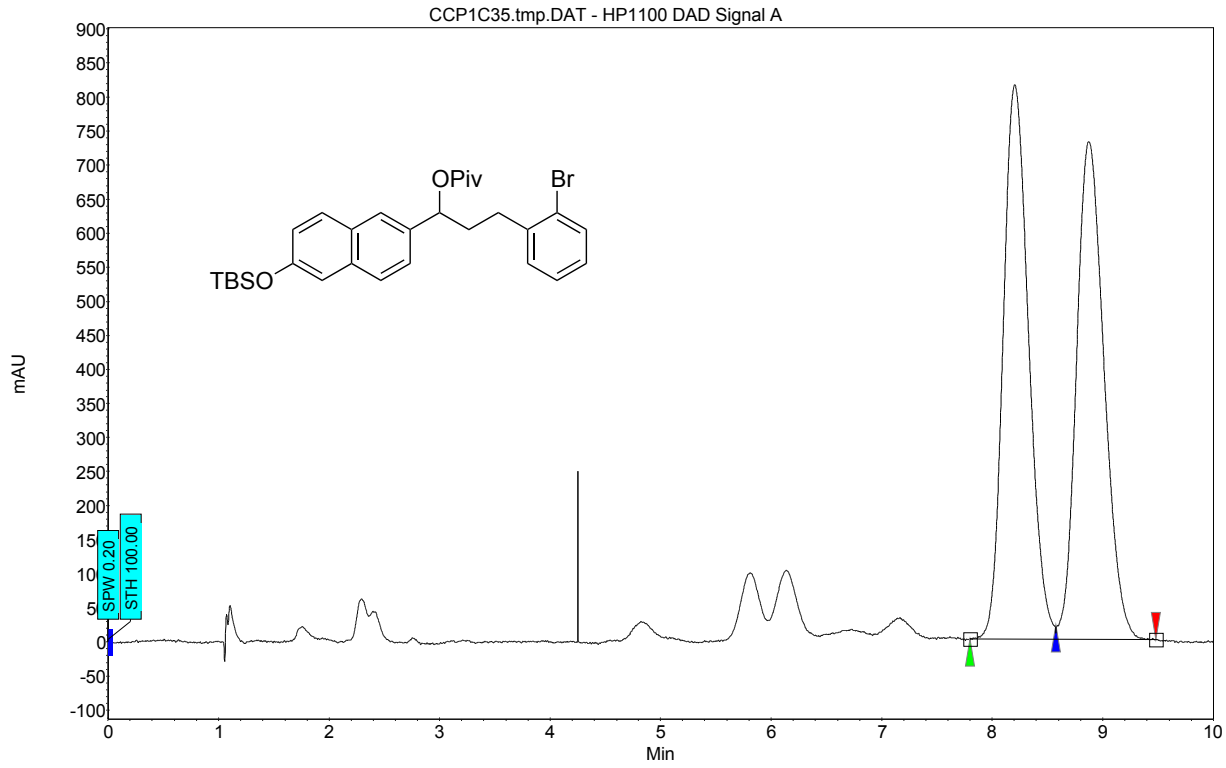
Date:11/24/2015
Time:7:13:20 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	17.57	18.20	19.67	0.00	96.84	138.8	94.2	96.842
2	UNKNOWN	21.01	21.63	22.76	0.00	3.16	5.9	3.1	3.158
Total						100.00	144.7	97.3	100.000

Method Name:MOK-IV-5otbspiv
Run Name:MOK-IV-racopiv5tbs3

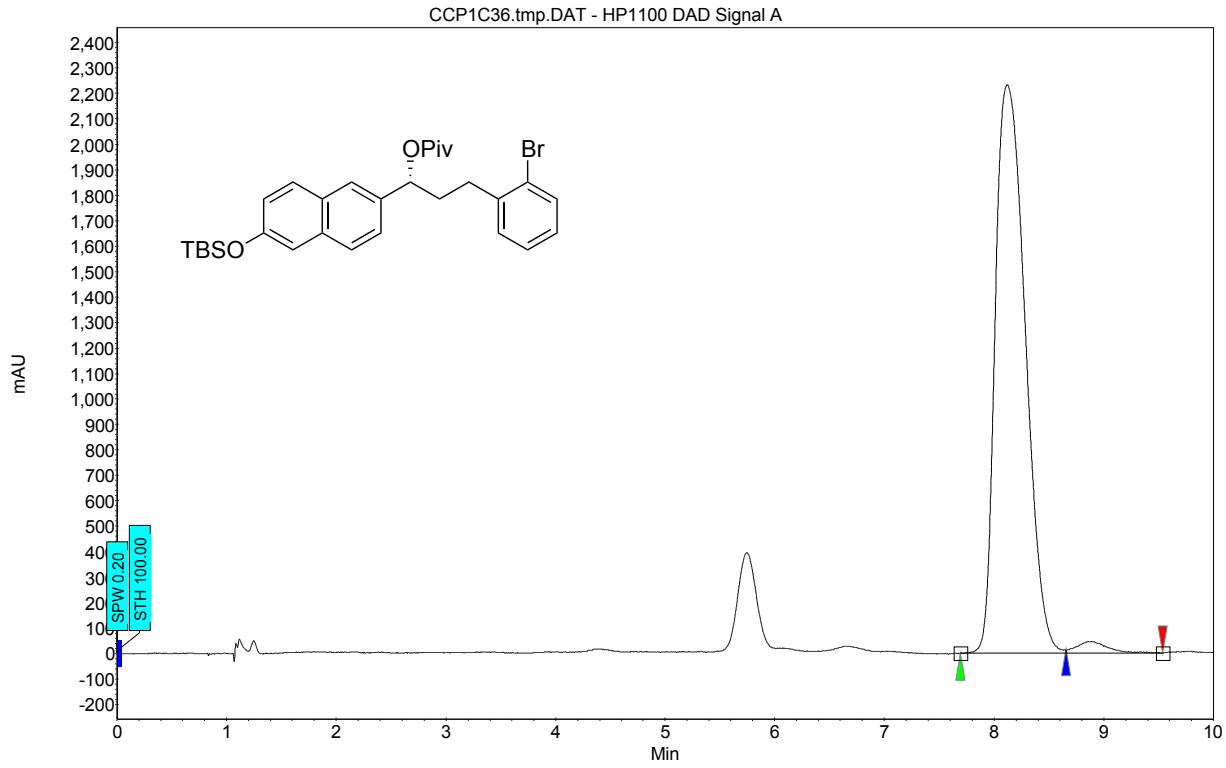
Date:11/24/2015
Time:7:00:17 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.80	8.20	8.58	0.00	50.65	813.6	216.6	50.651
2	UNKNOWN	8.58	8.87	9.48	0.00	49.35	730.6	211.1	49.349
Total						100.00	1544.2	427.7	100.000

Method Name:MOK-IV-5otbspiv
Run Name:MOK-IV-eeopiv5tbs2

Date:11/24/2015
Time:7:01:31 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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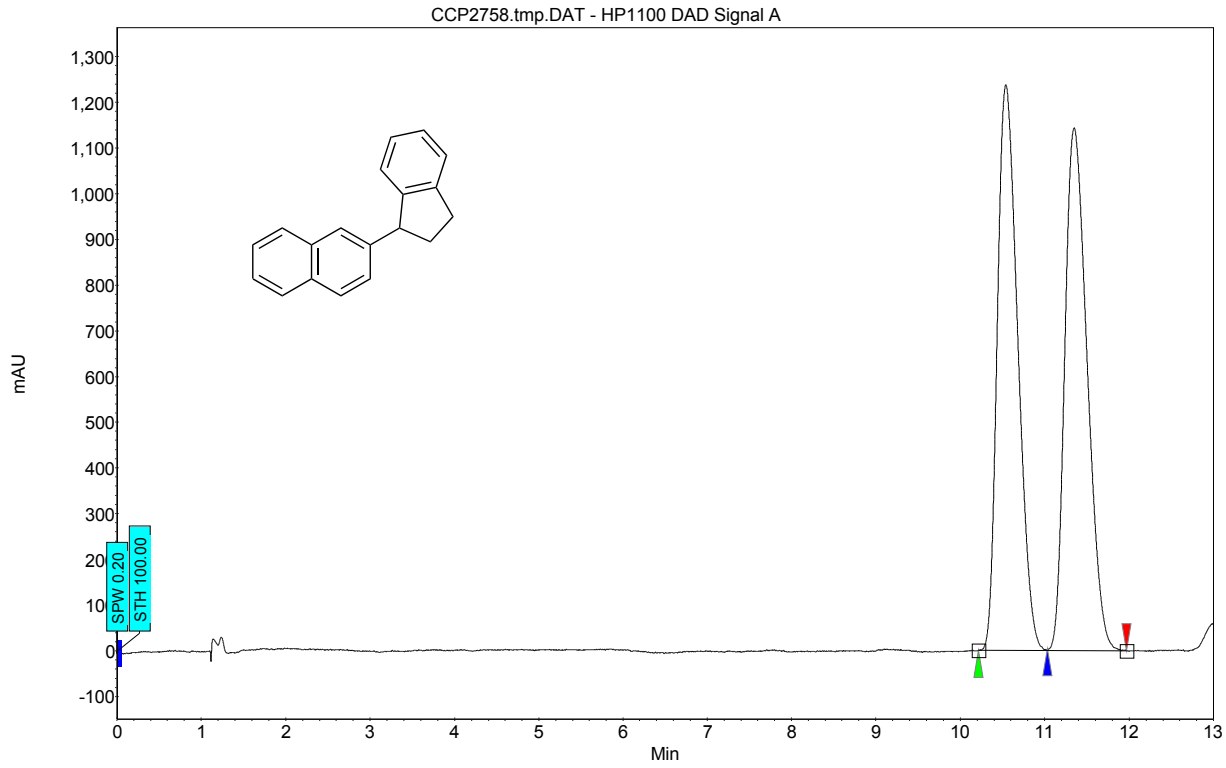
1	UNKNOWN	7.69	8.12	8.65	0.00	97.92	2231.6	705.5	97.918
2	UNKNOWN	8.65	8.86	9.54	0.00	2.08	45.6	15.0	2.082
Total						100.00	2277.2	720.5	100.000

Method Name:MOK-II-266rac
Run Name:MOK-2-266rac3

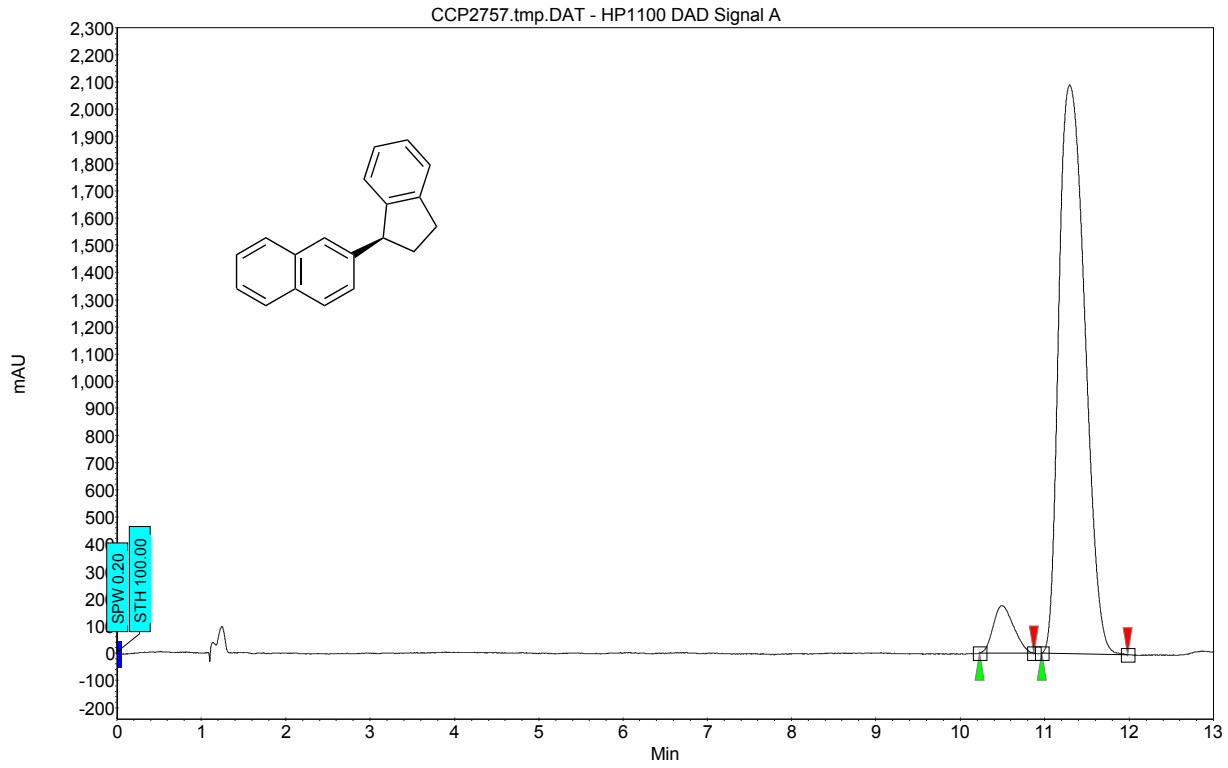
Date:9/7/2015
Time:3:04:42 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.21	10.54	11.04	0.00	49.76	1237.1	347.4	49.764
2	UNKNOWN	11.04	11.35	11.97	0.00	50.24	1143.6	350.7	50.236
Total						100.00	2380.8	698.2	100.000

Method Name:MOK-II-266rac
Run Name:LEH-5-151-pub3

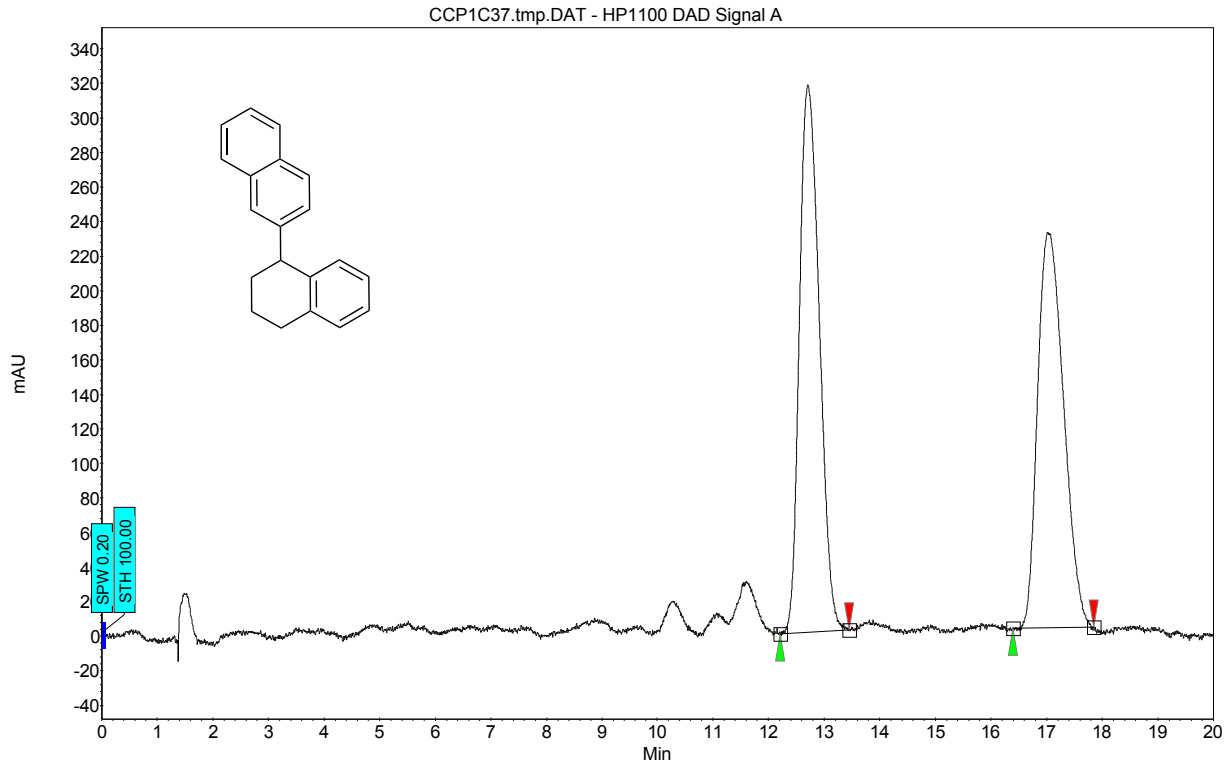
Date:9/7/2015
Time:3:03:27 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	10.23	10.50	10.87	0.00	6.12	174.1	48.8	6.121
1	UNKNOWN	10.96	11.30	11.98	0.00	93.88	2091.5	747.8	93.879
Total						100.00	2265.6	796.6	100.000

Method Name:MOK-266-NAP-6-Pdt
Run Name:LEH-6-083-F1

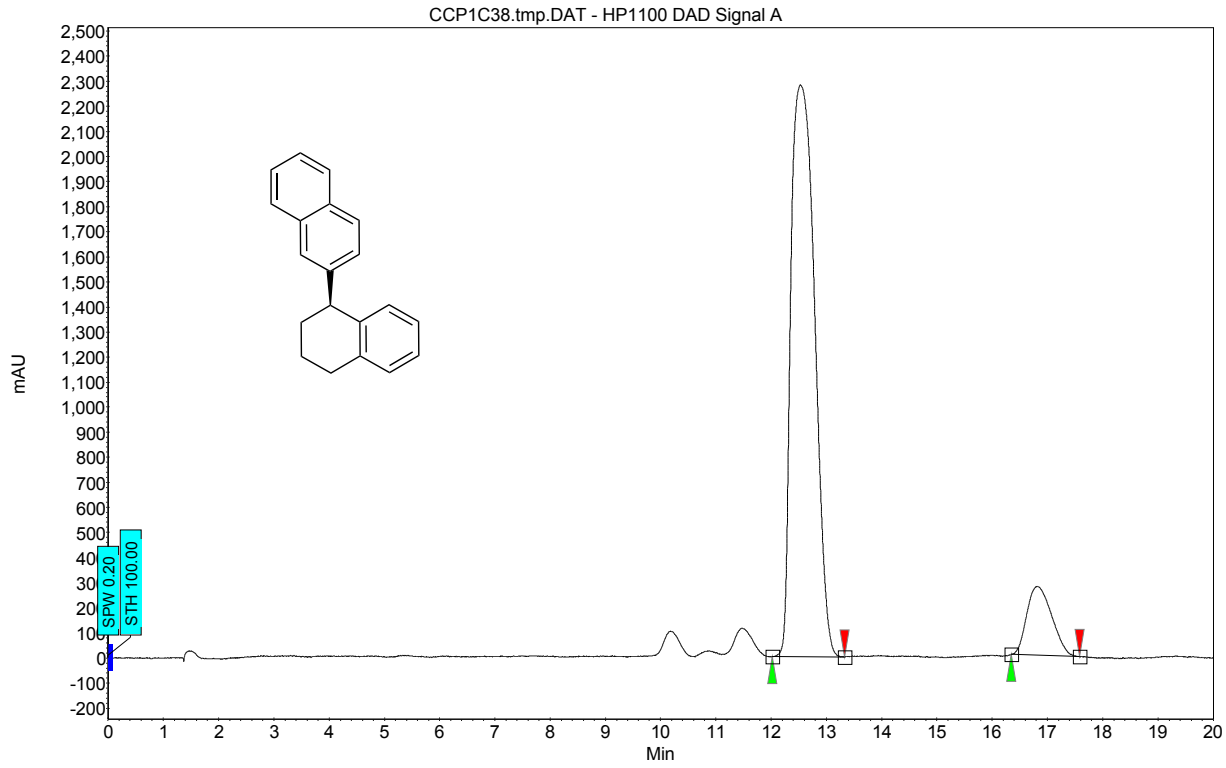
Date:11/24/2015
Time:7:04:23 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.21	12.71	13.45	0.00	51.53	316.7	131.5	51.529
2	UNKNOWN	16.40	17.02	17.84	0.00	48.47	228.9	123.7	48.471
Total						100.00	545.7	255.3	100.000

Method Name:MOK-266-NAP-6-Pdt
Run Name:LEH-6-085-A1

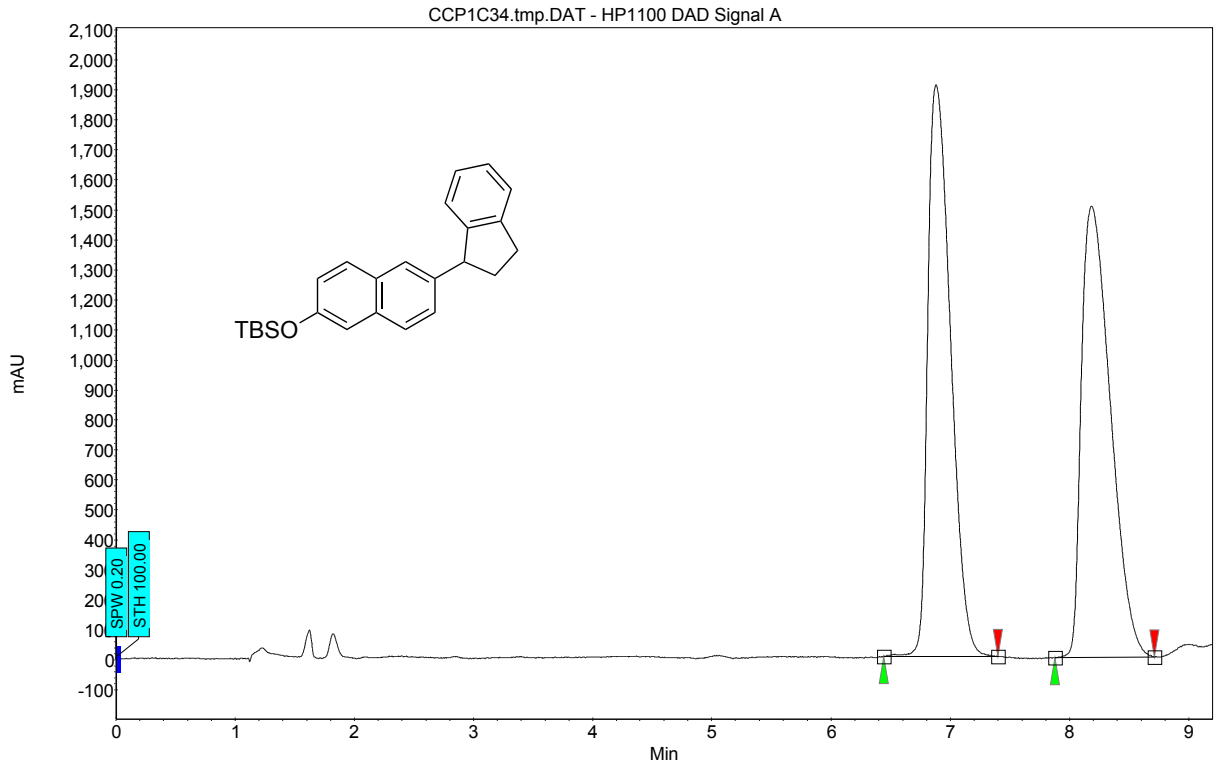
Date:11/24/2015
Time:7:06:01 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.02	12.53	13.33	0.00	88.75	2280.2	1130.7	88.750
2	UNKNOWN	16.35	16.81	17.57	0.00	11.25	273.6	143.3	11.250
Total						100.00	2553.9	1274.1	100.000

Method Name:MOK-IV-ractbsprodMETH
Run Name:MOK-IV-otbsracprod1

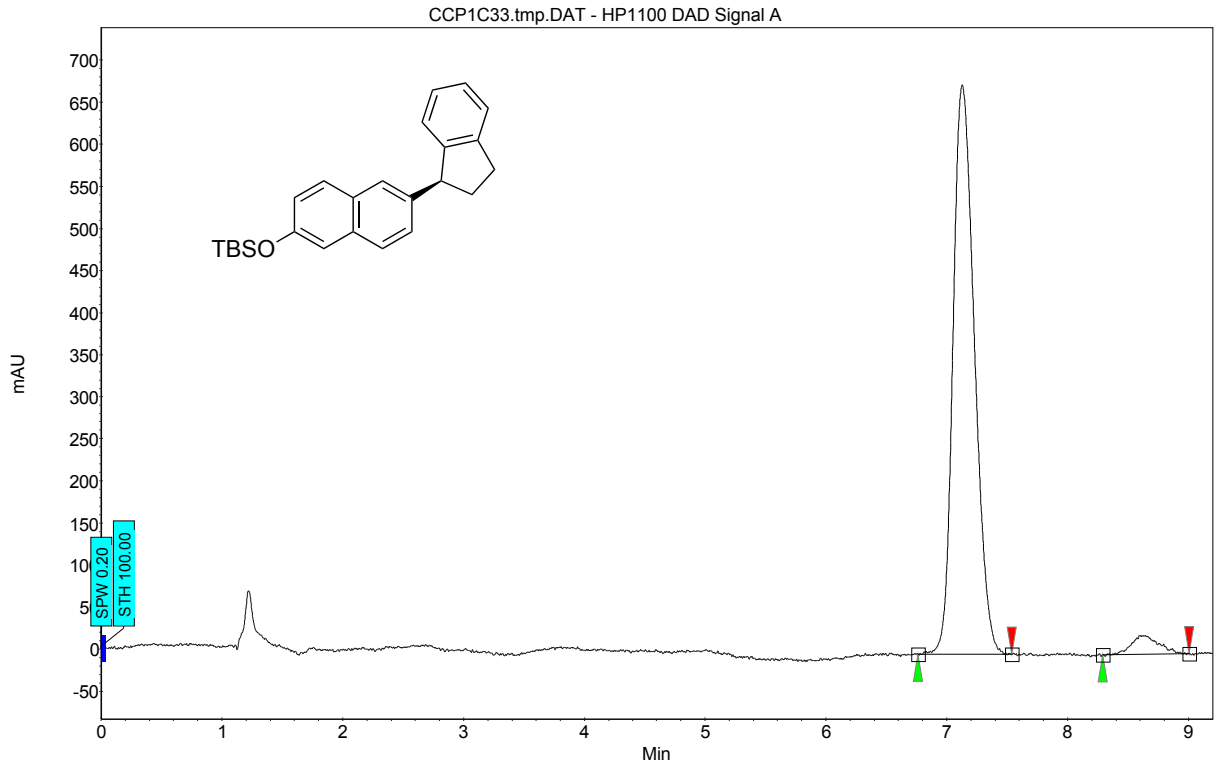
Date:11/24/2015
Time:6:58:37 PM



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.44	6.88	7.40	0.00	49.85	1904.4	421.7	49.849
2	UNKNOWN	7.88	8.19	8.71	0.00	50.15	1503.7	424.3	50.151
Total						100.00	3408.1	846.0	100.000

Method Name:MOK-IV-ractbsprodMETH
Run Name:MOK-IV-otbseeprod2

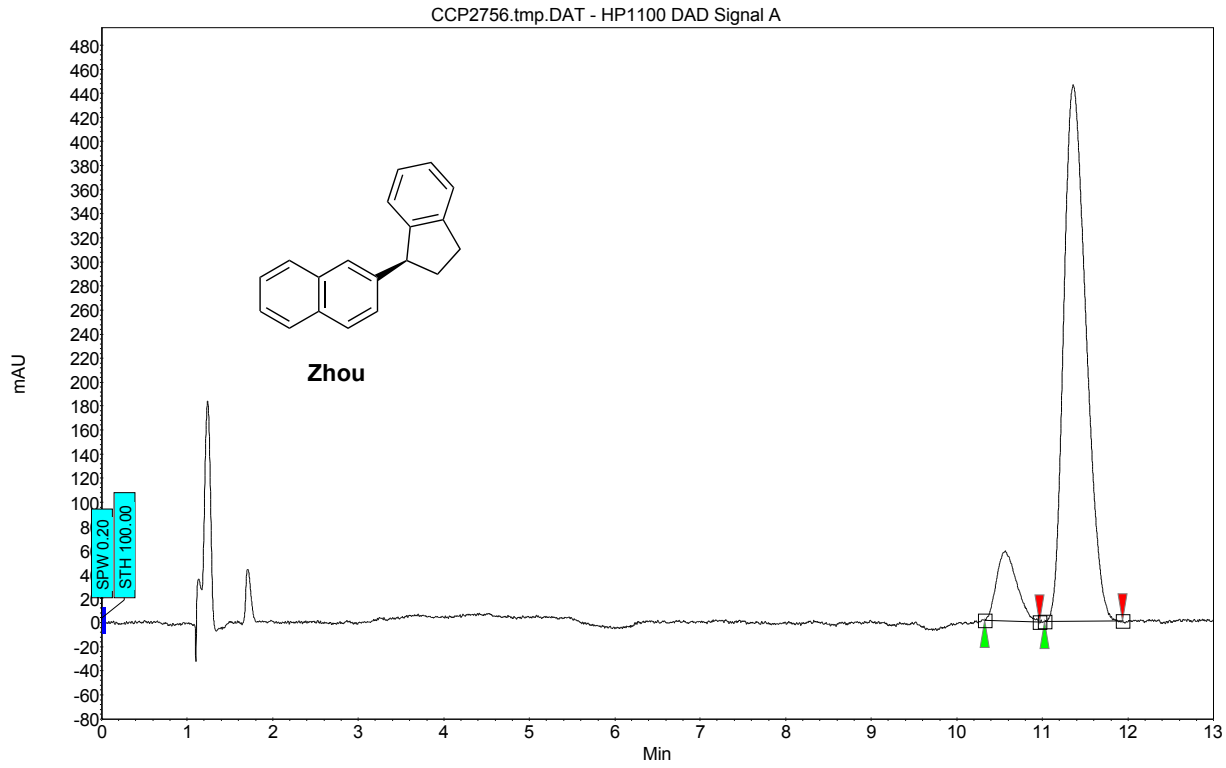
Date:11/24/2015
Time:6:57:22 PM



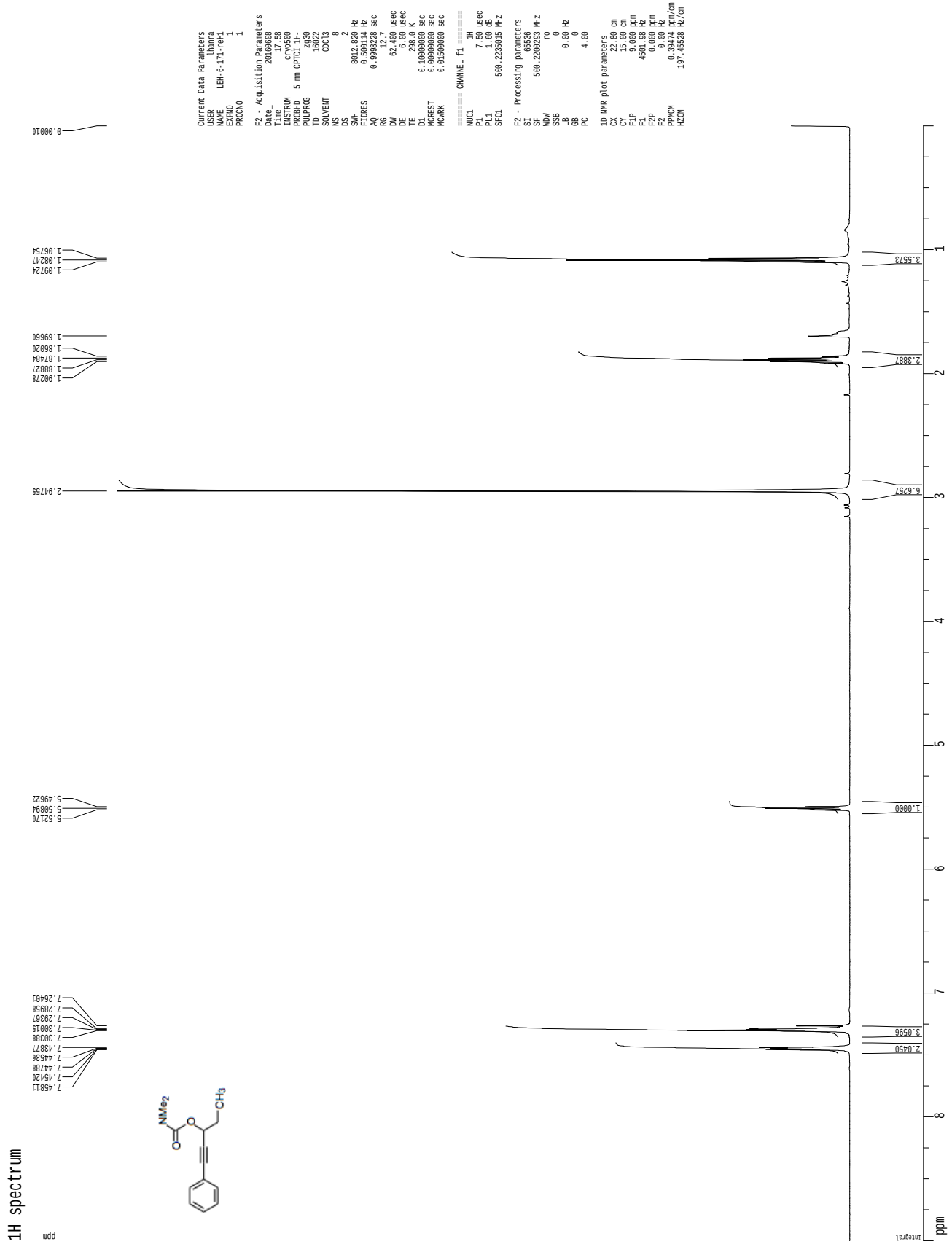
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.76	7.13	7.53	0.00	95.85	676.0	138.3	95.849
2	UNKNOWN	8.29	8.61	9.00	0.00	4.15	22.6	6.0	4.151
Total						100.00	698.6	144.3	100.000

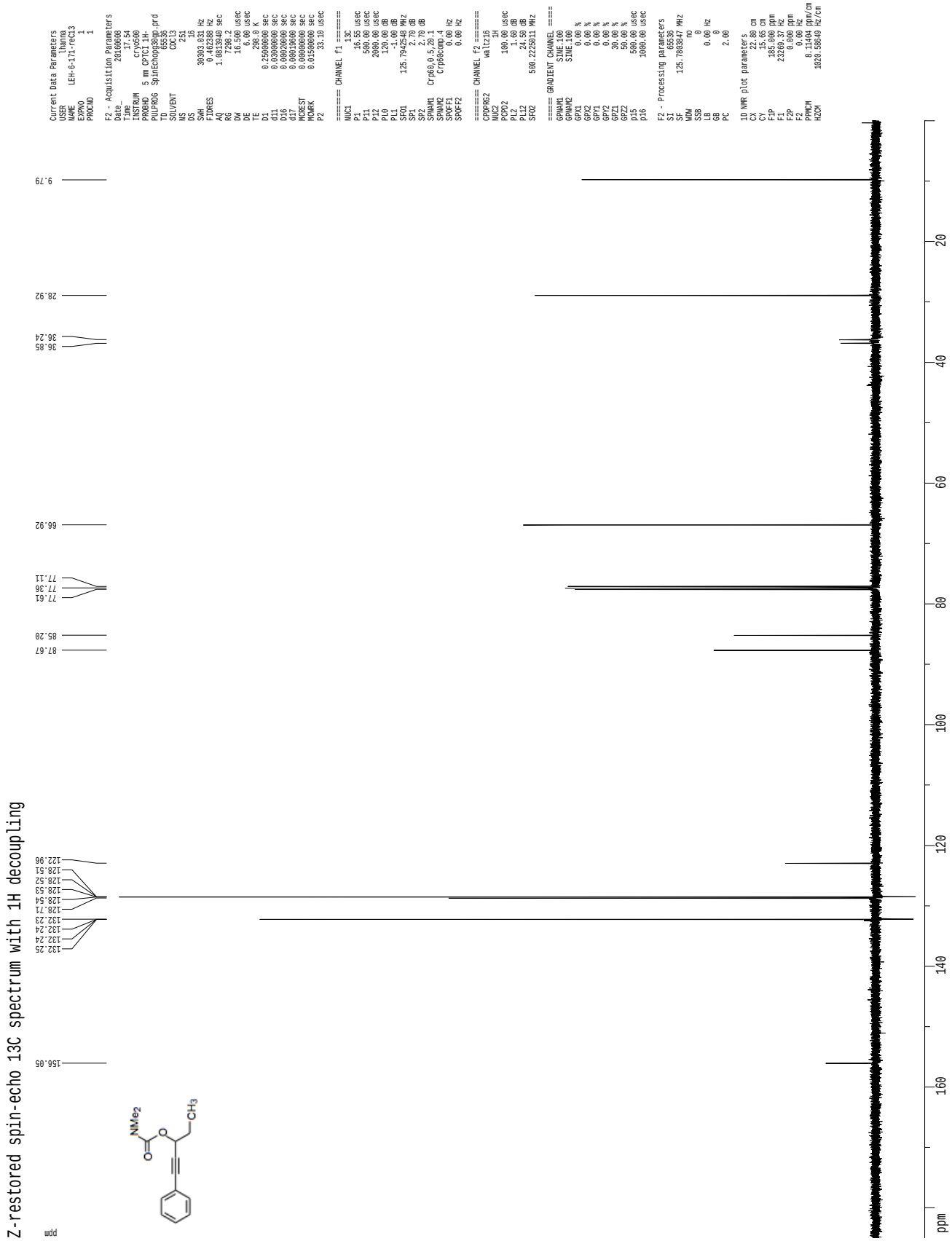
Method Name:MOK-II-266rac
Run Name:LEH-5-036-realeezhou3

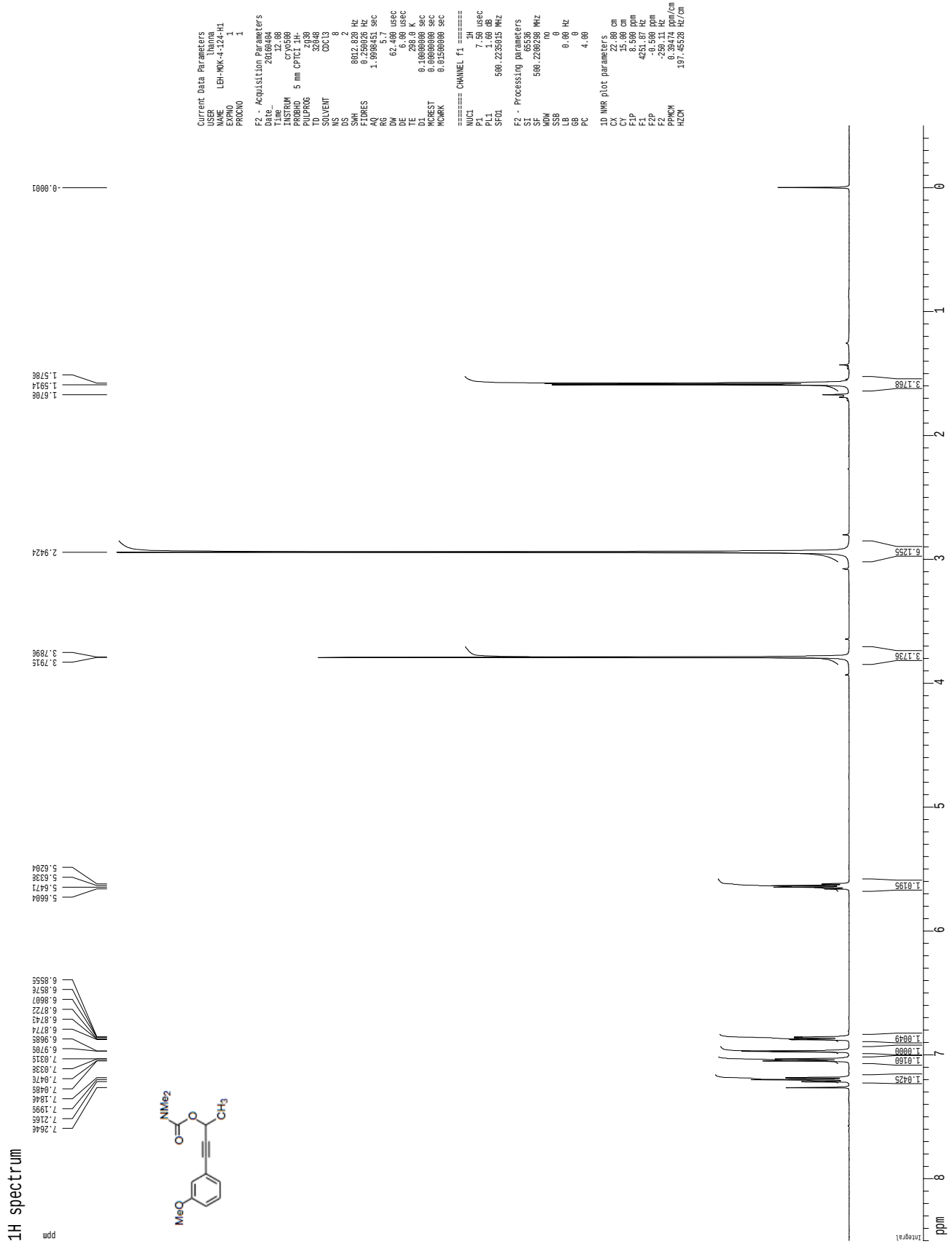
Date:9/7/2015
Time:2:49:52 PM

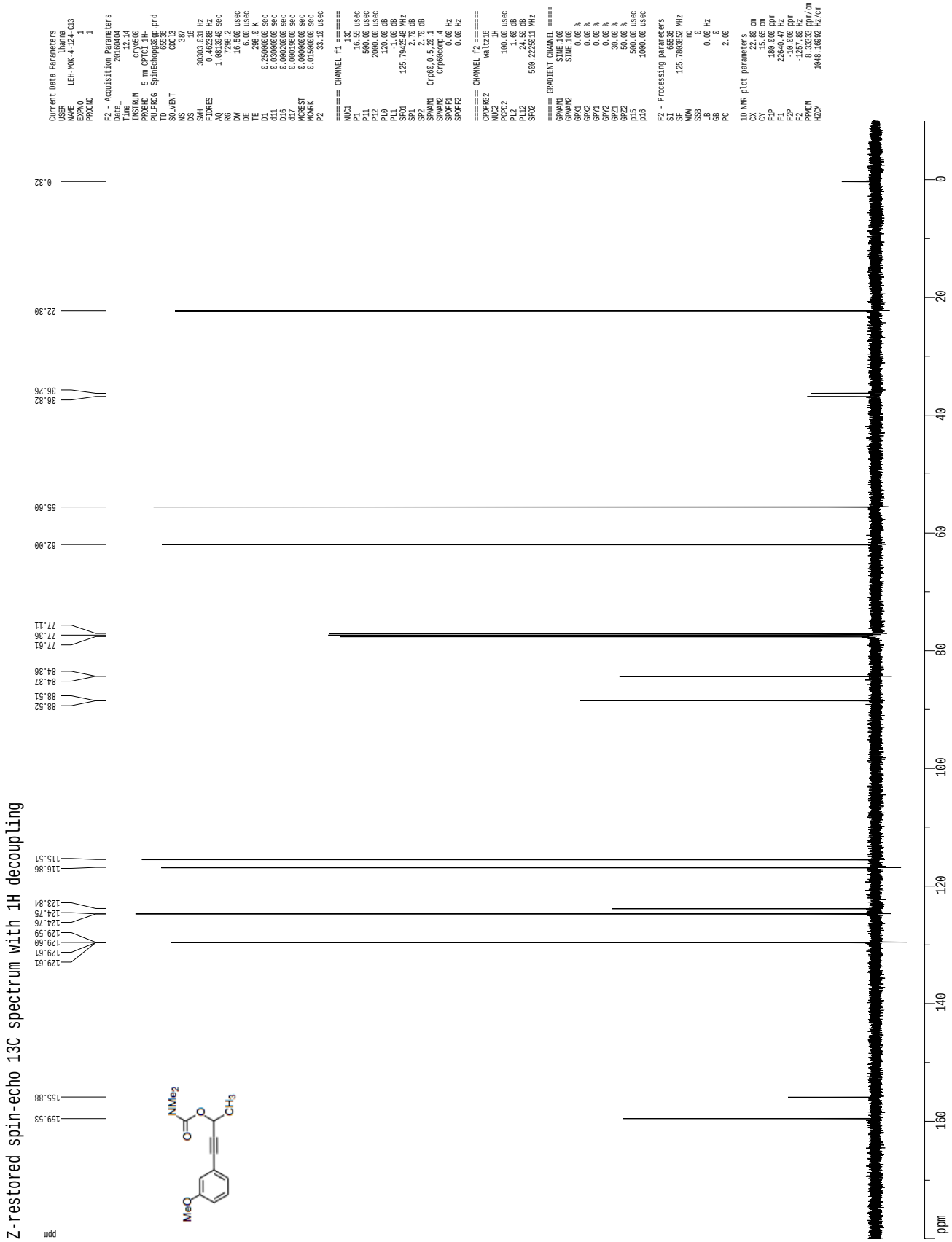


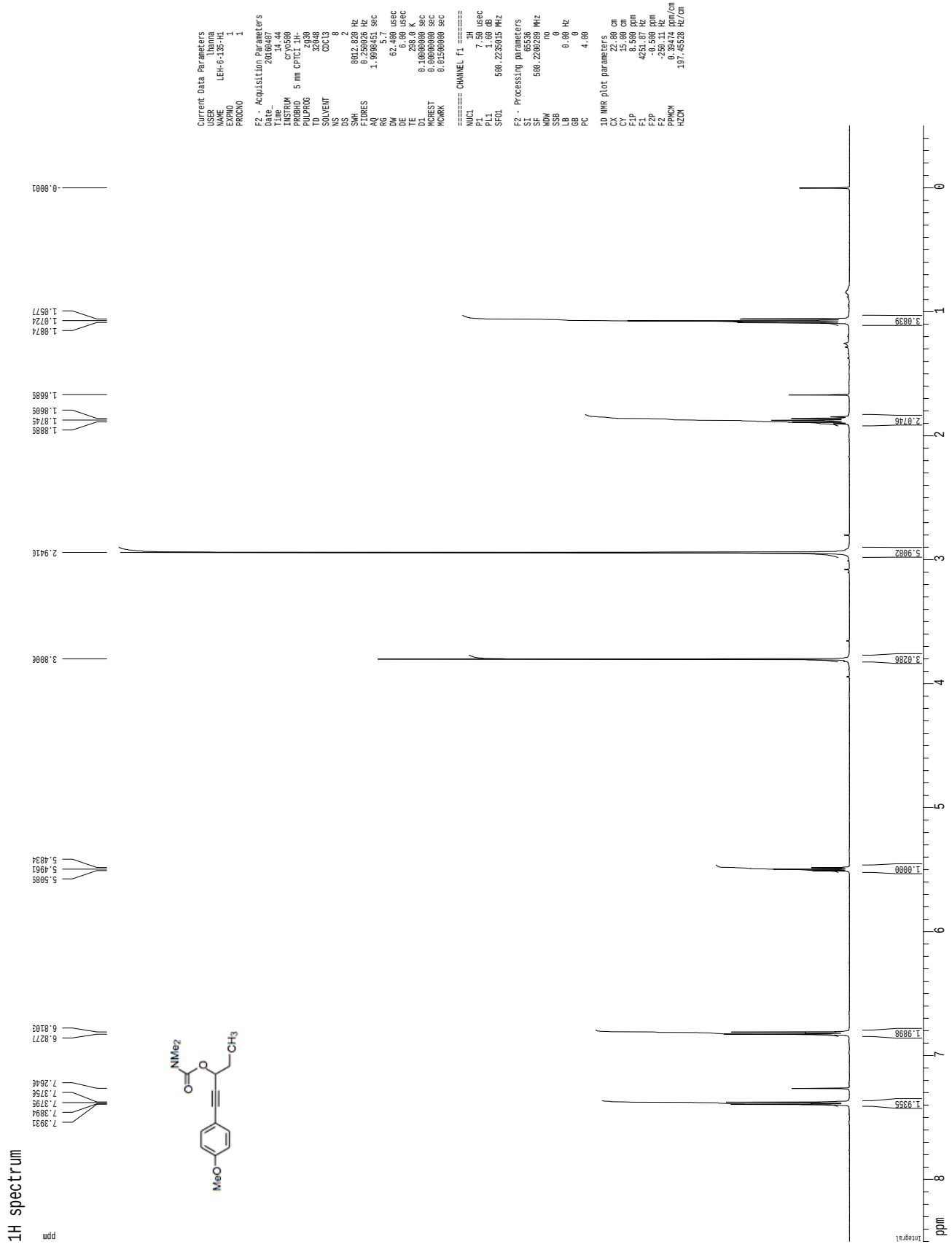
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.33	10.57	10.96	0.00	10.54	58.2	15.8	10.536
2	UNKNOWN	11.03	11.36	11.93	0.00	89.46	446.0	134.4	89.464
Total						100.00	504.2	150.3	100.000


1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-6-171-reH1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
20160608
Time 17.58
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65022
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.123114 Hz
AQ 0.9998228 sec
RG 12.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200203 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
7.45811
7.45426
7.44788
7.44536
7.43877
7.30388
7.30015
7.29367
7.28958
7.26401
5.52176
5.50894
5.49622
2.94755
1.90278
1.88827
1.87484
1.86026
1.69666
1.09724
1.08247
1.06754
0.00016
NMe2
O
O
CH3
3.5573
2.3887
6.6257
1.0000
3.0596
2.0450
ppm
Integral

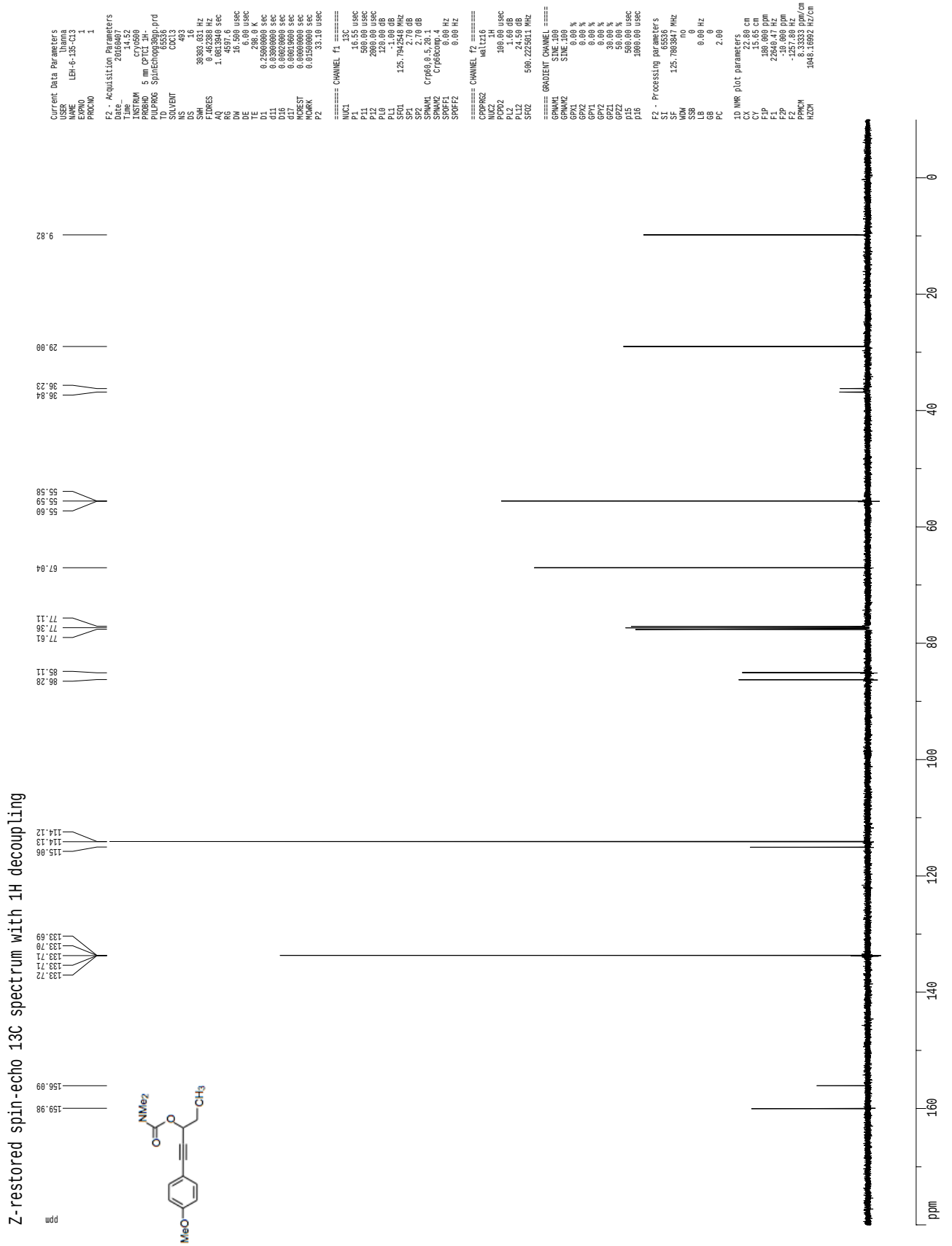

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
156.05
132.25
132.24
132.23
128.71
128.54
128.53
128.52
128.51
122.96
87.67
85.20
77.61
77.36
77.11
66.92
36.85
36.24
28.92
9.79
NMe2
CH3

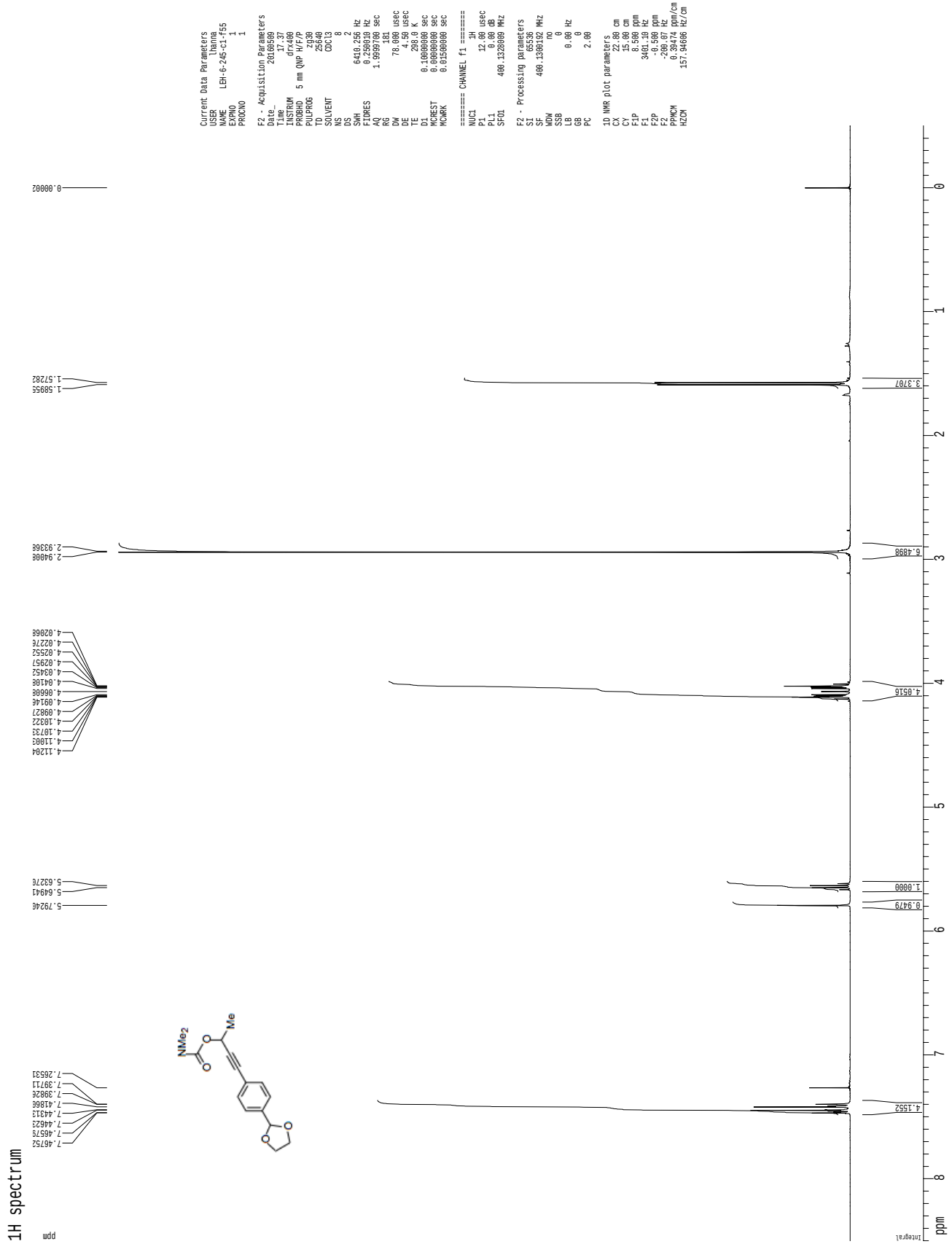

1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-MOK-4-124-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time 12.08
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200298 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.0001
1.5786
1.5914
1.6708
2.9424
3.7815
3.7896
5.6204
5.6338
5.6473
5.6604
6.8555
6.8576
6.8607
6.8722
6.8743
6.8774
6.9686
6.9705
7.0315
7.0338
7.0476
7.0485
7.1846
7.1995
7.2165
7.2646
3.1768
6.1255
3.1736
1.0195
1.0049
1.0000
1.0160
1.0425
Integral
ppm
NMe2
O
O
CH3
MeO

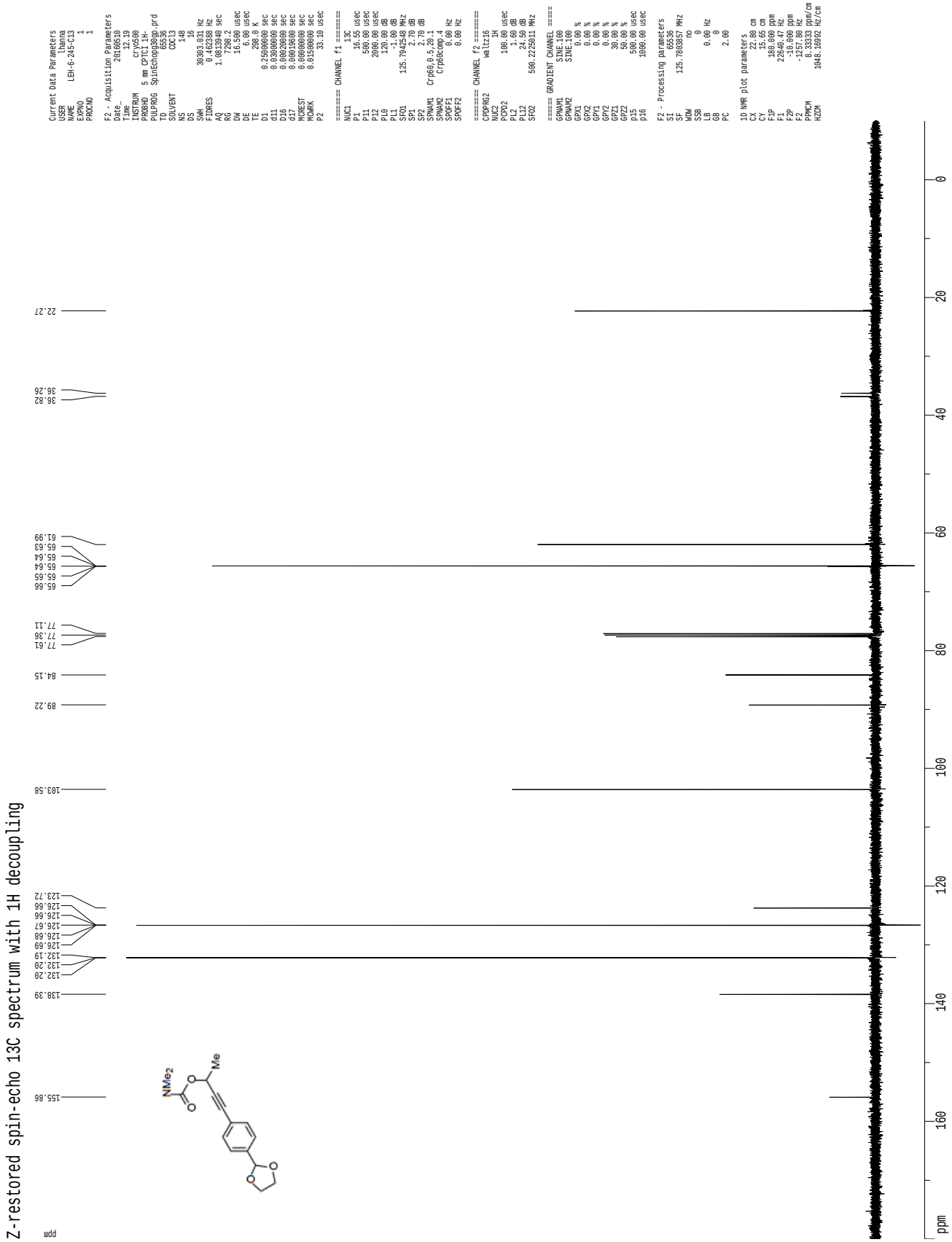

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
O
O
CH3
MeO
155.88
159.53
129.61
129.61
129.60
129.59
124.76
124.75
123.84
116.86
115.51
88.52
88.51
84.37
84.36
77.61
77.36
77.11
62.00
55.60
36.82
36.26
22.30
0.32
Current Data Parameters
USER Thanna
NAME LEH-MOK-4-124-c13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time 12.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 387
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803852 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm

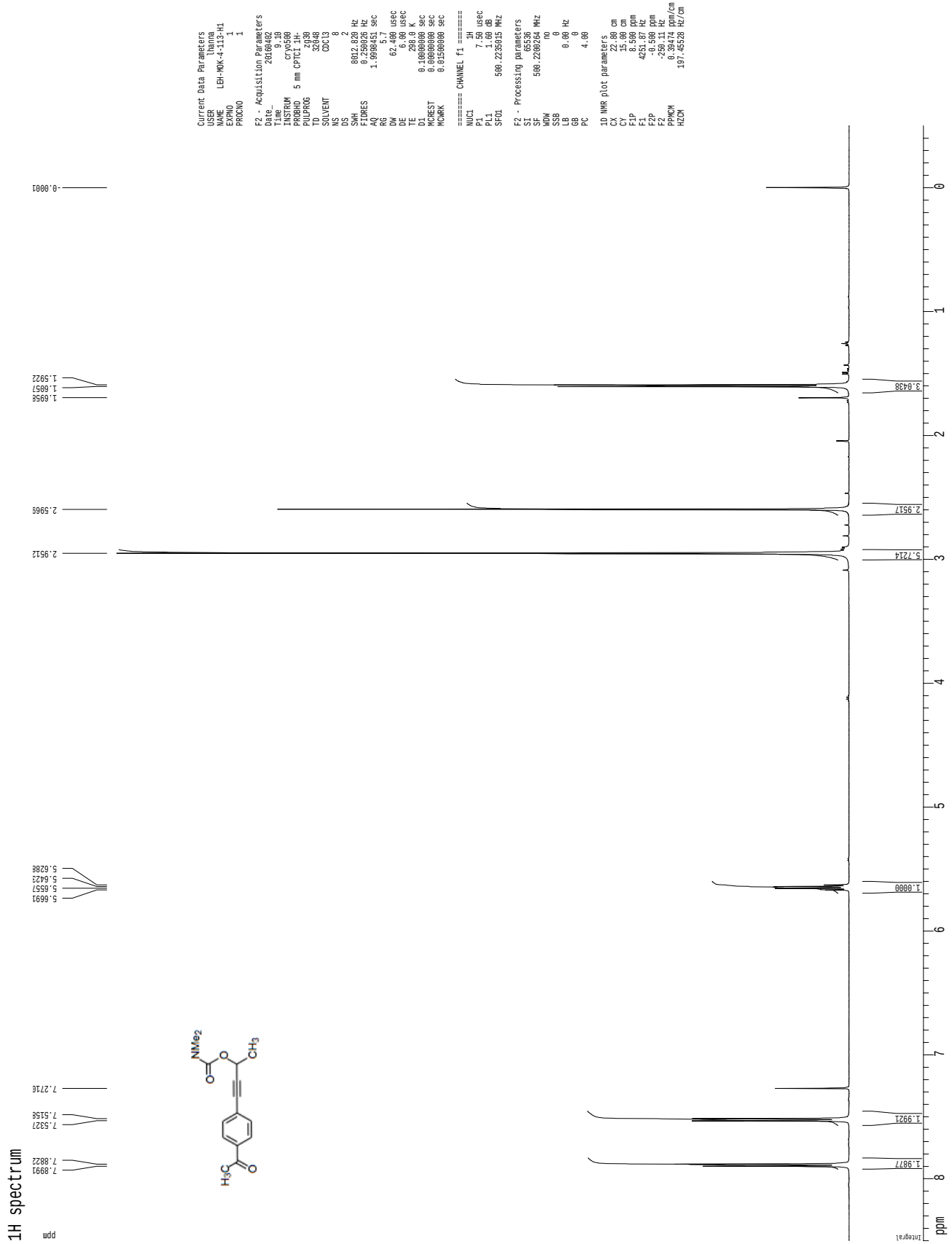

1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-6-135-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160407
Time 14.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200280 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.0001
1.0577
1.0724
1.0874
1.6685
1.8609
1.8745
1.8885
2.9416
3.8006
5.4834
5.4961
5.5089
6.8103
6.8277
7.2646
7.3736
7.3795
7.3894
7.3931
3.0839
2.0746
5.9882
3.0286
1.0000
1.9898
1.9355
NMe2
O
O
CH3
MeO
ppm
Integral

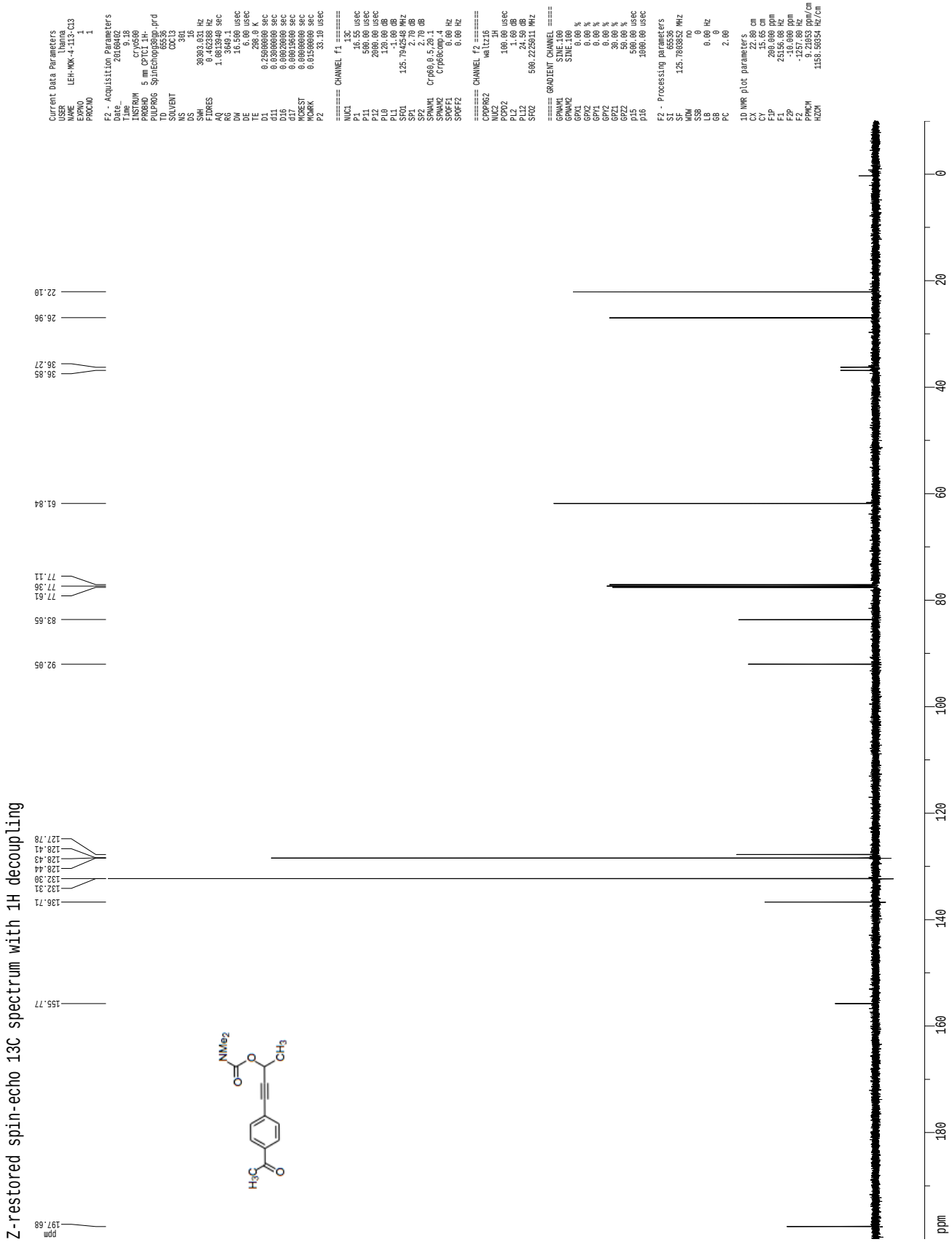
Z-restored spin-echo 13C spectrum with 1H decoupling

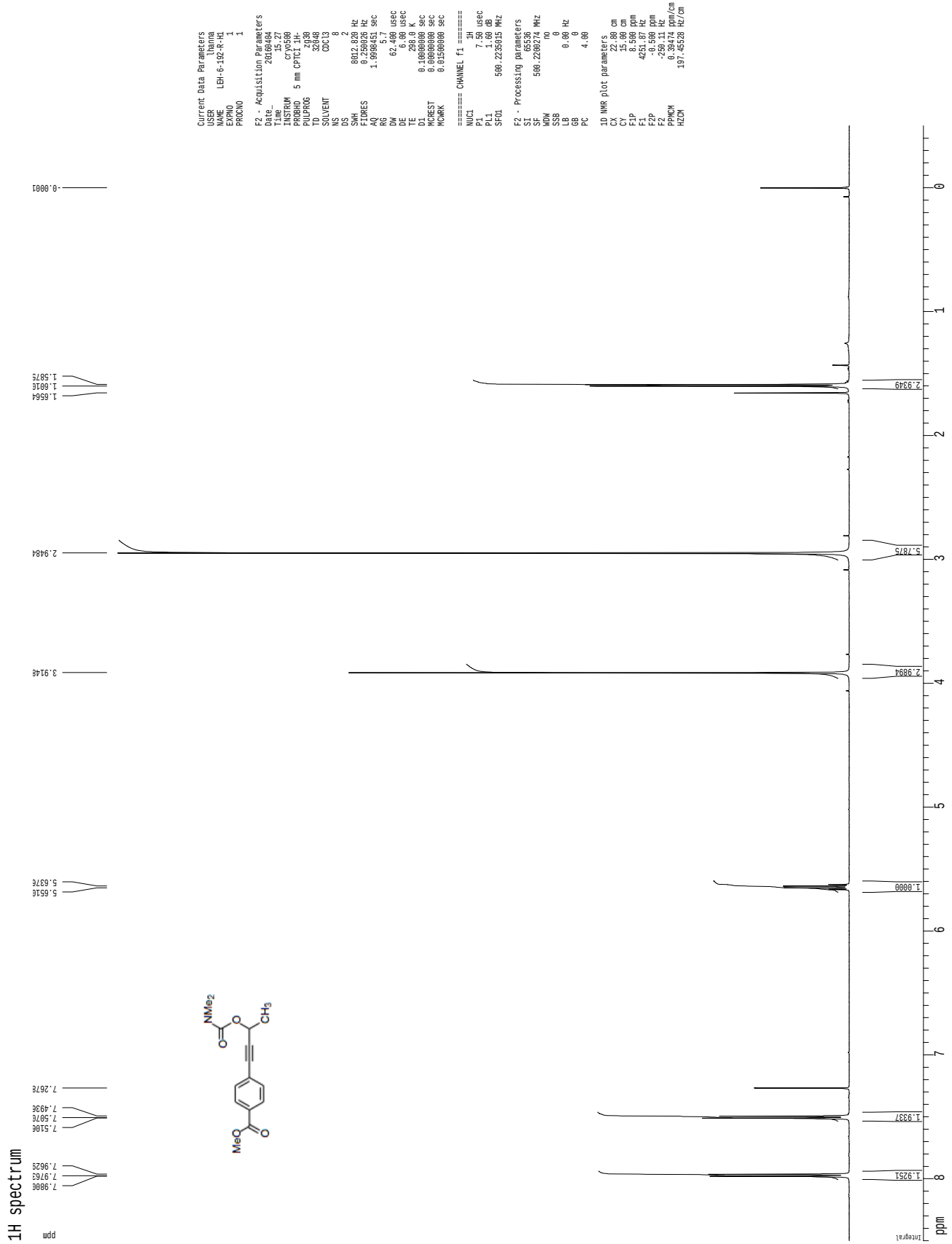


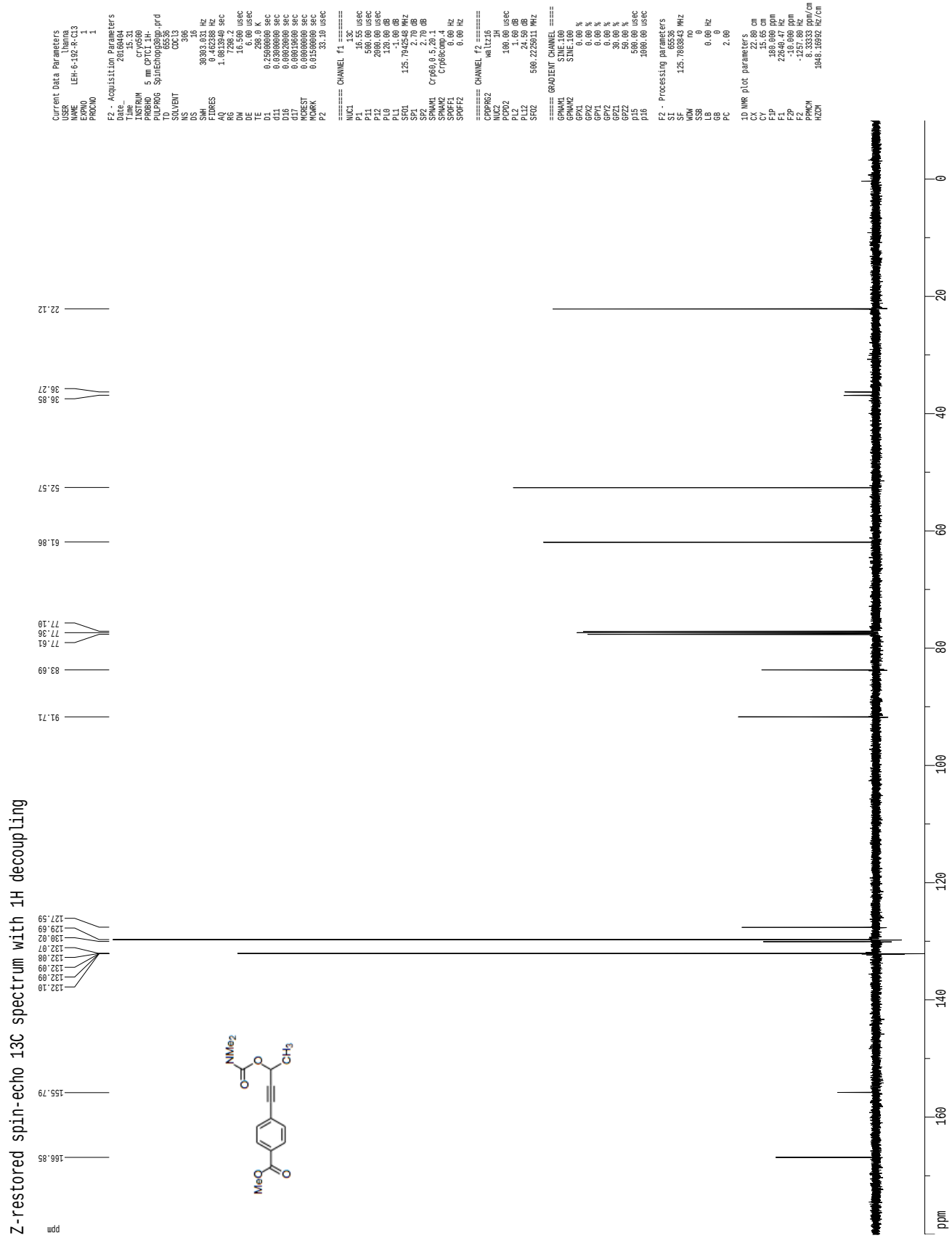

1H spectrum
Current Data Parameters
USER Thomas
NAME LEH-6-245-d1-f55
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160509
Time 17.37
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 181
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300132 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
NMe2
Me
0.00002
1.57282
1.58955
2.93368
2.94008
4.02068
4.02276
4.02552
4.02957
4.03452
4.04108
4.06608
4.09146
4.09827
4.10322
4.10733
4.11003
4.11204
5.63276
5.64941
5.79246
7.26531
7.39711
7.39826
7.41866
7.44913
7.44623
7.46579
7.46752
3.3707
6.4898
4.0516
1.0000
0.9479
4.1552
ppm
Integral

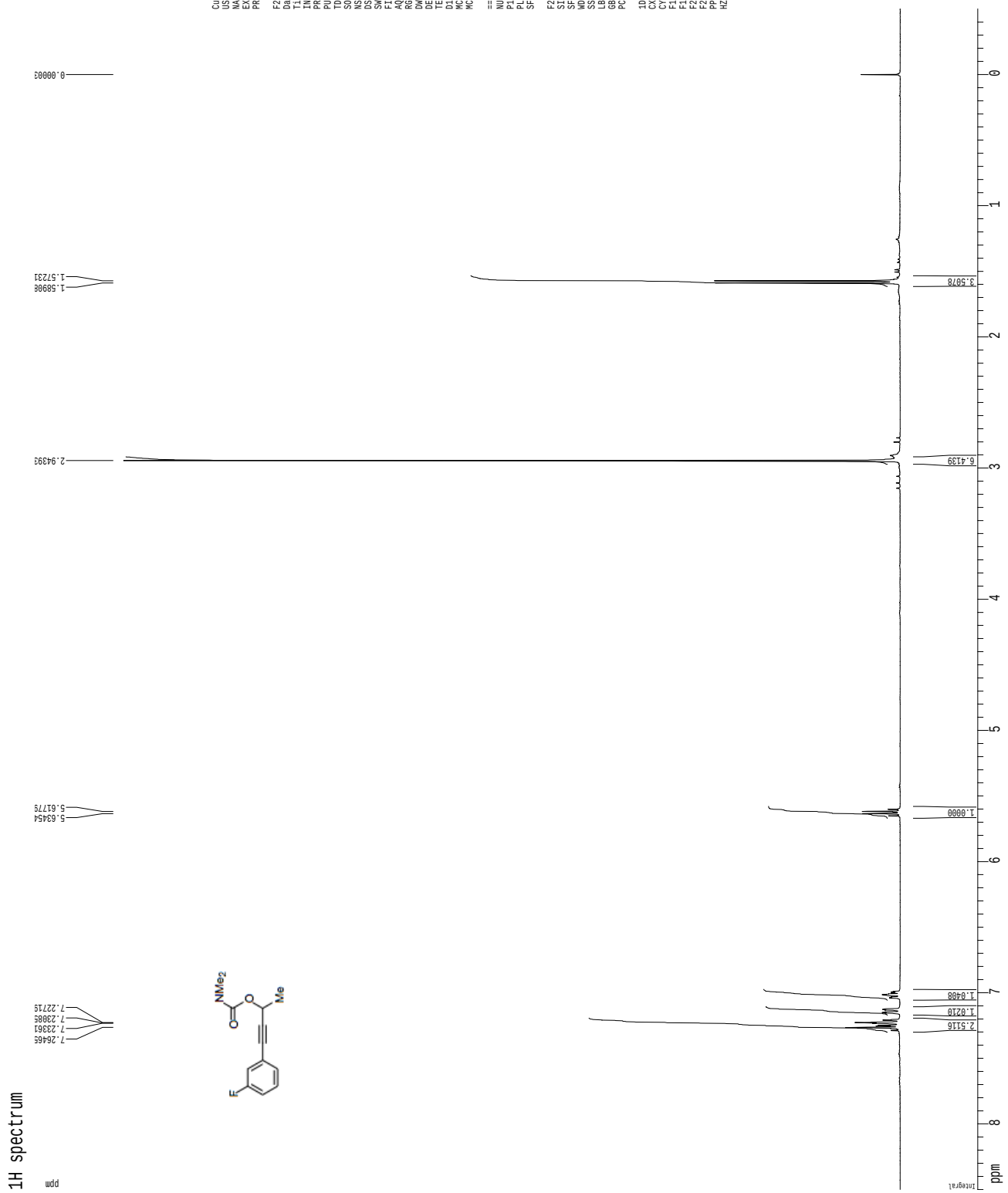

Z-restored spin-echo 13C spectrum with 1H decoupling
Current Data Parameters
USER thanna
NAME LEH-6-245-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160510
Time 12.19
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 148
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 5000.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803857 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
22.27
36.82
36.26
61.99
65.63
65.64
65.64
65.66
65.65
77.11
77.36
77.61
84.15
89.22
103.58
123.72
126.66
126.66
126.67
126.68
126.69
132.19
132.20
132.20
138.39
155.86
NMe2
Me
O
O
O
O
ppm
0
20
40
60
80
100
120
140
160

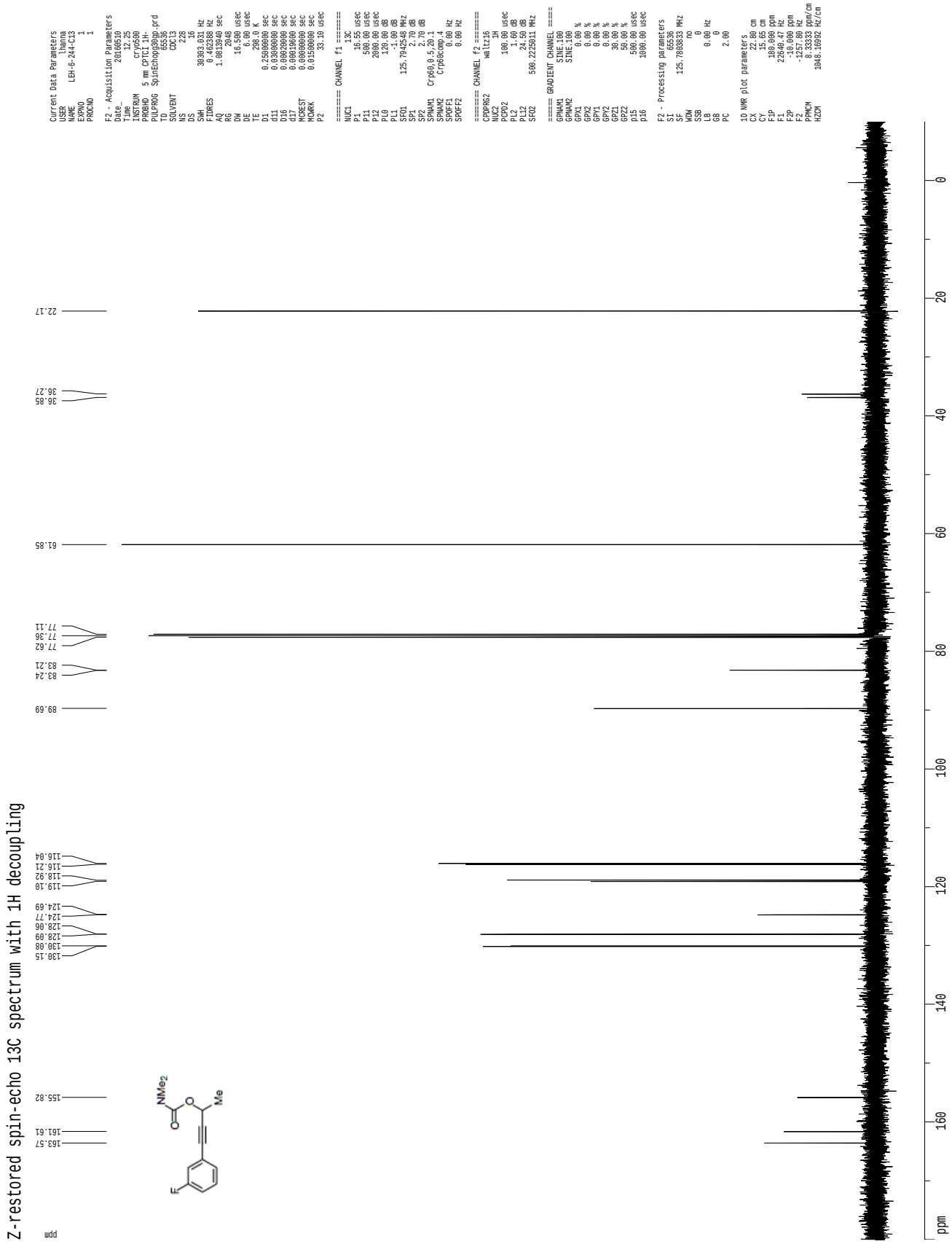

1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-MOK-4-113-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160402
Time 9.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200264 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
-0.0001
1.5922
1.6057
1.6958
2.5965
2.9512
5.6288
5.6423
5.6557
5.6691
7.2716
7.5156
7.5321
7.8822
7.8993
3.0438
2.9517
5.7214
1.0000
1.9921
1.9877
integral
ppm
NMe2
CH3
H3C

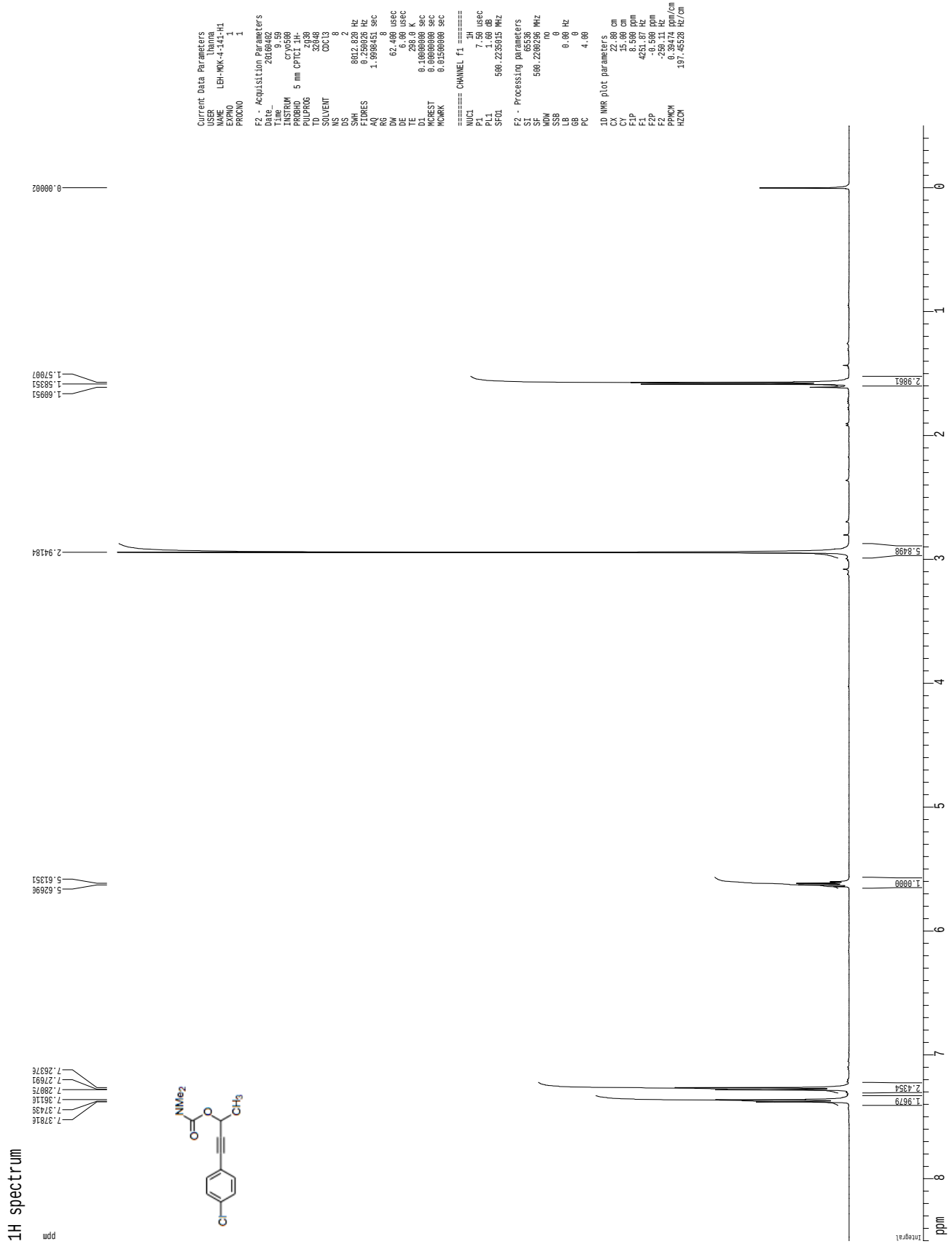

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
197.68
155.77
136.71
132.31
132.30
128.44
128.43
128.41
127.78
92.05
83.65
77.61
77.36
77.11
61.84
36.85
36.27
26.96
22.10
NMe2
O
O
CH3
H3C
O
Current Data Parameters
USER Thanna
NAME LEH-MOK-4-113-c13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160402
Time 9.18
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 301
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3649.1
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803852 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 200.000 ppm
F1 25156.08 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.50354 Hz/cm
ppm
0
20
40
60
80
100
120
140
160
180


1H spectrum
ppm
Integral
Current Data Parameters
USER lhanna
NAME LEH-6-192-R-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time 15.27
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200274 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
7.9806
7.9763
7.9629
7.5106
7.4936
7.4876
7.2678
5.6516
5.6376
3.9148
2.9484
1.6564
1.6016
1.5875
-0.0001
1.9251
1.9337
1.0000
2.9894
5.7875
2.9349
NMe2
O
O
CH3
MeO
O

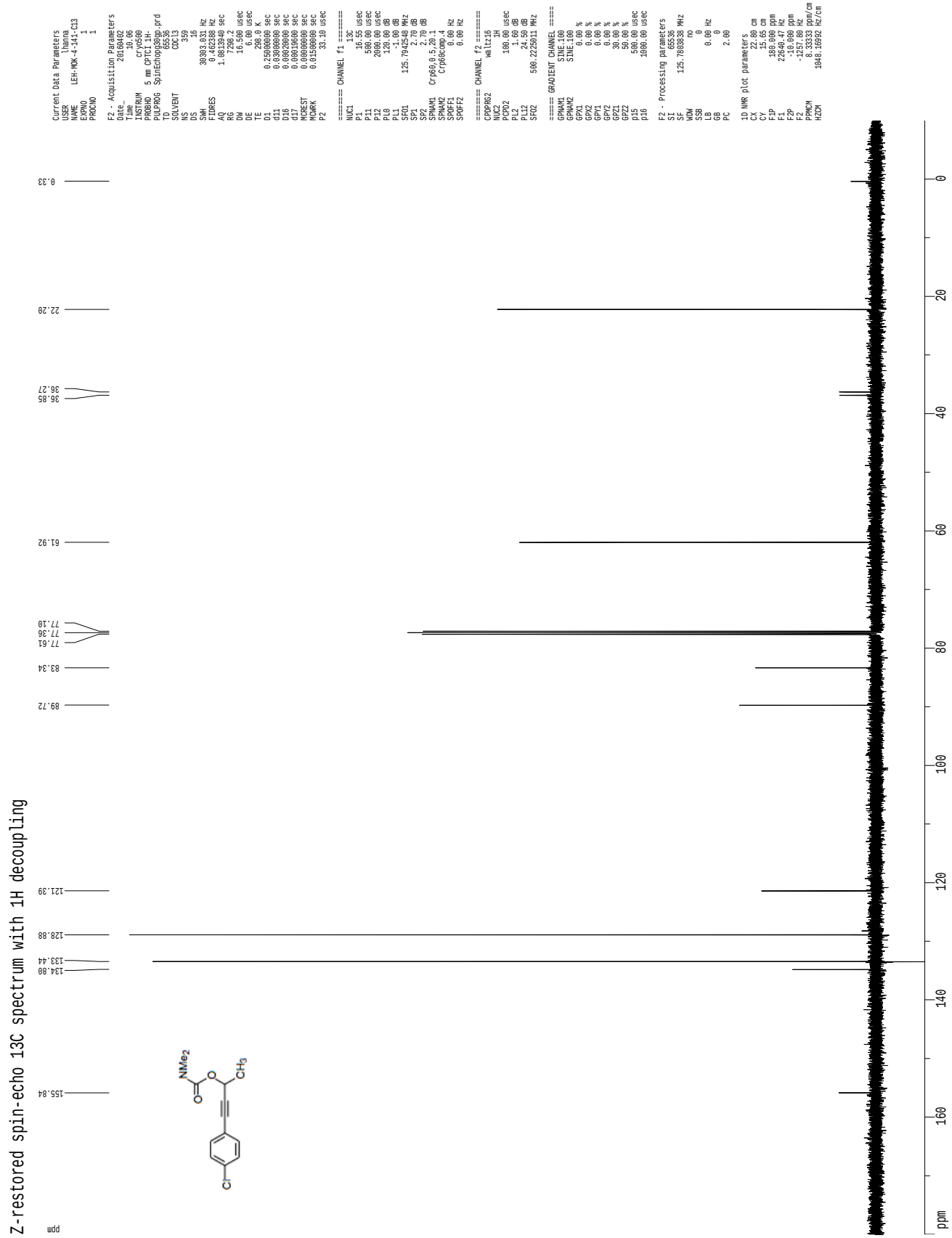

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
166.85
155.79
132.10
132.09
132.09
132.08
132.07
130.02
129.69
127.59
91.71
83.69
77.61
77.36
77.10
61.86
52.57
36.85
36.27
22.12
NMe2
CH3
MeO
Current Data Parameters
USER Thanna
NAME LEH-6-192-M-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time 15.31
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
NS 336
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883843 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm

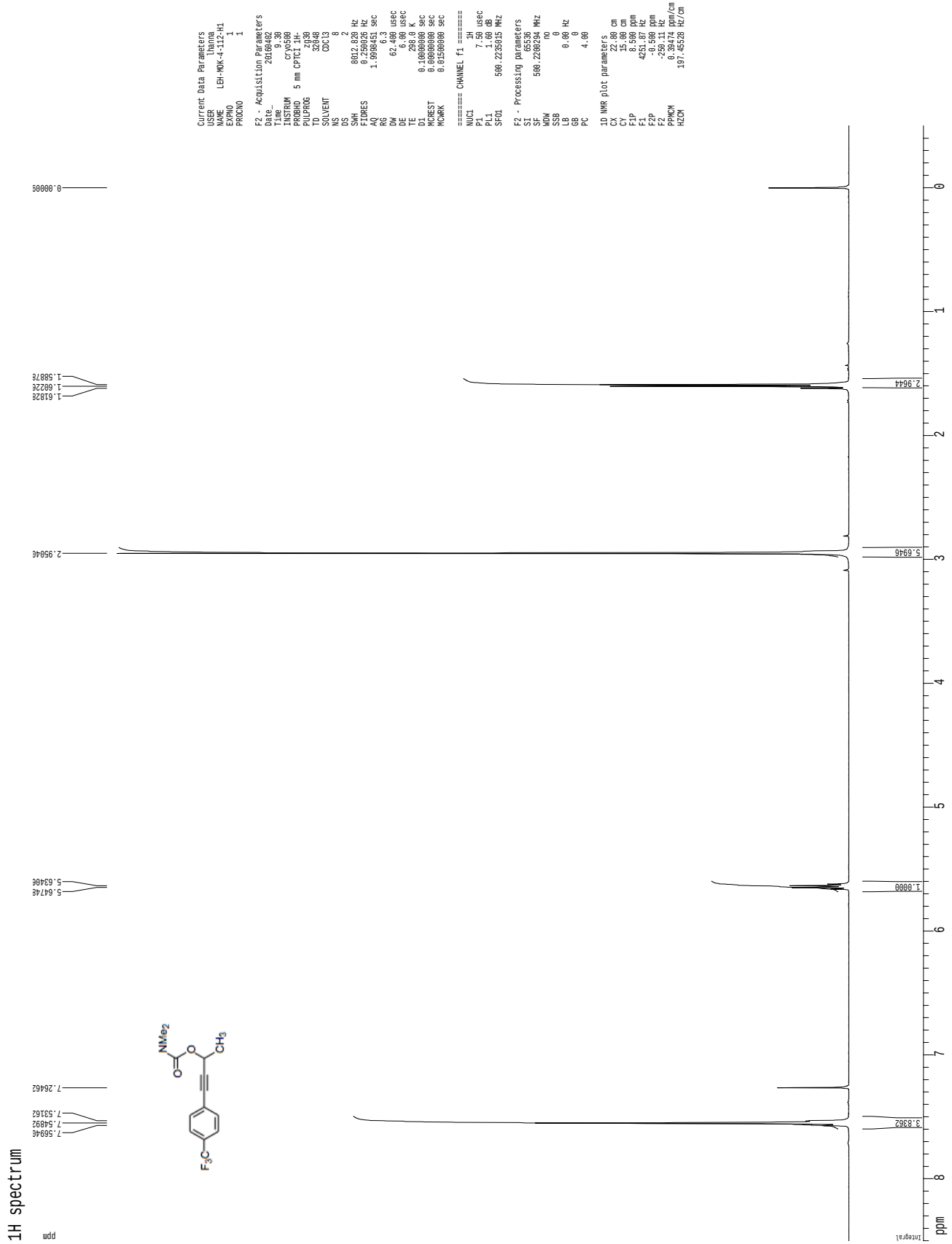

1H spectrum
Current Data Parameters
USER Thomas
NAME LEH-6-244-crude
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160506
Time 14.56
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250078 Hz
AQ 1.9990780 sec
RG 228.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300135 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm
NMe2
O
O
Me
F
7.26465
7.23361
7.23086
7.22719
5.63454
5.61779
2.94393
1.58908
1.57231
0.00003
1.0408
1.0210
2.5116
1.0000
6.4139
3.5078
ppm
Integral

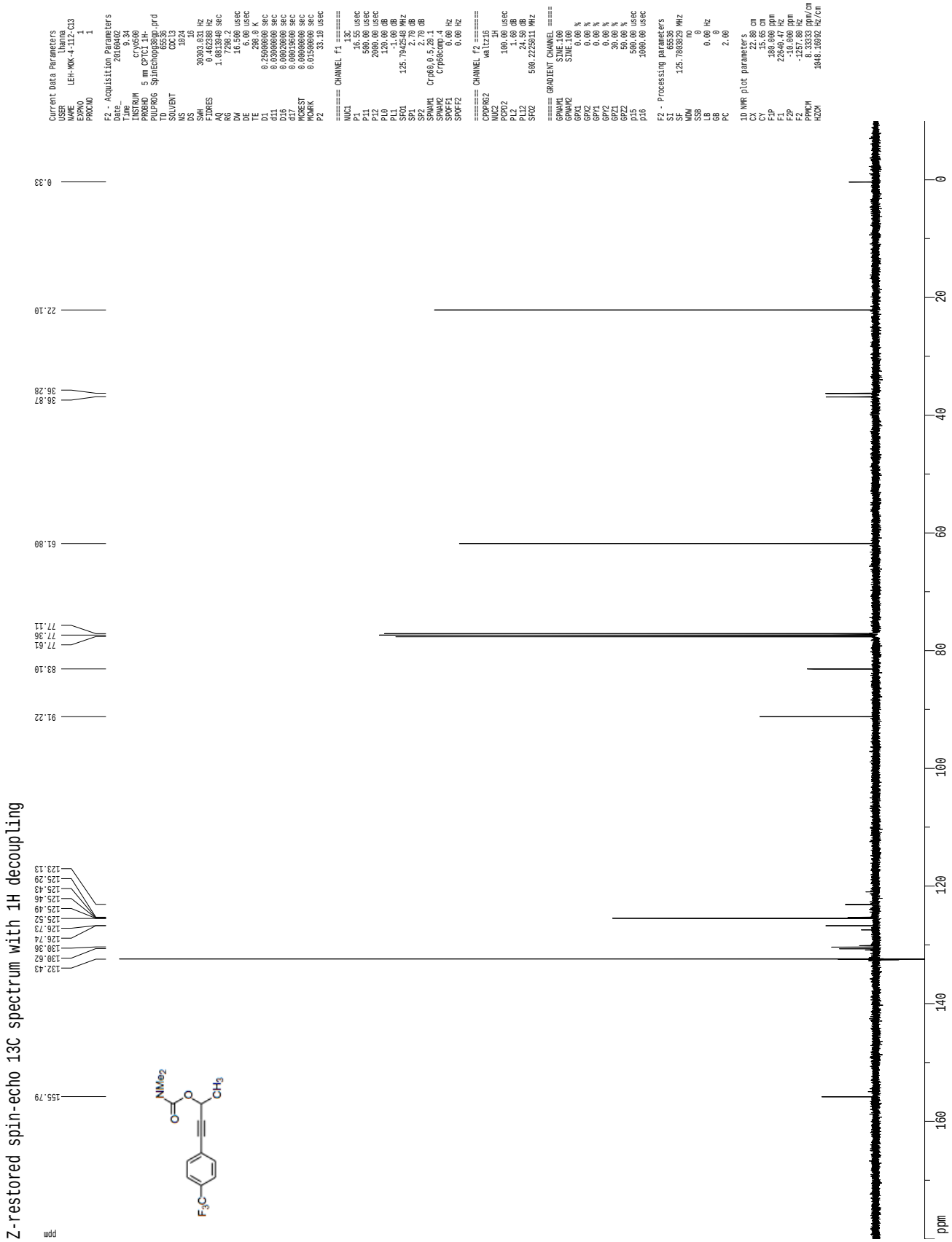

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
O
O
Me
F
22.17
36.85
36.27
61.85
77.11
77.36
77.62
83.21
83.24
89.69
116.04
116.21
118.92
119.10
124.69
124.77
128.06
128.09
130.08
130.15
155.82
161.61
163.57
Current Data Parameters
USER Thanna
NAME LEH-6-244-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160510
Time 12.25
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 238
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.08139940 sec
RG 2048
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019060 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 5000.00 usec
p16 1000.000 usec
F2 - Processing parameters
SI 65536
SF 125.7803833 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
ppm
0
20
40
60
80
100
120
140
160

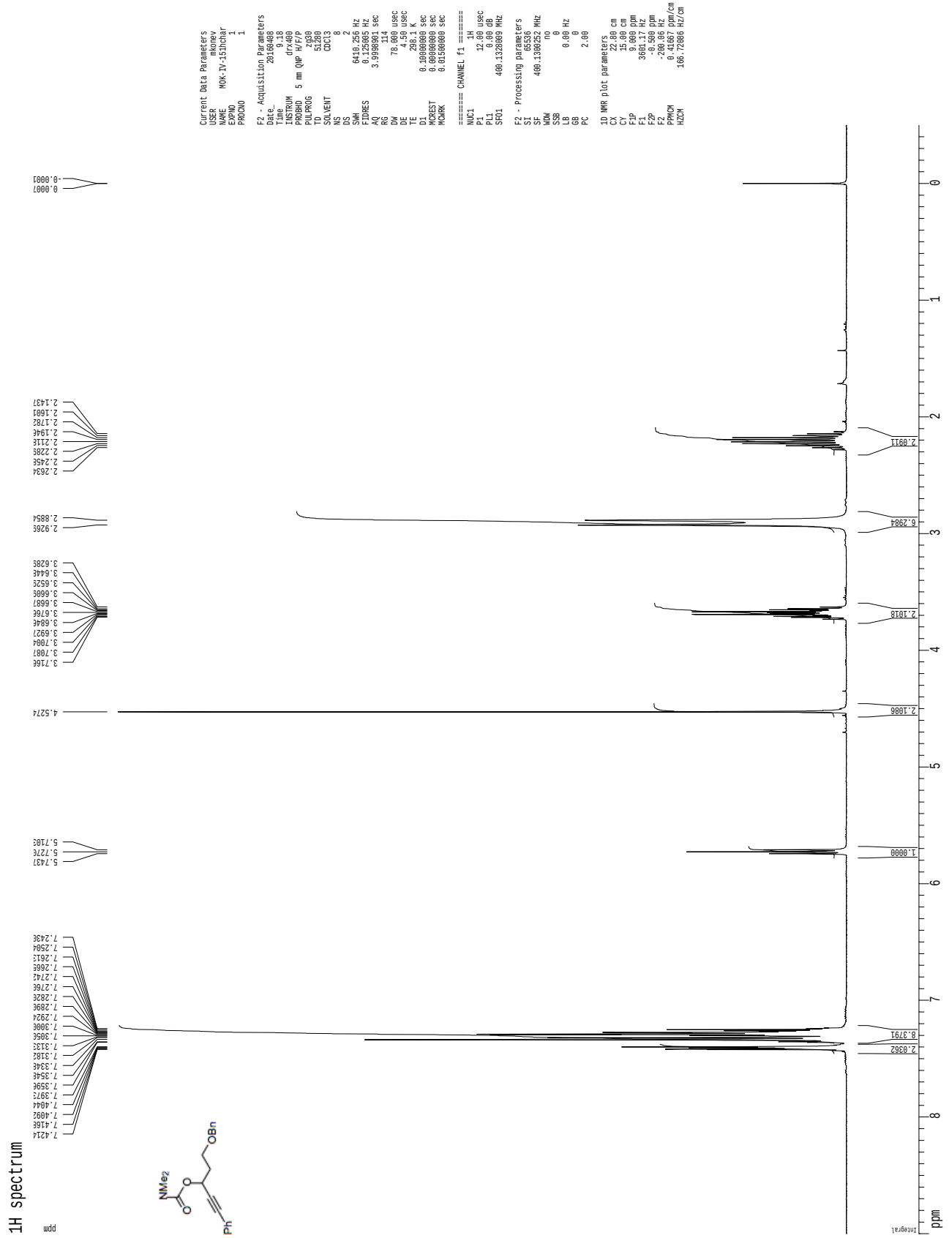

1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-MOK-4-141-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160402
Time 9.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200296 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.38474 ppm/cm
HZCM 197.45520 Hz/cm
0.00002
1.57007
1.58351
1.60951
2.94184
5.62696
5.61351
7.26376
7.27691
7.28075
7.36116
7.37436
7.37816
2.9861
5.8498
1.0000
2.4354
1.9679
integral
ppm
NMe2
O
O
CH3
Cl

Z-restored spin-echo 13C spectrum with 1H decoupling

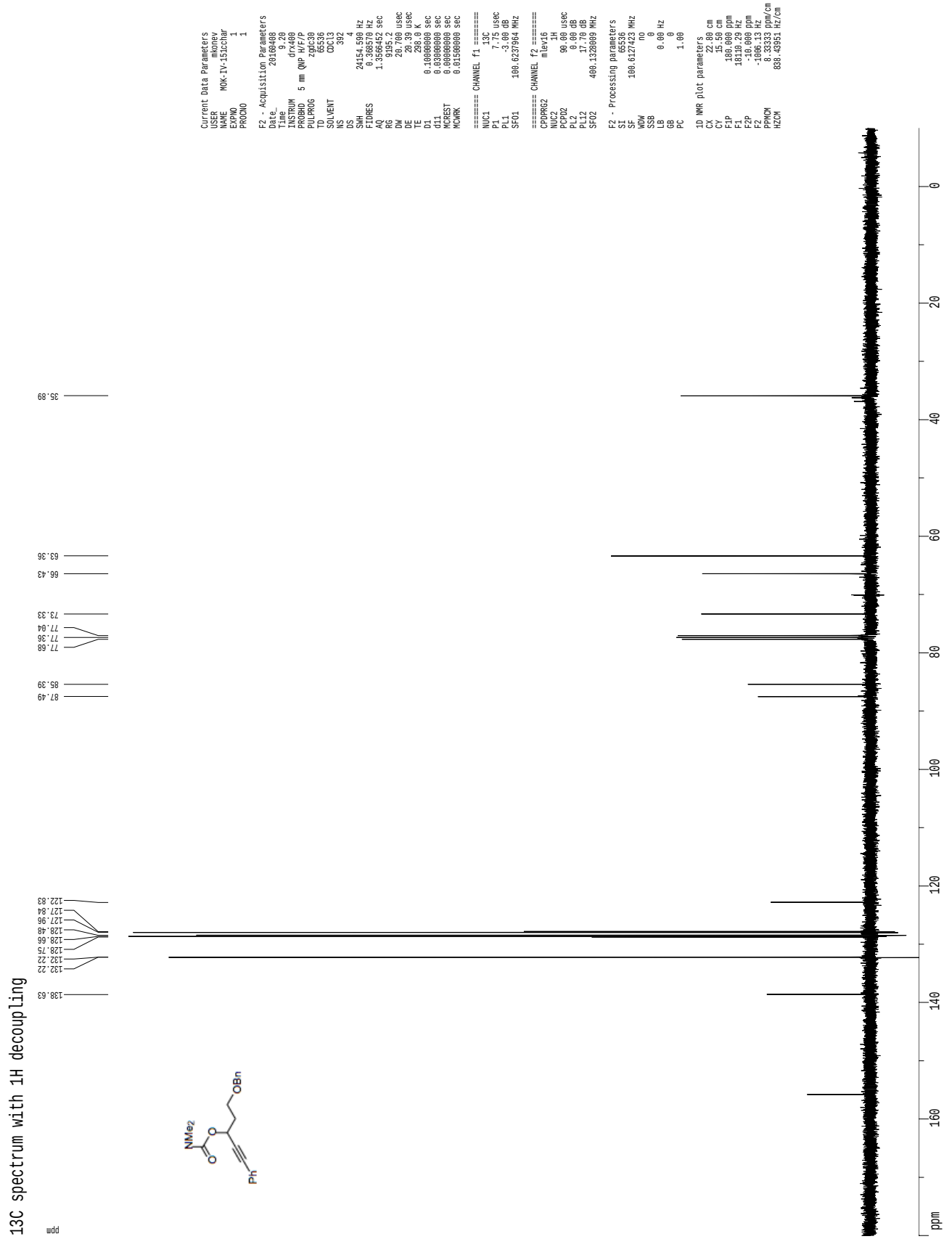


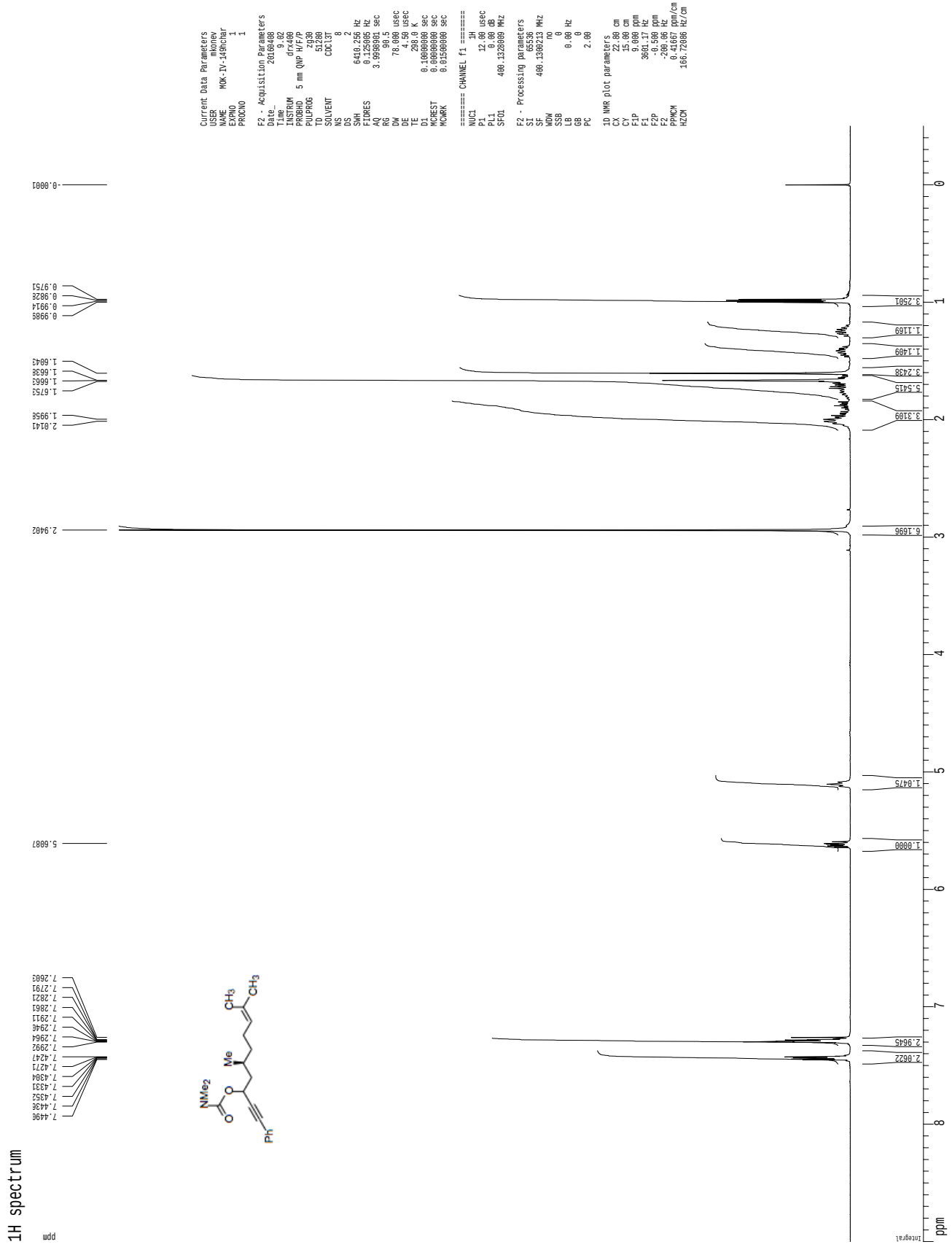

1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-MOK-4-112-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160402
Time 9.30
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200294 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.00000
1.58778
1.60226
1.61826
2.95046
5.63406
5.64748
7.26462
7.53162
7.54892
7.56946
2.9644
5.6946
1.0000
3.8362
NMe2
O
O
CH3
F3C
ppm
Integral


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
O
O
CH3
F3C
155.79
132.43
130.62
130.36
126.74
126.73
125.52
125.49
125.46
125.43
125.29
123.13
91.22
83.10
77.61
77.36
77.11
61.80
36.87
36.28
22.10
0.33
Current Data Parameters
USER Thanna
NAME LEH-MOK-4-112-c13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160402
Time 9.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
NS 1024
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803829 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1040.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm

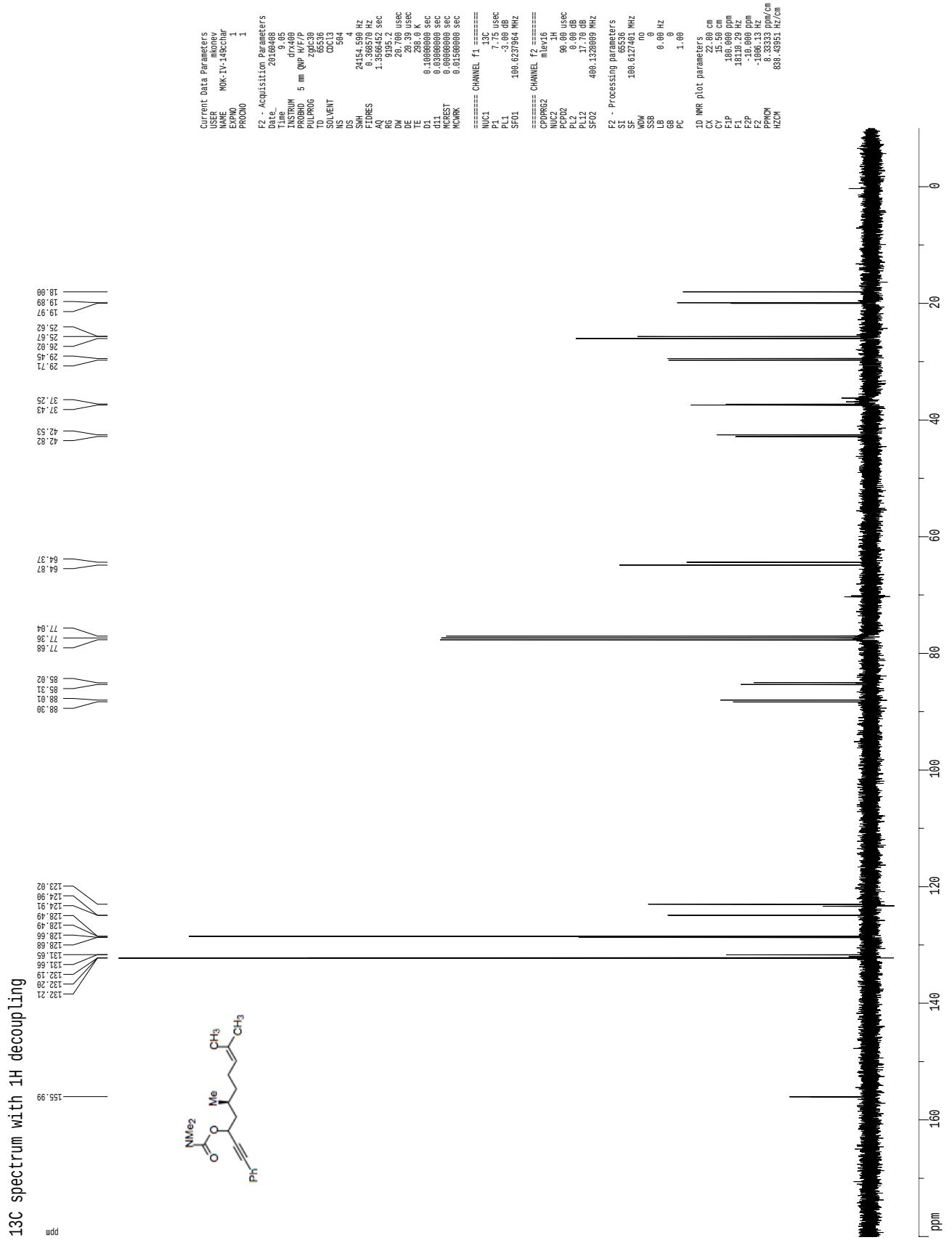

1H spectrum
ppm
integral
NMe2
OBn
Ph

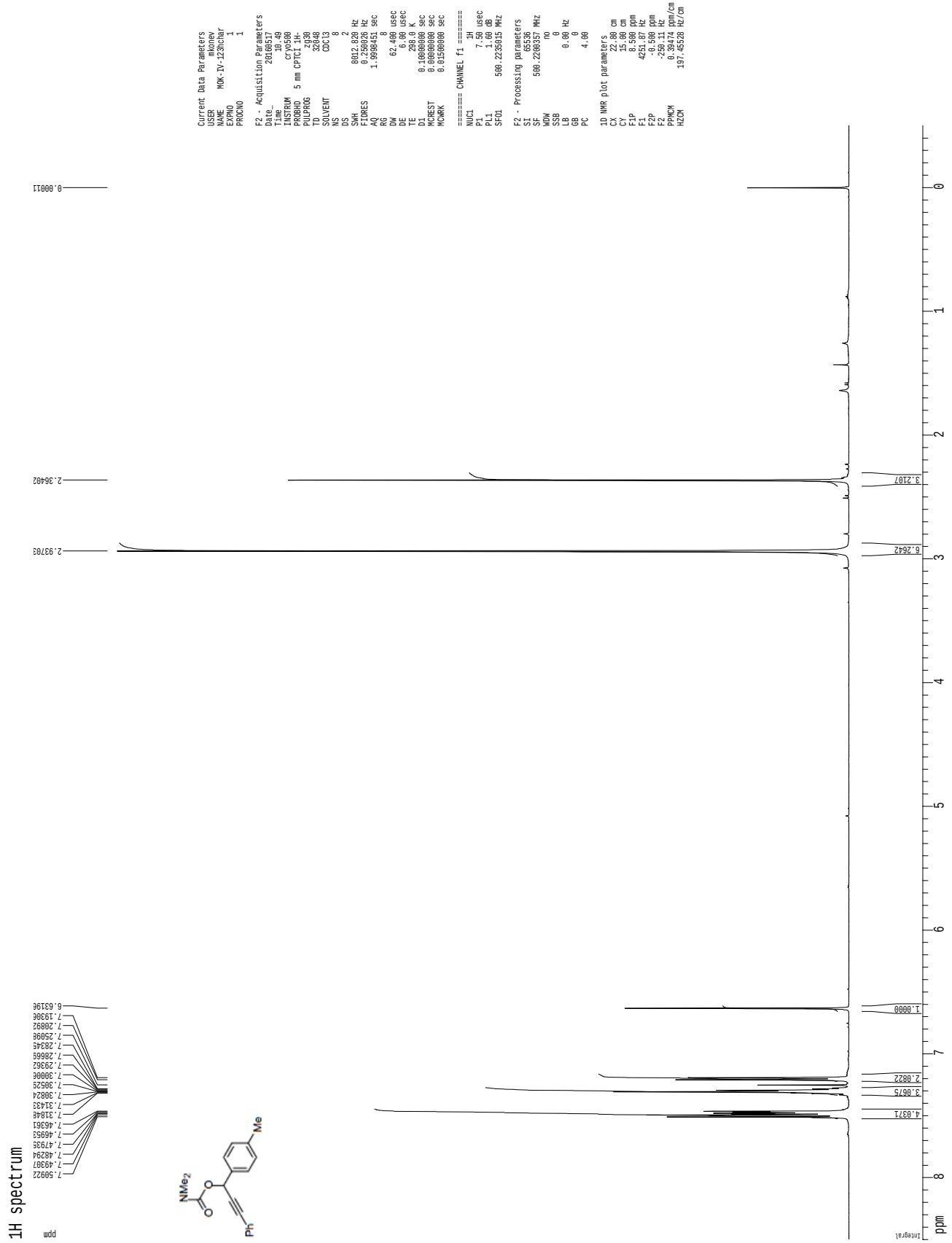
13C spectrum with 1H decoupling

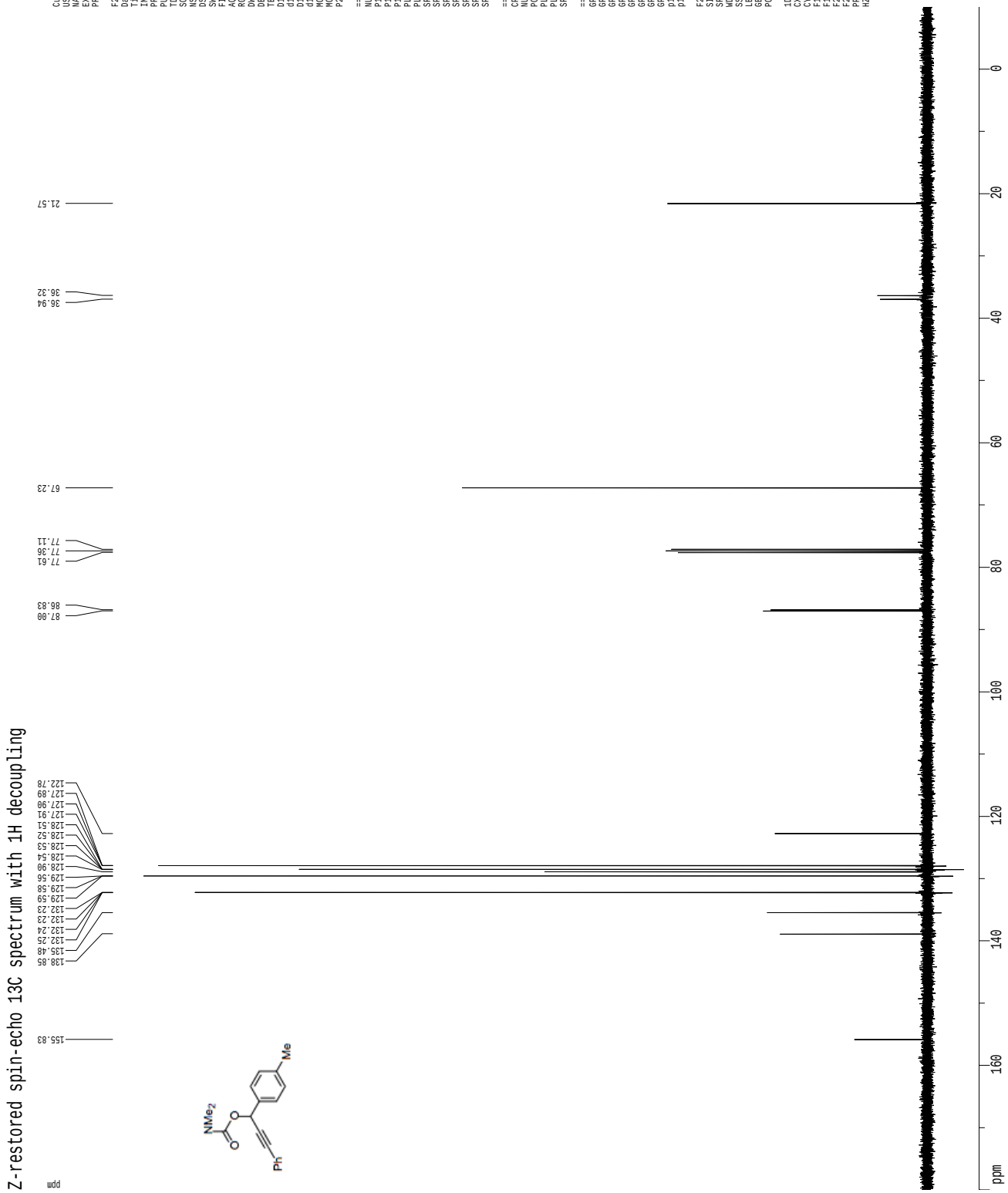


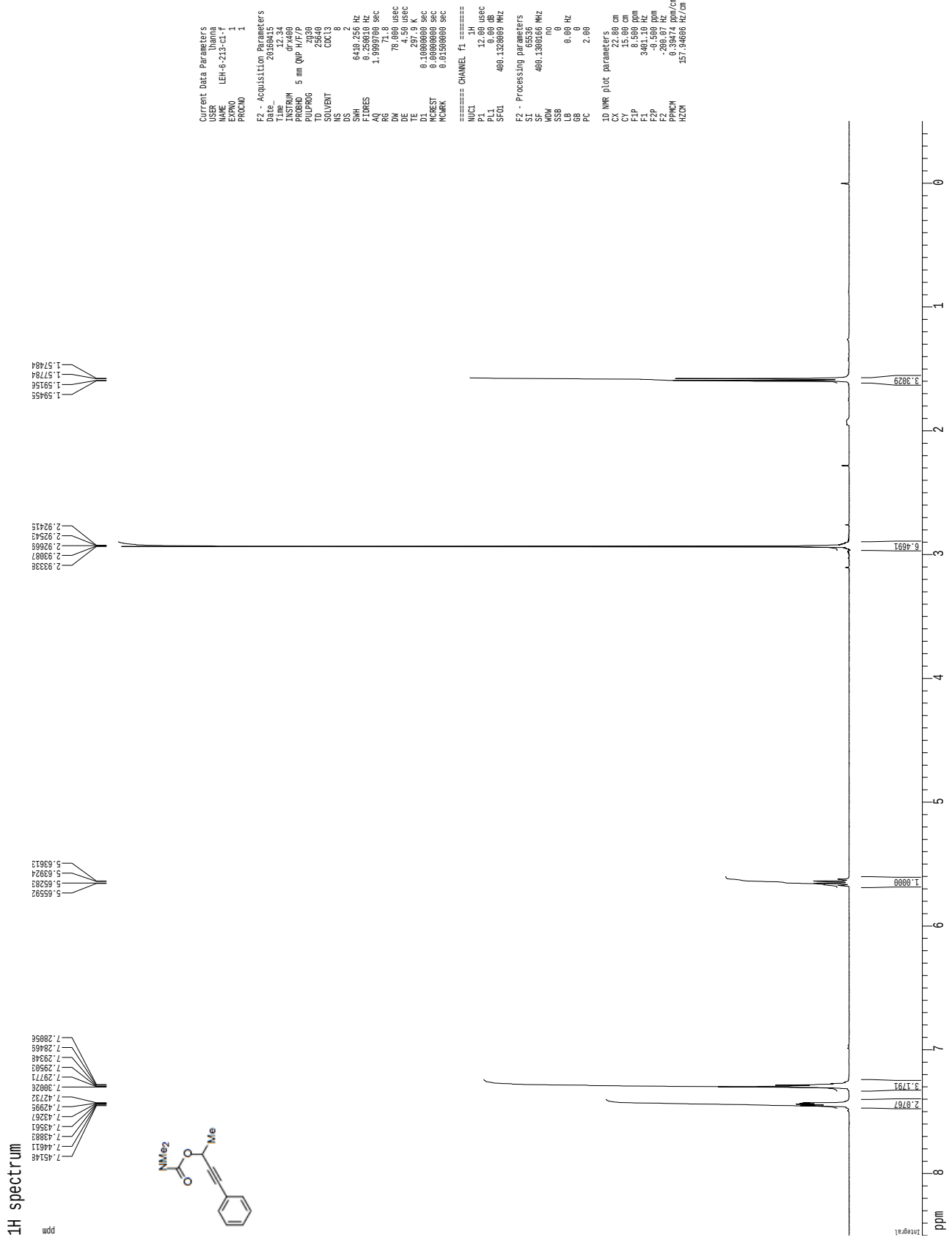

1H spectrum
Current Data Parameters
USER mvoney
NAME MOK-IV-149Pchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160408
Time 9.02
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 51200
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.125095 Hz
AQ 3.9908801 sec
RG 90.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300213 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -208.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72080 Hz/cm
0.0001
0.9751
0.9828
0.9914
0.9985
1.6043
1.6638
1.6663
1.6752
1.9958
2.0141
2.9402
5.6087
7.2603
7.2791
7.2821
7.2861
7.2911
7.2946
7.2964
7.2992
7.4247
7.4271
7.4304
7.4331
7.4352
7.4436
7.4496
3.2501
1.1169
1.1409
3.2438
5.5415
3.3109
6.1696
1.0475
1.0000
2.9645
2.0622
integral
ppm
NMe2
O
O
Me
CH3
CH3
Ph

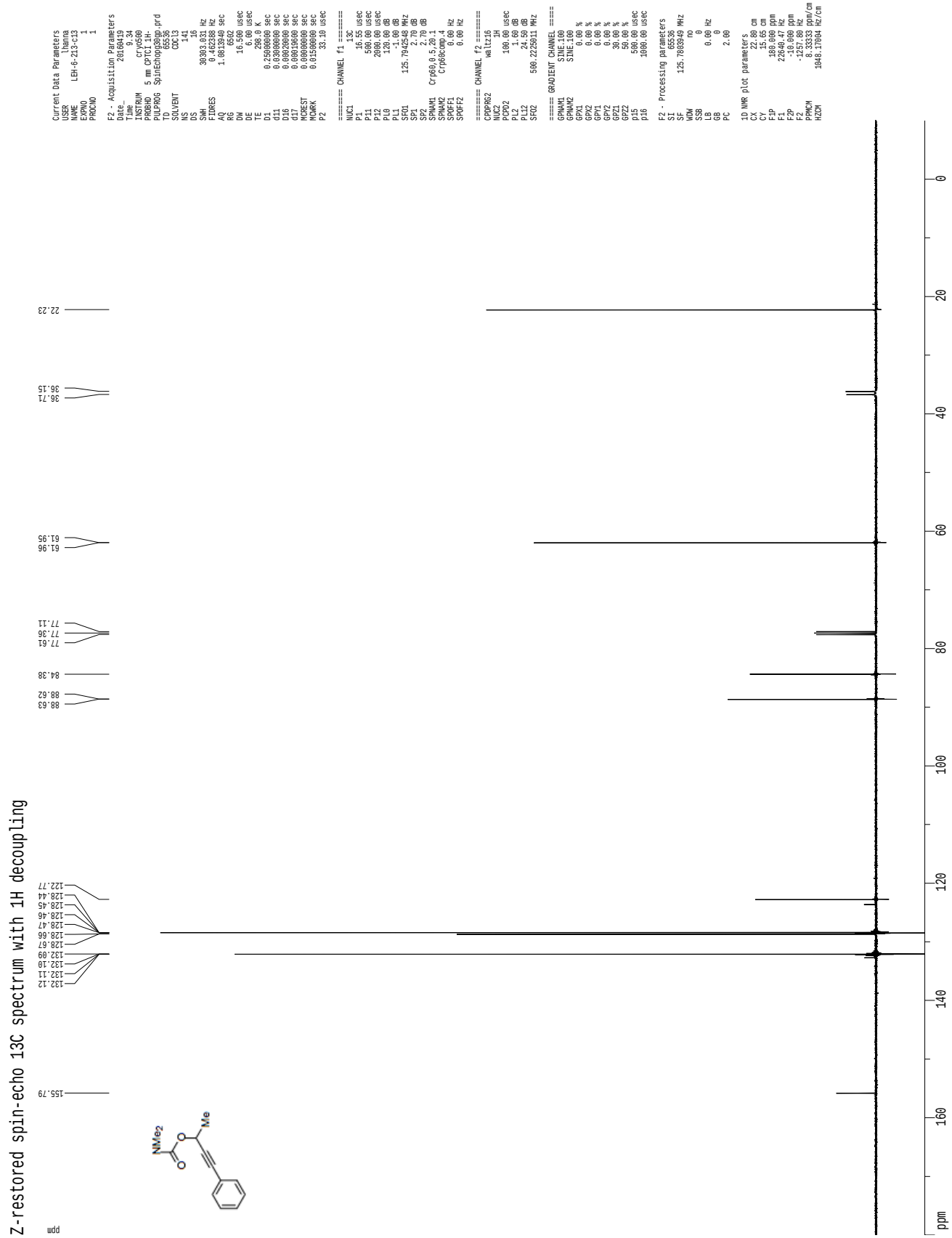
13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-123nchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160517
Time 10.49
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200357 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.596 ppm
F1 4251.87 Hz
F2P -0.560 ppm
F2 -280.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.00011
2.36402
2.93703
6.63196
7.19306
7.20892
7.25098
7.28345
7.28665
7.29362
7.30006
7.30524
7.30824
7.31433
7.31848
7.46361
7.46953
7.47935
7.48294
7.49307
7.50922
3.2107
6.2642
1.0000
2.0822
3.0675
4.0371
NMe2
O
O
Ph
Me
ppm
Integral


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
O
O
Ph
Me
155.83
138.85
135.48
132.25
132.24
132.23
132.23
129.59
129.58
129.56
128.90
128.54
128.53
128.52
128.51
127.91
127.90
127.89
122.78
87.00
86.83
77.61
77.36
77.11
67.23
36.94
36.32
21.57
Current Data Parameters
NAME MOK-IV-123cther
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160517
Time 10.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 152
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803866 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER Dharma
NAME LEH-6-213-t1-f
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160415
Time 12.34
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999708 sec
RG 71.3
DW 78.000 usec
DE 4.50 usec
TE 297.9 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300016 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39477 ppm/cm
HZCM 157.94608 Hz/cm
7.45148
7.44611
7.43561
7.43883
7.43267
7.42995
7.42732
7.38026
7.29771
7.29503
7.29348
7.28469
7.28056
5.65592
5.65283
5.63924
5.63613
2.93338
2.93087
2.92669
2.92543
2.92415
1.59456
1.59156
1.57784
1.57484
NMe2
O
O
Me
3.3029
6.4691
1.0000
3.1791
2.0767
ppm
Integral

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Current Data Parameters
USER      thanna
NAME      LEH-6-213-c13
EXPNO           1
PROCNO          1

F2 - Acquisition Parameters
Date_      20160419
Time           9.34
INSTRUM      cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchoDig30gp.prd
TD          65536
SOLVENT     CDCl3
NS             144
DS              16
SWH        30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG               6502
DW            16.500 usec
DE             6.00 usec
TE             298.0 K
D1          0.25000000 sec
D11         0.03000000 sec
D16         0.00020000 sec
d17         0.00019000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec
P2             33.10 usec

====== CHANNEL f1 ======
NUC1             13C
P1             16.55 usec
P11           500.00 usec
P12          2000.00 usec
PL0           120.00 dB
PL1            -1.00 dB
SFO1     125.7942548 MHz
SP1            2.70 dB
SP2            2.70 dB
SPNAM1    Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1         0.00 Hz
SPOFF2         0.00 Hz

======= CHANNEL f2 =======
CPDPRG2      waltz16
NUC2             1H
PCPD2       100.00 usec
PL2            1.60 dB
PL12          24.50 dB
SFO2     500.2229911 MHz

===== GRADIENT CHANNEL =====
GPNAM1       SINE.100
GPNAM2       SINE.100
GPX1             0.00 %
GPX2             0.00 %
GPY1             0.00 %
GPY2             0.00 %
GPZ1            30.00 %
GPZ2            50.00 %
p15           5000.00 usec
p16           1000.00 usec

F2 - Processing parameters
SI            65536
SF       125.7803949 MHz
WDW              no
SSB               0
LB             0.00 Hz
GB                0
PC             2.00

1D NMR plot parameters
CX            22.80 cm
CY            15.65 cm
F1P          180.000 ppm
F1         22640.47 Hz
F2P          -10.000 ppm
F2         -1257.80 Hz
PPMCM         8.33333 ppm/cm
HZCM      1048.17084 Hz/cm
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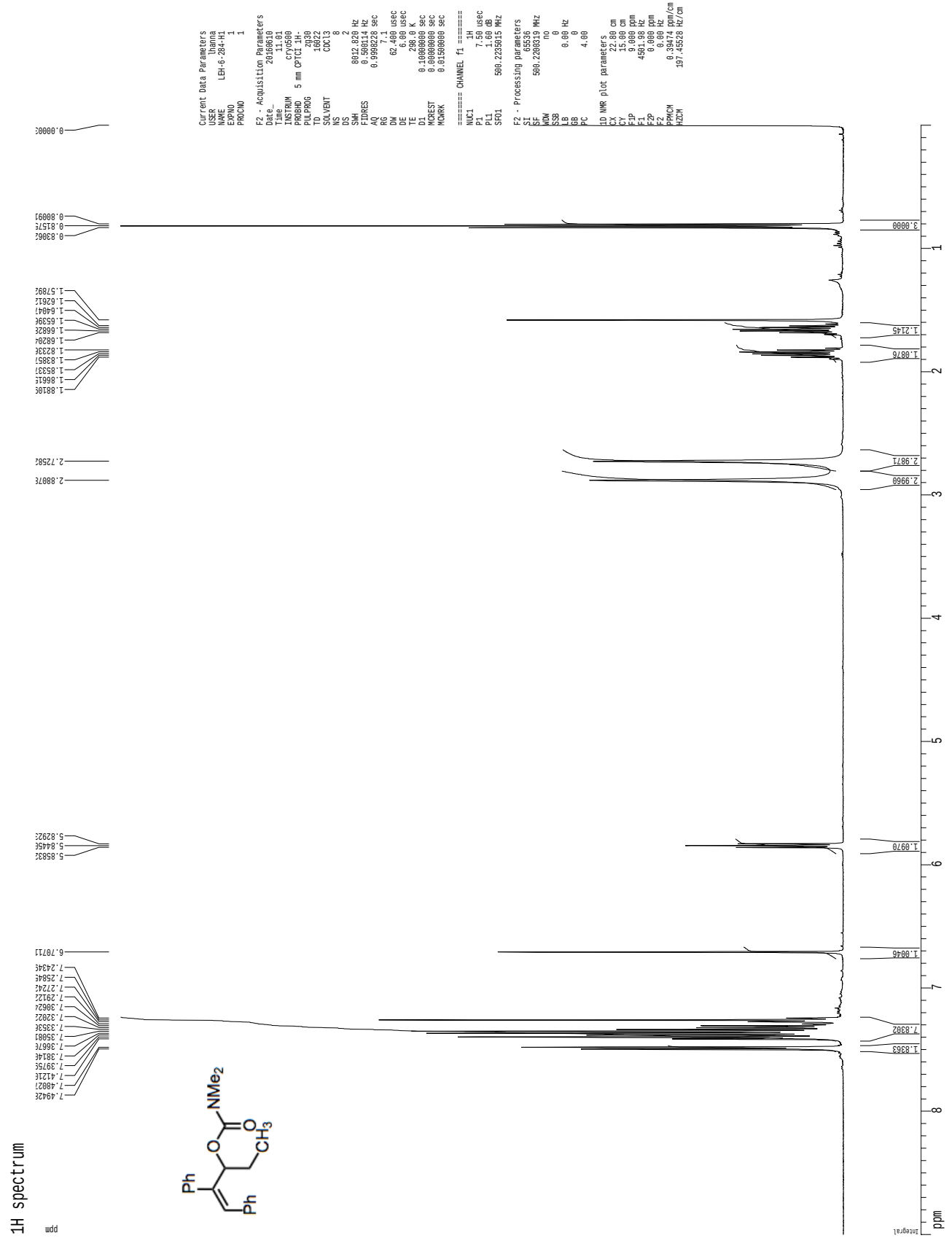
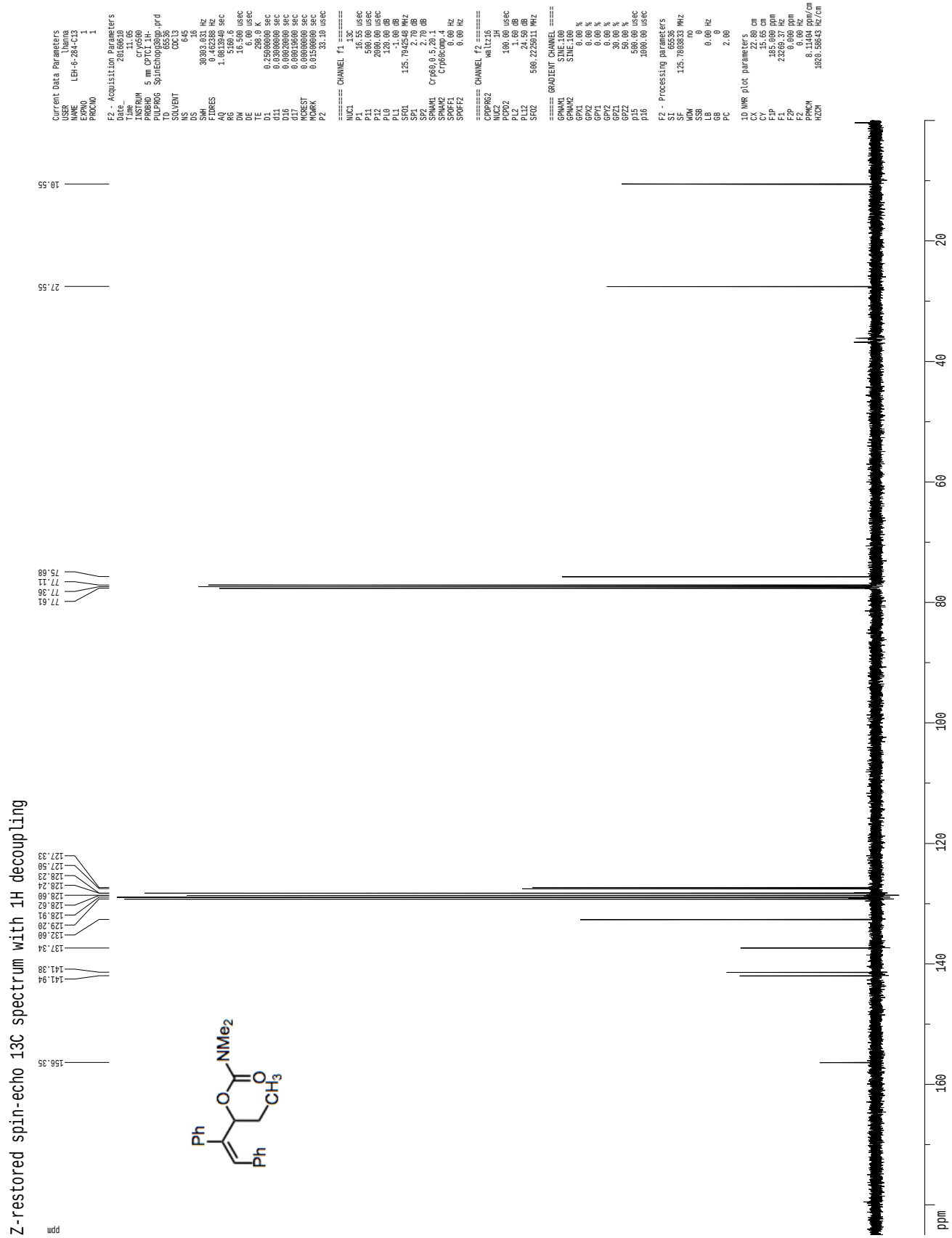
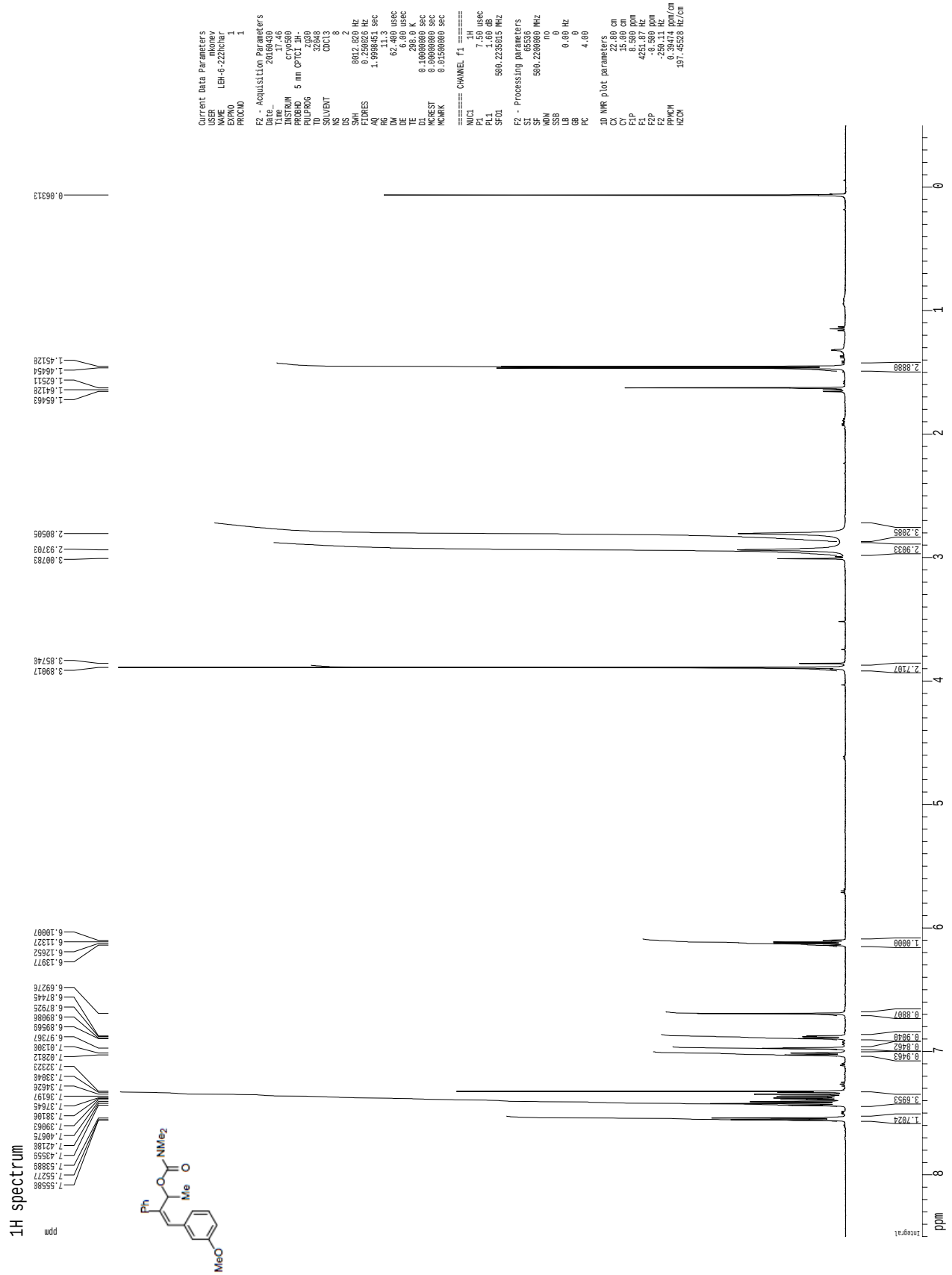
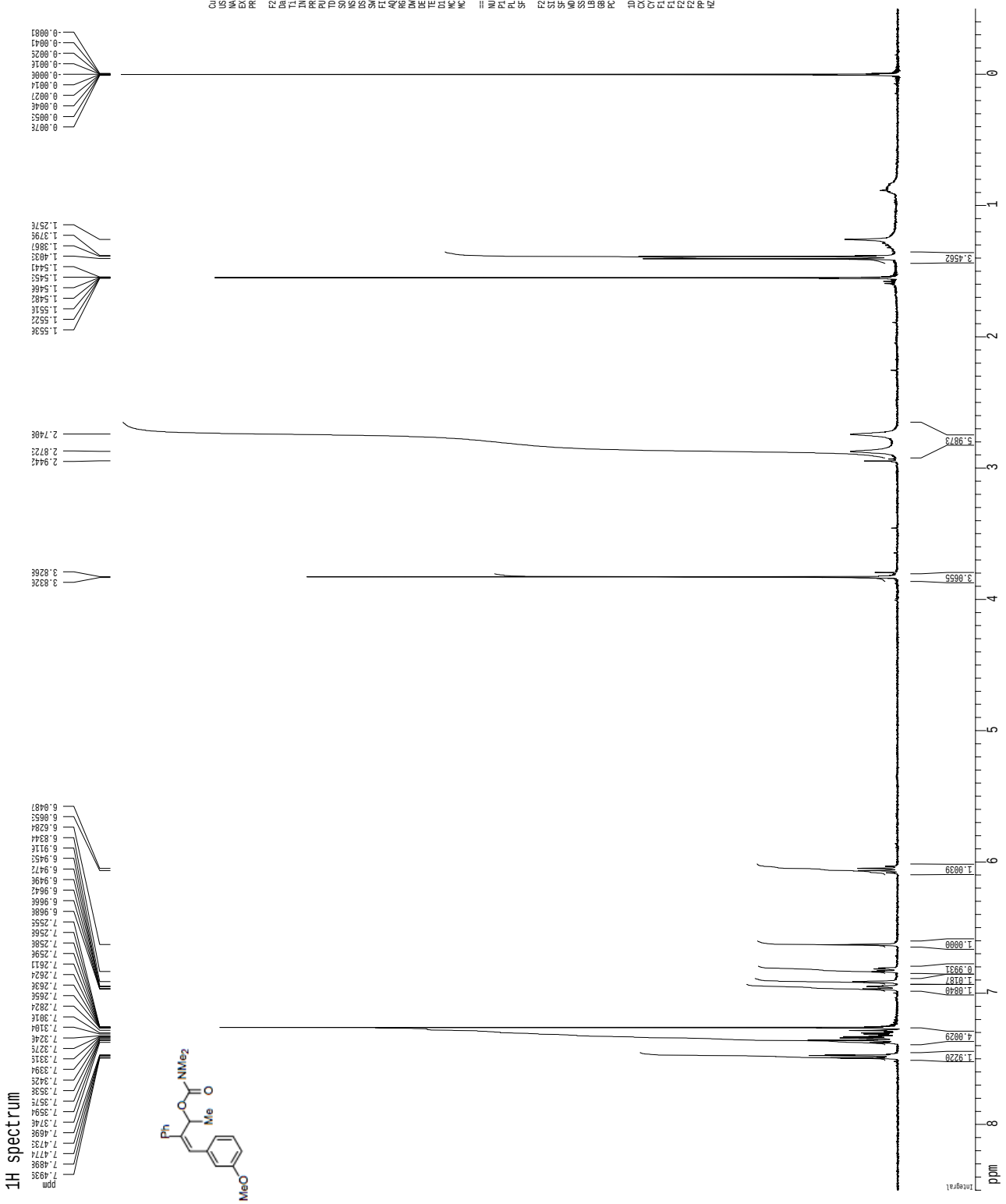
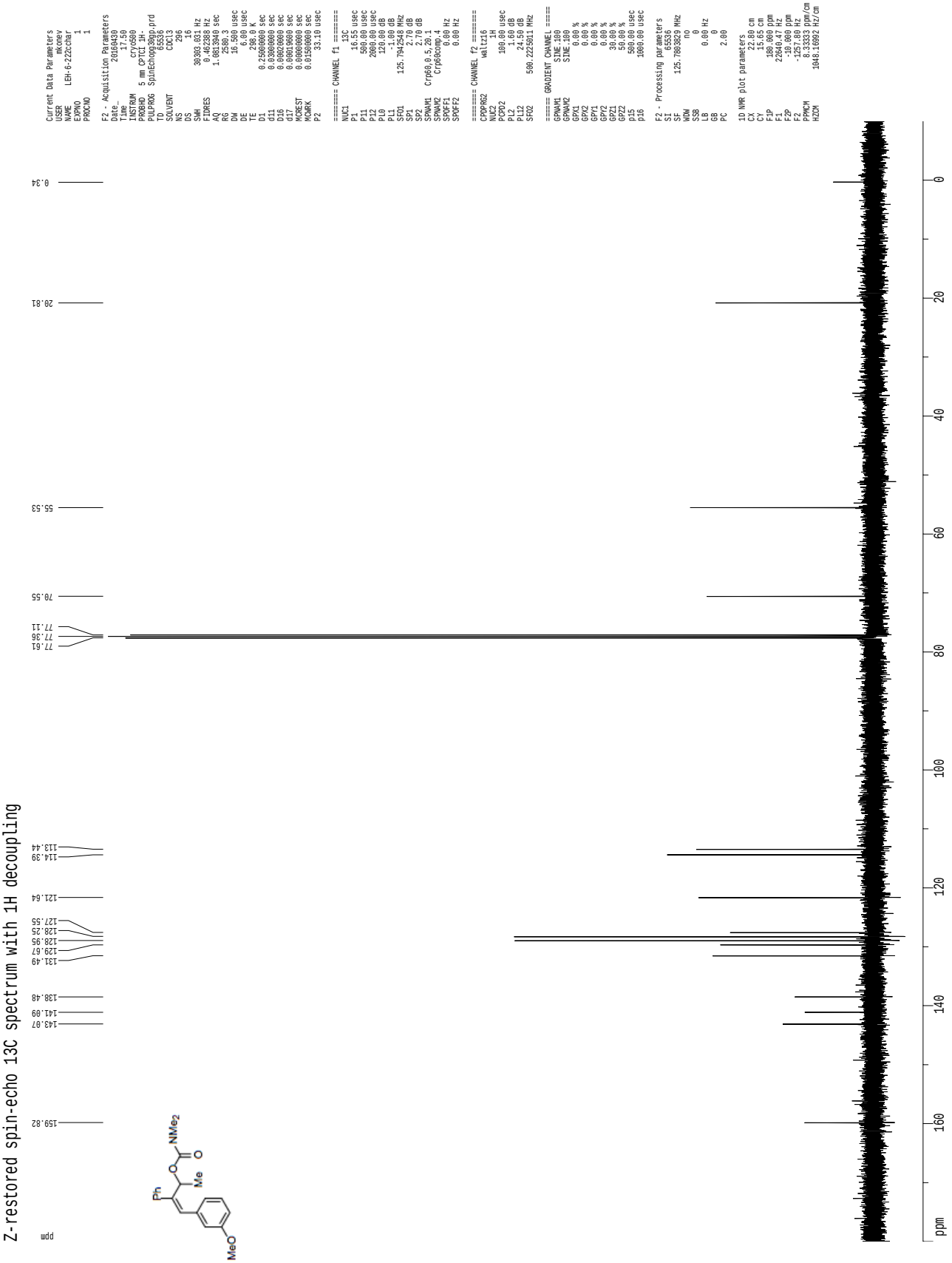

1H spectrum
Ph
NMe2
O
O
CH3
Ph
ppm

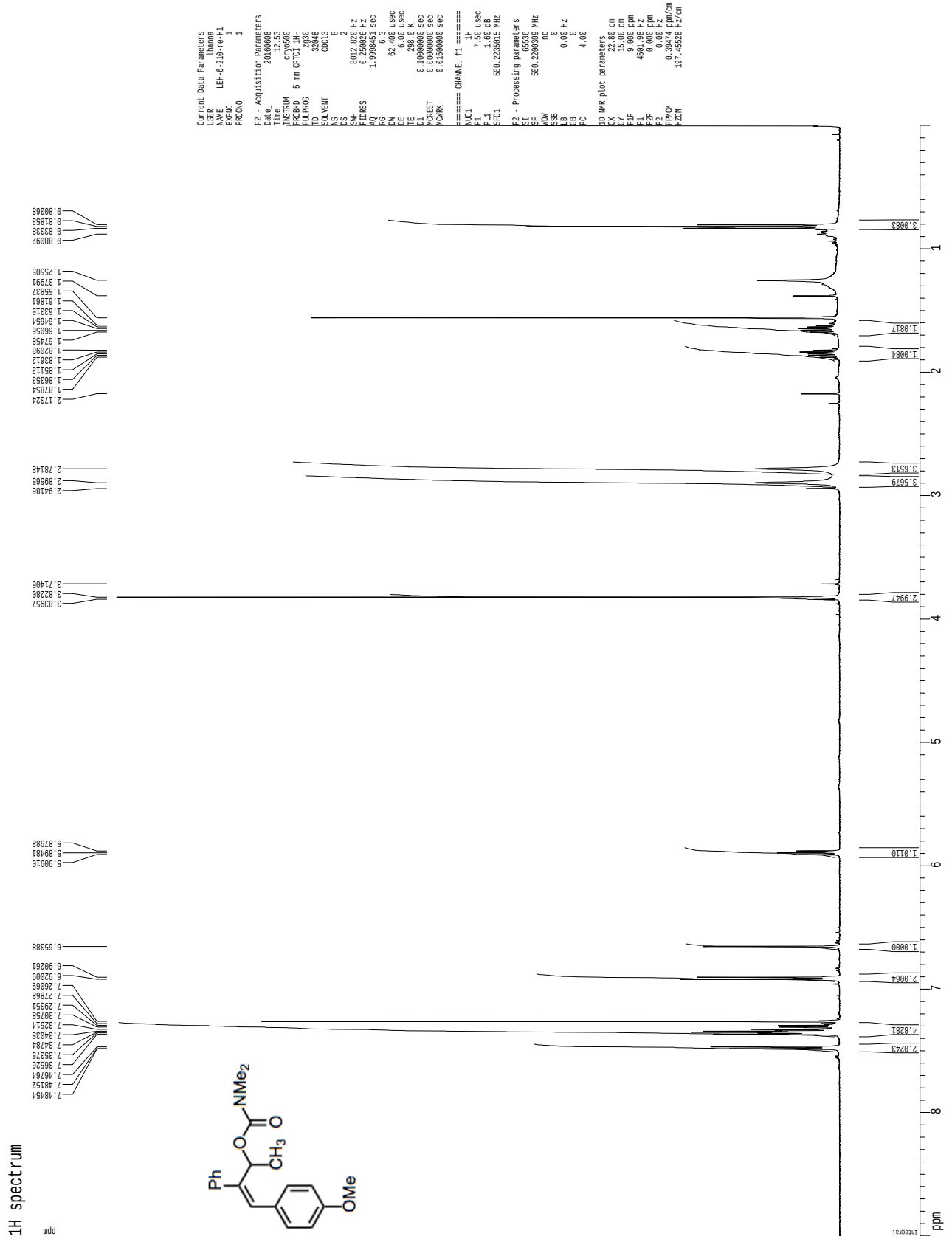

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
NAME LEH-6-284-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160610
Time 11.05
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 640
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803833 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 185.000 ppm
F1 23260.37 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.11494 ppm/cm
HZCM 1020.58643 Hz/cm
10.55
27.55
77.61
77.36
77.11
75.68
156.35
141.94
141.38
137.34
132.60
129.20
128.91
128.62
128.60
128.24
128.23
127.50
127.33
Ph
O
NMe2
O
CH3
Ph
20
40
60
80
100
120
140
160

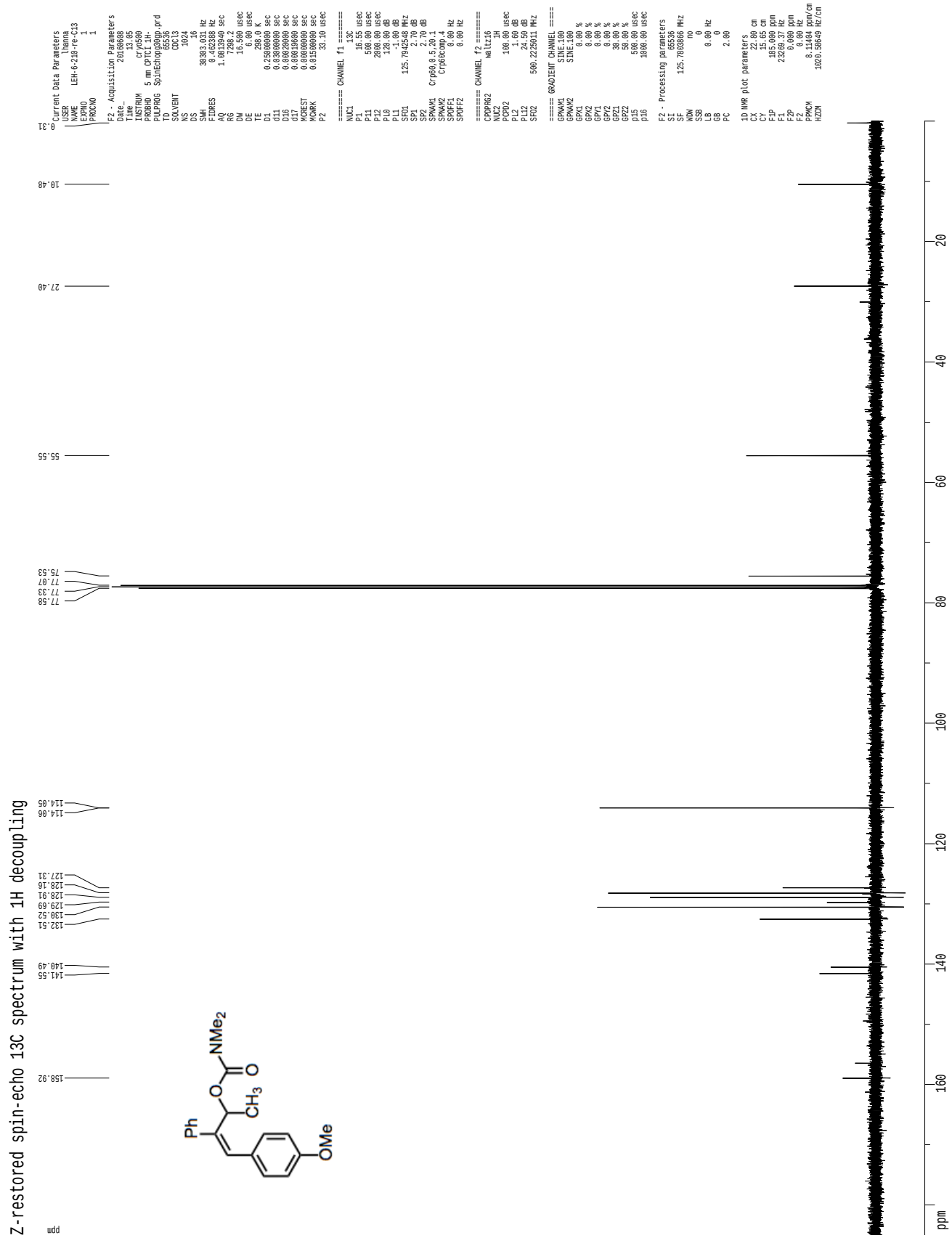

1H spectrum
ppm
Integral
Ph
NMe2
O
Me
MeO


1H spectrum
Current Data Parameters
USER Thanna
NAME LEH-6-222-c1-f-16
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time_ 13.27
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 0
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 1149.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.09000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300213 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.11 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.35774 ppm/cm
HZCM 157.94608 Hz/cm
0.0081
0.0041
0.0025
0.0016
0.0000
0.0014
0.0027
0.0046
0.0053
0.0076
1.2576
1.3796
1.3867
1.4033
1.5441
1.5453
1.5466
1.5482
1.5516
1.5522
1.5536
2.7406
2.8723
2.9442
3.8236
6.0487
6.0653
6.0804
6.2824
6.8344
6.9116
6.9453
6.9472
6.9496
6.9642
6.9666
6.9686
7.2556
7.2566
7.2586
7.2596
7.2613
7.2624
7.2636
7.2656
7.2824
7.3016
7.3104
7.3246
7.3275
7.3516
7.3924
7.3926
7.3576
7.3636
7.3594
7.3746
7.4696
7.4733
7.4774
7.4896
7.4936
3.4562
3.8655
5.9873
1.0039
1.0000
0.9931
1.0187
1.0840
4.0029
1.9220
Integral
ppm
Ph
Me
O
NMe2
MeO

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER Thanna
NAME LEH-6-210-re-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160608
Time 12.53
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200389 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 27.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
0.80366
0.81853
0.83336
0.88092
1.25506
1.57991
1.56857
1.61861
1.63316
1.64654
1.66056
1.67456
1.82096
1.83612
1.85113
1.86352
1.87854
2.17324
2.78148
2.89566
2.94186
3.71406
3.82864
3.83957
5.87986
5.89483
5.90916
6.65386
6.90261
6.92065
7.26066
7.27866
7.29351
7.30756
7.32514
7.34835
7.34784
7.35375
7.36526
7.46764
7.48152
7.48454
3.0083
1.0817
1.0084
3.6513
3.5679
2.9947
1.0110
1.0000
2.0064
4.8281
2.0243
Integral
ppm
Ph
NMe2
O
O
CH3
OMe


Z-restored spin-echo 13C spectrum with 1H decoupling
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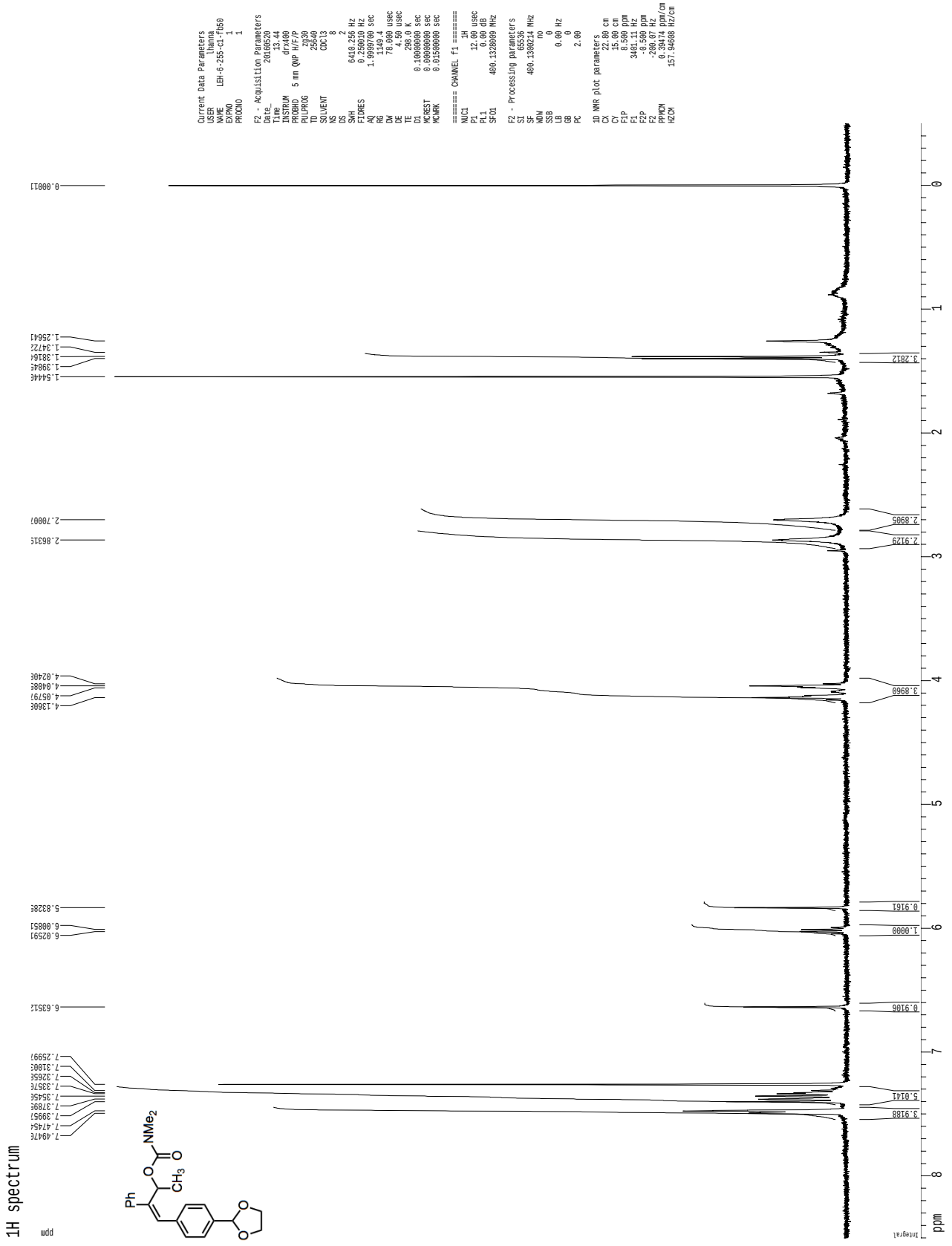
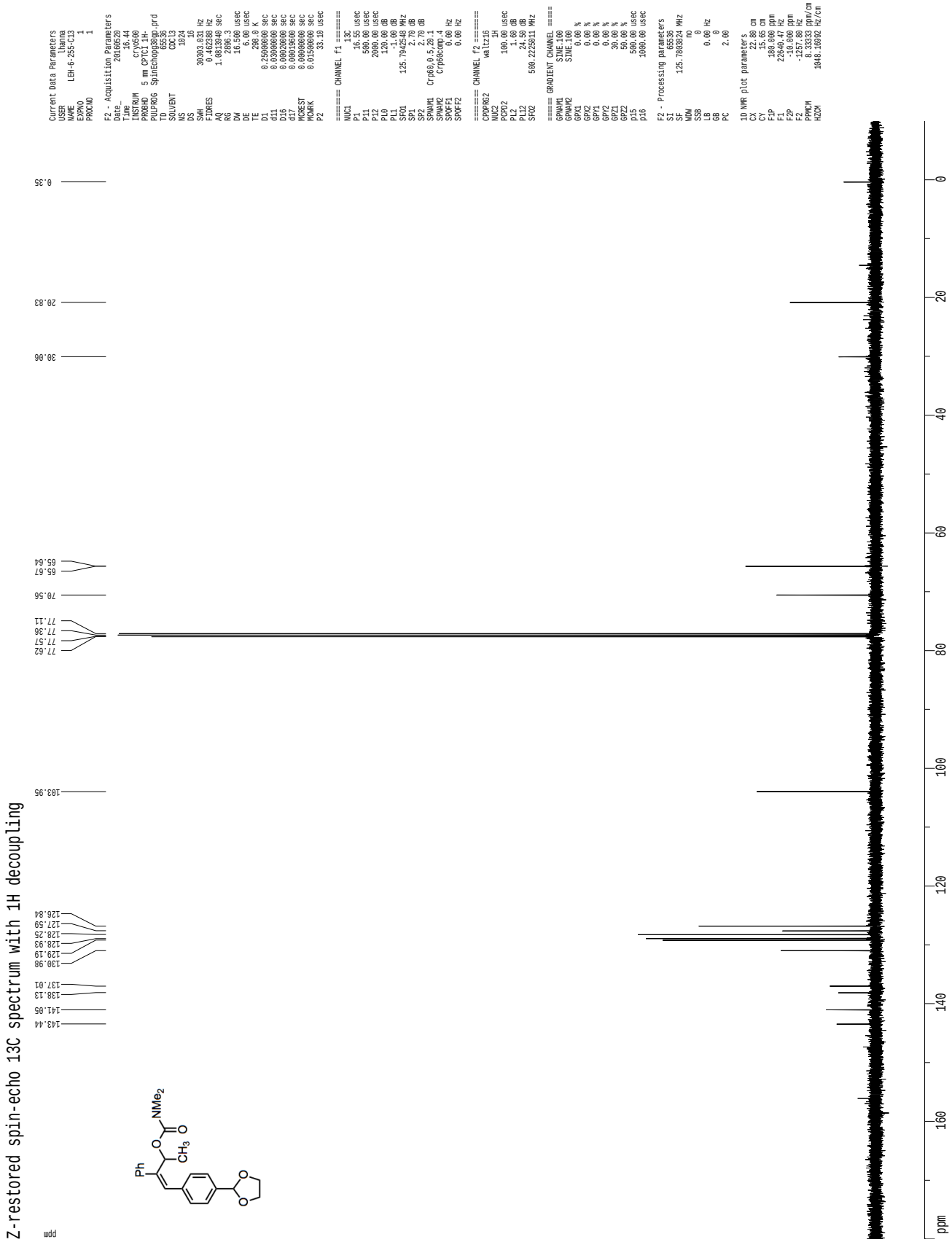
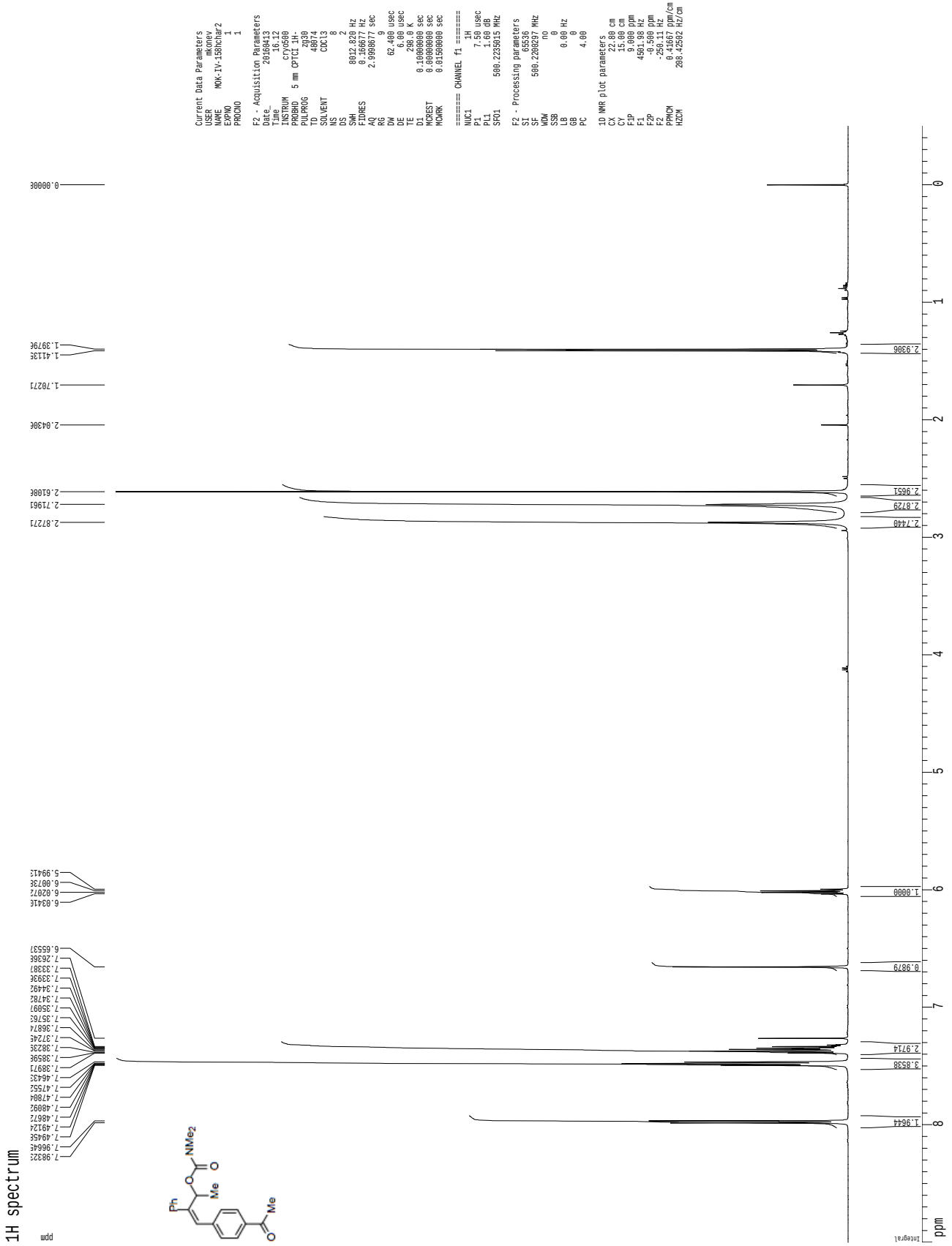
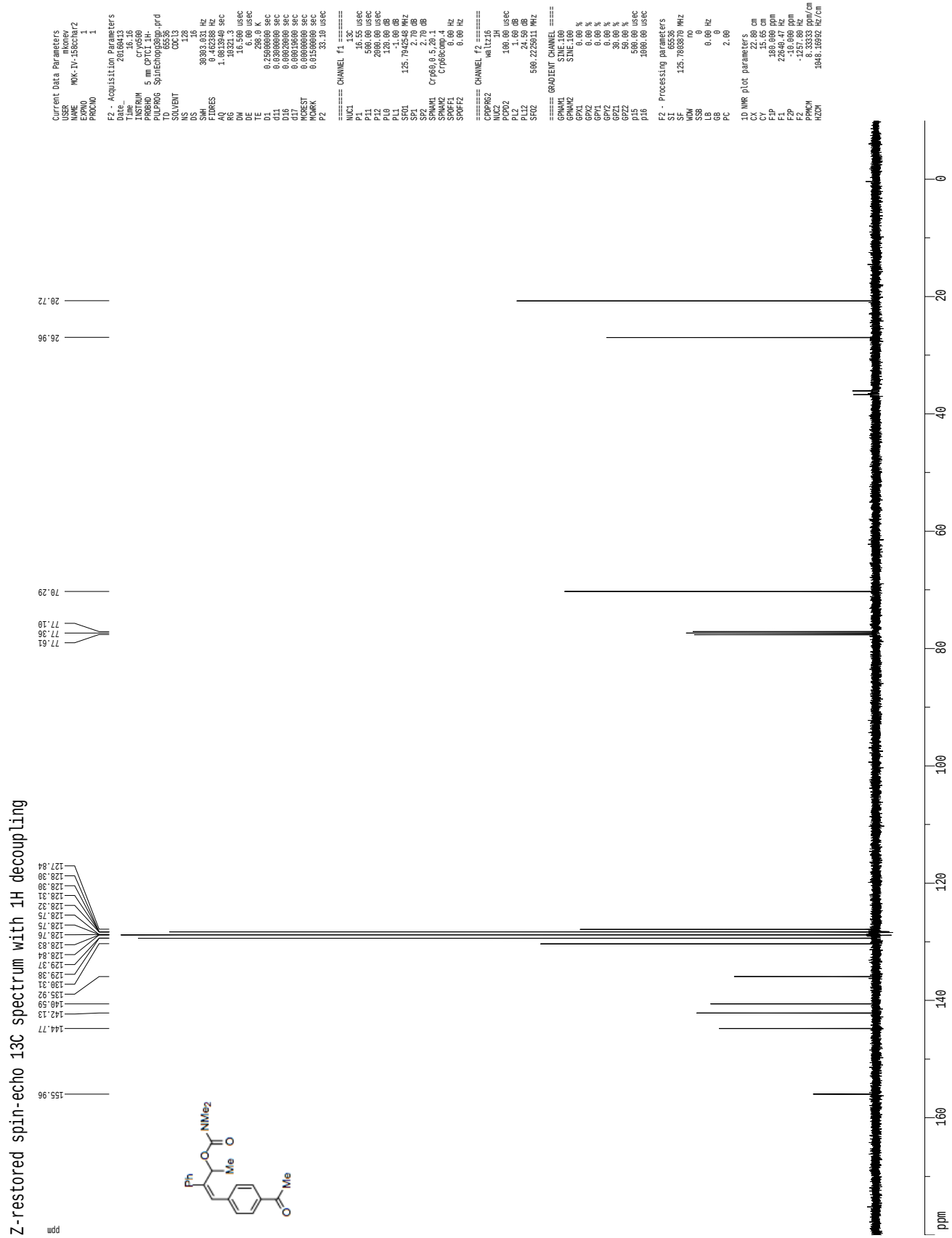
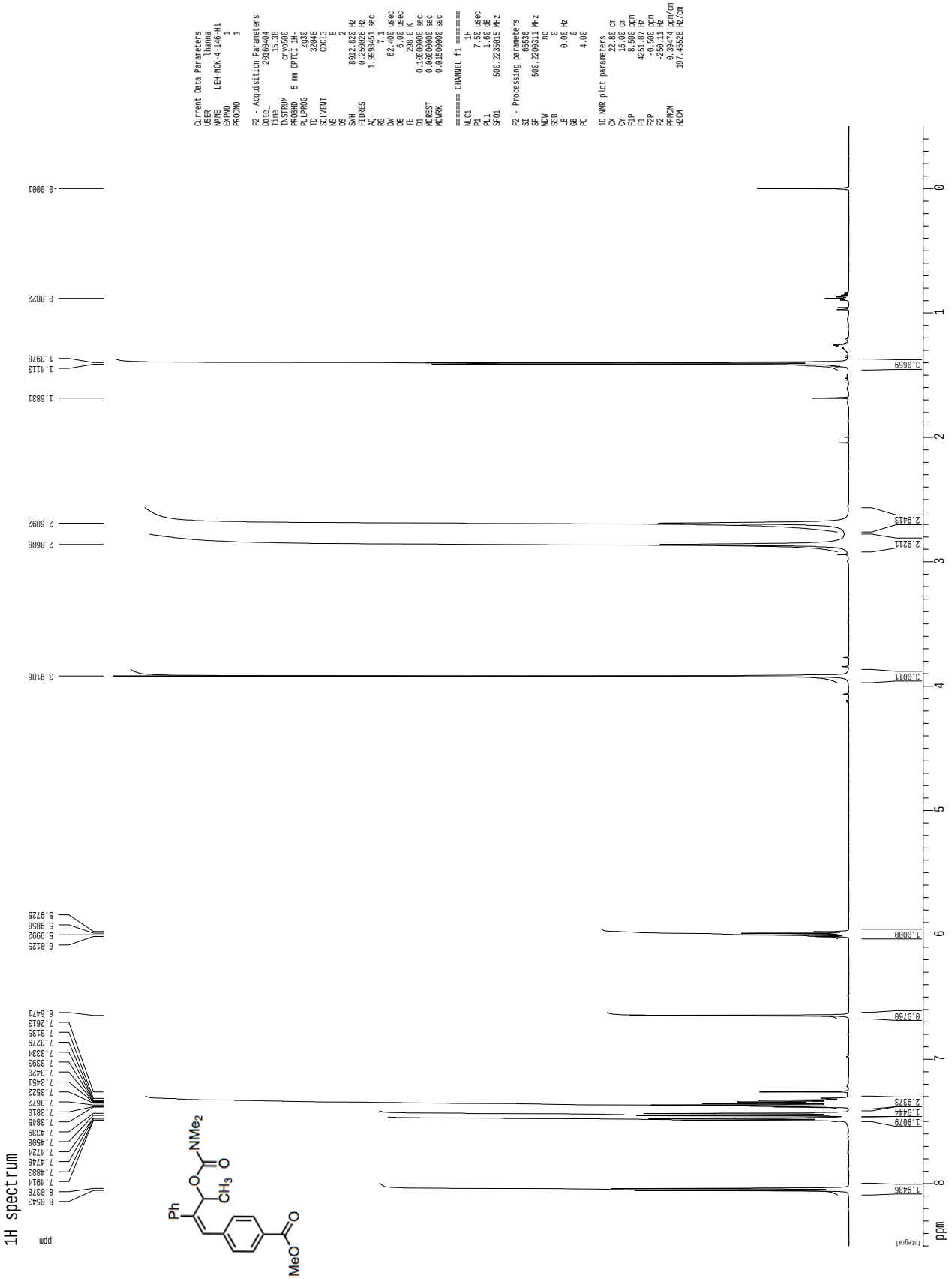
158.92
141.55
140.49
132.51
130.52
129.69
128.91
128.16
127.31
114.06
114.05
77.58
77.33
77.07
75.53
55.55
27.40
10.48
Ph
O
NMe2
CH3
O
OMe


1H spectrum
ppm
integral
Ph
NMe2
O
CH3
O
O
O


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
NMe2
O
CH3
O
O
O
143.44
141.05
138.13
137.01
130.98
129.19
128.93
128.25
127.59
126.84
103.95
77.62
77.57
77.36
77.11
70.56
65.67
65.64
30.06
20.83
0.35
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER mkoney
NAME MOK-IV-158hchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160413
Time 16.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 40074
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.199677 Hz
AQ 2.9998677 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200297 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5001.96 Hz
F2P -0.560 ppm
F2 -280.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00000
1.39796
1.41136
1.70271
2.04306
2.61086
2.71961
2.87271
5.99413
6.03416
6.02072
6.00736
6.65537
7.26366
7.33387
7.33936
7.34492
7.34782
7.35097
7.35763
7.36874
7.37245
7.38236
7.38598
7.38973
7.46435
7.47552
7.47864
7.48092
7.48672
7.49124
7.49456
7.96646
7.98325
2.9306
2.9651
2.8729
2.7448
1.0000
0.9879
2.9714
3.8538
1.9644
Integral
ppm
Ph
NMe2
O
O
Me
O
Me
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
NMe2
O
Me
O
Me
O
20.72
26.96
70.29
77.10
77.36
77.61
127.84
128.30
128.30
128.31
128.32
128.75
128.75
128.76
128.83
128.84
129.37
129.38
130.31
135.92
140.59
142.13
144.77
155.96
Current Data Parameters
USER mkonev
NAME MOK-IV-156ether2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160413
Time 16.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoDig30gp.prd
TD 65536
SOLVENT CDCl3
NS 128
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803870 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
NAME LEH-MOK-4-146-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time_ 15.38
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9990451 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.08000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SF01 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200311 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.87 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
8.0543
8.0378
7.4914
7.4883
7.4748
7.4724
7.4508
7.4335
7.3846
7.3816
7.3672
7.3522
7.3451
7.3426
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7.3362
7.3334
7.3275
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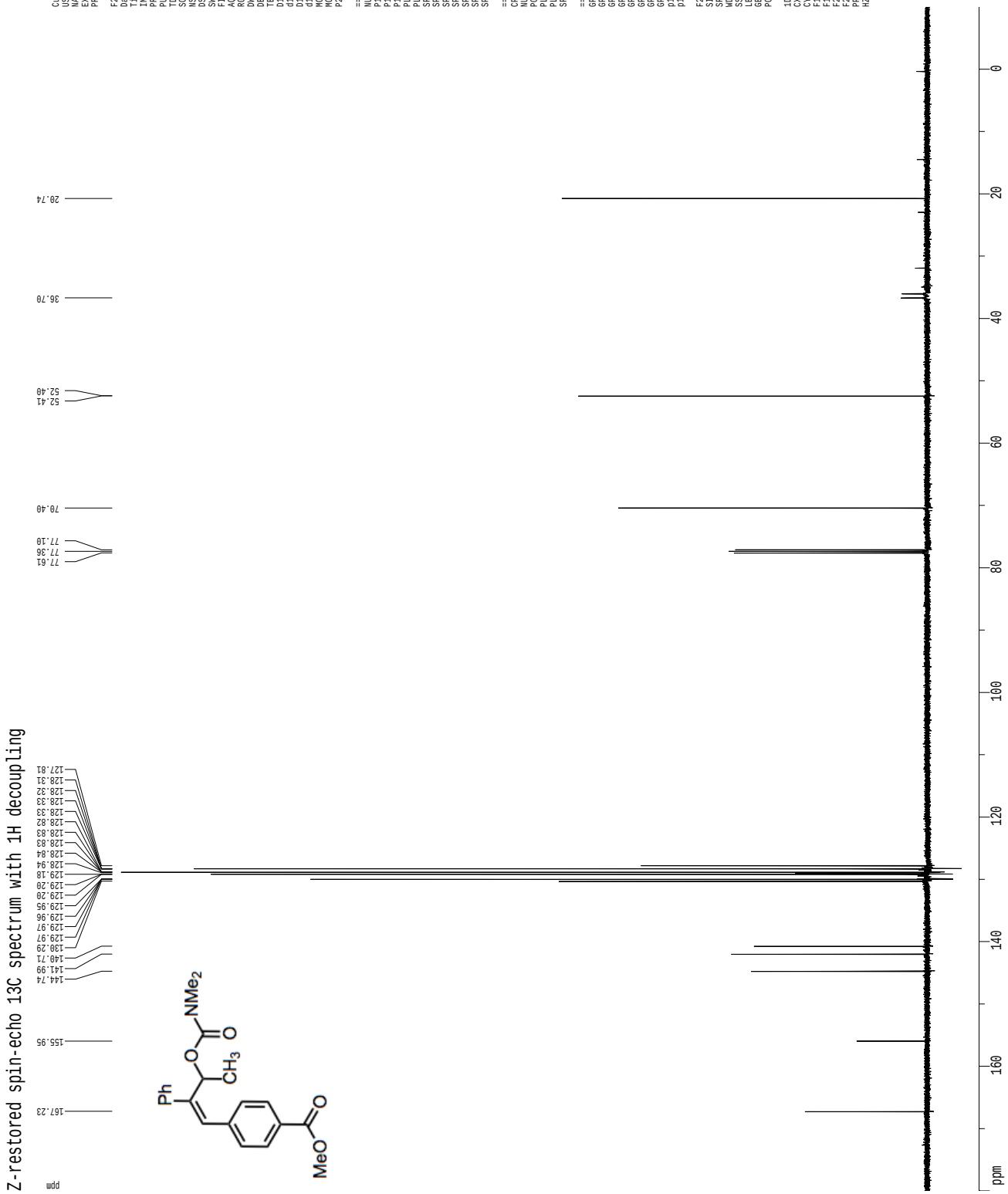
5.9992
5.9856
5.9725
3.9186
2.8808
2.8692
1.6833
1.4113
1.3976
0.8822
0.0001
1.9436
1.9444
1.9879
2.9373
0.9760
1.0000
3.0011
2.9211
2.9413
3.0659
integral
ppm
Ph
NMe2
O
O
CH3
MeO
O


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
NMe2
O
CH3
O
MeO
O
Current Data Parameters
USER Ilanna
NAME LEH-MOK-4-146-c13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160404
Time 15.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 336
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 15.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803866 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
20.74
36.70
52.41
52.40
70.40
77.61
77.36
77.10
127.81
128.31
128.32
128.33
128.33
128.82
128.83
128.83
128.84
128.94
129.18
129.20
129.20
129.95
129.96
129.97
129.97
130.29
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144.74
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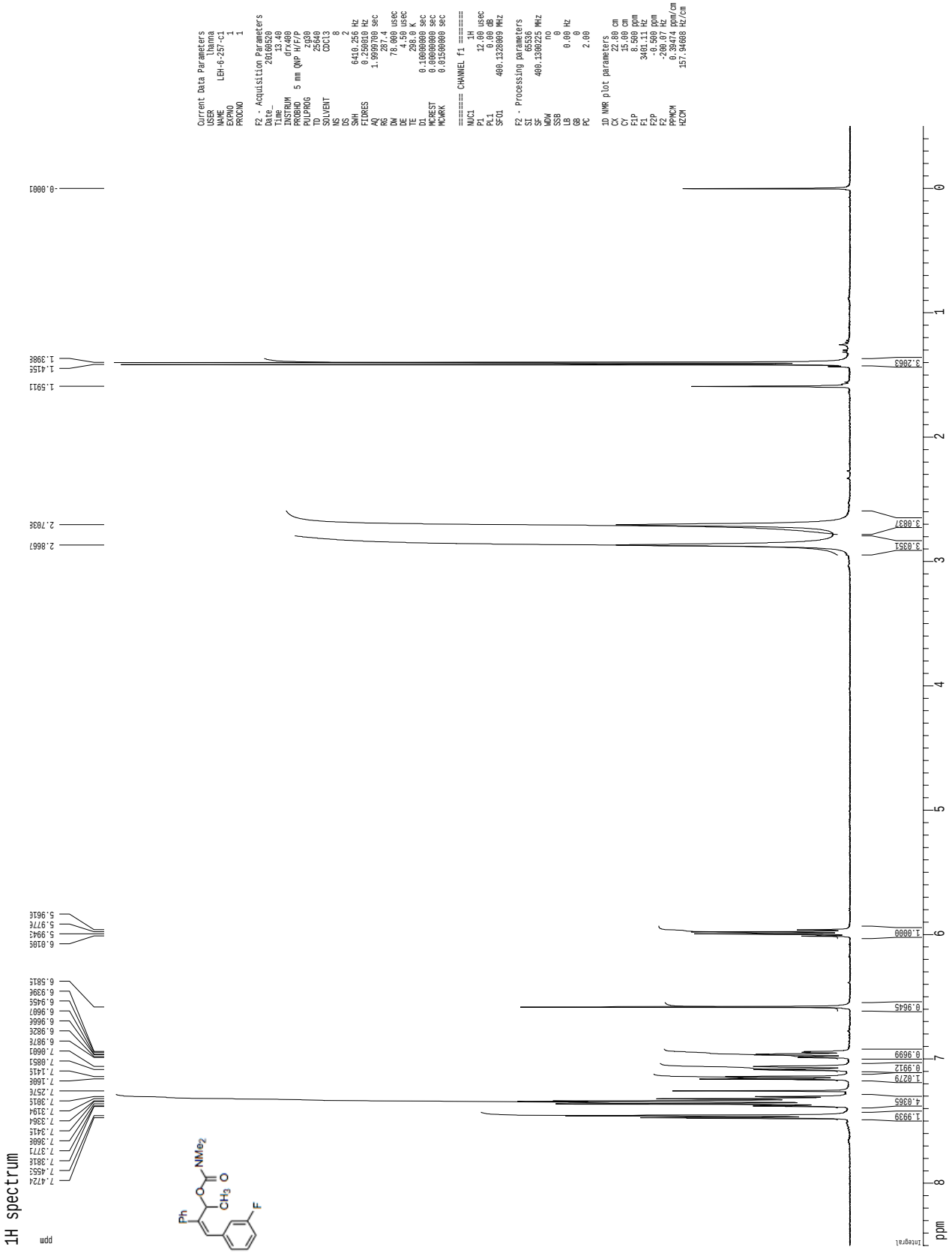
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-6-257-c1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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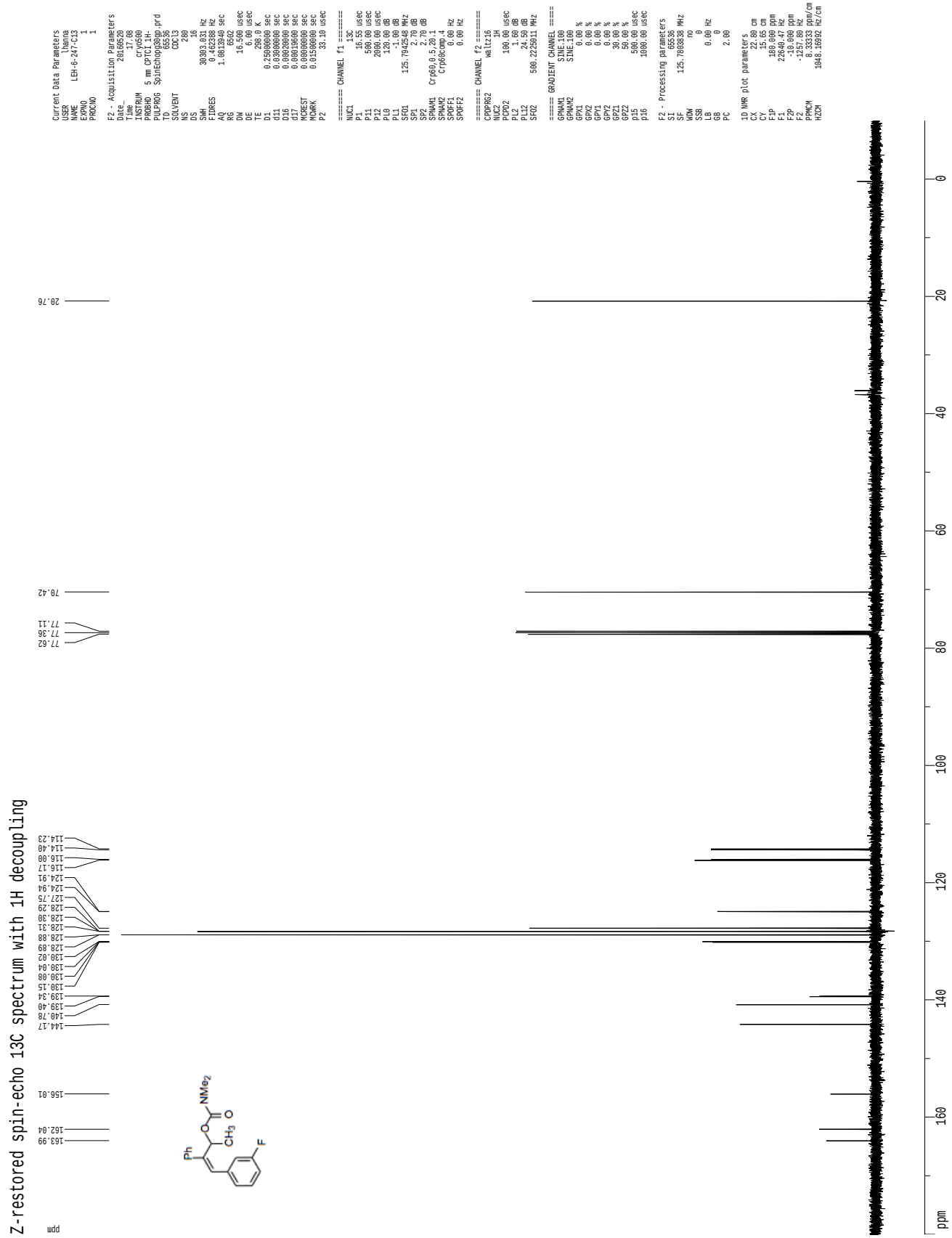
Time 13.40
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999709 sec
RG 287.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300225 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.11 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94608 Hz/cm
ppm
Integral
Ph
NMe2
O
O
CH3
F


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
NMe2
O
O
CH3
O
F
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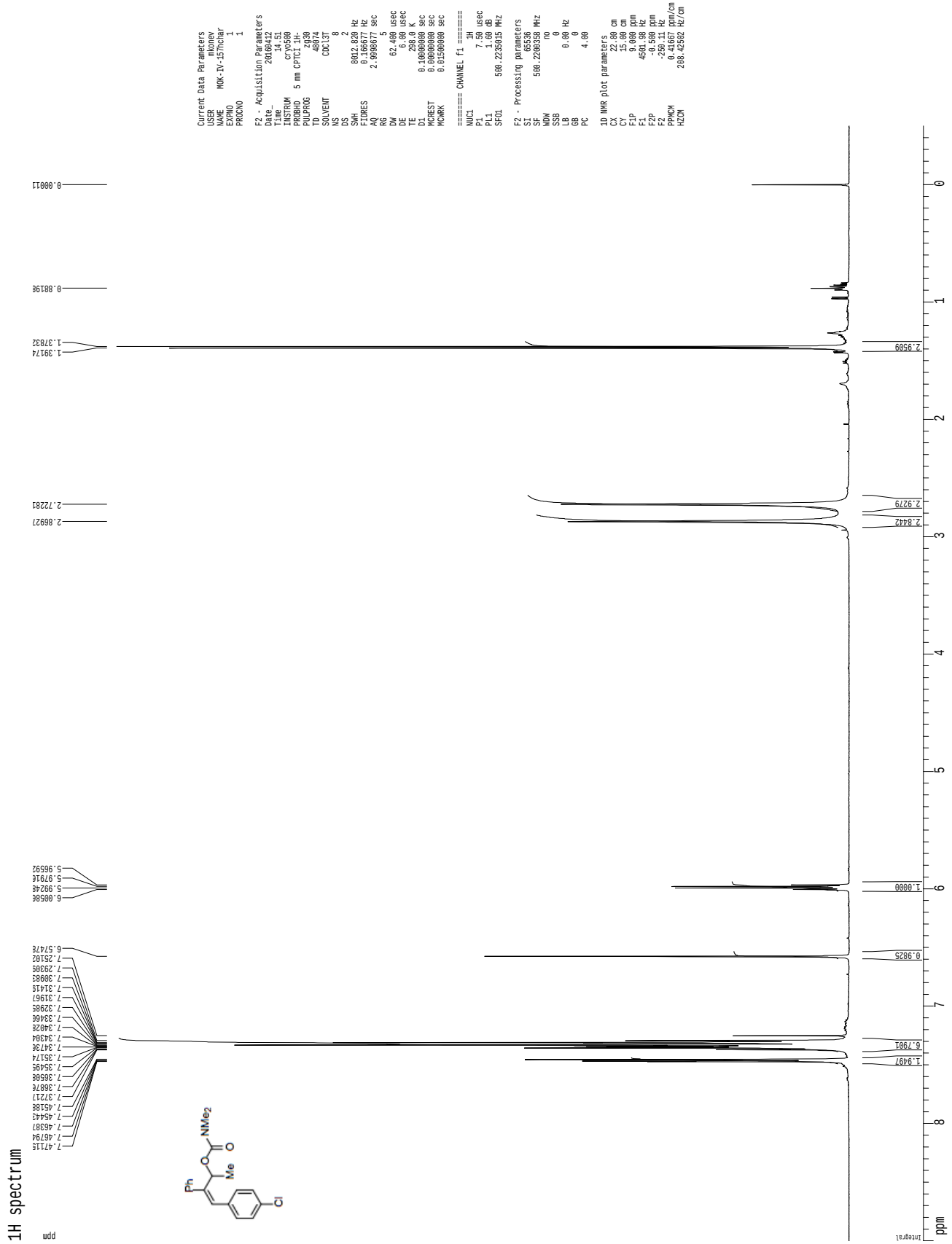
70.42
77.62
77.36
77.11
114.23
114.40
116.00
116.17
124.91
124.94
127.75
128.29
128.30
128.31
128.88
128.89
130.02
130.04
130.08
130.15
139.34
139.40
140.78
144.17
156.01
162.04
163.99
Current Data Parameters
USER tbanna
NAME LEH-6-247-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160520
Time 17.08
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoPg30gp.prd
TD 65536
SOLVENT CDCl3
NS 240
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 6502
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883838 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1040.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-157hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160412
Time 14.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 40974
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.196677 Hz
AQ 2.9998677 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200358 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.696 ppm
F2 -298.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00011
0.88198
1.37832
1.39174
2.72281
2.86927
2.95096
2.92796
2.84426
5.96592
5.97916
5.99248
6.00586
1.00000
6.57478
7.25102
7.29305
7.30983
7.31415
7.31967
7.32986
7.33466
7.34028
7.34304
7.34736
7.35174
7.35495
7.36508
7.36876
7.37217
7.45188
7.45443
7.46387
7.46794
7.47115
0.98255
6.79015
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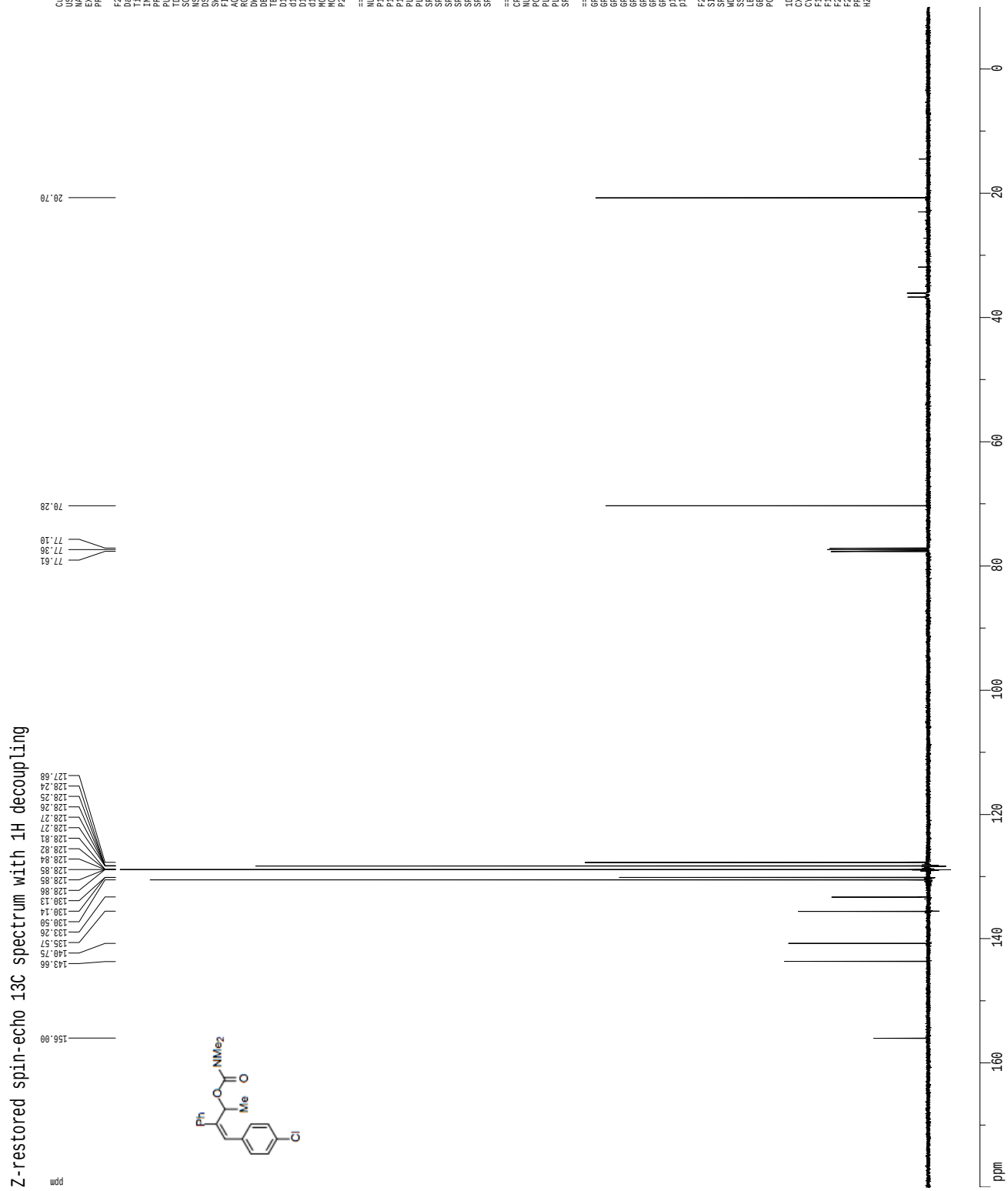
integral
ppm
NMe2
O
O
Me
Ph
Cl


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
O
Me
NMe2
Cl
Current Data Parameters
USER mkonev
NAME MOK-IV-157cther2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160412
Time 14.56
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
NS 240
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3251
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019060 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 165.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883889 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1040.10992 Hz/cm
156.00
143.66
140.75
135.57
133.26
130.50
130.14
130.13
128.86
128.85
128.85
128.84
128.82
128.81
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128.27
128.26
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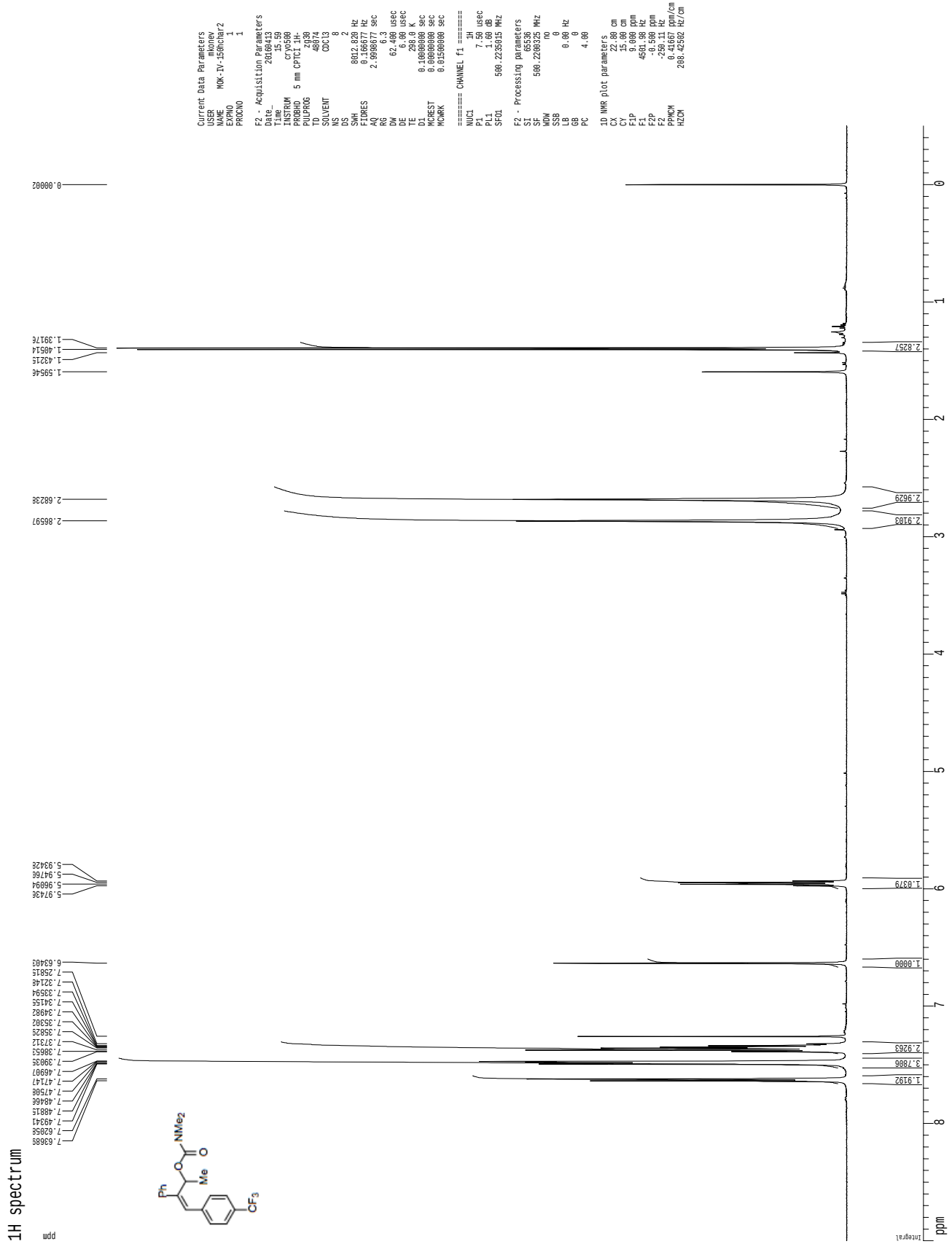
128.24
127.68
77.61
77.36
77.10
70.28
20.70
ppm


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-150rchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160413
Time 15.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 48074
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.166677 Hz
AQ 2.9998677 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200325 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.466 ppm
F2 -233.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.00002
1.39176
1.40514
1.43215
1.59546
2.68238
2.86597
5.97436
5.96094
5.94766
5.93428
6.63482
7.25815
7.32148
7.33594
7.34155
7.34982
7.35302
7.35829
7.37312
7.38653
7.39035
7.46907
7.47147
7.47508
7.48466
7.48815
7.49341
7.62058
7.63685
2.8257
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1.0000
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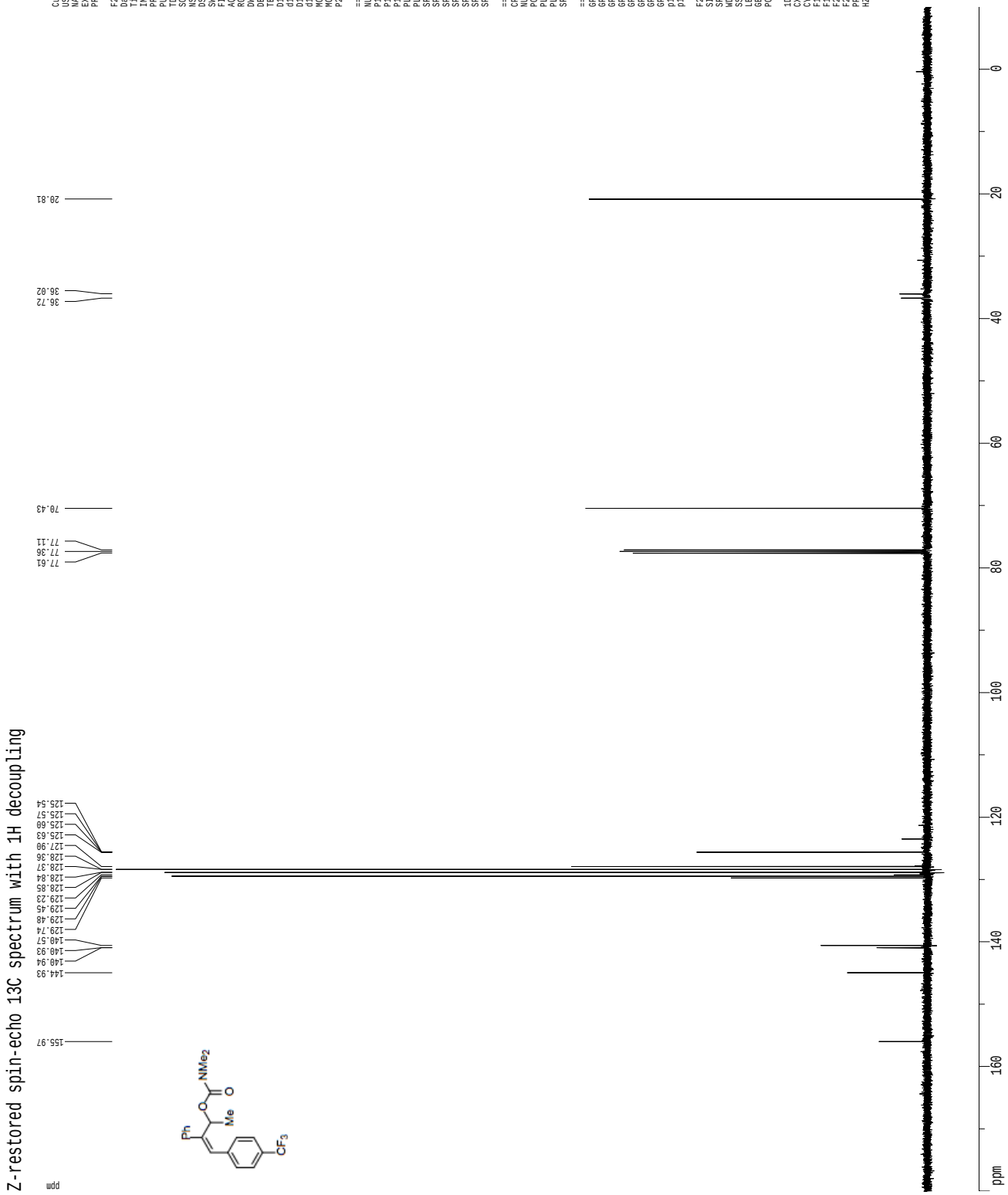
3.7806
1.9192
Integral
ppm
Ph
Me
NMe2
CF3


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
Me
O
NMe2
O
CF3
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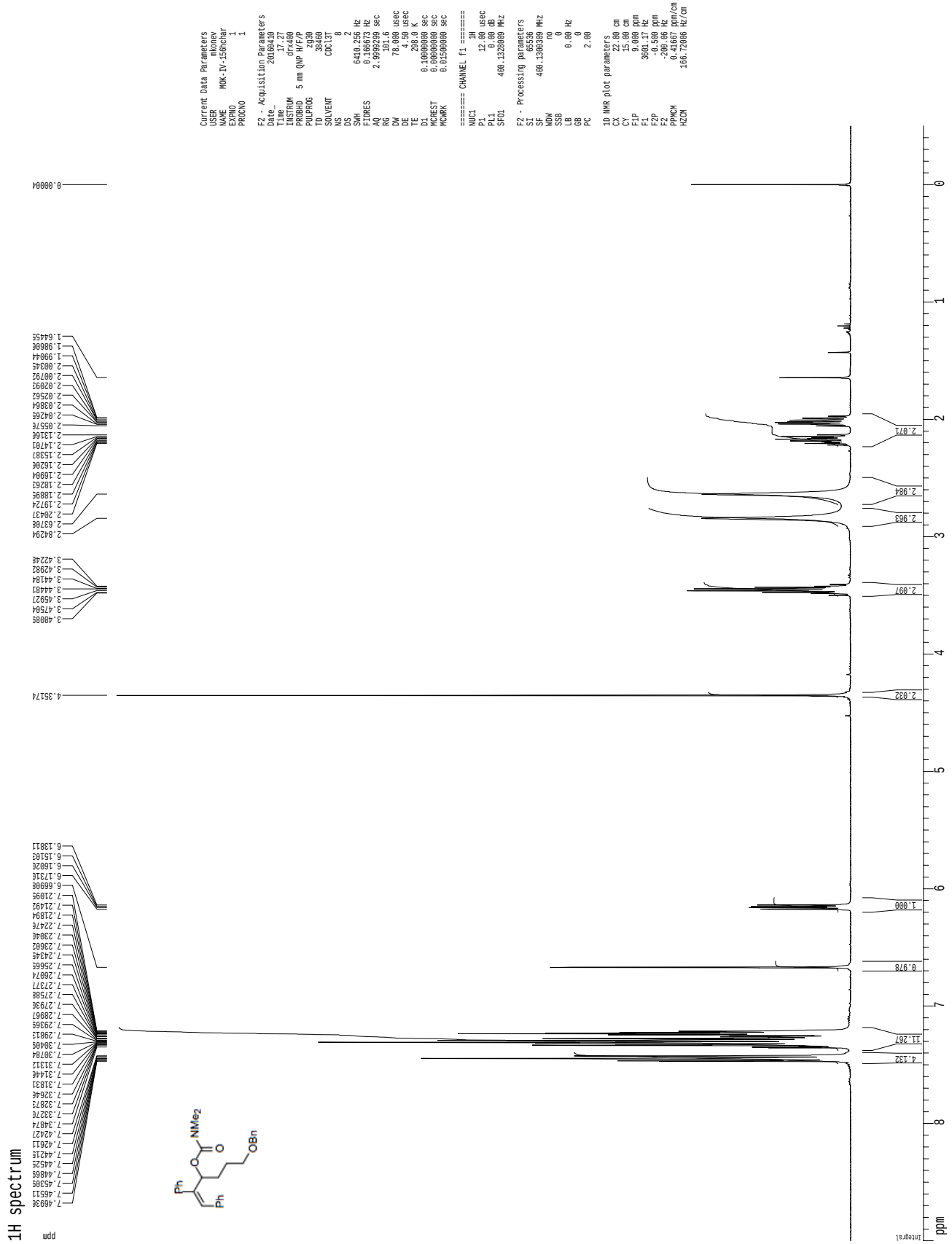
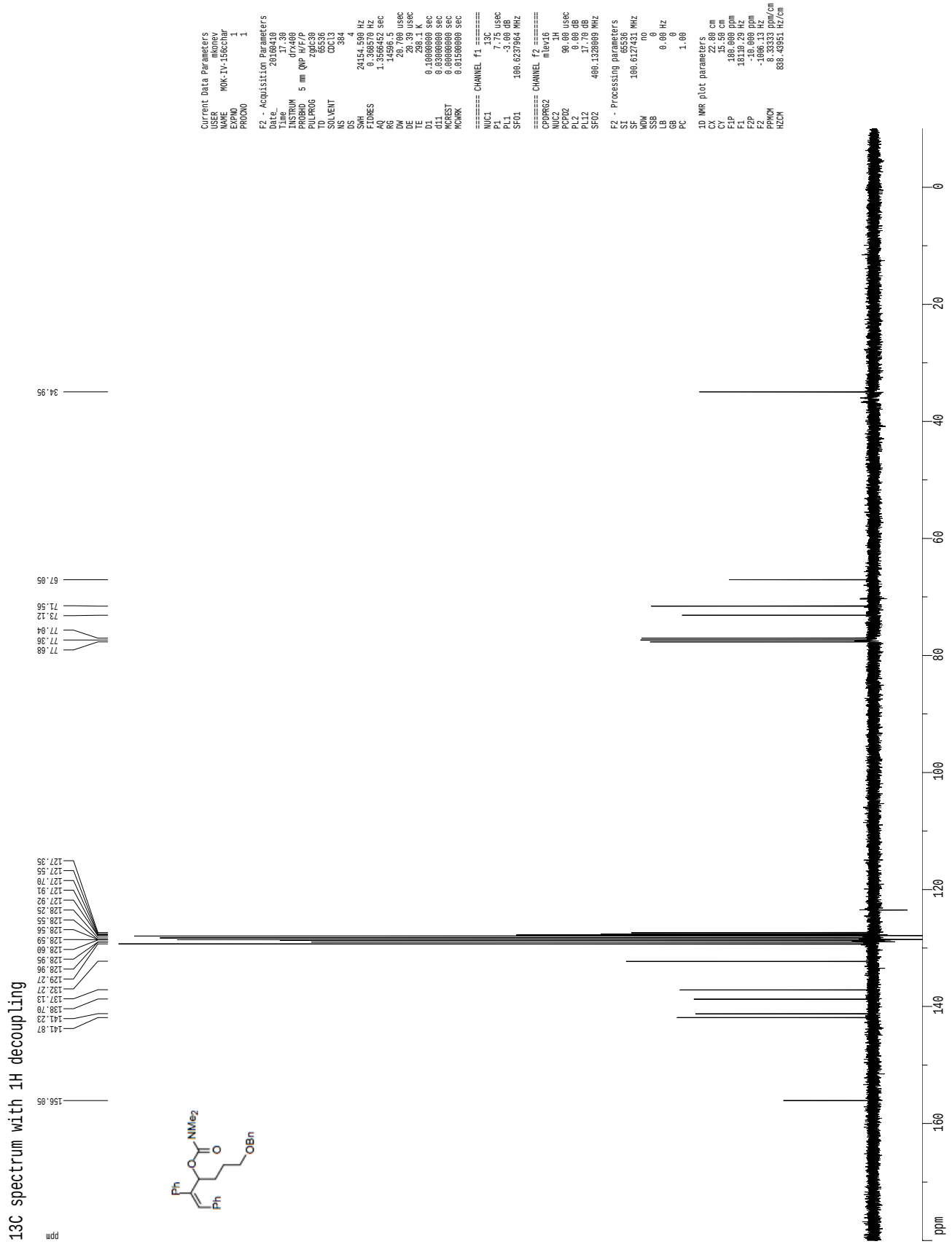
140.94
140.93
129.74
129.48
129.45
129.23
128.85
128.84
128.37
128.36
127.90
125.63
125.60
125.57
125.54
77.61
77.36
77.11
70.43
36.72
36.02
20.81
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER mickey
NAME MOK-IV-150/Char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160410
Time 17.27
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 38460
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.166573 Hz
AQ 2.9984176 sec
RG 101.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300300 MHz
WDW no
SSB 0.00 Hz
LB 0
GB 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.560 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72080 Hz/cm
NMe2
O
O
OBn
Ph
Ph
ppm
Integral


13C spectrum with 1H decoupling
Current Data Parameters
USER mkoney
NAME MOK-IV-156cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160410
Time 17.30
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 14596.5
DW 20.700 usec
DE 20.39 usec
TE 298.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.76 dB
SFO2 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 100.6127431 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 180.000 ppm
F1 18110.29 Hz
F2P -10.000 ppm
F2 -1806.13 Hz
PPMCM 8.33333 ppm/cm
HZCM 838.43951 Hz/cm
156.05
141.87
141.23
138.70
137.13
132.27
129.27
128.96
128.95
128.60
128.59
128.56
128.55
128.25
127.92
127.91
127.70
127.55
127.35
77.68
77.36
77.04
73.12
71.56
67.05
34.95
NMe2
O
O
OBn
Ph
Ph
ppm
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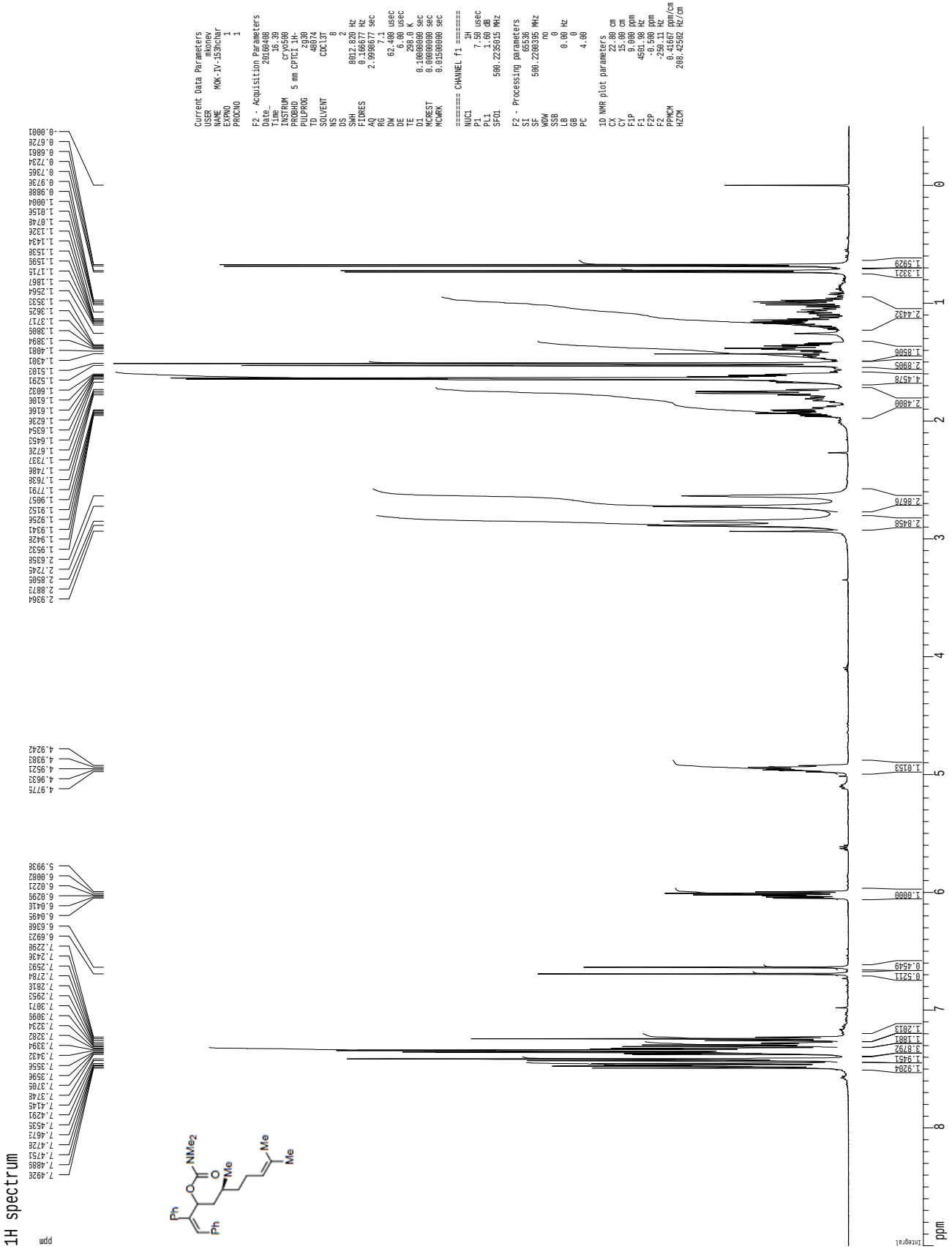
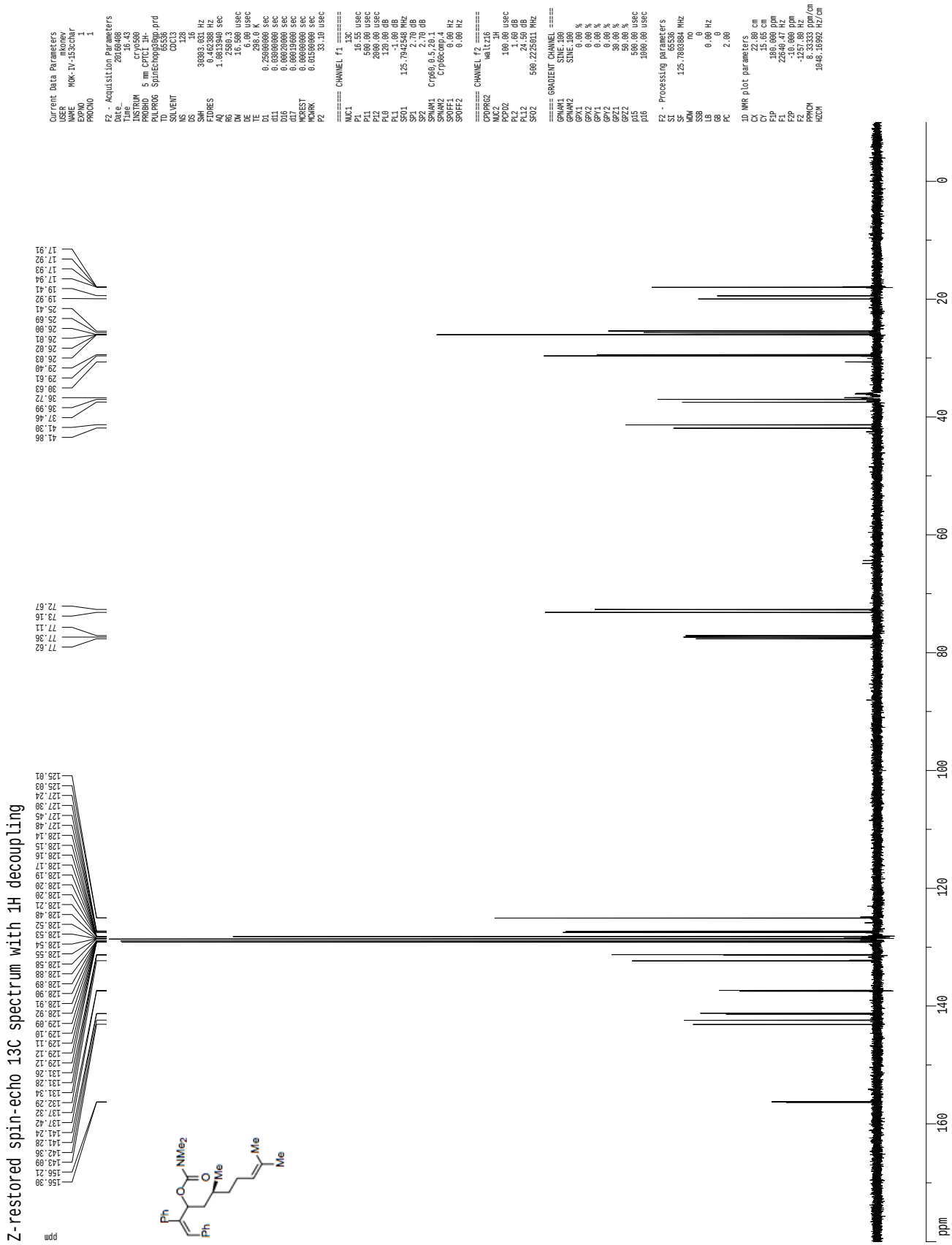
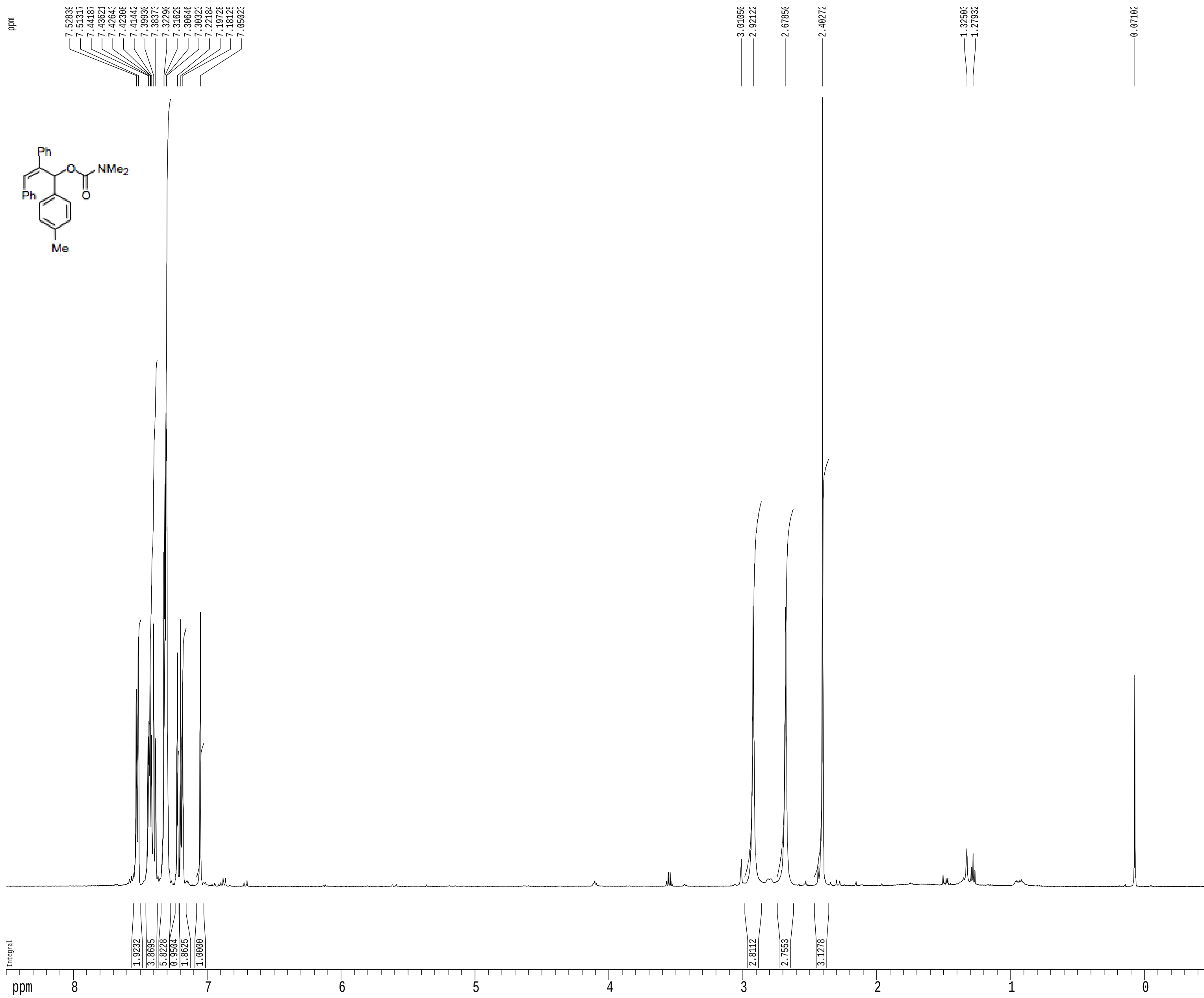
40
60
80
100
120
140
160


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-153nchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160408
Time 16.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 48074
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.166677 Hz
AQ 2.9998677 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200395 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.466 ppm
F2 -233.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
NMe2
O
O
Me
Me
Me
Ph
Ph
ppm
Integral


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
Ph
Ph
Me
Me
Me

1H spectrum

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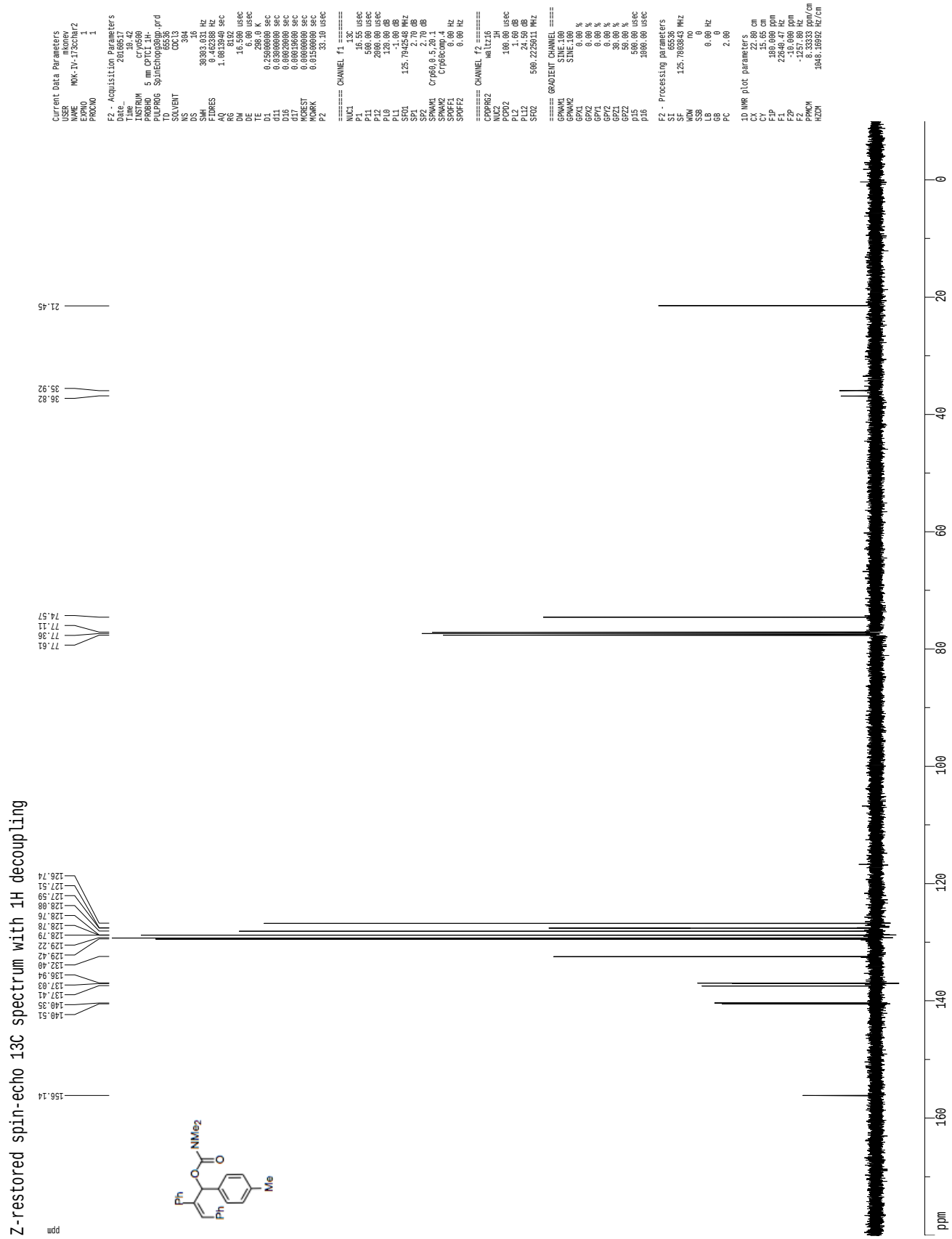
Current Data Parameters
USER              mkonev
NAME     MOK-IV-173hchar
EXPNO                  1
PROCNO                 1

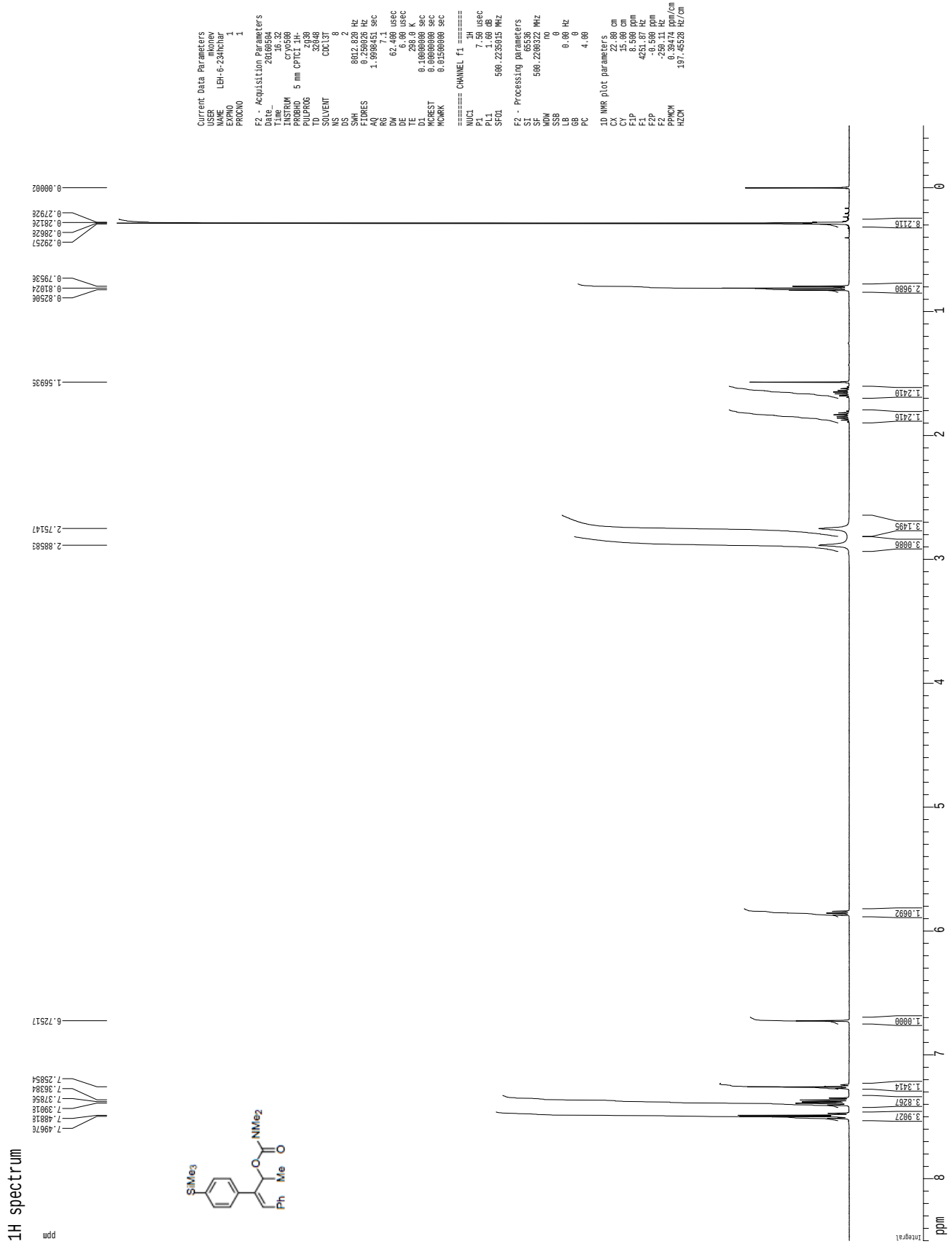
F2 - Acquisition Parameters
Date_          20160517
Time              10.35
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                32048
SOLVENT          CDCl3T
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.250026 Hz
AQ            1.9998451 sec
RG                  6.3
DW               62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

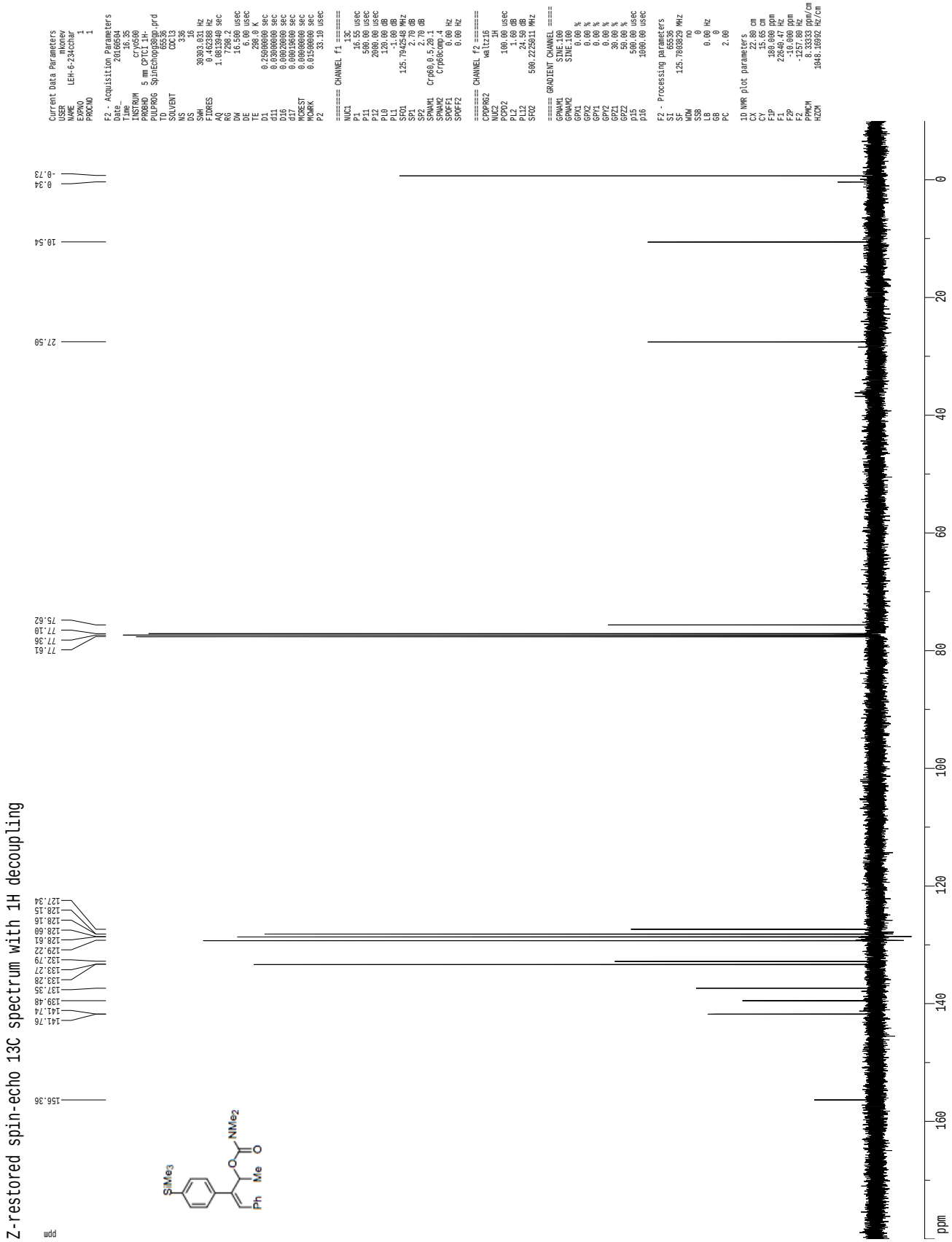
======== CHANNEL f1 ========
NUC1                 1H
P1                 7.50 usec
PL1                1.60 dB
SFO1        500.2235015 MHz

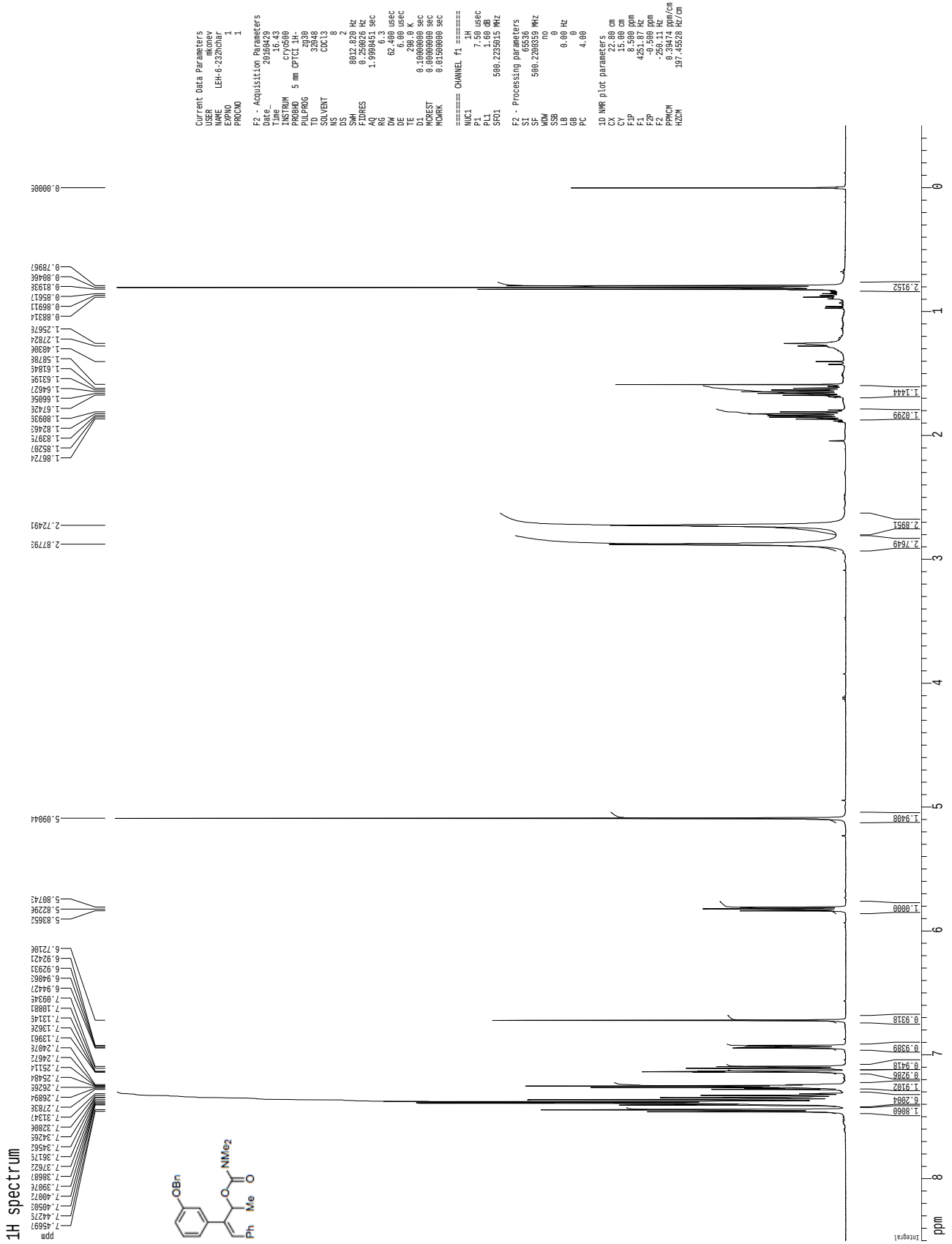
F2 - Processing parameters
SI                65536
SF          500.2200000 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 4.00

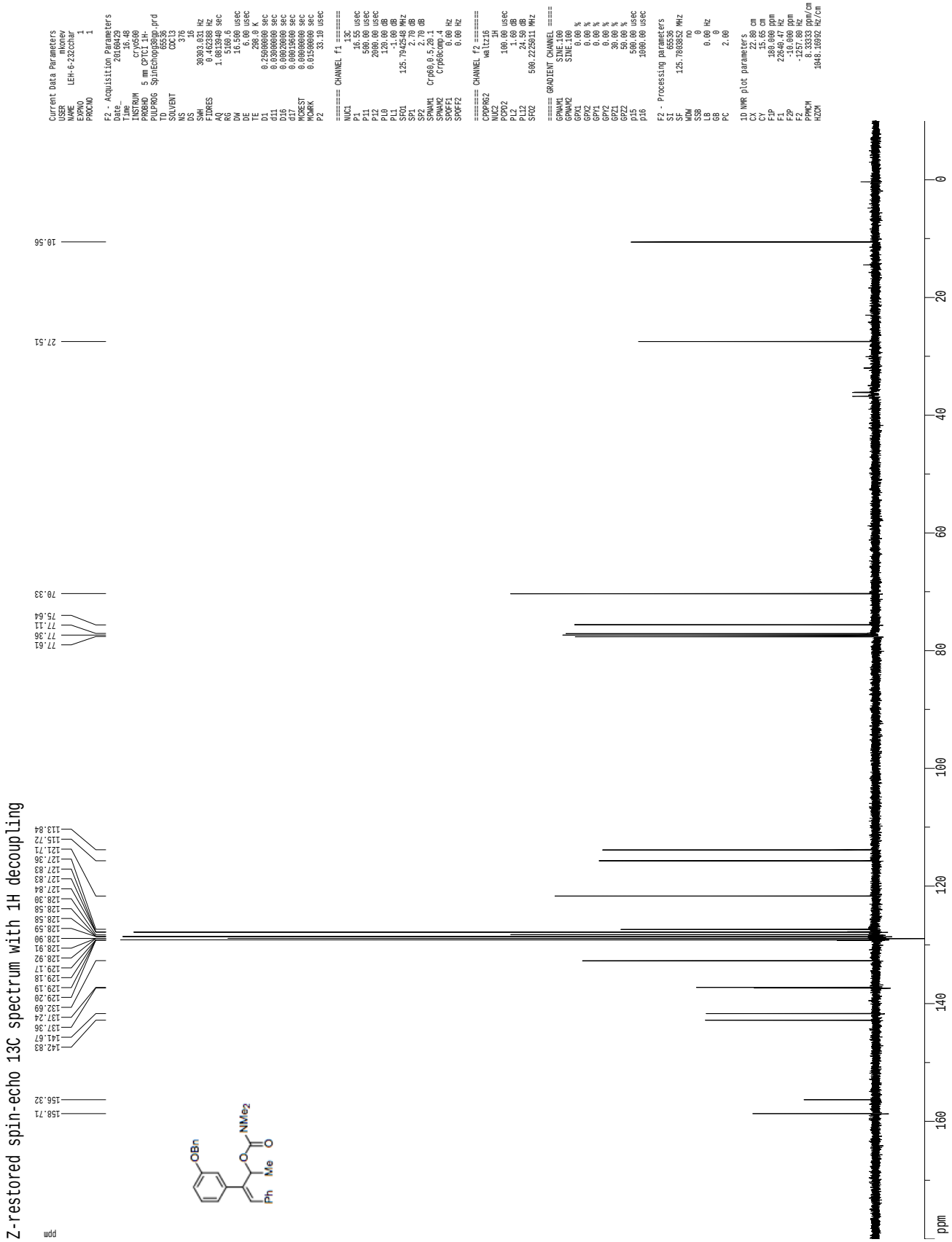
1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               8.500 ppm
F1              4251.87 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.39474 ppm/cm
HZCM          197.45528 Hz/cm
```

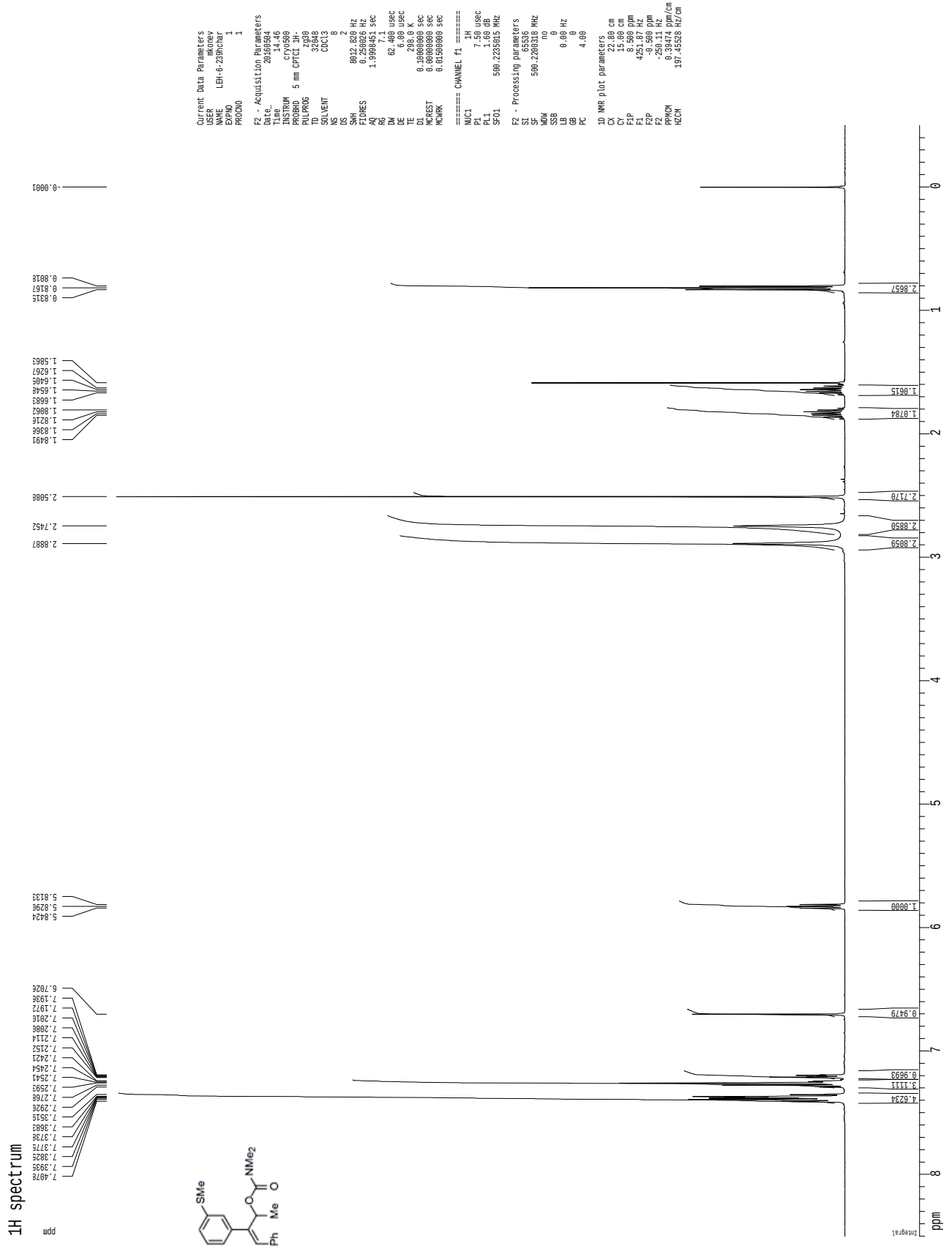

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
156.14
140.51
140.35
137.41
137.03
136.94
132.40
129.42
129.22
128.79
128.78
128.76
128.08
127.59
127.51
126.74
77.61
77.36
77.11
74.57
35.92
36.82
21.45
NMe2
O
O
Ph
Ph
Me
Current Data Parameters
USER mkonev
NAME MOK-IV-173cther2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160517
Time 10.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 304
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883843 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER mkonev
NAME LEH-6-234hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160504
Time 16.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200322 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
0.00002
0.27928
0.28126
0.28628
0.29257
0.79536
0.81024
0.82506
1.56935
2.75147
2.88583
6.72517
7.25854
7.36384
7.37856
7.39018
7.48818
7.49676
8.2116
7.9880
1.2410
1.2416
3.1495
3.0086
1.0692
1.0000
1.3414
3.8267
3.9027
SiMe3
NMe2
O
O
Me
Ph
ppm
Integral


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
SiMe3
Ph
Me
NMe2
O
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
-0.73
0.34
10.54
27.50
75.62
77.10
77.36
77.61
127.34
128.15
128.16
128.60
128.61
129.22
132.79
133.27
133.28
137.35
139.48
141.74
141.76
156.36
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
ppm
integral
OBn
NMe2
O
Me
Ph


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
OBn
NMe2
O
O
Ph
Me
Current Data Parameters
USER mkonev
NAME LEH-6-232cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time 16.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 376
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803852 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
158.71
156.32
142.83
141.67
137.36
137.24
132.69
129.20
129.19
129.18
129.17
128.92
128.91
128.90
128.59
128.58
128.58
128.30
127.84
127.83
127.83
127.36
121.71
115.72
113.84
77.61
77.36
77.11
75.64
70.33
27.51
10.56
ppm


1H spectrum
Current Data Parameters
USER mkoney
NAME LEH-6-239nchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160504
Time 14.46
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200318 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.669 ppm
F1 4251.87 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
0.0001
0.8018
0.8167
0.8315
1.5863
1.6267
1.6405
1.6546
1.6683
1.8062
1.8216
1.8366
1.8491
2.5088
2.7452
2.8887
5.8133
5.8296
5.8424
6.7026
7.1936
7.1972
7.2016
7.2086
7.2114
7.2162
7.2421
7.2454
7.2541
7.2593
7.2768
7.2926
7.3519
7.3683
7.3738
7.3775
7.3825
7.3935
7.4078
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2.7170
2.8850
2.8059
1.0000
0.9479
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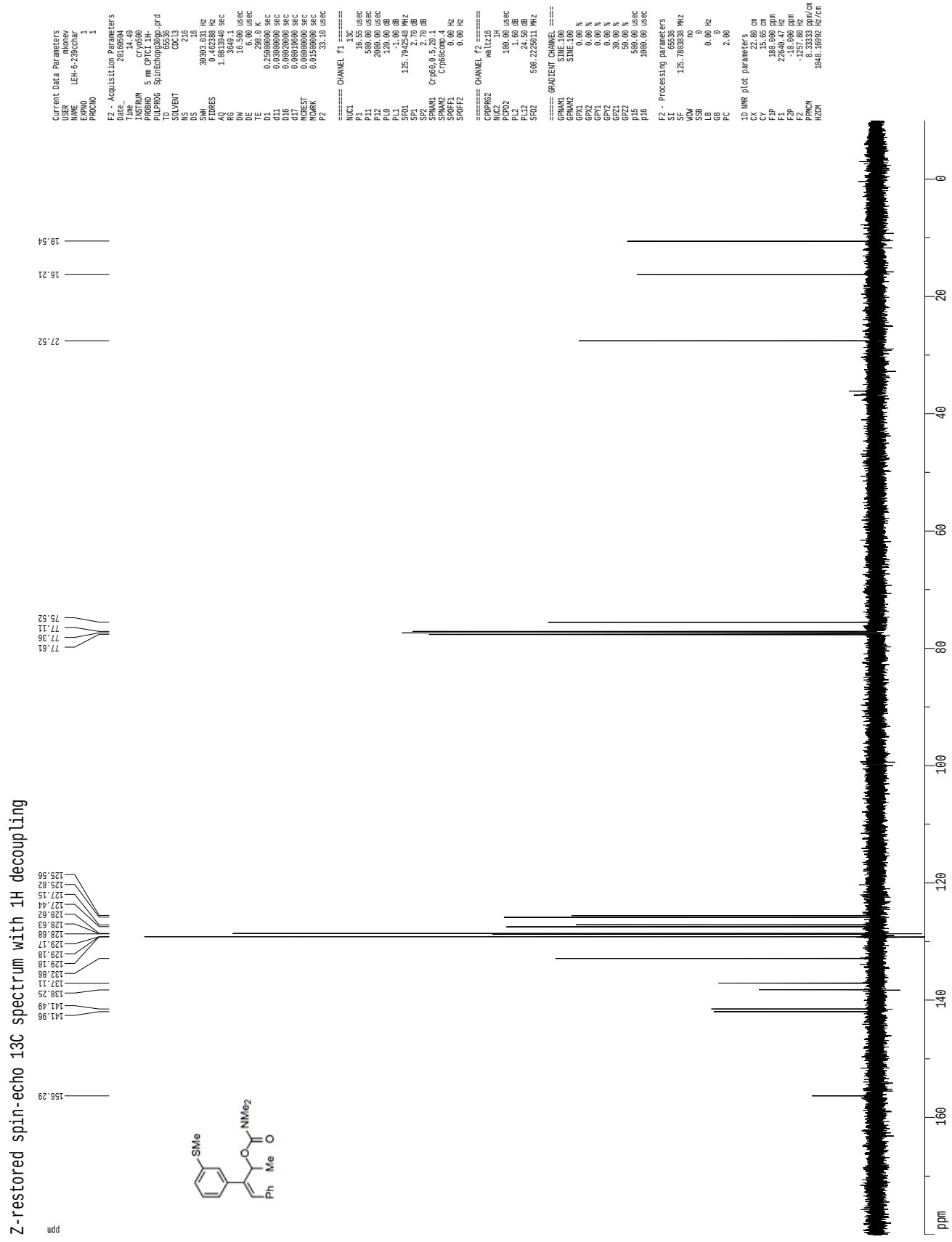
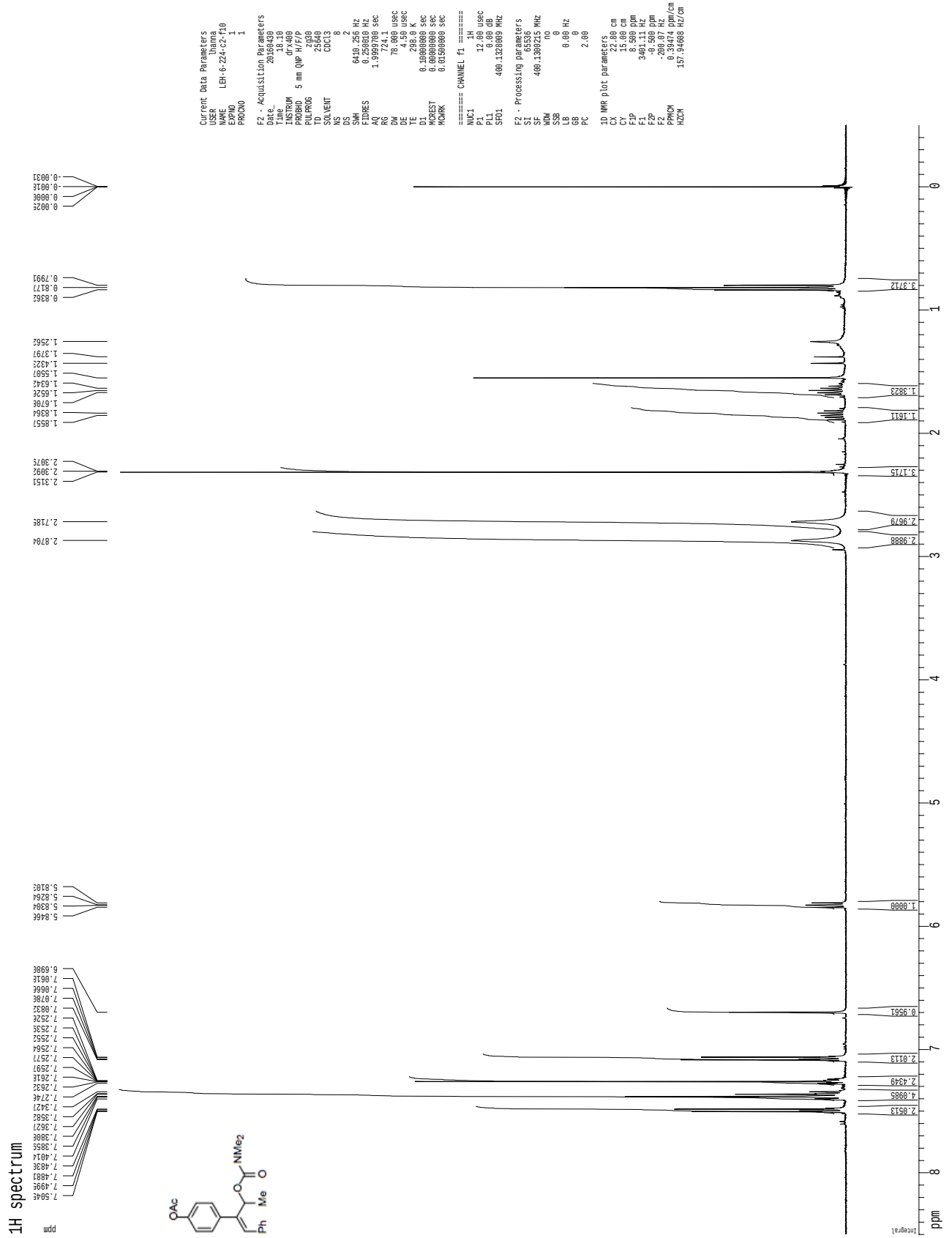
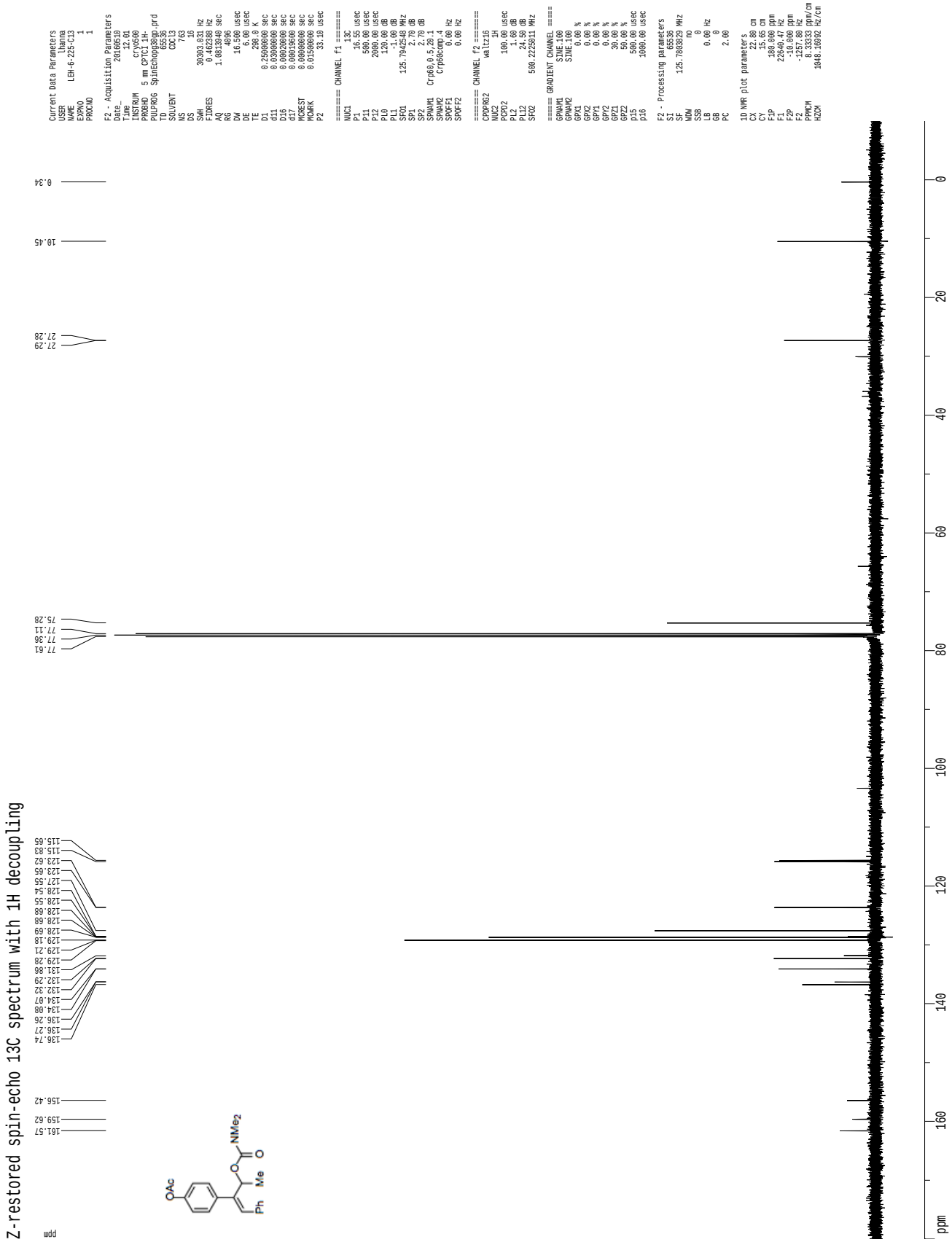
4.6234
SMe
NMe2
O
Me
Ph
ppm
Integral

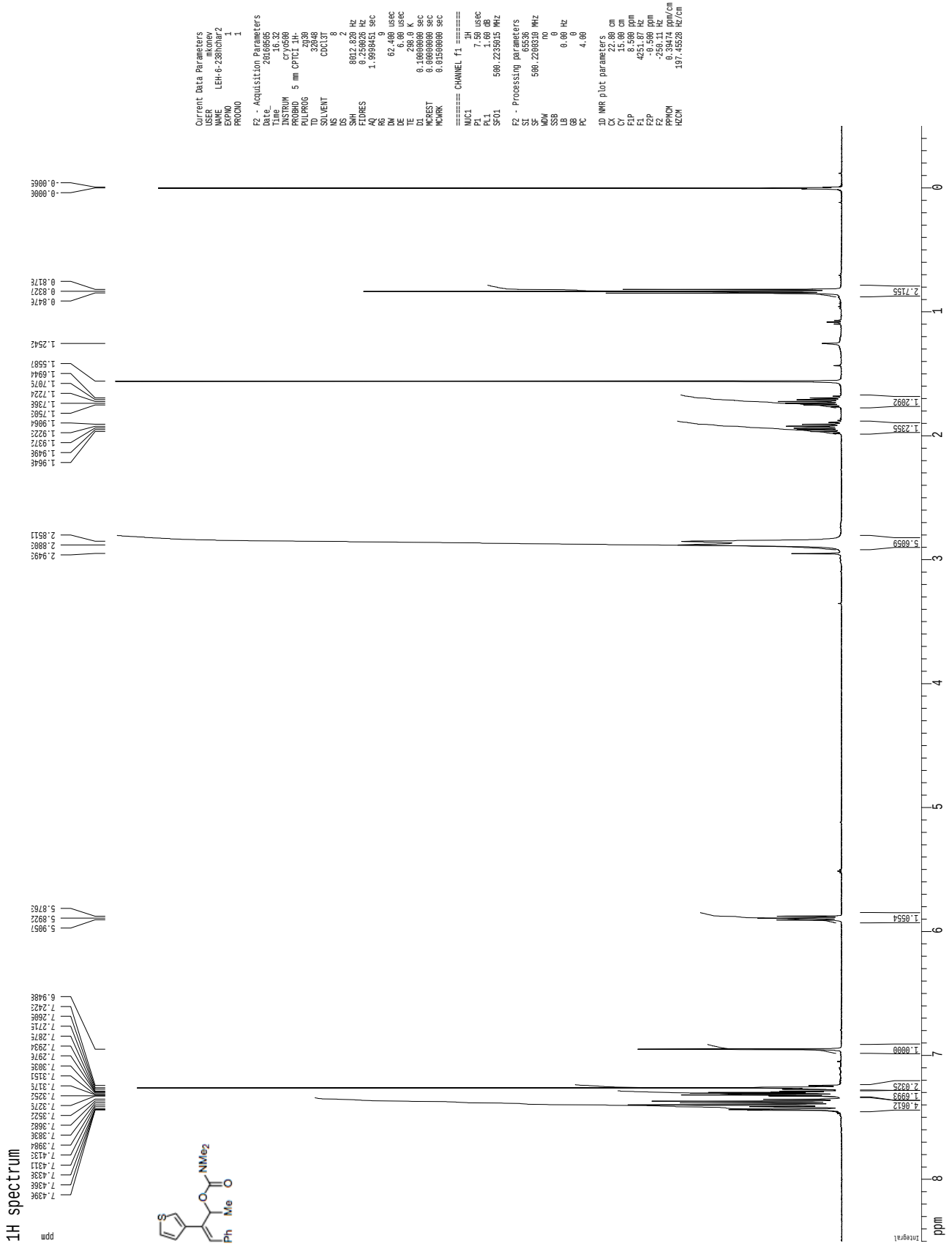
Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER Dhanna
NAME LEH-6-224-C2-Tf0
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160430
Time 18.10
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999709 sec
RG 724.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300215 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.560 ppm
F1 3401.11 Hz
F2P -0.569 ppm
F2 -200.80 Hz
PPMCM 0.33947 ppm/cm
HZCM 157.94608 Hz/cm
OAc
NMe2
Me
Ph
ppm
Integral

Z-restored spin-echo 13C spectrum with 1H decoupling




1H spectrum
Current Data Parameters
USER mkoney
NAME LEH-6-238hchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20166505
Time 16.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200310 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.359 ppm
F1 4215.87 Hz
F2P -0.150 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
0.8476
0.8327
1.2542
1.5587
1.6944
1.7075
1.7224
1.7366
1.7508
1.9064
1.9223
1.9372
1.9498
1.9646
2.8513
2.8806
2.9493
5.8763
5.8922
5.9057
6.9486
7.2423
7.2806
7.2974
7.2879
7.2994
7.2976
7.3036
7.3153
7.3179
7.3252
7.3279
7.3522
7.3682
7.3836
7.3984
7.4133
7.4313
7.4338
7.4366
7.4396
2.7155
1.2092
1.2355
5.6059
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1.0000
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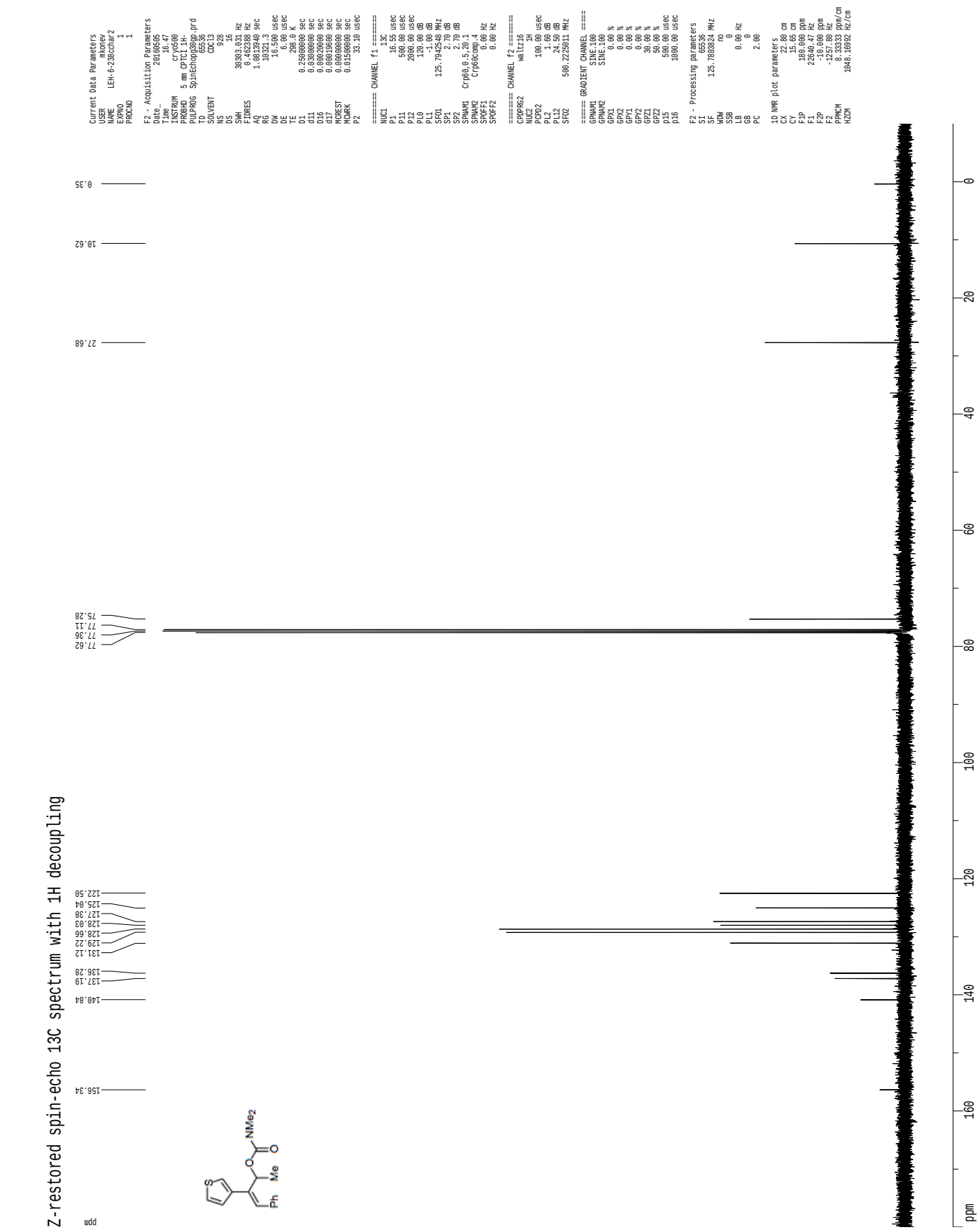
integral
ppm
NMe2
O
Me
Ph
S


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
NMe2
O
O
Me
Ph
S
156.34
140.84
137.19
136.28
131.12
129.22
128.66
128.03
127.38
125.04
122.50
77.62
77.36
77.11
75.28
27.68
18.62
0.35
Current Data Parameters
NAME LEH-6-23Cchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160505
Time 16.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog30gp.prd
TD 65536
SOLVENT CDCl3
NS 928
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803824 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
ppm
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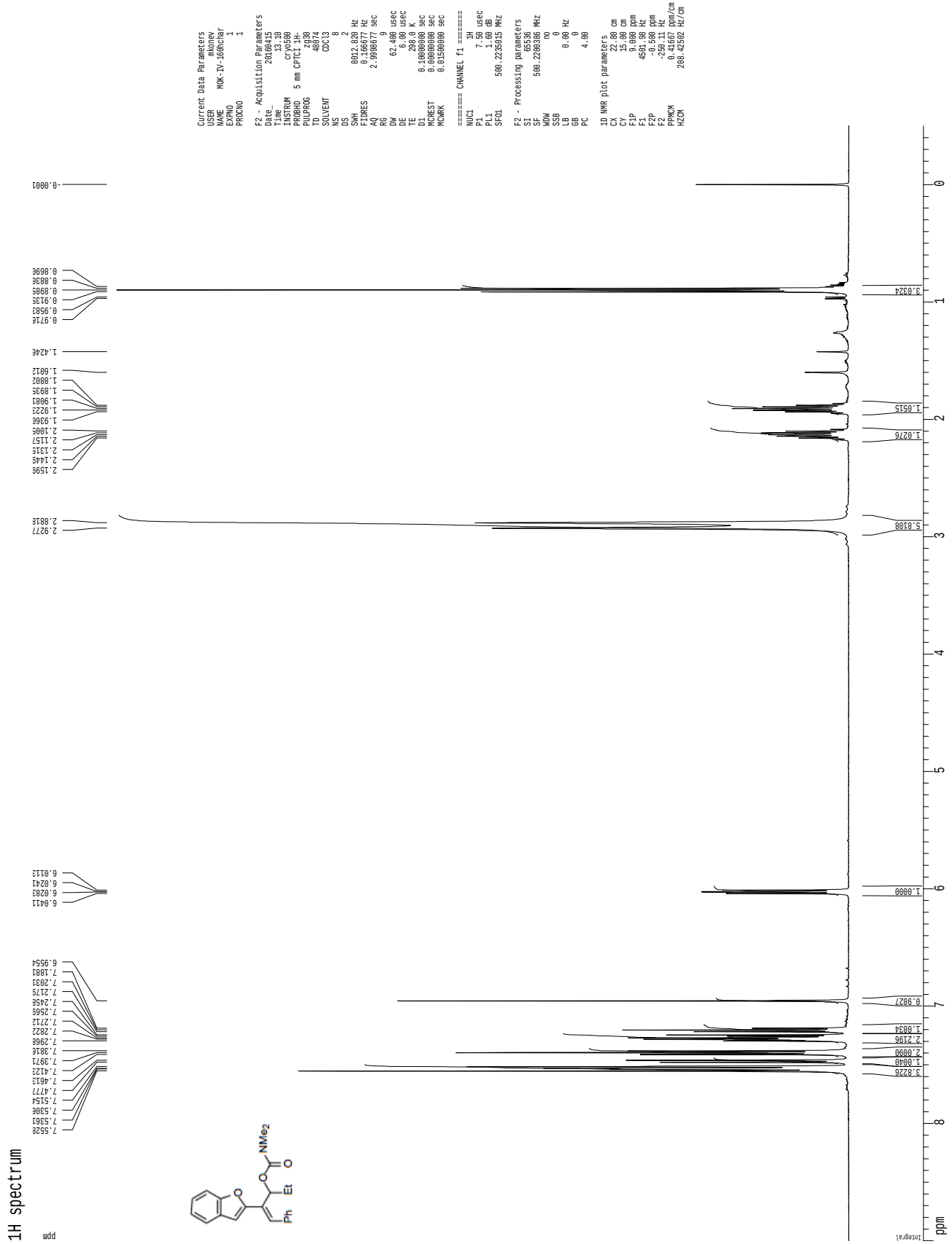
20
40
60
80
100
120
140
160


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-160nchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160415
Time 13.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 48074
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.166677 Hz
AQ 2.9998677 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200386 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.460 ppm
F2 -230.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
0.0001
0.8696
0.8936
0.8985
0.9135
0.9583
0.9746
1.4246
1.6012
1.8802
1.8936
1.9061
1.9223
1.9366
2.1005
2.1157
2.1315
2.1449
2.1599
2.8818
2.9277
6.0413
6.0583
6.0241
6.0113
7.1881
7.2051
7.2179
7.2458
7.2569
7.2712
7.2822
7.2966
7.3816
7.3971
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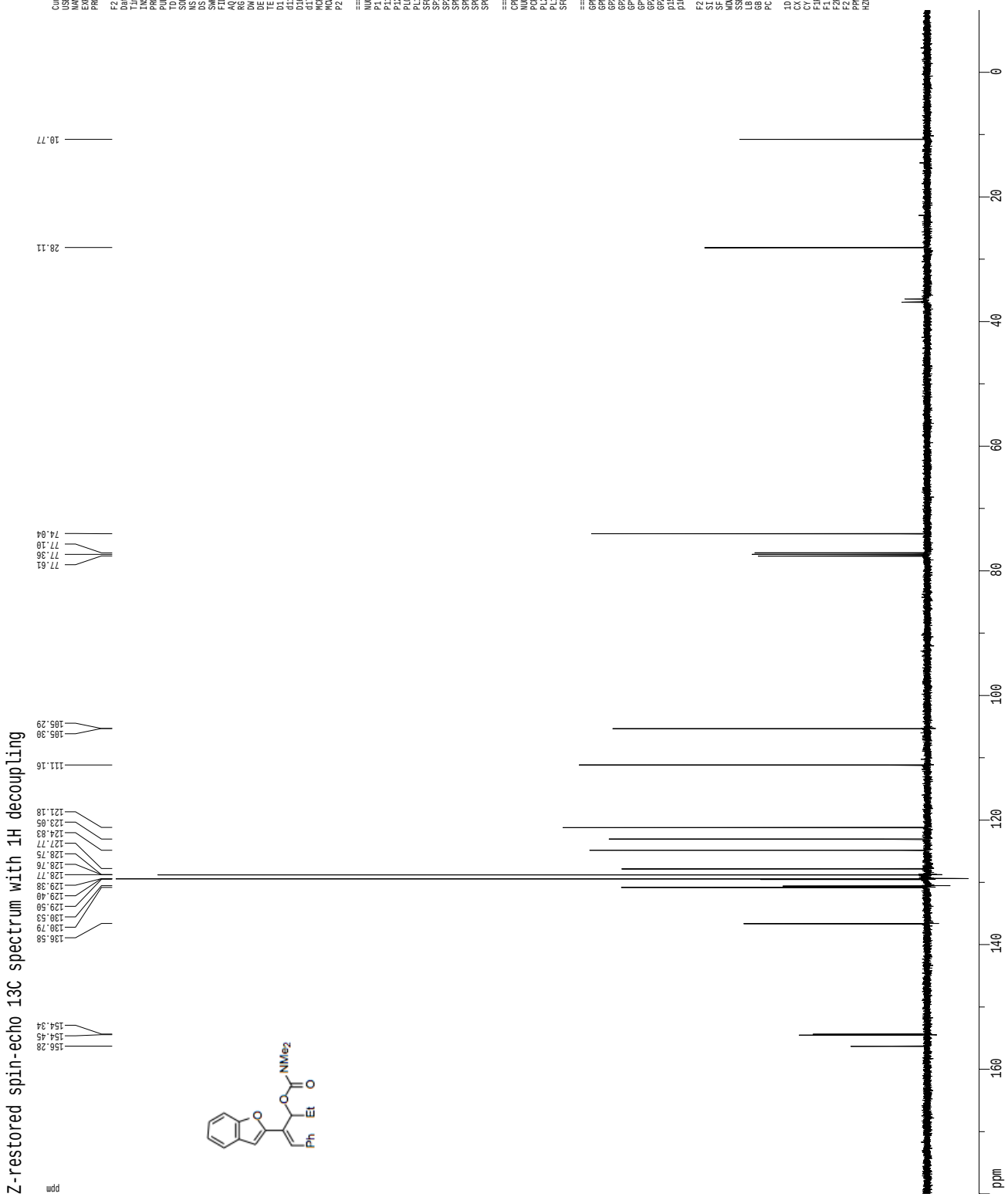
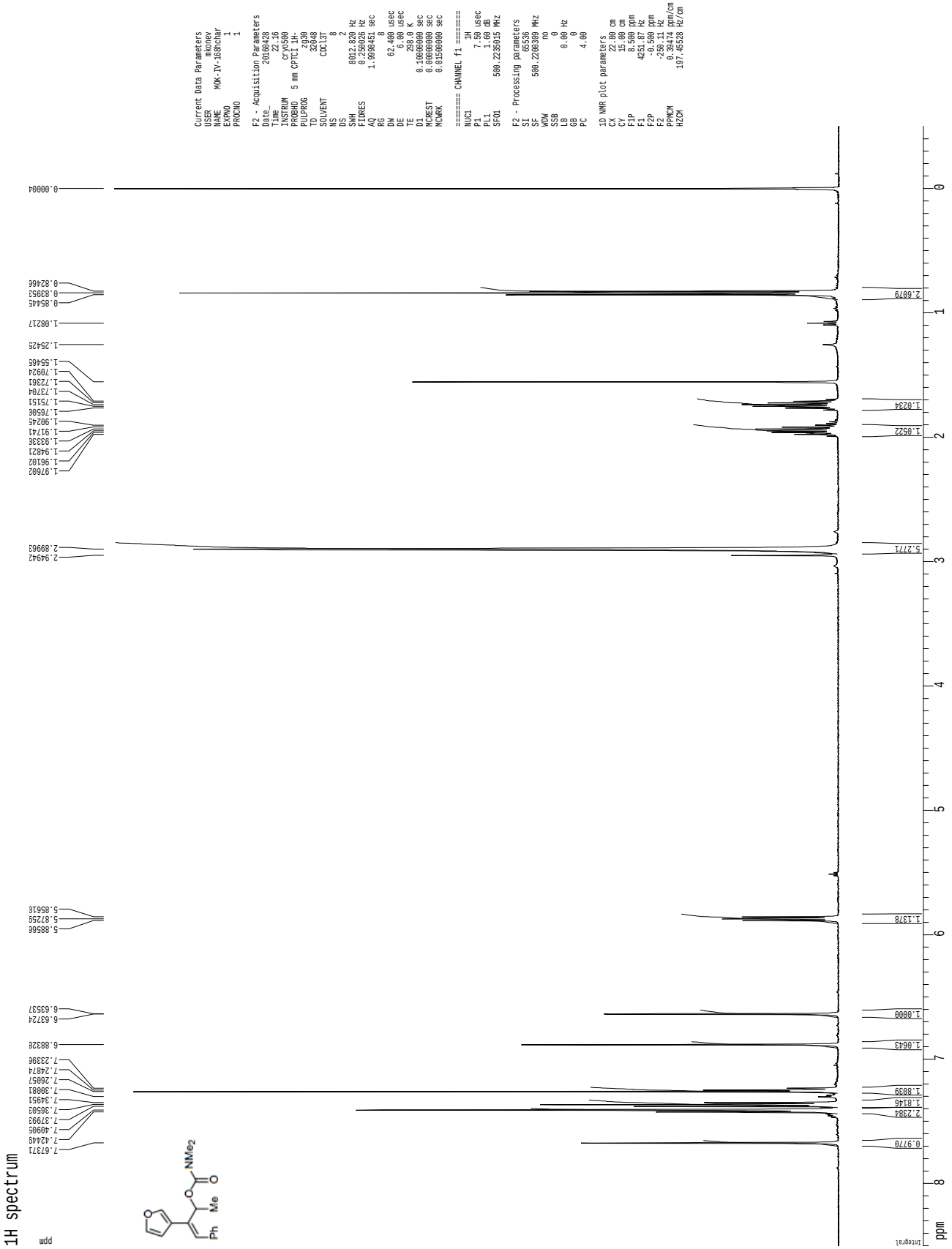
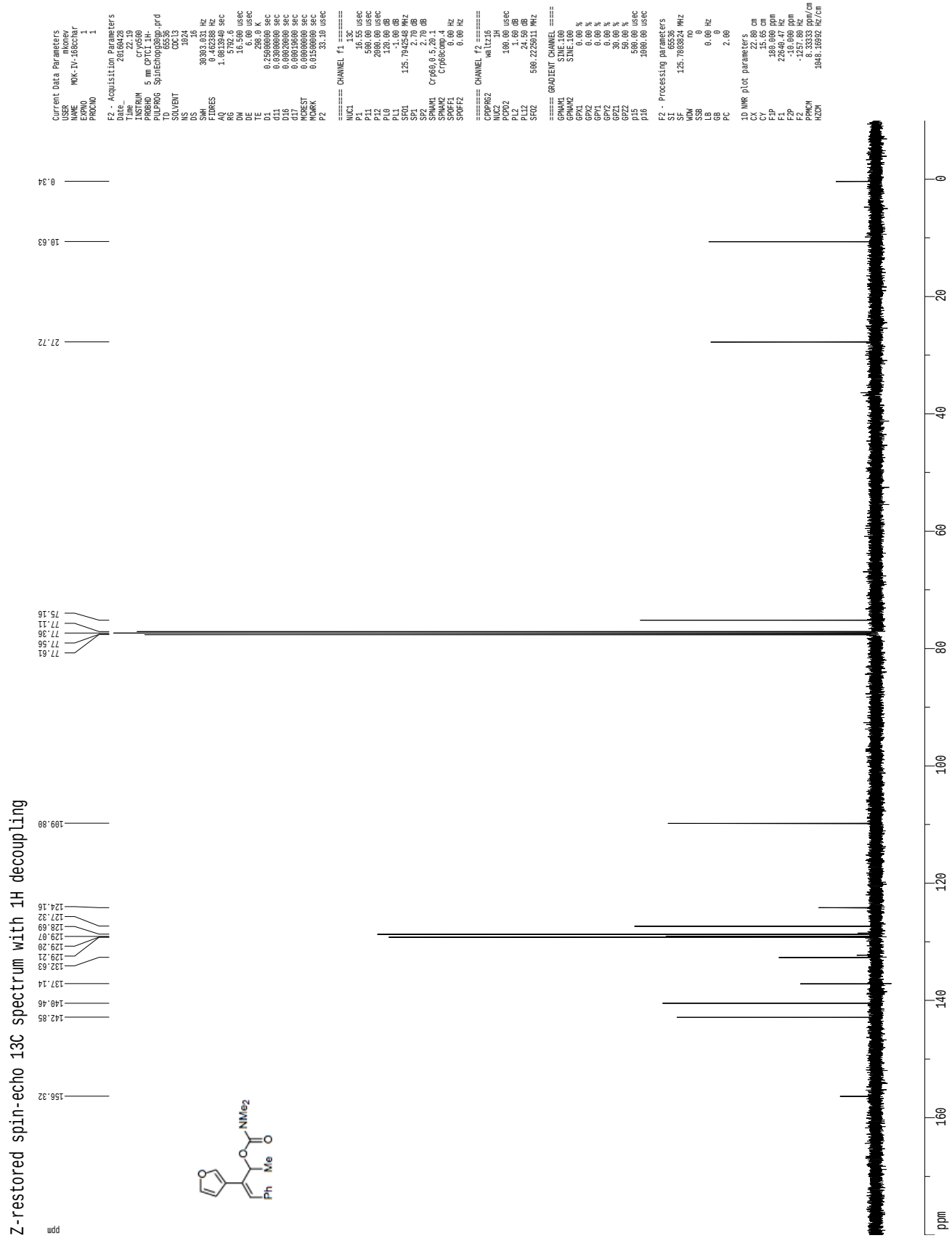
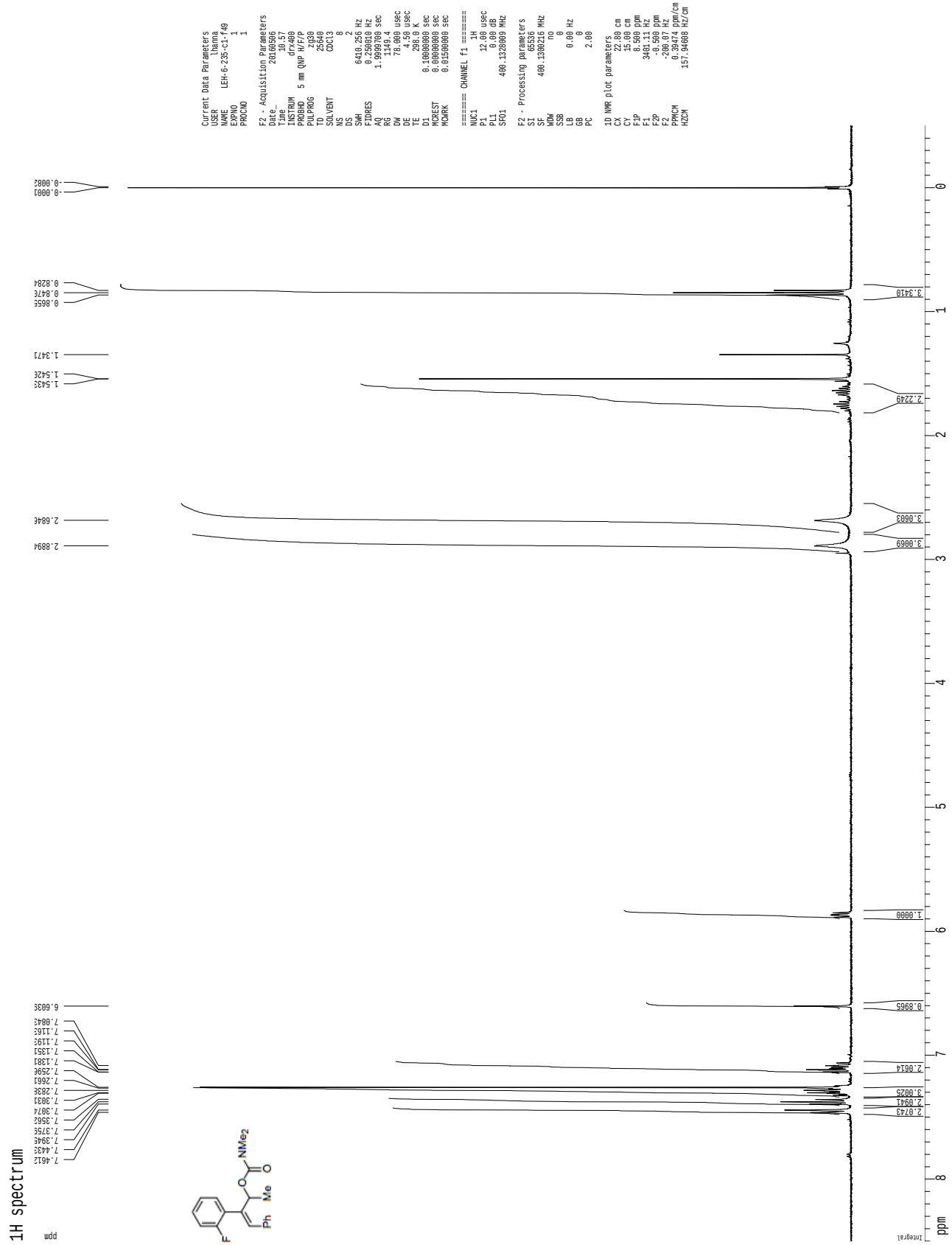
7.4613
7.4777
7.5154
7.5306
7.5361
7.5528
3.8324
1.0515
1.0276
5.8108
1.0000
0.9827
1.0834
2.2196
2.0890
1.0040
3.8226
Integral
ppm
0
1
2
3
4
5
6
7
8
ppm
NMe2
O
O
Et
Ph
O


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
156.28
154.45
154.34
136.58
130.79
130.53
129.50
129.40
129.38
128.77
128.76
128.75
127.77
124.83
123.05
121.18
111.16
105.30
105.29
77.61
77.36
77.10
74.04
28.11
10.77
NMe2
O
Et
Ph
Current Data Parameters
USER mkonev
NAME MOK-IV-106cther
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160415
Time 13.15
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchoDig30gp.prd
TD 65536
SOLVENT CDCl3
NS 160
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883870 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm

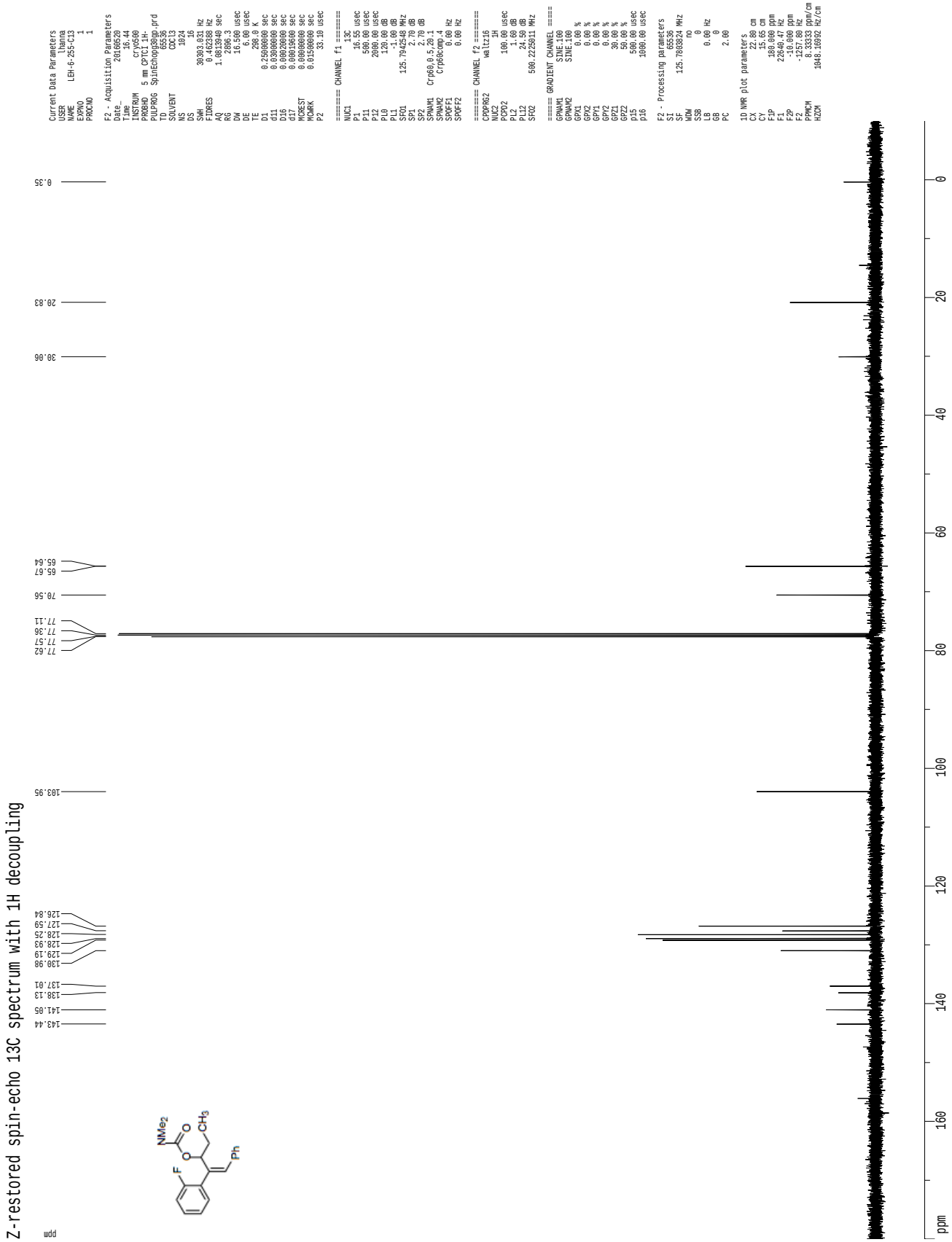

1H spectrum
Current Data Parameters
USER mkoney
NAME MOK-IV-168nchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160428
Time 22.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SF01 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200380 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.00004
0.82466
0.83952
0.85445
1.08217
1.25425
1.55465
1.70924
1.72361
1.73704
1.75151
1.76506
1.90245
1.91741
1.93336
1.94821
1.96102
1.97602
2.94942
2.99963
5.85616
5.87259
5.88566
6.63524
6.63537
6.88328
7.23396
7.24874
7.26057
7.30081
7.34951
7.36502
7.37993
7.40906
7.42445
7.61373
NMe2
O
Me
Ph
2.68079
1.08234
1.05222
5.27171
1.13178
1.00000
1.06443
1.88339
1.81446
2.23844
0.97706
ppm
Integral

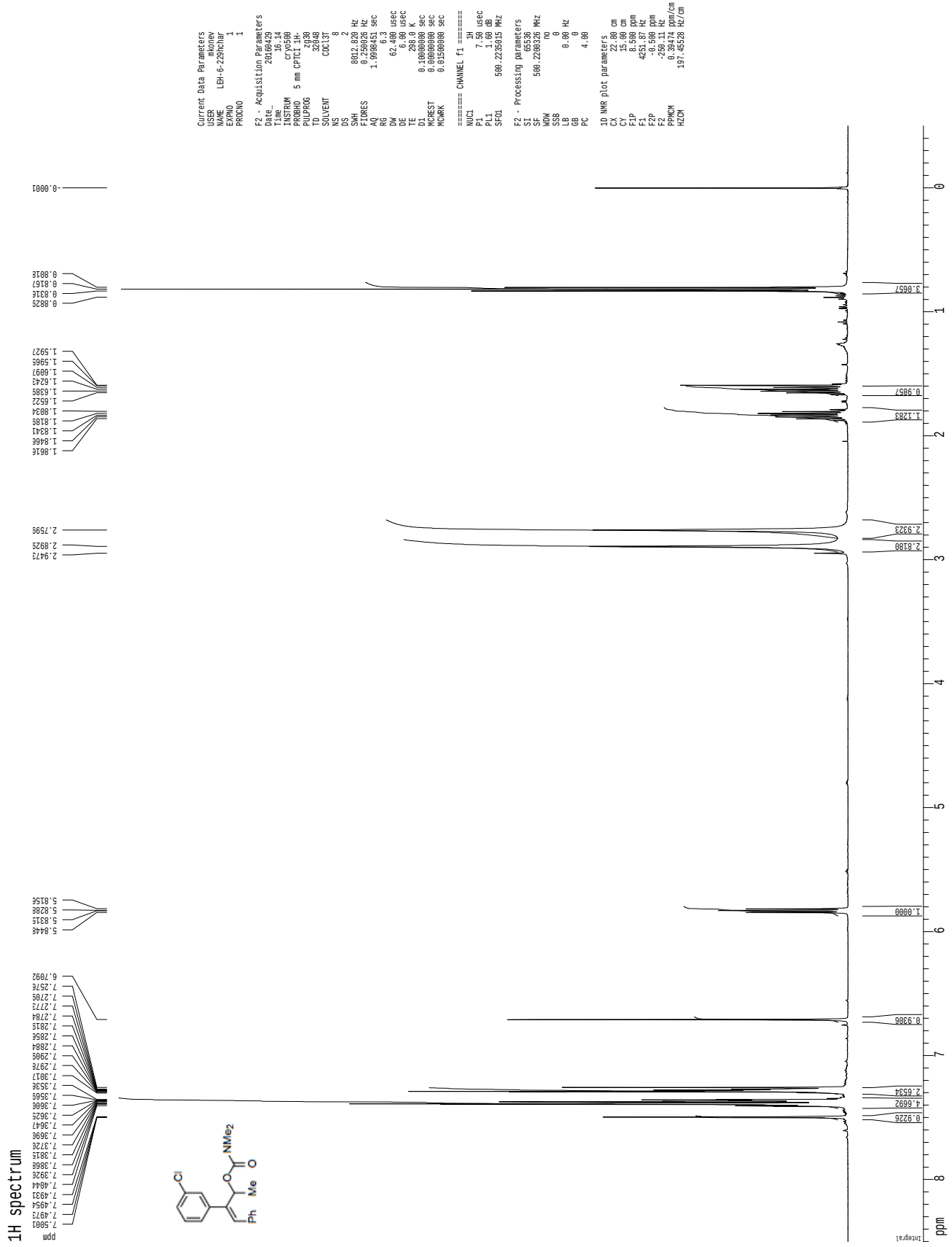
Z-restored spin-echo 13C spectrum with 1H decoupling

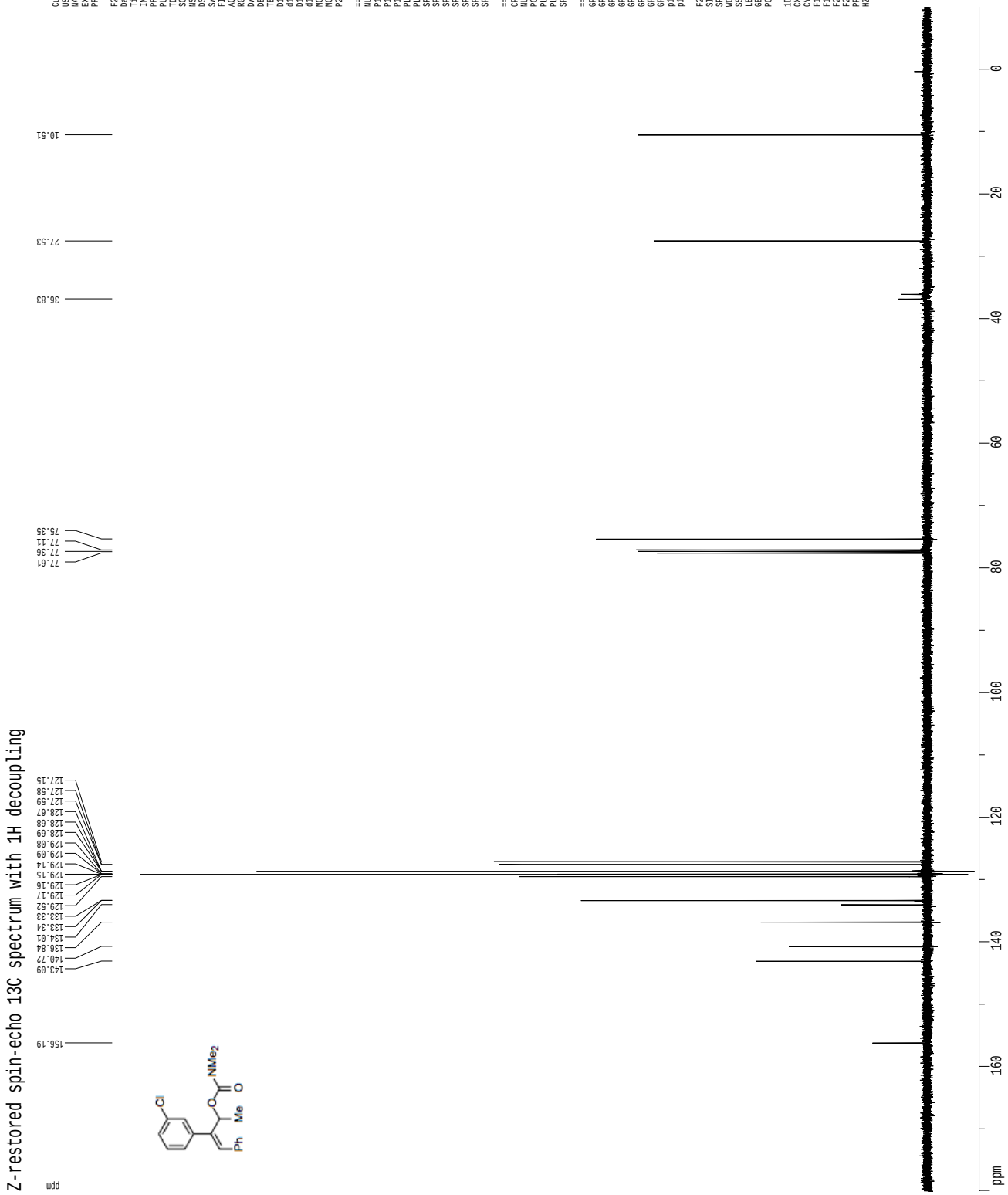


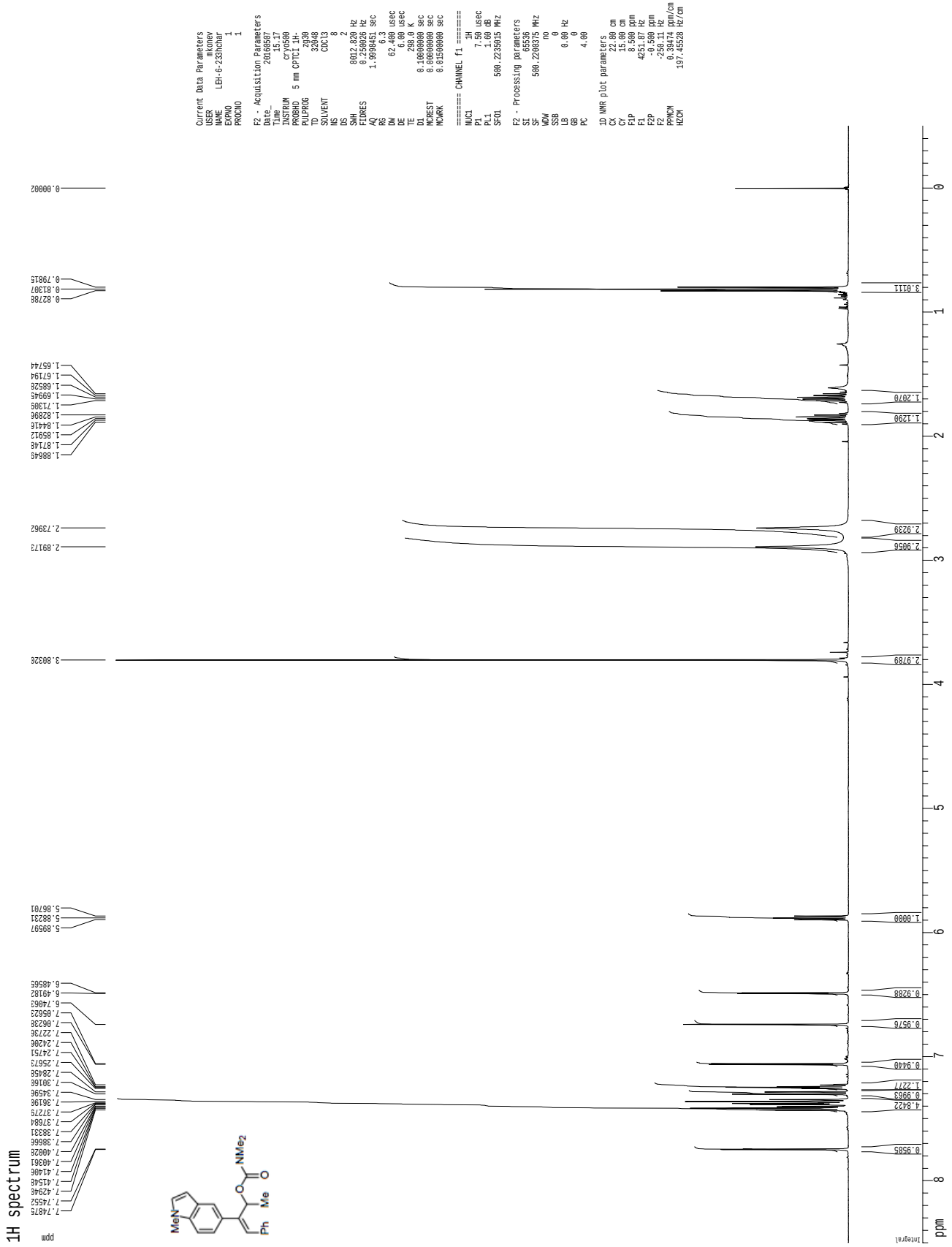

1H spectrum
Current Data Parameters
USER thanna
NAME LEH-6-235-c1-f40
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160506
Time_ 10.57
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999709 sec
RG 71.414
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300216 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.560 ppm
F1 3401.11 Hz
F2P -0.669 ppm
F2 -268.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94603 Hz/cm
0.8282
0.8476
0.8656
1.3471
1.5426
1.5435
2.6846
2.8894
6.6036
7.0843
7.1163
7.1193
7.1353
7.1383
7.2596
7.2661
7.2836
7.3033
7.3074
7.3562
7.3756
7.3946
7.4436
7.4612
3.3410
2.2249
3.0603
3.0669
1.0000
0.8965
2.0614
3.0025
2.0941
2.0743
NMe2
O
O
Me
Ph
F
ppm
Integral

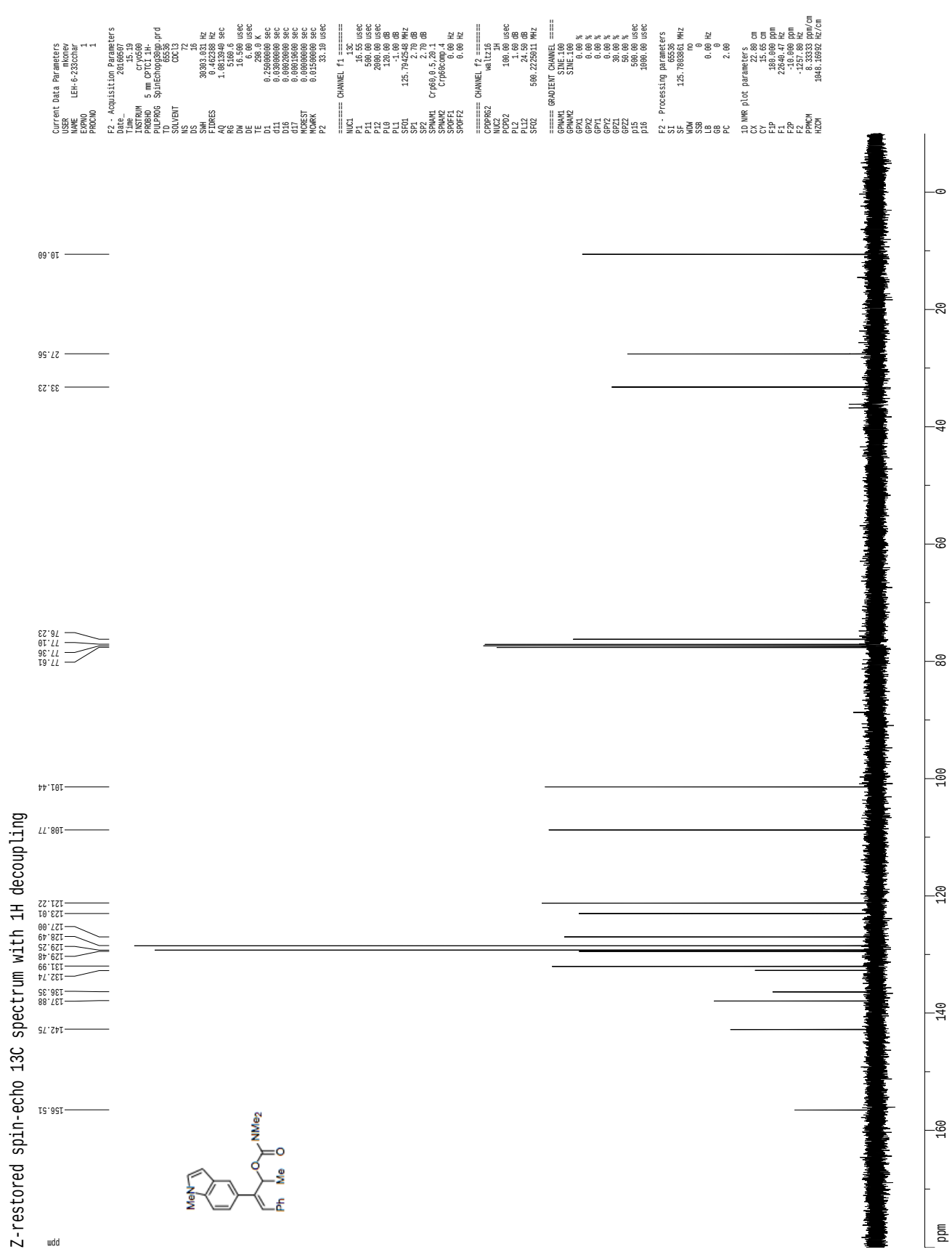
Z-restored spin-echo 13C spectrum with 1H decoupling

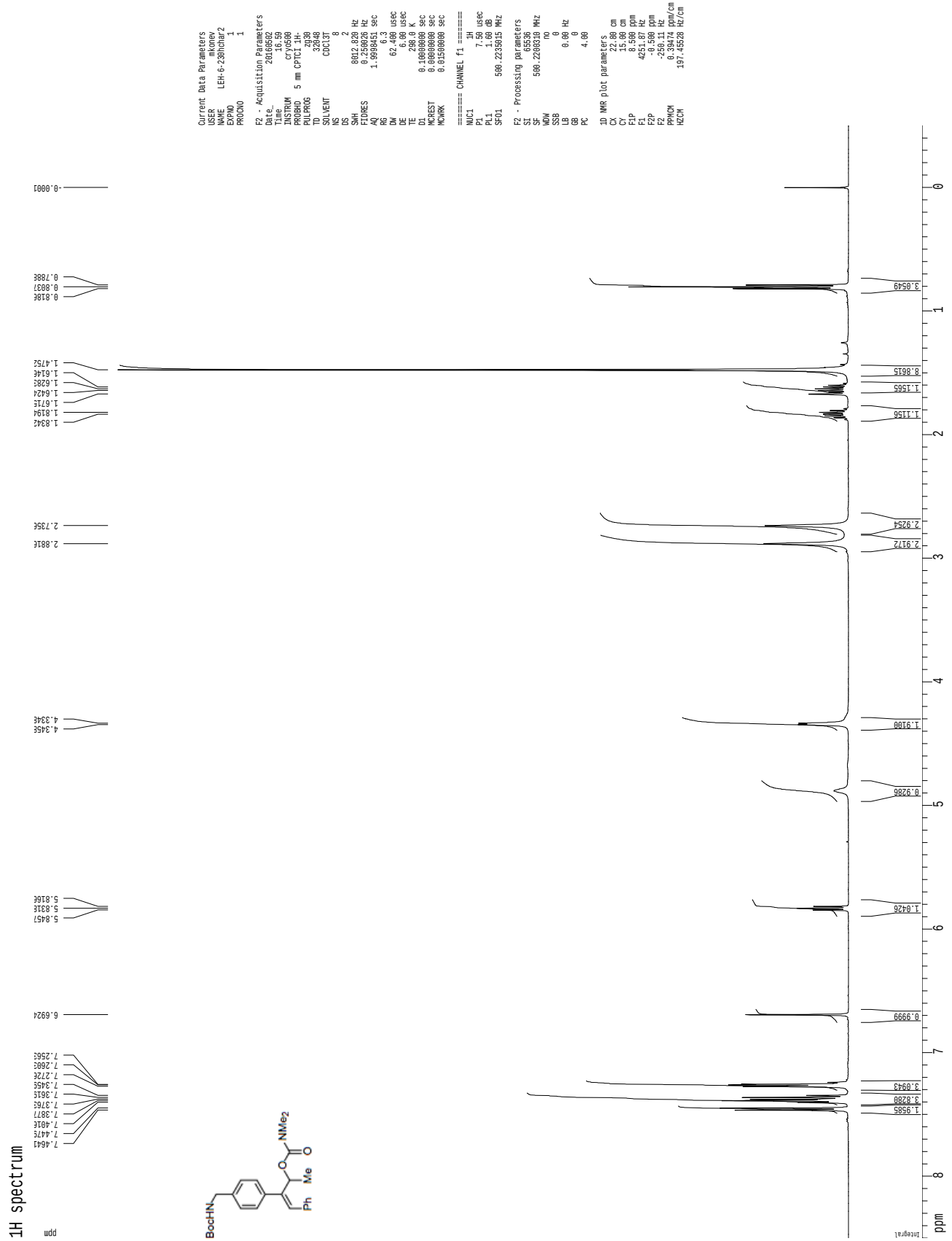


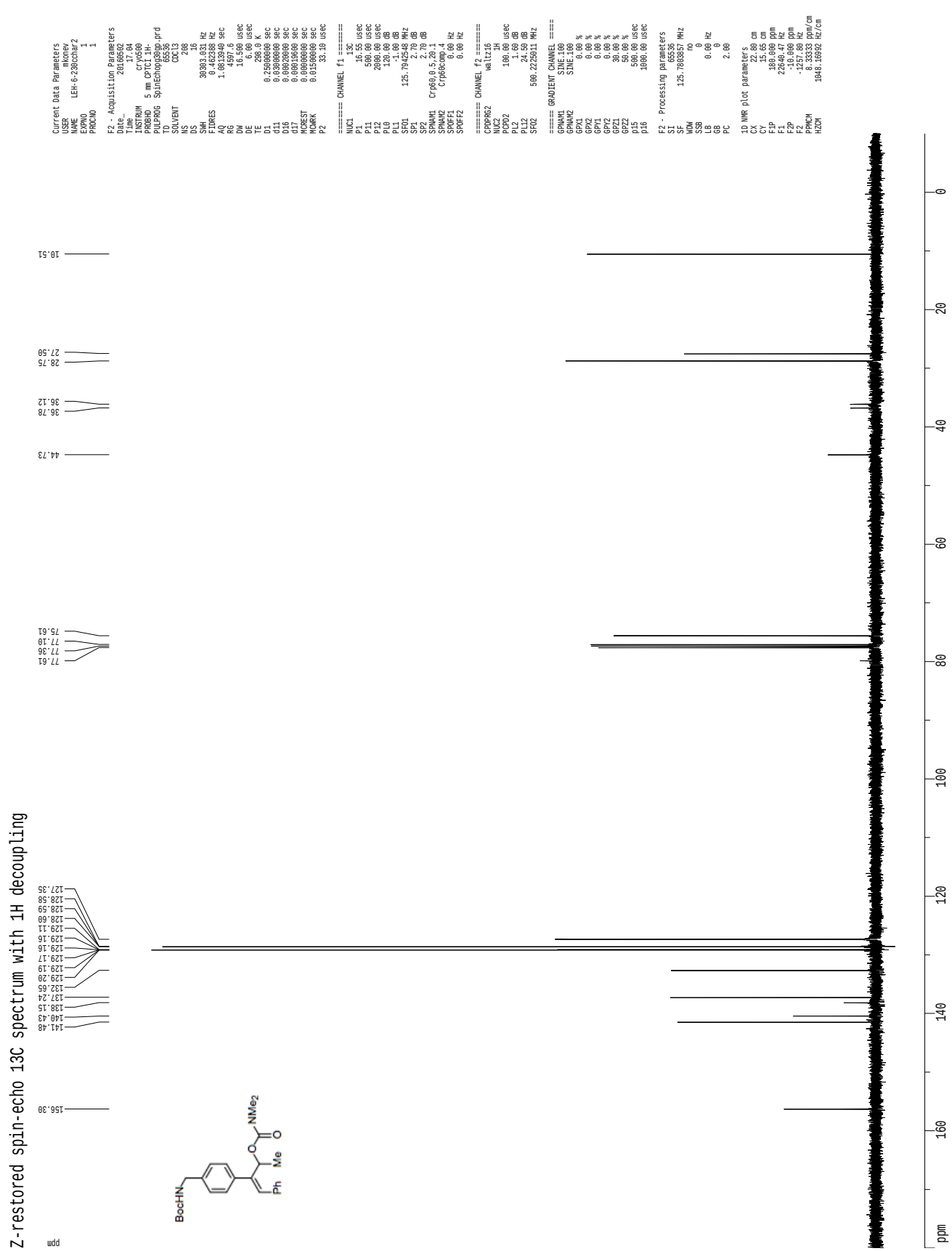

1H spectrum
Current Data Parameters
USER mkonev
NAME LEH-6-229hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time 16.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200326 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.4552 Hz/cm
0.0001
0.8018
0.8167
0.8316
0.8825
1.5927
1.5965
1.6097
1.6243
1.6385
1.6522
1.8034
1.8188
1.8341
1.8466
1.8616
2.7595
2.8925
2.9473
5.8156
5.8288
5.8315
5.8448
6.7092
7.2576
7.2706
7.2773
7.2784
7.2819
7.2856
7.2884
7.2906
7.2978
7.3017
7.3536
7.3565
7.3606
7.3625
7.3647
7.3696
7.3726
7.3815
7.3868
7.3926
7.4044
7.4933
7.4954
7.4973
7.5081
ppm
3.0657
0.9857
1.1283
2.9323
2.8180
1.0000
0.9306
2.6534
4.6692
0.9226
Integral
ppm
Cl
O
O
NMe2
Me
Ph

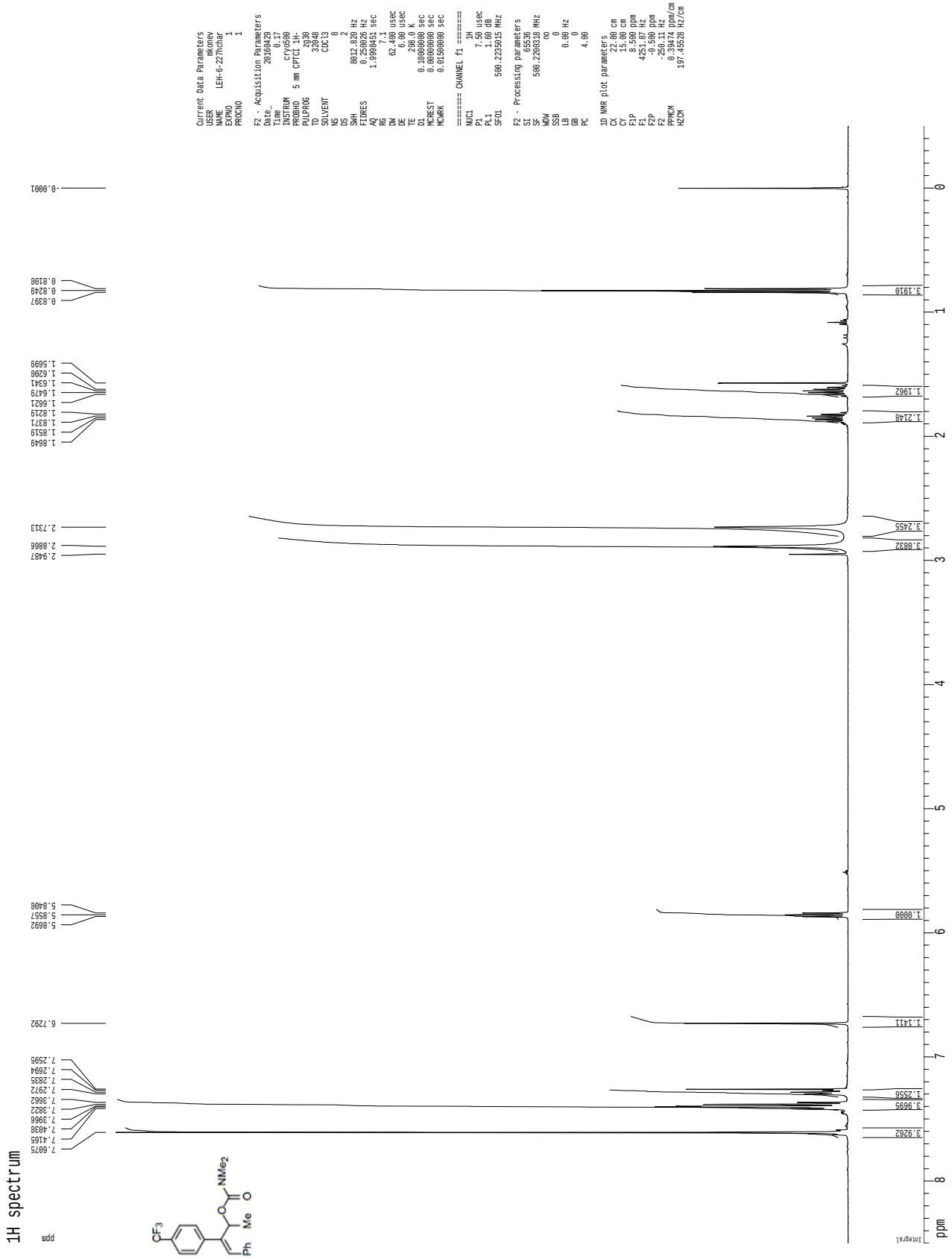

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Cl
NMe2
O
O
Ph
Me
156.19
143.09
140.72
136.84
134.01
133.34
133.33
129.52
129.17
129.16
129.15
129.14
129.08
128.69
128.68
128.67
127.59
127.58
127.15
77.61
77.36
77.11
75.35
36.83
27.53
10.51
Current Data Parameters
USER mkonev
NAME LEH-6-229cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time 16.18
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 368
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.000 usec
F2 - Processing parameters
SI 65536
SF 125.7803838 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.00 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm

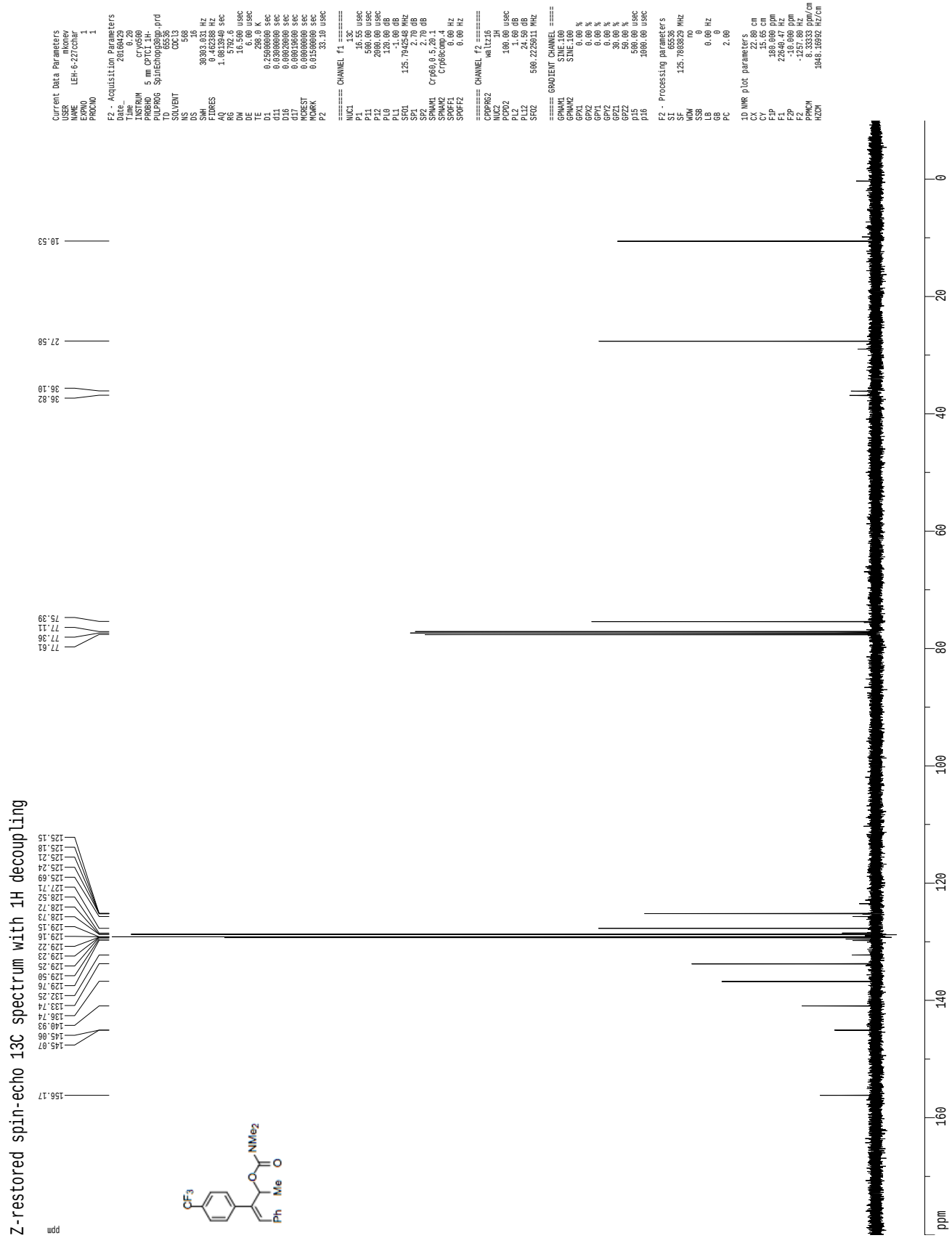

1H spectrum
Current Data Parameters
USER mkonev
NAME LEH-6-233hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160507
Time 15.17
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200375 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.596 ppm
F1 4251.87 Hz
F2P -0.560 ppm
F2 -280.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
0.00002
0.79815
0.81307
0.82788
1.65744
1.67194
1.68528
1.69945
1.71305
1.82896
1.84416
1.85912
1.87148
1.88649
2.73962
2.89173
3.80326
5.86701
5.88231
5.89597
6.48565
6.49182
6.74063
7.05623
7.06238
7.22736
7.24206
7.24751
7.25673
7.28458
7.30166
7.34596
7.36196
7.37279
7.37684
7.38331
7.38666
7.40028
7.40361
7.41406
7.41548
7.42946
7.74562
7.74875
3.0111
1.2070
1.1290
2.9239
2.9056
2.9789
1.0000
0.9288
0.9576
0.9440
1.2277
0.9963
4.8422
0.9585
Integral
ppm
MeN
NMe2
O
Me
Ph


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
MeN
Ph
Me
O
O
NMe2
156.51
142.75
137.88
136.35
132.74
131.99
129.48
129.25
128.49
127.00
123.01
121.22
108.77
101.44
77.61
77.36
77.10
76.23
33.23
27.56
10.60
Current Data Parameters
USER mkonev
NAME LEH-6-23Cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160507
Time 15.19
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 72
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 15.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803861 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
ppm
0
20
40
60
80
100
120
140
160


1H spectrum
Current Data Parameters
USER mkoney
NAME LEH-6-230hchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160502
Time 16.59
INSTRUM cryo600
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200310 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.1560 ppm
F1 4075.87 Hz
F2P -0.1500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
0.0001
0.7886
0.8037
0.8186
1.4752
1.6146
1.6283
1.6424
1.6715
1.8194
1.8342
2.7356
2.8814
4.3346
4.3456
5.8166
5.8318
5.8457
6.6924
7.2563
7.2682
7.2726
7.3456
7.3615
7.3763
7.3871
7.4016
7.4475
7.4641
3.0549
0.8615
1.1565
1.1156
2.9254
2.9172
1.9100
0.9286
1.0426
0.9999
3.0943
3.8280
1.9585
integral
ppm
BocHN
Ph
Me
O
O
NMe2


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
BocHN
Ph
Me
O
NMe2
156.30
141.48
140.43
138.15
137.24
132.65
129.20
129.19
129.17
129.16
129.16
129.11
128.60
128.59
128.58
127.35
77.61
77.36
77.10
75.61
44.73
36.78
36.12
28.75
27.50
10.51
Current Data Parameters
USER mkonev
NAME LEH-6-23DCchar2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160502
Time 17.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 208
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4597.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 165.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803857 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
ppm
0
20
40
60
80
100
120
140
160


1H spectrum
ppm
Integral
Current Data Parameters
USER mkonev
NAME LEH-6-227hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time_ 0.17
INSTRUM cryQ600
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250088 Hz
AQ 1.9998451 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 7.65 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200318 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.87 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
-0.0001
0.8100
0.8249
0.8397
1.5699
1.6200
1.6341
1.6479
1.6621
1.8219
1.8371
1.8519
1.8649
2.7313
2.8866
2.9487
5.8400
5.8557
5.8692
6.7292
7.2595
7.2694
7.2835
7.2972
7.3662
7.3822
7.3966
7.4838
7.4165
7.6075
3.1910
1.1962
1.2148
3.2455
3.0832
1.0000
1.1411
1.2556
3.9695
3.9262
CF3
NMe2
O
O
Me
Ph



Current Data Parameters
USER mkonev
NAME LEH-6-227cchar
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160429
Time 0.20
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 568
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec

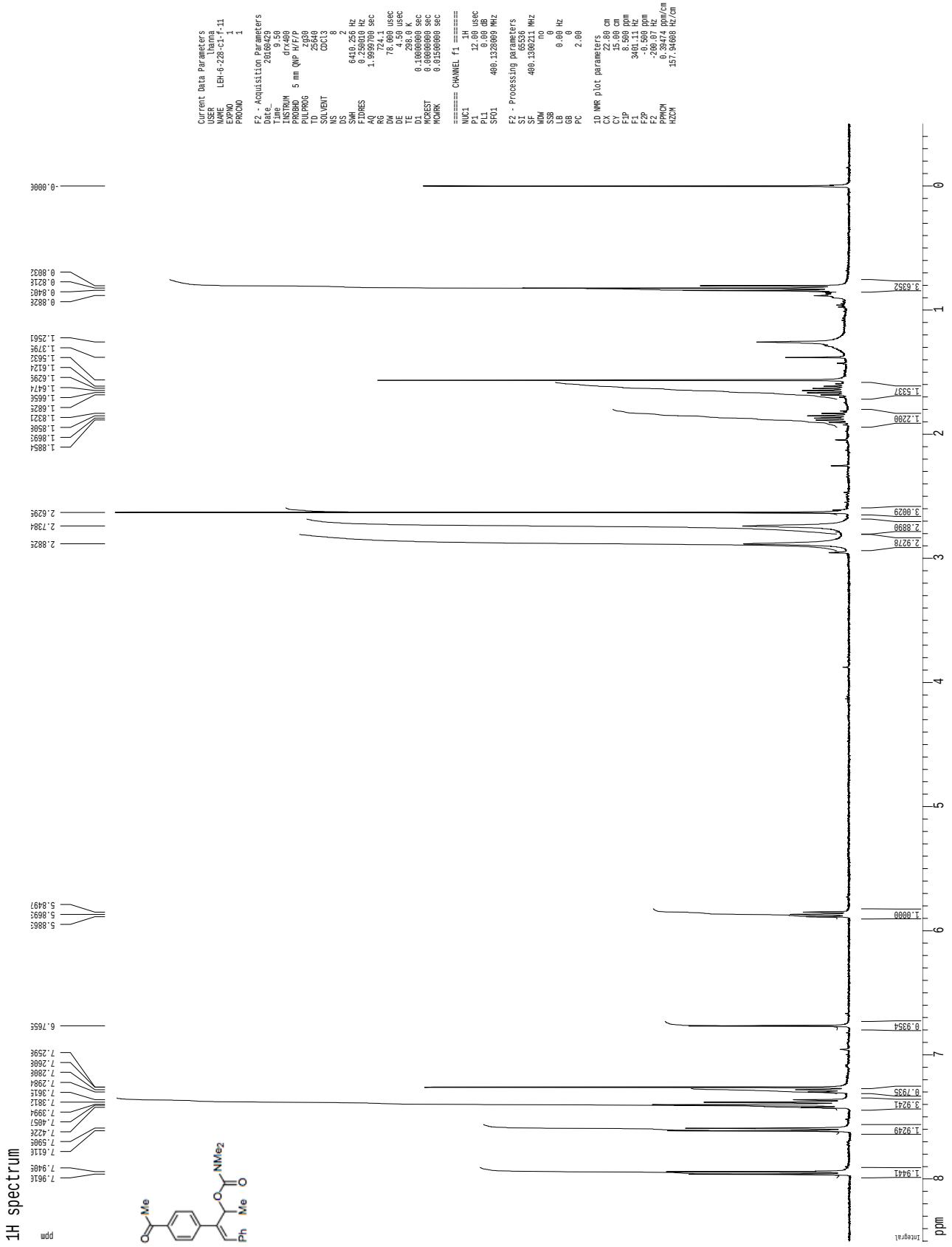
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

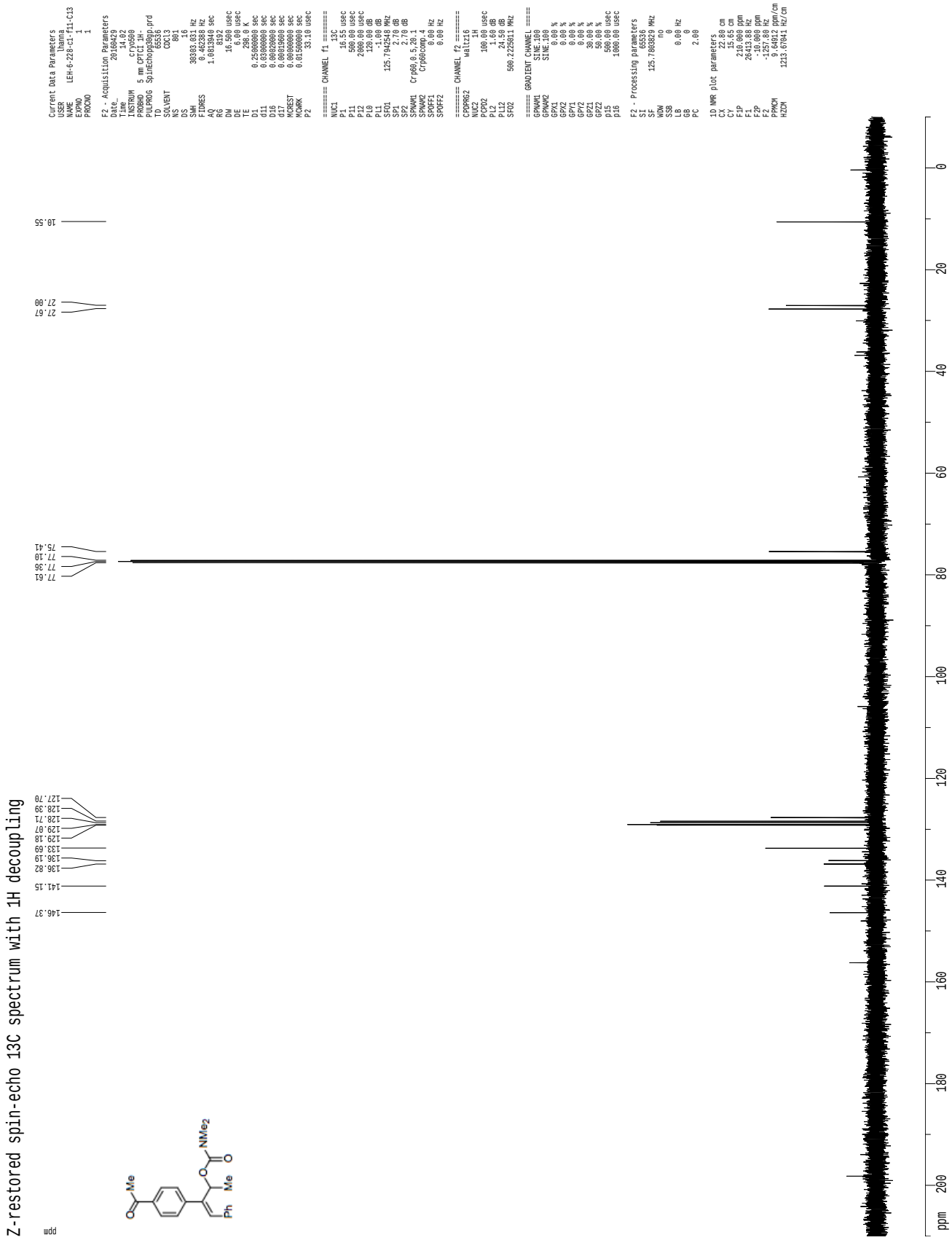
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz

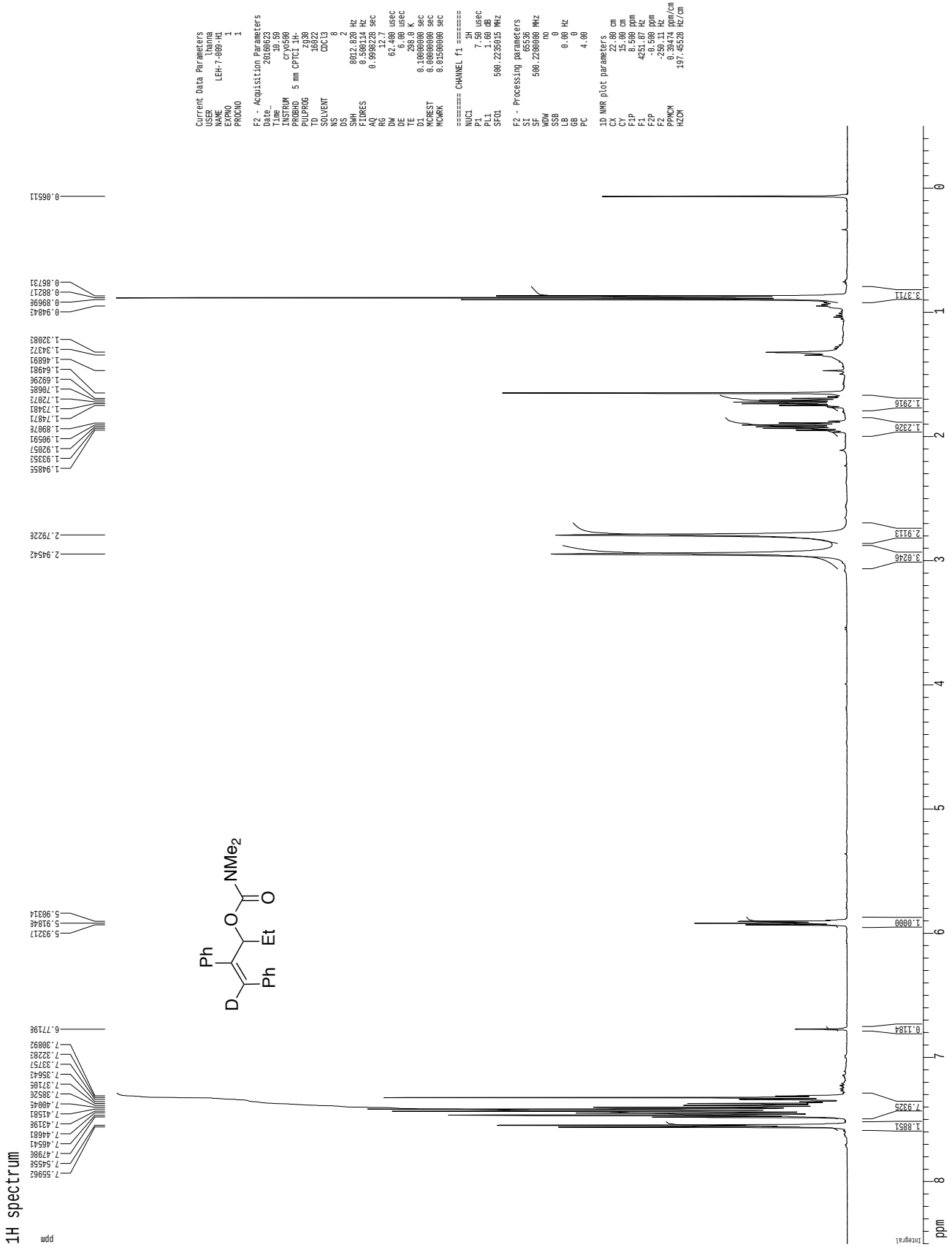
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

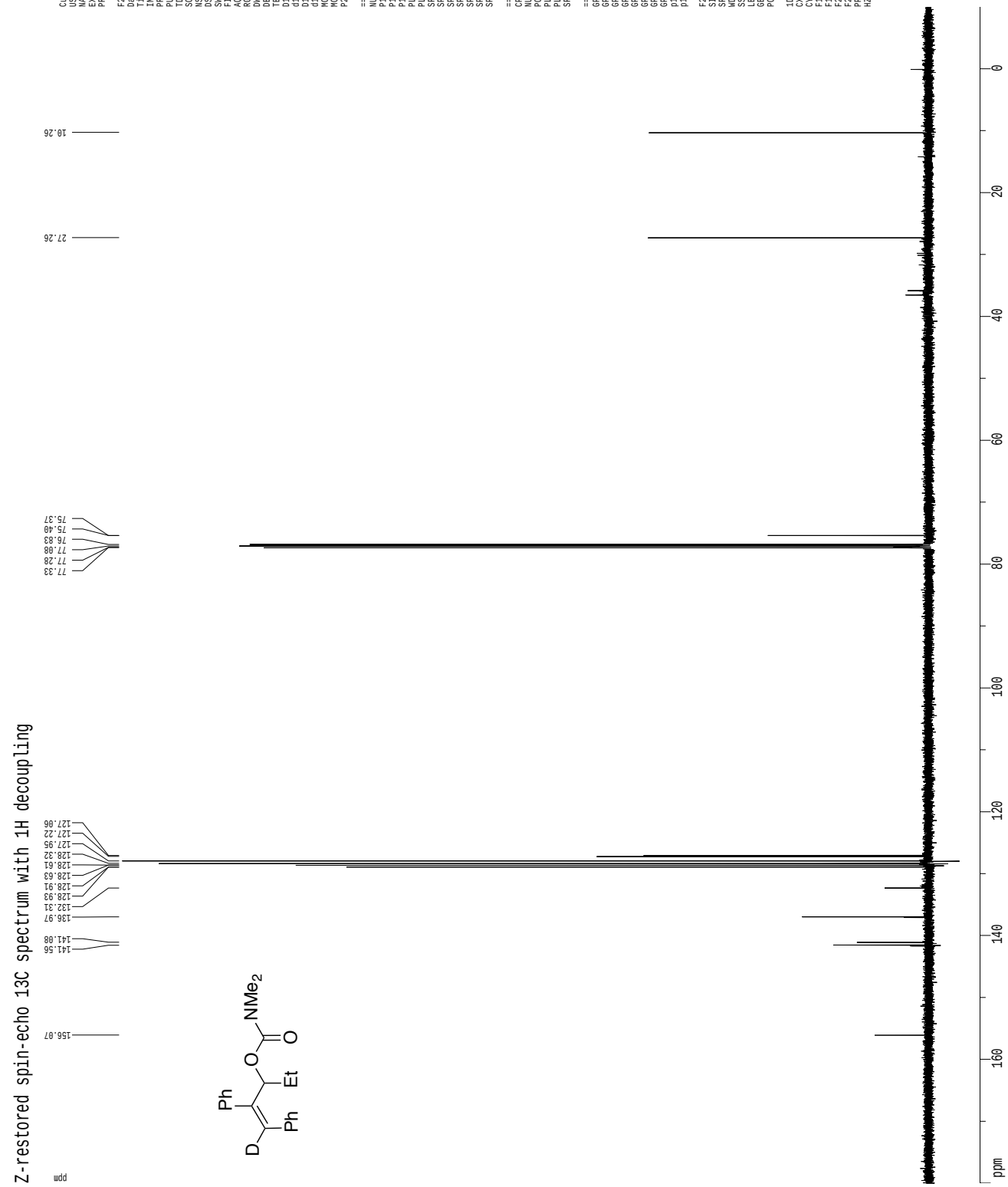
F2 - Processing parameters
SI 65536
SF 125.7803829 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

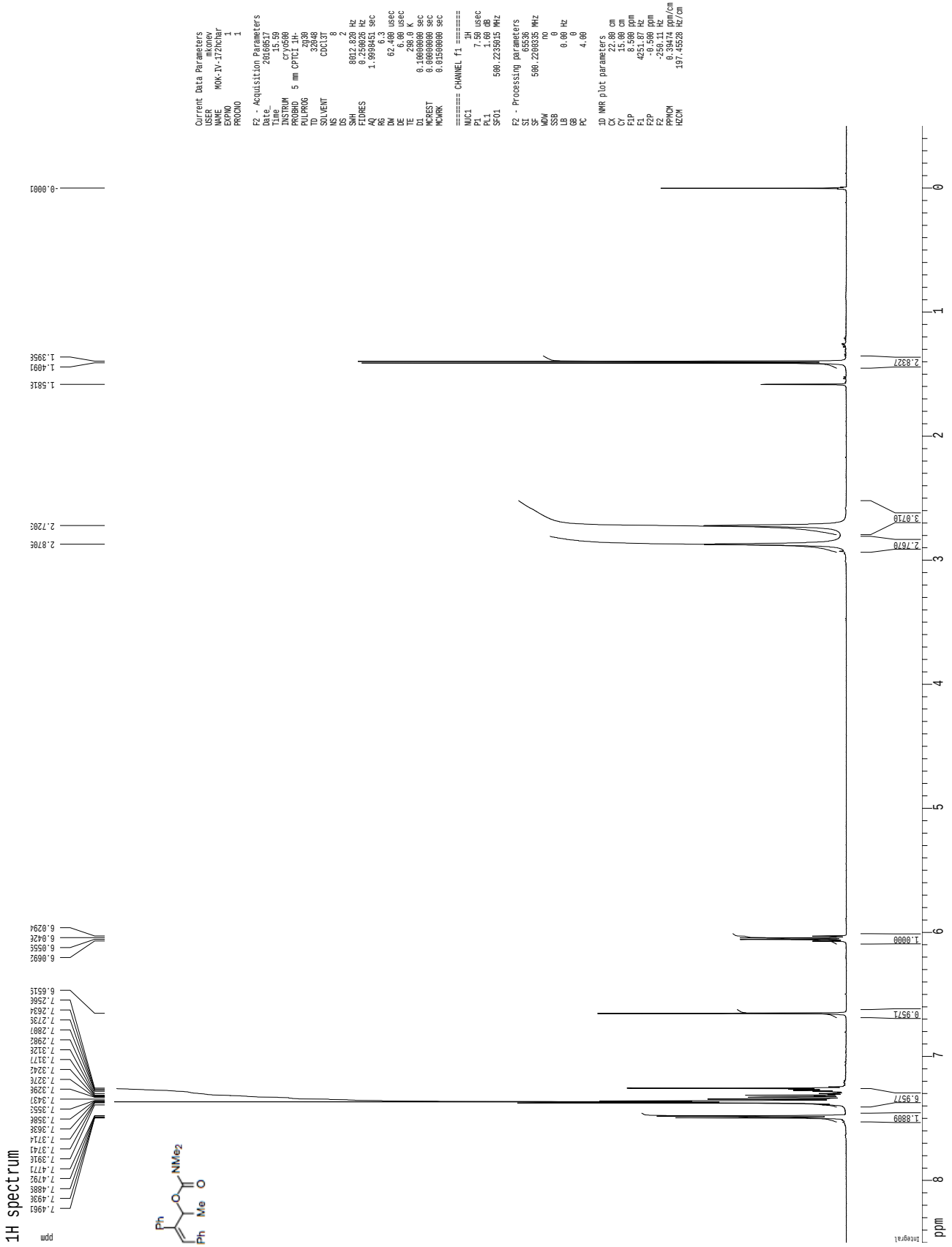
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm

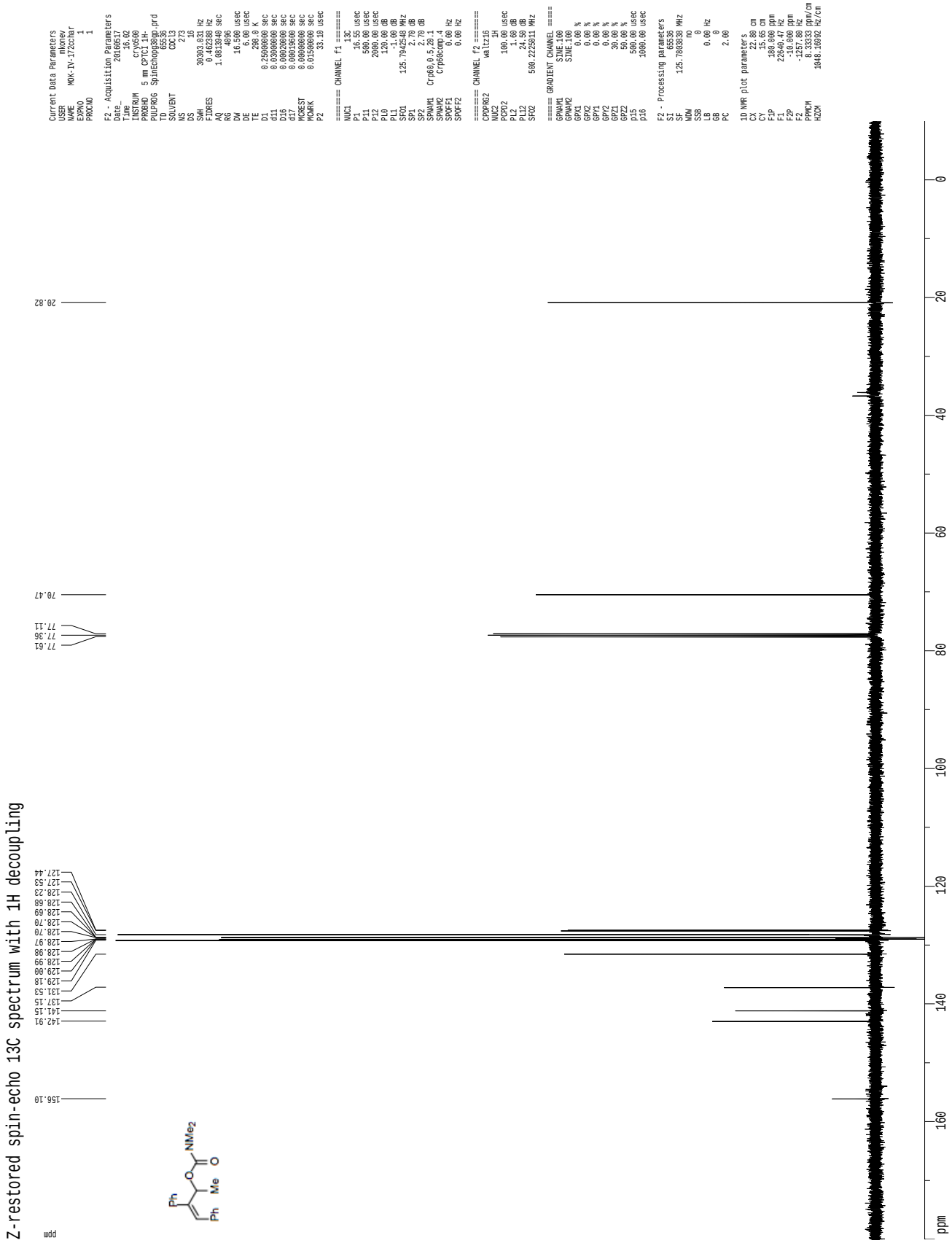

1H spectrum
Current Data Parameters
USER thanna
NAME LEH-6-228-C1-f-11
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160429
Time 9.50
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 724.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.09000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300211 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.11 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.35714 ppm/cm
HZCM 157.94608 Hz/cm
7.9616
7.9408
7.6116
7.5906
7.4226
7.4057
7.3994
7.3812
7.3616
7.2984
7.2806
7.2596
6.7656
5.8497
5.8693
5.8863
2.8825
2.7384
2.6295
1.8854
1.8693
1.8508
1.8321
1.6826
1.6656
1.6474
1.6296
1.6124
1.5652
1.3796
1.2561
0.8832
0.8716
0.8463
0.8824
1.9441
1.9249
3.9241
0.7935
0.9354
1.0000
2.9278
2.8890
3.0029
1.2200
1.5337
3.6352
Me
NMe2
O
Me
Ph
integral
ppm

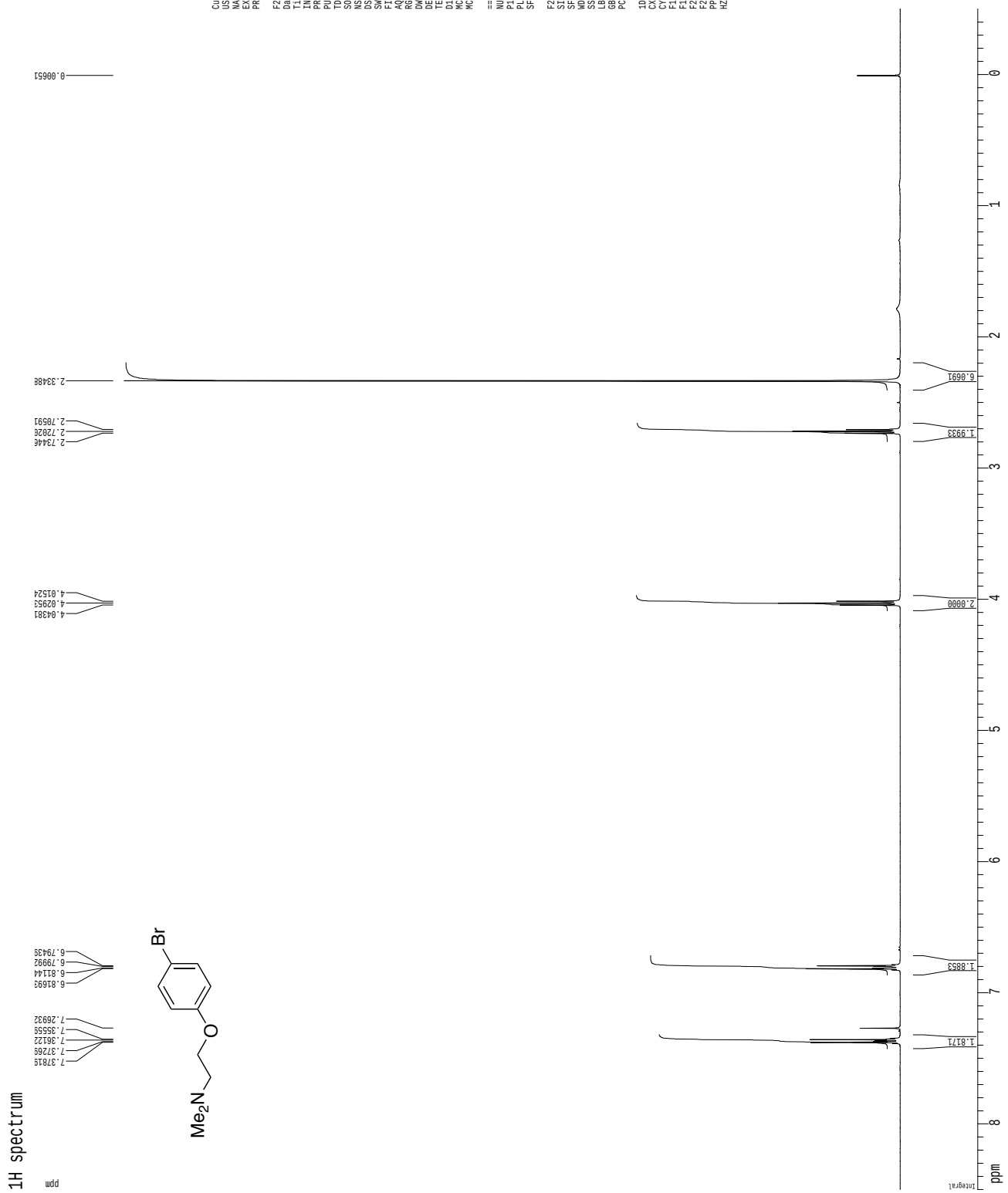

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Me
O
O
NMe2
O
Me
Ph
127.70
128.39
128.71
129.07
129.18
133.69
136.19
136.82
141.15
146.37
10.55
27.67
27.00
75.41
77.10
77.36
77.61
Current Data Parameters
USER data
NAME LEH-6-228-c1-f11-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150429
Time 14.02
INSTRUM cryo600
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 881
DS 16
SWH 30303.031 Hz
FIDRES 0.462288 Hz
AQ 1.0813940 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019900 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
======== CHANNEL f1 ========
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 1.90 dB
SFO1 125.7942454 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803829 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.610 Hz
PPMCM 13.66455 ppm/cm
HZCM 1213.67041 Hz/cm
0
20
40
60
80
100
120
140
160
180
200
ppm


1H spectrum
Current Data Parameters
USER lhanna
NAME LEH-7-009-H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160623
Time 10.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65022
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.123114 Hz
AQ 4.0895228 sec
RG 12.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.560 ppm
F2 -280.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
0.06511
0.86731
0.88217
0.89698
0.94843
1.32083
1.34372
1.48091
1.64981
1.69296
1.70685
1.72073
1.73481
1.74871
1.89078
1.90591
1.92057
1.93353
1.94855
2.79228
2.94542
5.90314
5.91848
5.93217
6.77198
7.30892
7.32283
7.33757
7.35643
7.37105
7.38526
7.40045
7.41581
7.43198
7.44681
7.46541
7.47986
7.54556
7.55962
3.3711
1.2916
1.2326
2.9113
3.0246
1.0000
0.1184
7.9325
1.8851
Integral
ppm
Ph
O
NMe2
O
Et
Ph
D


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
D
Ph
Et
O
O
NMe2
Current Data Parameters
USER thanna
NAME LEH-7-089-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160623
Time 10.40
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 1024
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7208.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7884190 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.17017 Hz/cm
10.26
27.26
75.37
75.40
76.83
77.08
77.28
77.33
127.06
127.22
127.95
128.32
128.61
128.63
128.91
128.93
132.31
136.97
141.08
141.56
156.07
0
20
40
60
80
100
120
140
160
ppm


1H spectrum
Current Data Parameters
USER mkoney
NAME MOK-IV-172hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160517
Time 15.59
INSTRUM cryo600
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32048
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.250026 Hz
AQ 1.9998451 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200335 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.1560 ppm
F1 4075.87 Hz
F2P -0.1500 ppm
F2 -250.11 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45520 Hz/cm
6.0692
6.0556
6.0426
6.0294
6.6516
7.2566
7.2634
7.2736
7.2807
7.2982
7.3126
7.3177
7.3242
7.3276
7.3298
7.3437
7.3555
7.3586
7.3636
7.3714
7.3734
7.3914
7.4171
7.4792
7.4886
7.4936
7.4961
1.3956
1.4091
1.5816
2.7203
2.8706
6.9577
1.8809
1.0000
0.9571
3.0710
2.7670
2.8327
Integral
ppm
Ph
NMe2
O
Me
Ph


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Ph
NMe2
O
Me
O
Ph
156.10
142.91
141.15
137.15
133.53
129.18
129.00
128.99
128.98
128.97
128.70
128.70
128.69
128.68
128.23
127.53
127.44
77.61
77.36
77.11
70.47
20.82
Current Data Parameters
NAME MOK-IV-172ether
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160517
Time 16.02
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchog50gp.prd
TD 65536
SOLVENT CDCl3
NS 272
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec
====== CHANNEL f1 ======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2229911 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7883838 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.10992 Hz/cm
0
20
40
60
80
100
120
140
160
ppm



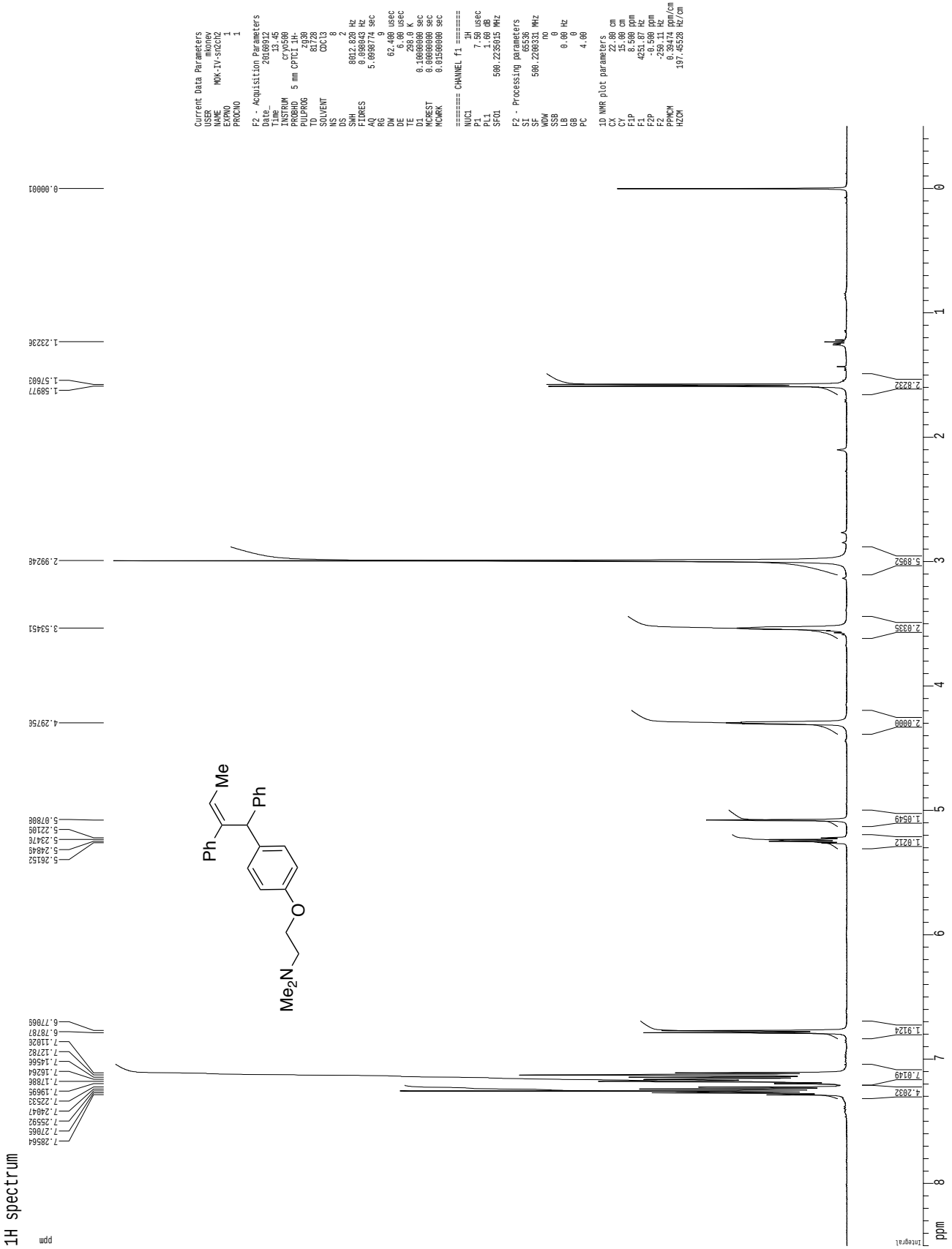
Current Data Parameters
USER mickey
NAME MOK-IV-171
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160509
Time 14.44
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250098 Hz
AQ 1.9990799 sec
RG 287.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

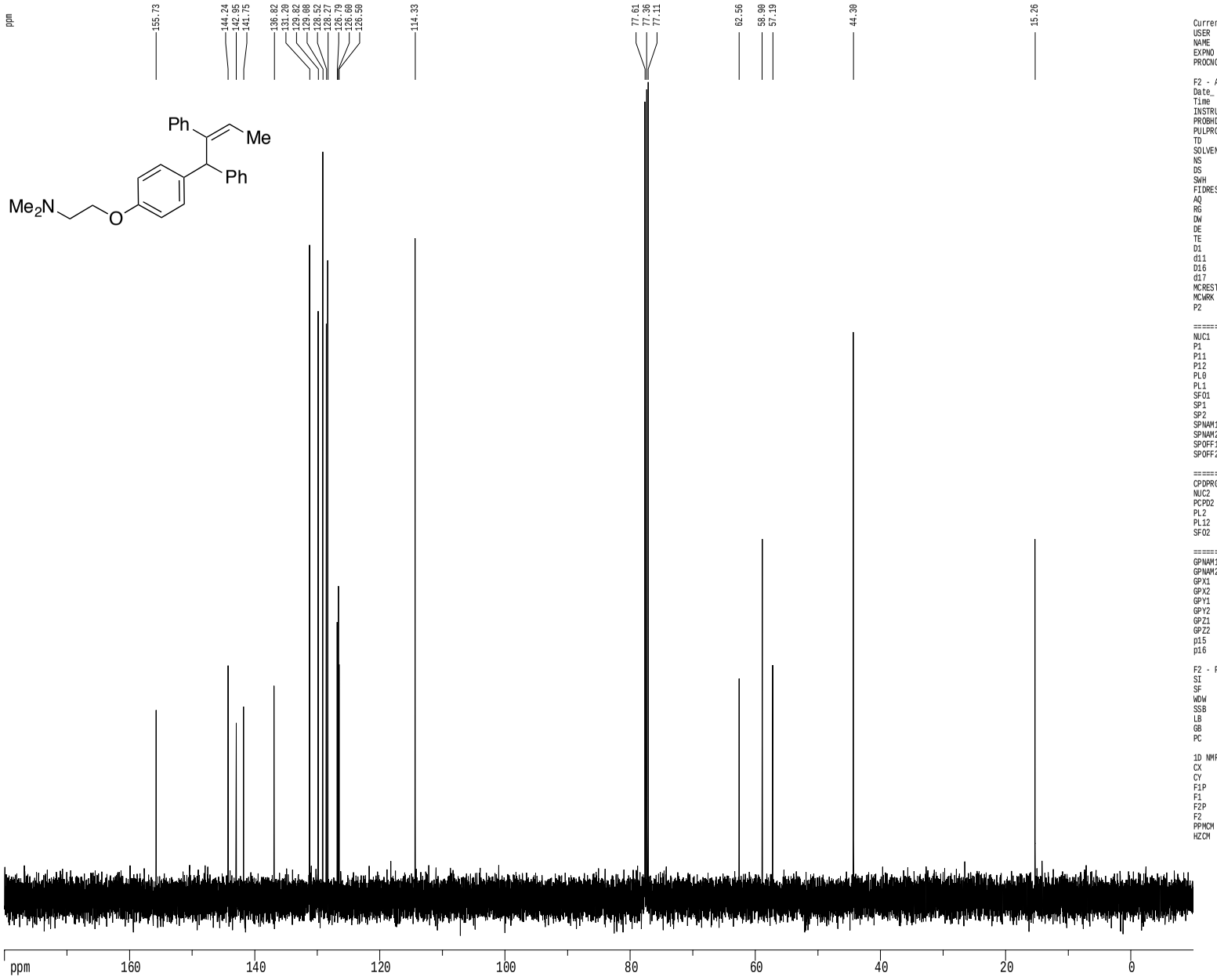
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm


1H spectrum
Current Data Parameters
USER mkonev
NAME MOK-IV-sn2ch2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160912
Time 13.45
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 7.50 usec
PL1 1.00 dB
SFO1 500.2235915 MHz
F2 - Processing parameters
SI 65536
SF 500.2200331 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 8.500 ppm
F1 4251.67 Hz
F2P -0.900 ppm
F2 -450.31 Hz
PPMCM 0.38474 ppm/cm
HZCM 197.45520 Hz/cm
0.00001
1.23236
1.57683
1.58977
2.99248
3.53451
4.29756
5.07808
5.22105
5.24776
5.24845
5.26152
6.77065
6.78787
7.11026
7.12782
7.14566
7.16264
7.17886
7.19695
7.22533
7.24047
7.25592
7.27065
7.28564
Me
Ph
Ph
O
Me2N
2.8232
5.8952
2.0335
2.0000
1.0549
1.0212
1.9124
7.0149
4.2832
integral
ppm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER mkonev
NAME MOK-IV-sn2c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160912
Time 13.43
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 79
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4597.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 33.10 usec

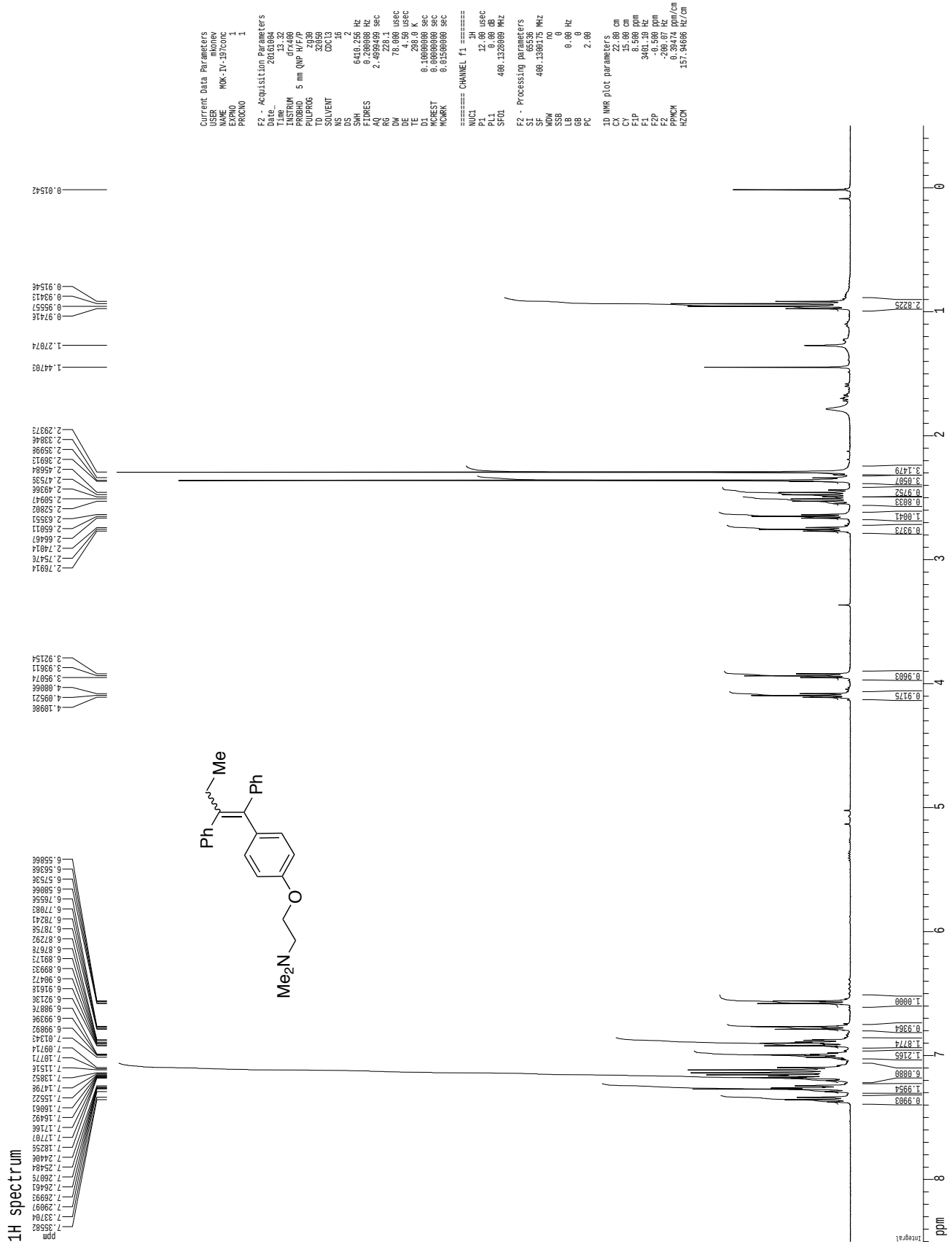
======= CHANNEL f1 =======
NUC1 13C
P1 16.55 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 2.70 dB
SP2 2.70 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803833 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 8.33333 ppm/cm
HZCM 1048.16992 Hz/cm


1H spectrum
Current Data Parameters
USER mvcorev
NAME MOK-IV-197conc
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161004
Time 13.32
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 32050
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.200008 Hz
AQ 2.4969149 sec
RG 228.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94406 Hz/cm
Me
Ph
Ph
O
Me2N
ppm
Integral