Title
SYNTHESIS AND STRUCTURE OF DICYCLOPENTENOU-RANOCENE, U[C8H6(CH2)3]2

Author
Zalkin, A.

Publication Date
1981-08-01

LBL-13216
Preprint

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

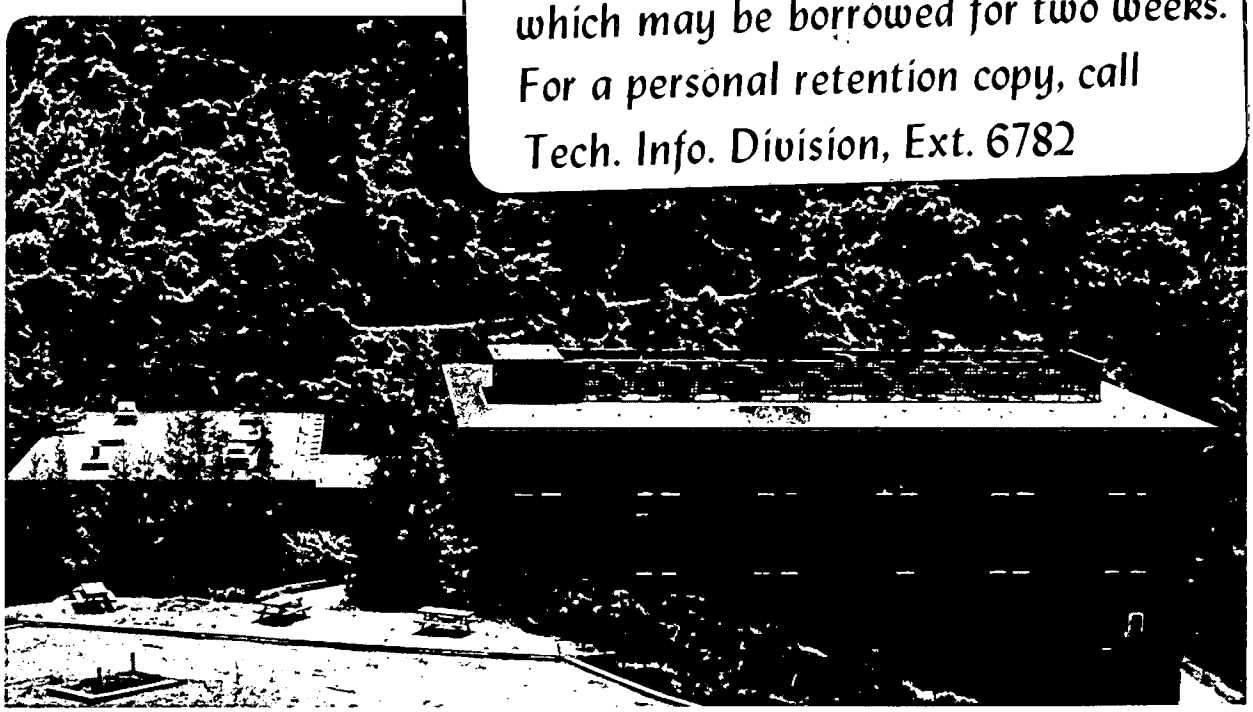
Materials & Molecular Research Division

Submitted to Organometallics

SYNTHESIS AND STRUCTURE OF DICYCLOPENTENOURANOCENE, $U[C_8H_6(CH_2)_3]_2$

Allan Zalkin, David H. Templeton, Wayne D. Luke, and Andrew Streitwieser, Jr.

August 1981



Prepared for the U.S. Department of Energy under Contract W-7405-ENG-48

DISCLAIMER

SYNTHESIS AND STRUCTURE OF DICYCLOPENTENOURANOCENE, $U[C_8H_6(CH_2)_3]_2$

Allan Zalkin,* David H. Templeton,* Wayne D. Luke and Andrew Streitwieser, Jr.*

Materials and Molecular Research Division
Lawrence Berkeley Laboratory
and
Department of Chemistry, University of California
Berkeley, California 94720

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy under Contract Number W-7405-ENG-48.

Abstract

The title compound 1 was synthesized by the reaction of dipotassium bicyclo[6.3.0]undeca-2,4,6-triene-1,8-diide, 3, and UCl_4, and its crystal and molecular structure was determined by single crystal X-ray diffraction. The compound crystallizes in the orthorhombic space group Pbca with 8 molecules in the unit cell with dimensions a = 17.393(8) Å, b = 22.468 (12) Å, c = 8.931 (4) Å. The uranium atom is located centrally between the two 8-membered rings with bond distances U-C = 2.64 ± 0.03 Å and C-C = 1.40 ± 0.02 Å. The effects of annulation on the physical properties of uranocene are discussed.

Introduction and Results

As part of a continuing study of annulated derivatives[1,2] of uranocene[3] we report the synthesis and X-ray structure determination of bis-π-(cyclopenteno-[8]annulene)uranium(IV) (dicyclopentenouranocene), 1.[4] This compound has significance for two reasons. First, it was expected to involve a conformation sufficiently well defined to assist nmr interpretation. Second, it would be the first X-ray structure of an unstrained uranocene in which the ring-U axis is not a C_2 symmetry axis; the position of the uranium was therefore expected to provide a significant indication of the relative roles of covalent and ionic bonding. The title compound was synthesized in a manner similar to dicyclobutenouranocene,[2] shown in Scheme 1. The bicyclotriene 2 was prepared by ether addition of 1,3-dibromopropane, or the dimethanesulfonate of 1,3-propanediol, to a solution of dilithium cyclooctatrienediide in liquid ammonia, affording distilled yields of 46.8% and 58.4%, respectively. This compound was contaminated by the tricyclic isomer 4; on standing, 2 rearranged completely to 4. Dideprotonation of 2 with potassium amide in THF/liquid ammonia formed a red solution of the dianion, 3, which could be isolated as an impure tan solid. Subsequent reaction of 3 with UCl_4 in THF produced the desired uranocene, 1, in 15.9% yield. The visible spectrum of this green air-sensitive material exhibited the typical uranocene cascade of four principal absorptions at 632, 656, 663 and 680 nm. The pricipal IR absorption bands are compared with those of uranocene in Table I.

The ^{1}H NMR (toluene-$\underline{d}_8$) spectrum of $\underline{1}$ at 30°C showed six sharp well-resolved resonances upfield from TMS, -8.3 ppm (m, 2H), -18.8 ppm (m, 4H), -23.1 ppm (s, 4H), -32.6 ppm (m, 2H), -34.2 ppm (s, 4H), -41.2 ppm (s, 4H), and one resonance downfield from TMS, +24.4 ppm (m, 4H). The ^{13}C NMR spectrum (dioxane-$\underline{d}_8$) at 39°C showed four broad peaks at 308.0, 296.9, 279,0, and 268.5 ppm downfield from TMS and two sharp peaks at 13.4 and -32.5 ppm.

Magnetic susceptibility measurements on the bulk solid from 2.4 to 95.6°K are shown in Fig. 1. Above 20°K the magnetic susceptibility follows the Curie-Weiss Law with C=0.743 ± 0.005 emu °K mol^{-1} μ=2.4 ±0.1 B.M. and θ=16.6° ± 0.5°. Below 10°K the magnetic moment was independent of temperature with $\chi m=2.56 \pm 0.03 \times 10^{-2}$ csu/mole. Using a diamagnetic correction of -187×10^{-6} emu mol^{-1} the corrected values are C=0.714 ± 0.005 emu °K mol^{-1}, μ=2.4 ± 0.1 B.M. and θ=16.1° ± 0.5°.[5]

The compound crystallizes in the orthorhombic space group Pbca with 8 molecules in the unit cell with dimensions $\underline{a}$=17.393(8) Å, $\underline{b}$=22.468(12) Å and $\underline{c}$=8.931(4) Å. With a molecular weight of 526.46 the calculated density is 2.004 g cm^{-3}. The structure was determined by conventional single crystal X-ray methods and was refined by full-matrix least-square to an R factor of 0.04 for 1000 data where $F^2>3\sigma$. Final positional parameters are given in Table II. Tables of calculated positional parameters for the hydrogen atoms, carbon-carbon distances, thermal parameters, and observed structure factors amplitudes are given in the Supplementary Material. Uranium-carbon distances are listed in Table III. The atom numbering is seen in Figures 2 and 3.

Discussion

The title compound exists as discrete molecules in the solid state. All atoms are in the general positions and no symmetry is imposed on the molecule by the space group; however, the molecule does have approximate C_2 symmetry (see Fig. 3). The uranium atom is centrally sandwiched between the two 8-membered rings with a U-ring distance of 1.92 Å, in good agreement with other uranocenes.[1,6,7,8] The COT rings of the molecule are rotated about 8° from a staggered configuration (see Fig. 3). In other uranocenes both staggered[11,12] and eclipsed[1,10,11] configurations have been reported. As in ferrocenes, the relative orientation of the rings in uranocene crystals results from crystal packing and not from significant steric interactions between the rings which are 3.85 Å apart. Bond angles and distances for the uranocene part of the molecule are similar to those reported for other uranocenes.[1,6,7,8] The mean planes of carbons, (C_{11}, C_9, C_1, C_8) and (C_{20}, C_{22}, C_{12}, C_{19}), are bent slightly inwards toward the uranium atom from the plane of the 8-membered ring as has been found in dicyclobutenouranocene[1] and in 1,3,5,7,1',3',5',7-octamethyluranocene.[7] Similar convex distortions in the carbocyclic rings have been observed in ferrocenes, chromacene[9] and in a variety of organometallic compounds with planar five, six- and eight-membered rings.[10]

Unrealistic bond distances (1.38 to 1.46 Å) and very large and anisotropic thermal parameters in the 5-membered rings indicate that we are observing the mean positions of atoms which have disorder up and down from the mean planes, a situation which is commonplace for aliphatic 5-membered rings.

This bending appears to be a general feature in sandwich structures where steric interactions between rings is small. Two possible explanations have been offered to account for this bending:[7] 1) bending the substituent toward the metal makes each carbon slightly more pyramidal with the π orbital of the C_nH_n ring bent inward toward the metal and providing greater directionality for overlap between π orbitals and metal orbitals; 2) contraction in volume of the electron density on the side of the ring adjacent to the highly charged metal ion. Non-bonding interaction between the substituted bond and the more diffuse electron density on the uncomplexed side of the ring would result in an inward bend of the substituent. This latter explanation suggests that the inward bending should be independent of ring size. However, it appears that for 3- and 4-membered rings the substituents bend outward away from the metal.[10] Theoretical calculations on d-transition metal compounds attribute this bending to the former explanation and predict an outward bend for 3- and 4-membered ring and an inward bend in the substituent for rings larger than 5.[10] This bending of the substituent in toward the metal appears to be a general feature in all substituted uranocenes in the absence of steric effects and may reflect the proposed covalency in ligand-metal bonding in these systems.[11]

An especially important point in this regard is the central position with respect to the C_8 rings of uranium in all uranocene structures thus far studied. This result is expected for a model involving important covalent ring-metal bonding. If ionic

character dominated we would expect some deviations from C_8-centrosymmetry, particularly in a case such as the present where the overall structure is so lacking in symmetry. This result makes the present structure an especially significant one.

The spectral properties of 1 show no significant differences from those of other uranocenes. In the visible spectrum the shift in λ_{max} from that of uranocene, (λ_{max} 616 nm), is between that of 1,1'-dialkyl substituted uranocenes, (λ_{max} 610-625 nm), and 1,3,5,7,1',3',5',7'-octamethyluranocene (λ_{max} 650 nm), in accord with the proposed charge transfer model for the visible spectrum.[12,13]

The paramagnetic shifts of the ^{1}H NMR resonances of 1 are similar to those reported for other substituted uranocenes.[11] Due to the paramagnetic center, the endo and exo protons in the α and β positions of the annulated rings are non-equivalent. The three non-equivalent ring proton positions could be assigned to the resonances at -32.1, -34.2 and -41.2 ppm, from the lack of splitting due to J-J coupling and their larger line widths (ca. 30 Hz). The remaining four resonances were partially resolved multiplets and could be easily differentiated into α (+24.4, -18.8 ppm) and (-8.3, -32.6 ppm) sets by integration. Further assignment into endo and exo sets was made in the following manner.

In uranocene[14] and 1,3,5,7,1',3',5',7'-octamethyluranocene[15] the paramagnetic shifts have been factored into contact and pseudo contact components.[11] The contact component affords an upfield shift to ring protons, and an alternating pattern of upfield and downfield shifts to substituent protons such that protons on the

β, δ , etc., carbons are shifted upfield and protons on the α, γ , etc., carbons are shifted downfield, with the magnitude of the contact interaction rapidly diminshing to zero along the substituent chain. Variable temperature studies on uranocene and octamethyl-uranocene have established that the total paramagnetic shift is linear with 1/T. Consequently, the pseudo-contact contribution can be expressed by

$$\Delta_{\text{Pseudo-contact}} = \frac{(\chi_z - 1/2\,\chi_x - 1/2\,\chi_y)}{3}\frac{(3\cos^2\theta-1)}{R^3} - \frac{(\chi_x - \chi_y)}{2}\frac{(\sin^2\theta\cos 2\psi)}{R^3} \quad (1)$$

Where symmetry requires $\chi x = \chi y$, the latter term is zero.[16]

For a number of unsymmetrical uranocenes it appears that $\chi_x = \chi_y$ and that the last term in eq. 1 can be generally neglected.[11] With this assumption, the sign of the pseudo-contact shift will be controlled by θ. Similar to dicyclobutenouranocene we expect the contact shift of the α_{exo} and α_{endo} protons to be approximately the same; calculation from the observed structure gives $\theta < 54.74°$ for the α_{exo} protons and thus, the pseudo-contact shift is downfield. Hence, the α proton resonances are assigned as α_{exo} + 24.4 ppm and α_{endo} -18.8 ppm; by analogy, the β-proton resonances are β_{exo} 8.3 ppm and β_{endo} -32.6 ppm.

For ^{13}C NMR the contact shift model for uranocene[15] predicts that the contact shifts for carbons should be just opposite those of the corresponding protons. Ring carbons are shifted to low field, and are assigned tentatively to the four resonances at 308.0, 296.9, 297.0, 268.5 ppm. Correspondingly, the α carbon is assigned to the upfield resonance (-32.5 ppm) and the β carbon to the 13.4 ppm resonance.

The similarity of the ^{1}H and ^{13}C resonances in 1 with those of other substituted uranocenes[12,13] and the parent compound[12] itself suggest that the electronic factors giving rise to the paramagnetic shifts are similar in all uranocenes. Further support for this generalization comes from the magnetic susceptability data which shows that the magnetic moment of 1 is equal within experimental error to that of uranocene.[5,17] Further analysis of the nmr spectra of 1 and related annulated uranocenes, their temperature dependences and dissection into contact and pseudocontact components will be detailed in another paper now in preparation.

Experimental

All reactions requiring air-free anhydrous conditions were conducted under an Ar atmosphere or in a Vacuum Atmospheres recirculating glove box. Solvents were distilled from CaH_2 and degassed prior to use. Visible spectra were obtained on a Cary 118 spectrometer, infra-red spectra on a Perkin Elmer 297 spectrometer, ^{1}H and NMR on Varian T-60 (60 MHz) or the V.C. Berkeley FT-NMR system (180 MHz), and ^{13}C NMR on a Brucker TT-23 spectrometer. Mass spectra and elemental analysis were performed by the Analytical Services Laboratory, University of California, Berkeley. Magnetic susceptability measurements were measured on a vibrating-sample magnetometer previously described in the literature.[17]

X-ray diffraction. Single crystals suitable for X-ray analysis were grown from hot hexane in an Ar atmosphere glove box. A crystal fragment approximately 0.10 x 0.15 x 0.35 mm was placed on a Picker FACS-I automated diffractometer equipped with graphite monochromated MoKα radiation, (λ=0.70930 Å). The setting angles of 12 manually centered reflections (35° < 2θ < 40°) were used to determine the cell parameters by least-squares.

Intensity data were collected using the θ-2θ scan technique with a scan speed of 2°/min on 2θ. Each reflection was scanned from 0.65° before the $K\alpha_1$ peak to 0.65° after the $K\alpha_2$ peak, and backgrounds were counted for 4s at each end of the scan range, offset by 0.5°. The temperature during data collection was 22 ± 1°C. Three standard reflections (6,0,0; 0,0,$\bar{4}$; 0,10,0) were measured after every 200th scan. At the beginning of the data collection ω scans of the 400, 060, and 002 reflections showed half-widths of 0.09°, 0.07°, and 0.09° respectively; at the end of the data collection the values were 0.18, 0.12 and 0.11, indicating significant deterioration of the crystal. Although 6043 scans were collected, only the first 3010 were used; the remaining half of the data, based on the declining intensities of the standards, were rejected. The decay was anisotropic with the largest amount recorded by the 600 standard reflection. An isotropic decay correction varying from 0.91 to 1.06 was applied to the 3010 data used. An absorption correction[18] was estimated and corrections between 1.5 and 1.8 were applied to the data. Because the crystal had an irregular shape with indistinct faces, a shape and size

were estimated, and the dimensions were tailored to fit the intensity variations obtained from three azimuthal scans taken after the data collection was concluded. The 3010 scans resulted in 2312 unique data 1000 of which have $F^2 > 3\sigma$.

A three-dimensional Patterson function calculation revealed the uranium position, and subsequent least-squares calculations and Fourier maps revealed all of the carbon atoms. A series of least squares in which the function $\Sigma w(|Fo|-|Fc|)^2/\Sigma wF_o^2$ was minimized converged rapidly to the final structure. The expressions that were used in processing the data and estimating the weights are given in the supplementary material; the "ignorance factor", p, was set to 0.06. Scattering factors from Doyle and Turner[19] were used, and anomalous dispersion corrections[20] were applied. The positions of all of the hydrogen atoms were estimated and included in the calculations with isotropic temperature factors, but were not refined. Anisotropic thermal parameters were applied to U and all of the carbon atoms.

Because of the low quality of the data, the cyclooctatetraene (COT) ring was not well resolved, and some of the C-C bond distances deviated from the expected values by as much as 0.2 Å. Restraints were imposed on the bond distances in the COT ring and the cyclopenteno group adjacent to the ring in the following manner:[21] Interatomic distances between selected atoms were introduced into the least-squares calculations and treated as observations; estimated standard deviations of these distances were also introduced and used to calculate the weights. The derivatives of these

distances with respect to the positional parameters were calculated by a special patch and these "observations" were not included in the least-squares calculation in the same manner as the observed structure factors. This procedure allows the structure to adjust to the electron density with a flexibility governed by the weighting. The C-C bond distances within the ring were restrained to 1.40± 0.02 Å and the C-C bonds from the cyclopento carbon to the ring carbons were restrained to 1.54 ± 0.02 Å.

The discrepancy indices for 1000 data where $F^2 > 3\sigma$ are

$$R = \Sigma||F_o|-|F_o||/\Sigma|F_o| = 0.040$$

$$R_w = [\Sigma w|F_o|-|F_o|)^2/\Sigma w|F_o|^2]^{1/2} = 0.046$$

R for all 2332 data is 0.14. The error in an observation of unit weight is 1.07. In the last cycle no parameter changed more than 0.13 σ. The top three peaks in the final difference Fourier map are 1.0 to 1.3 e A^{-3} and are all ripples about the uranium atom.

cis-Bicyclo[6.3.0]undeca-2,4,6-triene, 2. Under an Ar atmosphere, 1.4 g (0.2 mol) of lithium wire (1% sodium) was added to 300 mL of anhydrous liquid ammonia in a 500 mL round bottom flask. To this deep blue solution was added 10.4 g (0.1 mol) of freshly distilled cyclooctatetraene (33°C/14 mm Hg) (BASF), at -40°C, via syringe. The resulting yellow suspension of dianion was stirred for 1.5 hr at -40°C followed by dropwise addition (1 drop every 2 seconds) of 20.2 g (0.1 mol) of 1,3-dibromopropane

(Aldrich) in 20mL of ether. The reaction mixture was stirred for 4.5 h at ca. -40°C followed by overnight evaporation of ammonia to afford a red brown solid which was suspended in 200 mL of saturated ammonia chloride and extracted with ether (4x100 mL). The ether extracts were washed with water (3x100 mL) and dried over $MgSO_4$. Removal of solvent followed by vacuum distillation (25°C/0.1 mm Hg) yielded 6.83 g (46.8%) of a clear yellow liquid; mass spectrum parent m/e = 156. The ^1H and ^{13}C NMR ($CDCl_3$) of this material indicated that it was mixture of both the bicyclic (2) and tricyclic (4) valence isomers; bicyclic ^1H NMR: δ 5.80 (s, 6H, vinyl), 2.77 (br m. 1.4H, bridgehead), and 1.65 (complex m, 5.3H cyclopentyl); ^{13}C NMR: δ 135.4, 127.7, 126.2 (vinyl), 43.5 (bridgehead), 32.3, 19.5 (cyclopentyl); tricyclic ^1H NMR ($CDCl_3$): δ 5.57 (s, 4H, vinyl), 2.77 and 2.43 (br m, 2H, bridgehead), 1.67 (complex multiplet, 5.3H, cyclopentyl); ^{13}C NMR: δ 126.6, 120.3 (vinyl), 51.0, 35.6 (bridgehead), 32.9, 24.7 (cyclopentyl). After standing for 4 days at room temperature the material had rearranged completely to the tricyclic isomer, 4.

Following the same procedure but using 23.2 g (0.1 mol) of 5 in 100 mL of THF instead of 1,3-dibromopropane in 10 mL of ether, gave the same product in 58.4% yield.

<u>$AgNO_3$ adduct of cis-bicyclo[6.3.0]undeca-2,4,6-triene</u>. To a boiling solution of 3.4 g (0.02 mol) of silver nitrate and 15 mL of abs ethanol was added 2.9 g (0.02 mol) of 2. Most of the silver nitrate dissolved upon addition of the hydrocarbon. Cooling of the solutuion is a refrigerator for several hrs afforded

off-white crystals which were recrystallized from absolute ethanol; mp 135-136°C dec.

Anal. Calcd. for $C_{11}H_{14}NO_3Ag$: C, 41.79; H, 4.46; N, 4.43. Found: C, 41.48; H, 4.35; N, 4.29.

1,3-Bis(methylsulfonyloxy)propane. To a stirred 0°C solution of 15.22 g (0.2 mol) of 1,3-propanediol (Aldrich), 70 mL (0.5 mol) of triethylamine and 1 L of CH_2Cl_2 in a 2 L round bottom flask was added dropwise 32.5 mL (0.42 mol) of methanesulfonyl chloride (Eastman) over 5 min. The resulting reaction mixture was stirred for 0.5h during which time a white ppt formed. After sequential extractions with 200 mL of ice water, 200 mL of cold 10% HCl, 200 mL of saturated sodium bicarbonate and 200 mL of brine, the organic layer was dried over $MgSO_4$ and stripped of solvent to afford a white solid which was recrystallized from hot methanol; yield 38.4 g (82.7%), mp 40.5-41.5°C; NMR ($CDCl_3$): δ 4.37 (t, 4H, $-OCH_2-$), 3.07 (s, 6H, $-CH_3$), 2.19 (p, 2H, $-CH_2-$).

Anal. Calcd. for $C_5H_{12}O_6S_2$: C, 25.85; H, 5.21; S, 27.61. Found: C, 26.04, H, 5.26, S, 27.42.

Dipotassium Bicyclo[6.3.0]undeca-2,4,6-triene-1,8-diide, 3. Under Ar a suspension of potassium amide in liquid ammonia was prepared by distilling 300 mL of ammonia from a lithium metal-ammonia solution into a 500 mL round bottom flask containing several mg of anhydrous $FeCl_3$. Subsequent addition of 2.14 g (0.055 mol) of potassium metal at -40°C afforded a blue solution which was stirred (ca 5 min) until the blue color disappeared indicating formation of the amide. A 4.0 g (0.027 mol) aliquot

of 2 was added via syringe and the resulting red brown solution was stirred for 1.5 h at -35°C. The solution was slowly warmed to room temperature and the ammonia was swept out with a steady Ar purge overnight, affording 4.66 g of the crude dianion as a highly air sensitive grey brown solid; NMR (THF-d_8): ^{1}H δ 5.67, 1.3, 0.93, broad singlets; ^{13}C δ 99.9 (quat), 96.9, 89.8, 87.0 (ring), 46.4 (α CH_2), 27.2 (β CH_2). No attempt was mady to purify this material.

Dicyclopentenouranocene, 1. In an Ar atmosphere glove box 2.56 g (0.0068 mol) of UCl_4 in 25 mL of THF was added to a solution of 3.0 g (0.0135 mol) of 3 in 100 mL of THF in a 500 mL round bottom flask, and the resulting green solution was stirred for 18 h. Removal of the solvent by vacuum transfer afforded a green solid which was purified by Soxhlet extraction with hexane; yield 0.57 g (15.9%); mass spectrum, parent peak m/e 526; visible spectrum in hexane, nm ($\varepsilon x10^3$) 632(2.1), 656(0.9), 663(0.9), 680(0.7). The ir spectrum is given in Table I and nmr spectra are discussed in the text.

Acknowledgment

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy under Contract Number W-7405-ENG-48.

References

(1) Zalkin, A.; Templeton, D. H.; Berryhill, S. R.; Luke, W. D. Inorg. Chem. 1979, 18, 2287.

(2) Luke, W. D.; Berryhill, S. R.; Streitwieser, Jr., A. Inorg. Chem., in press.

(3) Streitwieser, A. Jr.; Muller-Westerhoff, U.; Sonnischen, G.; Mares, F.; Morrell, D. G.; Hodgson, K. O.; Harmon, C. A. J. Am. Chem. Soc. 1973, 95, 8644.

(4) Controversy exists in naming sandwich organometallic actinide compounds of cyclooctatetraene. Other possible names are bis-(η^8-bicyclo-[6.3.0]-undeca-2,4,6-triene-1,8-diide)uranium (IV) and bis-(η^8-cyclopentenocyclooctatetraene)uranium(0). The former does not properly describe delocalization of charge in the ligand while the latter fails to convey the proper formal oxidation state of the metal or the delocalized nature of the cyclooctatetraene ligand. Thus, we have chosen to name these compounds organometallic derivatives of [8]-annulene or as substituted uranocenes in analogy with ferrocene nomenclature.

(5) Karraker, D.; Stone, J.; Jones, E. Jr.; Edelstein, N. J. Am. Chem. Soc. 1970, 92, 4841.

(6) Zalkin, A.; Raymond, K. N. J. Am. Chem. Soc. 1969, 91, 5667.

(7) Hodgson, K. O.; Raymond, K. N. Inorg. Chem. 1973, 12, 458.

(8) Templeton, L. K.; Templeton, D. H.; Walker, R. Inorg. Chem. 1976, 15, 3000.

(9) Haaland, A. Fortschr. Chem. Forsch. 1976, 53, 1, and references therein.

(10) Elian, M.; Chen, M. M. L.; Mingos, D. M. P.; Hoffman, R. Inorg. Chem. 1976, 15, 1148.

(11) Luke, W. D.; Streitwieser, A. Jr. ACS Symposium Series, No. 131, "Lanthanide and Actinide Chemistry and Spectroscopy", Edelstein, N. M. Ed., p. 93.

(12) Streitwieser, A. Jr.; Harmon, C. A. Inorg. Chem. 1973, 12, 1102.

(13) Harmon, C. A.; Bauer, D. P.; Berryhill, S. R. Hagiwara, K.; Streitwieser, A. Jr. Inorg. Chem. 1977, 16, 2143.

(14) Edelstein, N.; La Mar, G. N.; Mares, F.; Streitwieser, A. Jr. Chem. Phys. Lett. 1971, 8, 399.

(15) Streitwieser, A. Jr.; Dempf, D.; LaMar, G. N.; Karraker, D. G.; Edelstein, N. J. Am. Chem. Soc. 1971, 93, 7343.

(16) The symbols θ, ψ and R refer to the spherical polar coordinates of the resonating nucleus in a coordinate system whose origin is centered at the metal atom.

(17) Edelstein, N.; Streitwieser, A. Jr.; Morrell, D. G.; Walker, R. Inorg. Chem. 1976, 15, 1397.

(18) Templeton, L. K.; Templeton, D. H. Abstracts, American Crystallographic Association Proceedings, 1973, Series 2, Vol. 1, p. 143.

(19) Doyle, P. A.; Turner, P. S. Acta Crystallogr. 1968, 24, 390.

(20) Cromer, D. T.; Waber, J. T. Acta Crystallogr. 1965, 18, 104.

(21) Waser, J. Acta Crystallogr. 1963, 16, 1091.

(22) Goffart, J.; Fuger, J.; Gilbert, B.; Kanellakopulos, B.; Duychaerts, G. Inorg. Nucl. Chem. Lett. 1972, 8, 403.

(23) Streitwieser, A. Jr.; Muller-Westerhoff, U.; Sonnichsen, G.; Mares, F.; Morrell, D. G.; Hodgson, K. O.; Harmon, C. A. J. Am. Chem. Soc. 1973, 95, 8644.

(24) Hocks, L.; Goffart, J.; Duychaerts, G.; Teyssie, P. Spectrochimica Acta 1974, 30A, 907.

(25) Goffart, J.; Fuger, J.; Brown, D.; Duychaerts, G.; Inorg. Nucl. Chem. Lett. 1974, 10, 413.

Supplementary Material Available

Data processing formulas, calculated positional parameters for the hydrogen atoms, anisotropic thermal parameters, carbon-carbon distances, a table of selected antles, and a list of observed structure factors (14 pages). Ordering information is given on any current masthead.

Table I. Infrared Spectrum (cm^{-1}) in NUJOL

$(C_8H_8)_2U$ [≠]	$[C_8H_6(CH_2)_3]_2U$, [1] ≃
	1870 (w)
1730 (w)	1765 (w)
	1320 (m)
1262 (s)	1260 (m)
1095 (s)*	1090 (m)*
1018 (s)*	1020 (m)*
	900 (s)
799 (s)	790 (m)
	745 (s)
720 (s)	700 (vs)

(a) w = weak; m = medium; s = strong; vs = very strong

(b) Refs. 11, 12, 22-25.

(c) very broad

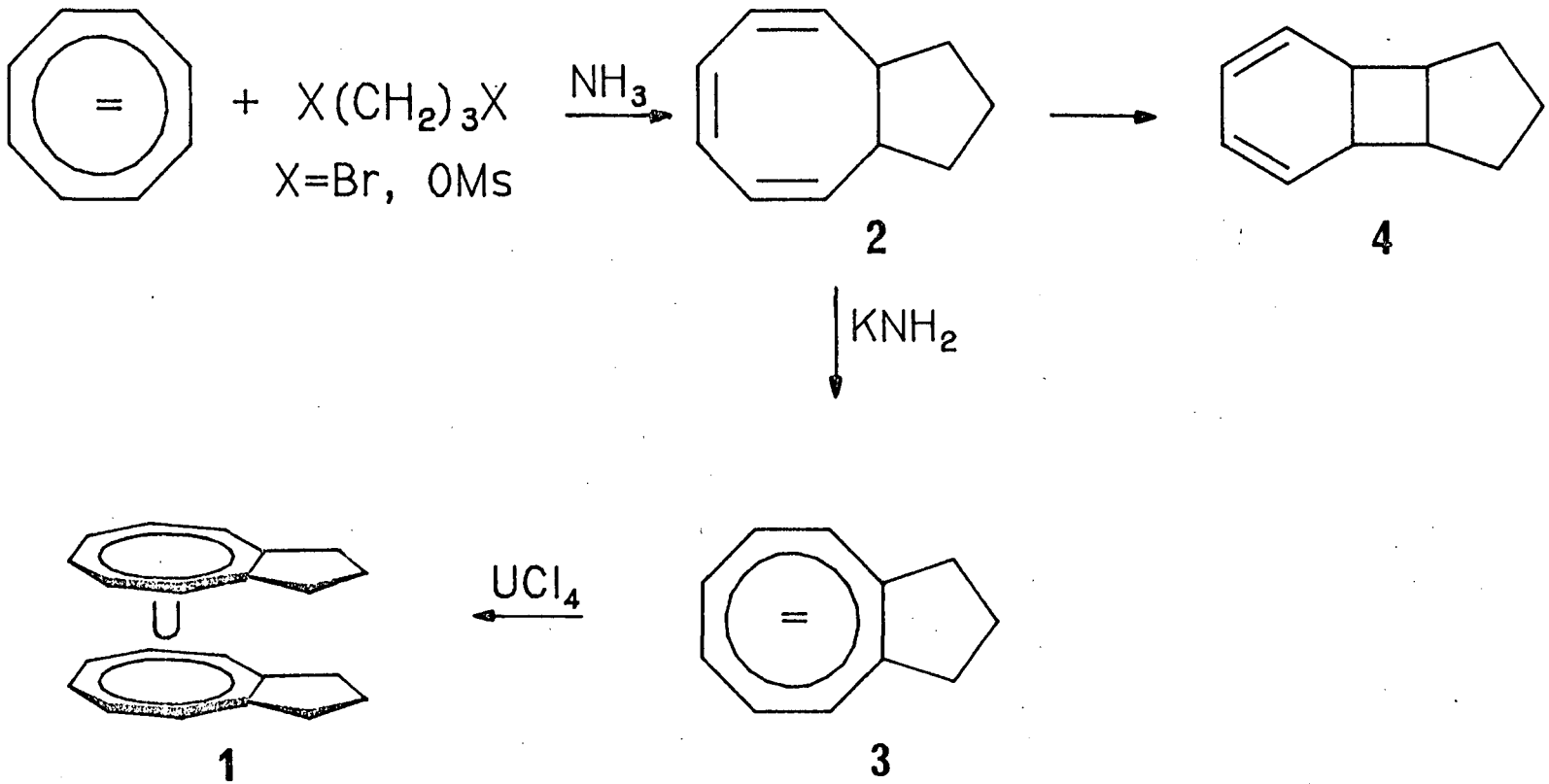
Table II. Positional Parameters[a]

Atom	x	y	z
C(1)	.047(2)	.163(1)	.159(3)
C(2)	.077(2)	.122(1)	.264(3)
C(3)	.147(2)	.109(2)	.329(4)
C(4)	.218(2)	.138(1)	.309(3)
C(5)	.254(2)	.182(2)	.225(4)
C(6)	.228(2)	.220(2)	.115(4)
C(7)	.153(2)	.2305(9)	.061(3)
C(8)	.079(2)	.209(1)	.074(3)
C(9)	.014(2)	.230(1)	-.032(3)
C(10)	-.051(3)	.205(3)	.029(6)
C(11)	-.038(2)	.152(2)	.122(6)
C(12)	.285(1)	.098(1)	-.160(3)
C(13)	.222(2)	.116(1)	-.245(3)
C(14)	.142(2)	.111(2)	-.258(4)
C(15)	.099(2)	.067(2)	-.184(4)
C(16)	.105(2)	.022(1)	-.078(4)
C(17)	.168(2)	-.003(1)	-.004(3)
C(18)	.245(1)	.014(1)	.016(2)
C(19)	.294(1)	.054(1)	-.052(2)
C(20)	.378(1)	.058(2)	.005(3)
C(21)	.407(2)	.111(2)	-.061(5)
C(22)	.361(2)	.132(1)	-.182(4)
U	.17237(5)	.11510(4)	.0369(1)

[a] Here and in the following tables the number in parenthesis is the estimated standard deviation in the least significant figure. The estimated standard deviations are a result of least squares refinement on a model in which C-C distances were restrained as described in the text. Hydrogen atoms were included but not refined.

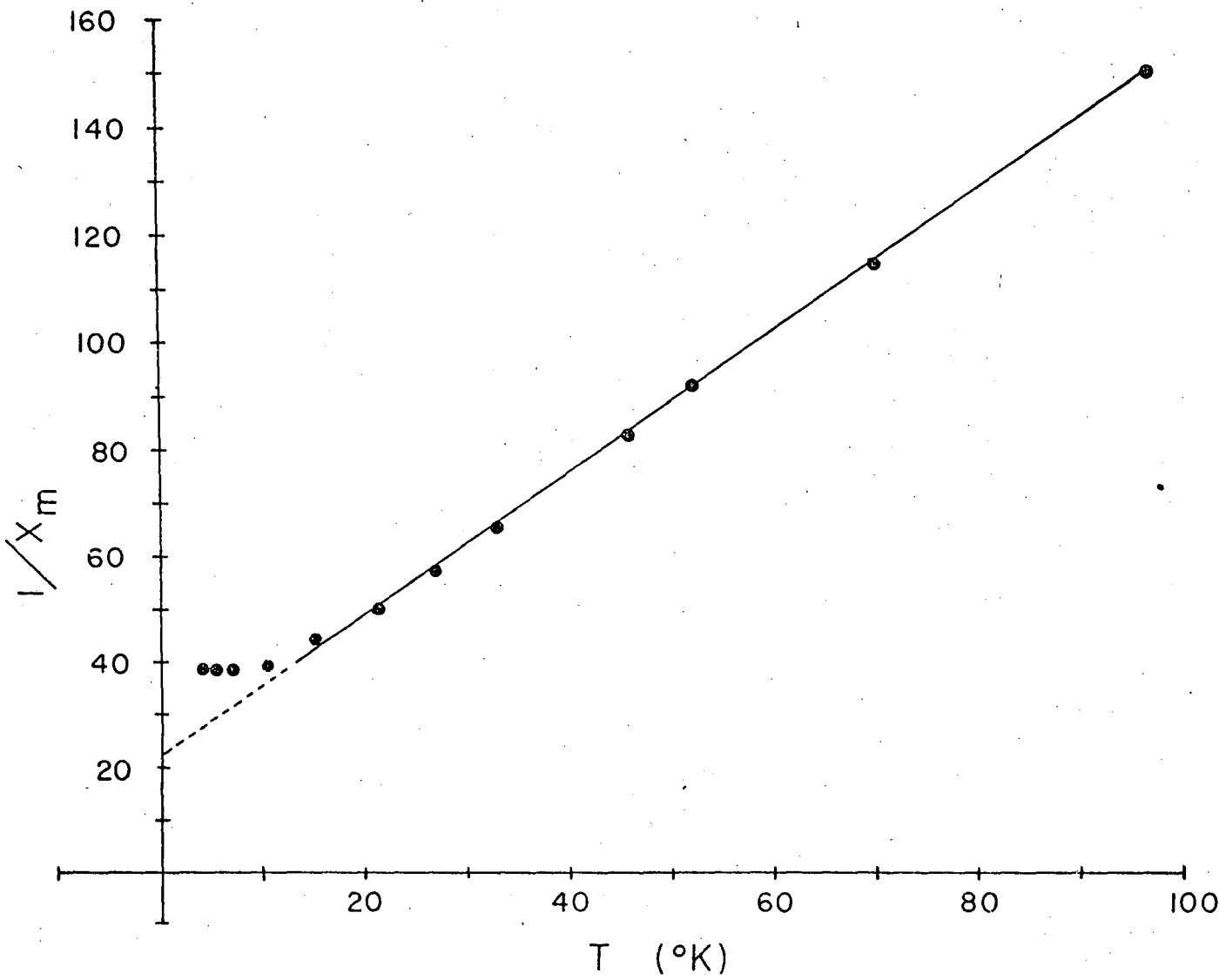
Table III. Uranium-Carbon Distances (A)

U -	C(1)	2.66(3)
	C(2)	2.63(3)
	C(3)	2.65(4)
	C(4)	2.60(3)
	C(5)	2.66(3)
	C(6)	2.64(3)
	C(7)	2.62(2)
	C(8)	2.68(2)
	C(12)	2.66(2)
	C(13)	2.66(3)
	C(14)	2.68(4)
	C(15)	2.58(3)
	C(16)	2.61(3)
	C(17)	2.67(2)
	C(18)	2.61(2)
	C(19)	2.66(2)



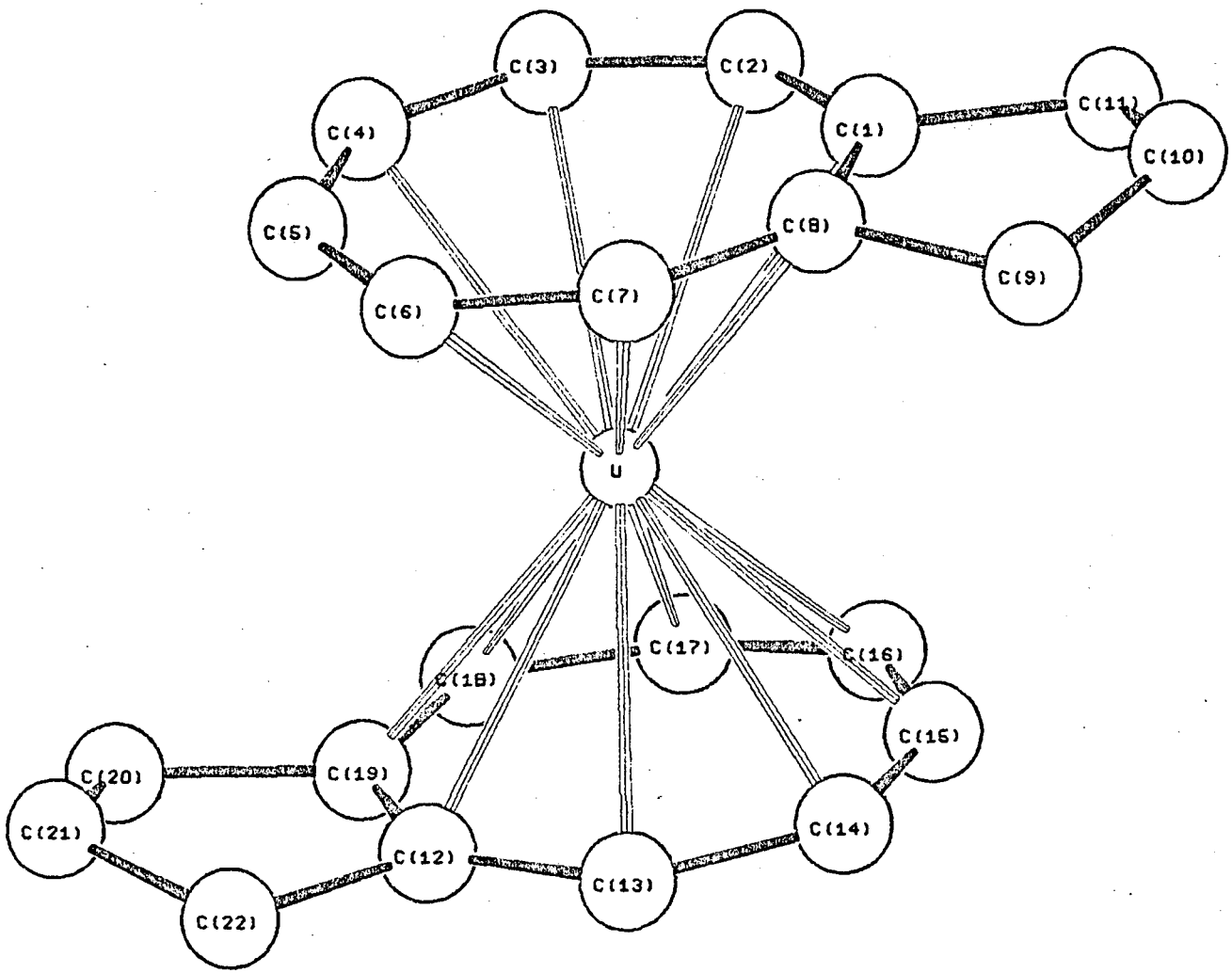
XBL 817-10783

Chart I.



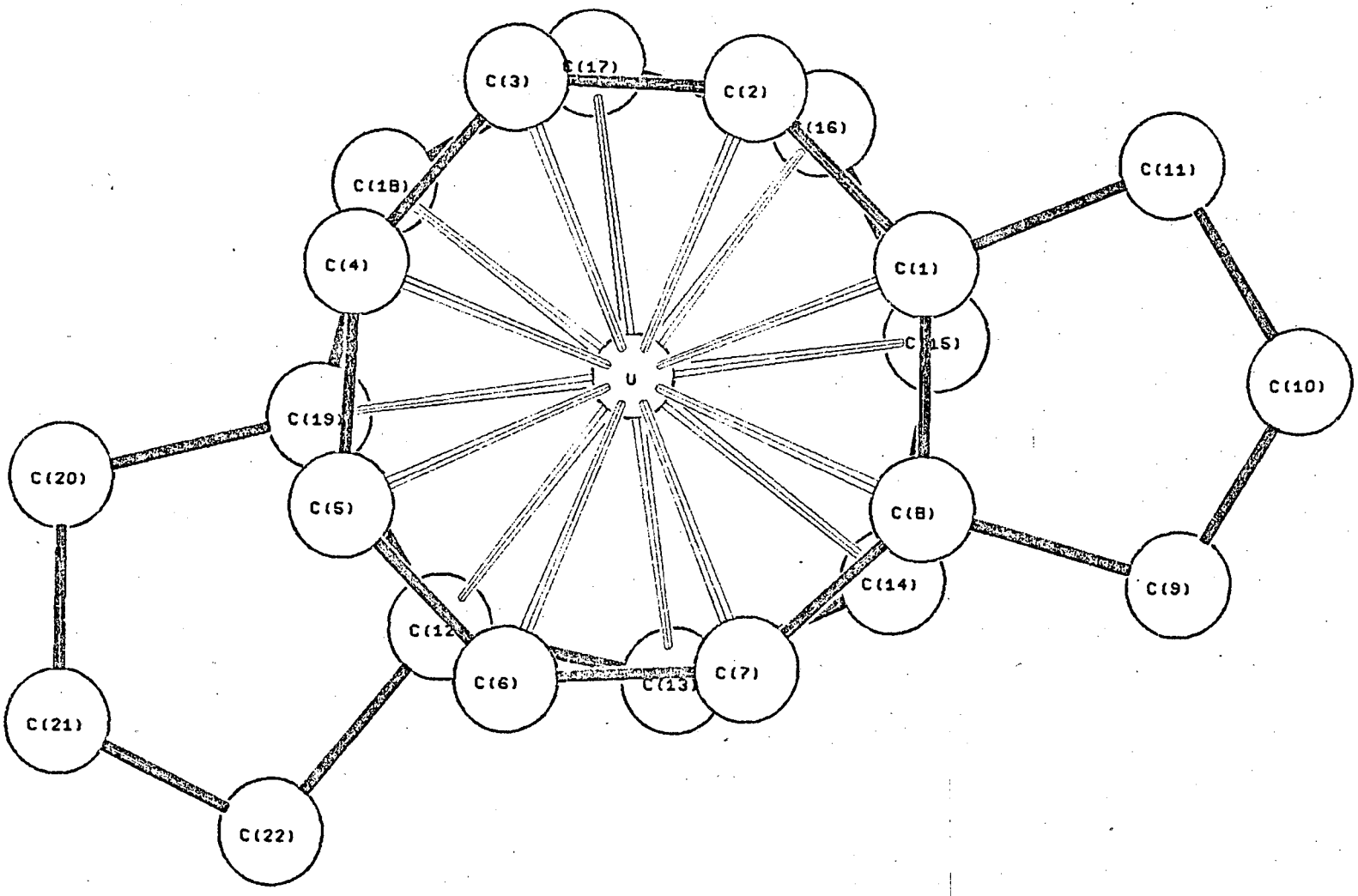
XBL 817-10782

Figure 1. Plot of $1/\chi$ vs. absolute temperature for dicyclopentenouranocene, 1.



XBL 7811-12981

Figure 2. Ortep ball and spoke view of the molecule. Hydrogen atoms are omitted for clarity.



XBL 7811-12980

Figure 3. Ball and spoke view of the molecule perpendicular to the cyclooctetraene rings. Hydrogen atoms omitted.

SYNTHESIS AND STRUCTURE OF DICYCLOPENTENOURANOCENE, $U[C_8H_6(CH_2)_3]_2$

Allan Zalkin,* David H. Templeton,* Wayne D. Luke
and Andrew Streitwieser, Jr.*

Materials and Molecular Research Division
Lawrence Berkeley Laboratory
and
Department of Chemistry, University of California
Berkeley, California 94720

SUPPLEMENTARY MATERIAL

Table IV. Thermal Parameters[a,b]

Atom	B11	B22	B33	B12	B13	B23
C(1)	7.0(19)	6.4(18)	6.3(18)	2.0(16)	1.3(15)	-1.7(15)
C(2)	10.8(24)	7.4(28)	7.3(22)	4.9(19)	4.7(17)	1.1(19)
C(3)	23.9(57)	3.4(19)	5.4(21)	3.6(25)	4.3(30)	.3(15)
C(4)	26.1(63)	4.7(21)	2.6(18)	8.2(28)	-5.8(27)	-1.2(15)
C(5)	6.9(21)	12.4(32)	11.1(34)	5.8(23)	-7.2(23)	-8.1(24)
C(6)	3.5(15)	16.5(38)	13.0(35)	-2.2(20)	3.8(21)	-10.9(28)
C(7)[c]	19.0(36)	1.1(10)	8.7(22)	- .5(16)	8.1(27)	-3.1(11)
C(8)	7.4(18)	3.3(13)	3.7(17)	1.9(12)	- .3(13)	- .6(10)
C(9)	9.1(20)	7.7(20)	9.8(28)	4.4(18)	2.3(22)	1.3(17)
C(10)	9.0(28)	17.1(42)	18.8(45)	2.4(29)	-7.7(30)	-5.6(36)
C(11)	4.7(21)	12.1(28)	25.7(52)	2.3(20)	7.2(26)	- .3(32)
C(12)	4.9(14)	6.2(17)	4.4(15)	-1.9(12)	3.4(13)	- .5(12)
C(13)	14.3(33)	6.4(18)	5.2(19)	6.6(23)	2.0(22)	- .4(15)
C(14)	21.9(56)	12.3(33)	2.7(17)	12.6(41)	1.9(25)	.2(18)
C(15)	8.4(26)	15.6(43)	8.2(30)	8.7(28)	-6.0(24)	-4.7(24)
C(16)	6.0(20)	6.5(22)	15.3(43)	-2.3(15)	2.5(23)	-5.0(22)
C(17)	12.6(24)	2.5(18)	9.3(25)	-1.4(18)	5.2(28)	-1.2(11)
C(18)	5.9(15)	4.8(13)	2.6(13)	.9(12)	.3(12)	1.2(9)
C(19)	7.2(15)	4.7(12)	1.8(12)	- .0(11)	.6(11)	- .7(11)
C(20)	4.6(17)	15.1(29)	7.6(24)	.9(18)	-2.6(15)	1.6(19)
C(21)	7.8(22)	21.7(47)	10.8(31)	-7.6(27)	3.4(22)	1.2(31)
C(22)	13.6(32)	6.9(20)	8.9(24)	-1.2(19)	3.4(23)	-2.7(17)
U	5.63(4)	3.44(3)	3.64(4)	1.47(5)	- .18(5)	- .60(4)

[a] The anisotropic temperature factor has the form $\exp(-.25(B_{11}H^2a^{*2} + 2\,B_{12}hka^*b^* + \ldots))$.

[b] Hydrogen isotropic thermal parameters of 8.0 $\mathring{A}^2$ for H(1)-H(12) and 12.0 $\mathring{A}^2$ for H(13)-H(24) were assigned but not refined.

[c] C(7) thermal tensor calculates non-positive definite.

Table V. Calculated Positional Parameters for the Hydrogen Atoms in Dicyclopentenouranocene.

H(1)	.0376	.0969	.3019
H(2)	.1485	.0756	.3938
H(3)	.2554	.1212	.3757
H(4)	.3070	.1877	.2478
H(5)	.2664	.2427	.066
H(6)	.1535	.2631	-.0079
H(7)	.2394	.1424	-.3217
H(8)	.1154	.138	-.3197
H(9)	.0465	.0692	-.2161
H(10)	.0576	.0038	-.0509
H(11)	.1561	-.0391	.0441
H(12)	.2692	-.0078	.0949
H(13)	.0222	.2159	-.1315
H(14)	.0099	.272	-.0334
H(15)	-.0841	.1930	-.051
H(16)	-.076	.2337	.0885
H(17)	-.069	.1523	.2094
H(18)	-.0453	.1162	.0687
H(19)	.4072	.0244	-.0276
H(20)	.3793	.0602	.1107
H(21)	.4578	.104	-.0972
H(22)	.4092	.1414	.014
H(23)	.3527	.1736	-.1759
H(24)	.3833	.1223	-.2756

Table VI. Carbon-carbon Bond Distances in Dicyclopentenouranocene[a]

C(1)	C(2)	1.41(2)
C(2)	C(3)	1.40(2)
C(3)	C(4)	1.40(2)
C(4)	C(5)	1.39(2)
C(5)	C(6)	1.38(2)
C(6)	C(7)	1.40(2)
C(7)	C(8)	1.38(2)
C(8)	C(1)	1.40(2)
C(12)	C(13)	1.39(2)
C(13)	C(14)	1.40(2)
C(14)	C(15)	1.40(2)
C(15)	C(16)	1.39(2)
C(16)	C(17)	1.39(2)
C(17)	C(18)	1.40(2)
C(18)	C(19)	1.39(2)
C(19)	C(20)	1.40(2)
C(8)	C(9)	1.55(2)
C(9)	C(10)	1.38(5)
C(10)	C(11)	1.46(6)
C(11)	C(1)	1.53(2)
C(19)	C(20)	1.54(2)
C(20)	C(21)	1.43(5)
C(21)	C(22)	1.43(5)
C(22)	C(12)	1.53(2)

[a]These distances are the result of a restrained structural refinement and are thus prejudiced.

Table VII. Selected Angles (deg.)

C(1)	- U -	C(2)	31.0(5)
C(2)	- U -	C(3)	30.7(5)
C(3)	- U -	C(4)	31.0(5)
C(4)	- U -	C(5)	30.6(5)
C(5)	- U -	C(6)	30.2(5)
C(6)	- U -	C(7)	31.0(5)
C(7)	- U -	C(8)	30.1(4)
C(8)	- U -	C(1)	30.3(4)
C(12)	- U -	C(13)	30.4(5)
C(13)	- U -	C(14)	30.4(5)
C(14)	- U -	C(15)	30.8(5)
C(15)	- U -	C(16)	31.1(5)
C(16)	- U -	C(17)	30.5(4)
C(17)	- U -	C(18)	30.7(4)
C(18)	- U -	C(19)	30.5(4)
C(19)	- U -	C(12)	30.6(5)

C(8)	- C(1) -	C(2)	134(3)
C(1)	- C(2) -	C(3)	138(4)
C(2)	- C(3) -	C(4)	128(4)
C(3)	- C(4) -	C(5)	143(3)
C(4)	- C(5) -	C(6)	133(3)
C(5)	- C(6) -	C(7)	131(3)
C(6)	- C(7) -	C(8)	140(3)
C(7)	- C(8) -	C(1)	133(3)
C(19)	- C(12) -	C(13)	133(2)
C(12)	- C(13) -	C(14)	143(3)
C(13)	- C(14) -	C(15)	124(4)
C(14)	- C(15) -	C(16)	142(3)
C(15)	- C(16) -	C(17)	132(3)
C(16)	- C(17) -	C(18)	135(2)
C(17)	- C(18) -	C(19)	135(2)
C(18)	- C(19) -	C(12)	134(2)
C(2)	- C(1) -	C(11)	113(3)
C(8)	- C(1) -	C(11)	113(3)
C(1)	- C(11) -	C(10)	98(3)
C(11)	- C(10) -	C(9)	115(4)
C(10)	- C(19) -	C(8)	104(3)
C(1)	- C(8) -	C(9)	105(2)
C(7)	- C(8) -	C(9)	122(3)
C(13)	- C(12) -	C(22)	118(3)
C(19)	- C(12) -	C(22)	110(2)
C(12)	- C(22) -	C(21)	104(3)
C(22)	- C(21) -	C(20)	112(3)
C(21)	- C(20) -	C(19)	105(3)
C(20)	- C(19) -	C(12)	107(2)
C(20)	- C(19) -	C(18)	119(3)

DATA PROCESSING FORMULAE

$$I = C - (t_c/2t_b)(B_1+B_2)$$

$$\sigma(B) = \text{Max}[(t_c/2t_b)(B_1+B_2)^{\frac{1}{2}}, (t_c/2t_b)|B_1-B_2|]$$

$$\sigma(I) = [C + \sigma^2(B)]^{\frac{1}{2}}$$

$$F^2 = (D \cdot A/Lp)I$$

$$\sigma(F^2) = (D \cdot A/Lp)\sigma(I)$$

$$F_a^2 = \Sigma F^2/n$$

$$\sigma(F_a^2) = [\Sigma\sigma^2(F^2)]^{\frac{1}{2}}/n$$

When $S(F_a^2) > 4\sigma(F_a^2)$, $\sigma(F_a^2)$ is replaced by $S(F_a^2)$.

$$S(F_a^2) = [\Sigma|F^2-F_a^2|^2/n(n-1)]^{\frac{1}{2}}$$

$$\sigma(F_o^2) = [\sigma^2(F_a^2) + (pF_a^2)^2 + q^2]^{\frac{1}{2}}$$

$$F_o = (F_a^2)^{\frac{1}{2}}$$

$$\sigma(F) = F_o - [F_a^2 - \sigma(F_o^2)]^{\frac{1}{2}} \text{ when } \sigma(F_o^2) \leq F_a^2 \text{ or } [\sigma(F_a^2]^{\frac{1}{2}} \text{ when } \sigma(F_a^2) > F_a^2$$

$$Lp = [\cos^2 2\theta_m + \cos^2 2\theta]/[\sin 2\theta\ (1 + \cos^2 2\theta_m)]$$

$$wtg = 1/\sigma^2(F)$$

C = counts recorded during a scan

I = individual raw intensity, background removed.

t_c = scan count time

t_b = background count time

B_1 = individual background count

$\sigma(B)$ = estimated standard deviation of the total background count

F = structure factor

D = decay correction; an empirically applied correction obtained from the fluctuations of the standard reflections.

A = absorption correction

Lp = Lorentz and polarization corrections

θ_m = monochromater angle

θ = crystal diffraction angle

S = scatter

a = average

q = additional uncertainty that affects the weak intensities

p = estimate of non-statistical errors

wtg = weighting factors in least squares

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 2.02
UC(22)H(24) F(0,0,0) = 3801

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.
SG = ESTIMATED STANDARD DEVIATION OF FOB. DEL = /FOB/ - /FCA/.
* INDICATES ZERO WEIGHTED DATA.

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
H,L=	0,	0		0	689	22	-5	2	235	19	16	22	90	30	-27*	2	241	9	2
2	110	115	-22*	2	127	9	-14	4	0	90	-60*	23	55	57	14*	3	69	14	29*
4	1351	41	-12	4	480	17	17	6	197	16	-8	H,L=		1,	3	4	73	15	-9*
6	441	14	15	6	124	12	-1	8	102	27	28*	1	271	9	4	5	104	13	3
8	1181	36	26	8	472	15	-8	H,L=		1,	1	2	712	23	14	6	193	8	-6
10	622	19	19	10	228	11	11	1	494	16	26	3	227	8	3	7	50	52	0*
12	647	20	6	12	263	16	4	21	256	40	34	4	230	8	12	8	73	16	20*
14	466	15	-1	14	213	11	5	3	406	13	11	5	280	9	4	9	79	17	-6*
16	329	12	-1	16	228	12	-4	4	366	12	18	6	604	19	3	10	130	11	-6
18	520	17	17	18	181	15	-19	5	103	6	4	7	133	7	11	11	52	54	48*
20	128	17	3	20	83	33	4*	6	634	20	-3	8	174	7	-2	12	122	11	-4
22	314	13	12	22	137	22	2	7	107	7	2	9	431	14	1	13	82	16	25*
24	0	73	-8*	H,L=		0,	5	8	428	13	12	10	463	15	-17	14	130	11	1
H,L=		0,	1	2	588	22	-5	9	495	16	5	11	0	42	-24*	15	47	66	46*
2	327	10	12	4	146	10	24	10	655	20	-9	12	245	10	-8	16	71	23	-16*
4	345	12	18	6	481	16	-32	11	22	39	-22*	13	217	9	-6	17	72	33	-10*
6	166	7	6	8	302	11	2	12	400	13	-4	14	250	9	-15	18	101	16	7
8	36	9	21	10	389	13	-8	13	230	8	-12	15	26	48	7*	19	0	71	-9*
10	142	8	6	12	310	12	-4	14	321	11	-13	16	258	10	-9	20	96	19	17*
12	81	37	-11*	14	221	14	5	15	54	24	-13*	17	181	10	5	H,L=		1,	6
14	70	21	1*	16	294	12	9	16	390	13	-17	18	170	20	4	0	471	15	-8
16	119	15	4	18	105	25	-19*	17	198	12	-22	19	0	52	-43*	1	182	11	6
18	0	60	-29*	20	202	16	-16	18	190	10	-11	20	211	11	-6	2	54	28	3*
20	77	35	-18*	H,L=		0,	6	19	94	15	5	21	94	21	12*	3	217	9	-1
22	0	66	-7*	0	134	13	2	20	313	14	-4	22	0	58	-39*	4	437	14	-4
24	68	83	6*	2	0	55	-60*	21	128	17	-7	23	41	70	-15*	5	89	25	-15*
H,L=		0,	2	4	87	23	-16*	22	0	63	-48*	H,L=		1,	4	6	165	9	-6
0	1110	34	0	6	50	58	24*	23	82	23	-3*	0	745	26	17	7	217	11	-7
2	201	7	7	8	86	25	-28*	24	189	12	-25	1	277	10	13	8	372	13	-14
4	253	8	-6	10	0	62	-13*	H,L=		1,	2	2	30	41	-9*	9	12	68	-33*
6	328	10	8	12	0	64	-46*	0	286	10	5	3	301	10	7	10	267	10	2
8	690	23	-1	14	55	64	42*	1	335	11	12	4	632	21	19	11	202	10	-3
10	415	15	6	16	13	69	-70*	2	159	6	2	5	86	10	5	12	224	10	-13
12	413	16	-1	18	0	66	-17*	3	153	8	10	6	272	10	10	13	31	60	26*
14	335	16	-4	H,L=		0,	7	4	147	7	-3	7	295	10	-9	14	247	11	-24
16	252	11	-10	2	486	16	-1	5	249	8	10	8	548	18	11	15	142	12	-15
18	380	14	-5	4	110	19	8	6	101	11	-4	9	21	52	10*	16	137	21	-8
20	137	20	34	6	444	15	-9	7	176	7	0	10	291	10	3	17	38	57	-16*
22	246	13	1	8	182	16	-7	8	243	8	-2	11	252	10	-8	18	208	11	2
H,L=		0,	3	10	316	14	-17	9	40	42	20*	12	281	10	-12	H,L=		1,	7
2	709	23	41	12	257	12	3	10	205	8	-1	13	32	50	23*	1	57	74	49*
4	62	14	-15*	14	180	16	-7	11	144	9	6	14	263	10	-4	2	0	53	-12*
6	526	18	4	16	221	15	9	12	172	8	-7	15	176	11	-8	3	52	48	52*
8	426	16	22	H,L=		0,	8	13	5	42	2*	16	129	12	-2	4	71	21	54*
10	417	14	0	0	136	18	11	14	177	8	12	17	34	52	-38*	5	42	52	37*
12	289	11	19	2	72	39	68*	15	95	12	-2	18	226	11	-13	6	0	60	-19*
14	191	12	-2	4	81	34	-19*	16	74	32	-3*	19	118	19	6	7	49	53	10*
16	278	12	0	6	0	68	-74*	17	71	19	8*	20	30	60	-42*	8	39	54	-13*
18	33	63	-55*	8	65	66	21*	18	167	10	-4	21	61	41	-9*	9	45	53	16*
20	229	13	3	10	92	29	45*	19	91	16	19	22	174	12	4	10	37	71	27*
22	61	73	35*	12	101	27	48*	20	21	53	-32*	H,L=		1,	5	11	37	53	24*
H,L=		0,	4	H,L=		0,	9	21	41	82	-5*	1	73	17	1*	12	37	54	12*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
13	8	71	0*	2	19	25	-10*	9	440	14	-4	19	77	24	-22*	2	126	14	8
14	28	56	18*	3	316	18	4	10	327	11	4	20	98	33	-15*	3	126	19	27
15	27	57	6*	4	40	19	7*	11	40	57	23*	H,L=		2,	6	4	52	57	25*
16	28	59	-22*	5	242	8	0	12	144	10	-19	0	88	13	11	5	160	15	-4
H,L=		1,	8	6	46	39	-6*	13	300	16	-9	1	46	47	17*	6	77	29	-21*
0	296	12	-5	7	96	7	-11	14	107	14	3	2	45	46	7*	7	69	33	2*
1	120	13	20	8	53	14	-10*	15	85	20	-9*	3	62	53	3*	8	42	58	-6*
2	39	55	-1*	9	241	9	-4	16	125	39	-41*	4	21	48	-34*	H,L=		3,	1
3	145	21	2	10	75	12	-1	17	201	29	-28	5	76	24	10*	1	688	21	-6
4	263	13	-23	11	0	43	-50*	18	0	218	-93*	6	33	55	21*	2	60	15	7*
5	96	41	15*	12	33	46	-4*	19	83	19	-40*	7	38	52	-9*	3	420	13	15
6	89	20	15*	13	138	17	-6	20	65	60	-51*	8	83	17	1*	4	55	16	7*
7	129	17	-5	14	52	32	5*	21	128	18	1	9	58	43	9*	5	790	24	9
8	248	11	-7	15	33	75	4*	22	54	64	28*	10	76	24	12*	6	71	23	-23*
9	77	24	39*	16	18	64	-19*	H,L=		2,	4	11	47	56	25*	7	380	12	-1
10	121	16	-6	17	62	38	-38*	0	349	11	1	12	84	22	29*	8	40	29	9*
11	81	26	-34*	18	0	113	-41*	1	298	10	7	13	74	46	51*	9	926	28	30
12	154	13	-10	19	45	57	-1*	2	106	7	6	14	32	58	25*	10	85	60	9*
13	36	57	24*	20	66	36	42*	3	371	12	14	15	0	63	-71*	11	0	42	-15*
H,L=		1,	9	21	76	30	23*	4	278	9	2	16	40	75	0*	12	0	50	-41*
1	66	74	14*	22	0	62	-4*	5	232	9	-2	17	0	83	-26*	13	584	19	-6
2	149	18	12	23	0	66	-31*	6	51	19	1*	18	0	91	-6*	14	0	50	-77*
3	46	69	6*	H,L=		2,	2	7	427	13	-14	H,L=		2,	7	15	76	20	-4*
4	0	60	-54*	0	383	12	-19	8	231	9	3	1	330	12	4	16	0	92	-31*
5	55	66	4*	1	439	22	18	9	91	17	-6*	2	275	11	4	17	461	15	-9
6	101	23	8*	2	326	11	9	10	63	24	-26*	3	226	10	-10	18	0	139	-80*
7	0	60	-5*	3	324	11	4	11	311	12	4	4	69	26	17*	19	136	14	-6
8	68	72	-3*	4	343	11	-7	12	110	14	-23	5	351	13	-9	20	0	83	-25*
H,L=		2,	0	5	311	10	-1	13	0	53	-12*	6	251	11	-3	21	278	11	3
0	739	51	-128	6	133	12	1	14	112	15	0	7	132	11	3	22	0	291	-28
1	765	73	-60	7	621	23	13	15	244	12	-4	8	72	36	-22*	23	136	18	-20
2	289	25	-10	8	389	12	-12	16	115	31	-15*	9	322	12	-8	H,L=		3,	2
3	723	52	-31	9	153	7	-7	17	0	80	-26*	10	198	28	-11	0	179	15	-20
4	625	39	-18	10	177	8	7	18	0	81	-101*	11	0	59	-36*	1	327	11	-1
5	339	22	-8	11	518	15	-3	19	142	15	-16	12	132	16	4	2	0	60	-45*
6	171	6	0	12	205	9	-7	20	20	83	-32*	13	254	12	-22	3	328	11	20
7	951	41	11	13	38	46	6*	21	32	70	-23*	14	137	17	8	4	14	50	-13*
8	592	23	-7	14	166	35	-31*	H,L=		2,	5	15	76	35	19*	5	69	23	-10*
9	164	6	-8	15	338	12	-19	1	373	12	3	16	108	22	14*	6	0	37	-1*
10	334	14	23	16	9	167	-171*	2	331	11	-1	H,L=		2,	8	7	457	14	24
11	739	23	-4	17	0	68	-89*	3	270	9	10	0	64	27	9*	8	90	9	0
12	322	11	-13	18	0	188	-206*	4	53	24	0*	1	72	28	13*	9	49	20	-7*
13	86	13	15	19	239	17	-25	5	408	13	9	2	0	71	-9*	10	0	52	-18*
14	263	21	-6	20	0	64	-70*	6	301	11	-2	3	93	20	34*	11	311	10	-11
15	460	15	-11	21	119	18	11	7	81	18	-10*	4	50	64	-15*	12	0	47	-35*
16	181	73	-68*	22	115	22	-23*	8	123	9	-1	5	61	30	49*	13	40	50	34*
17	87	24	-71*	23	125	21	-25	9	384	15	-11	6	24	54	-22*	14	0	64	-20*
18	290	13	-36	H,L=		2,	3	10	194	12	0	7	78	22	3*	15	221	10	-9
19	332	12	-25	1	458	20	19	11	50	55	-22*	8	44	54	20*	16	72	23	29*
20	52	83	-29*	2	365	12	11	12	168	11	-7	9	18	62	5*	17	110	15	19
21	138	17	-19	3	345	11	6	13	322	11	-4	10	0	64	-9*	18	0	57	-4*
22	148	16	5	4	128	6	3	14	90	63	19*	11	60	69	-6*	19	159	13	1
23	176	15	-12	5	487	15	24	15	46	58	-18*	12	49	64	22*	20	0	78	-37*
24	0	67	-16*	6	372	12	7	16	155	14	-7	13	0	80	-30*	21	81	27	10*
H,L=		2,	1	7	39	45	19*	17	241	11	-5	H,L=		2,	9	22	0	66	-14*
1	221	15	0	8	211	8	0	18	86	25	1*	1	127	15	-14	23	86	28	24*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
H,L=		3,	3	10	20	54	-31*	7	276	12	-6	17	70	28	-16*	0	175	8	-1
1	675	21	16	11	57	35	47*	8	94	23	47*	18	0	73	-1*	1	298	10	-5
2	76	12	4	12	84	23	-3*	9	52	63	-15*	19	35	57	15*	2	55	22	30*
3	502	16	23	13	146	14	-18	10	0	81	-16*	20	58	58	10*	3	367	12	8
4	22	39	20*	14	34	82	5*	11	228	13	-25	21	0	62	-37*	4	181	9	6
5	637	20	9	15	7	57	-20*	12	0	67	-41*	22	48	62	42*	5	217	9	3
6	0	42	-55*	16	0	60	-4*	H,L=		3,	9	23	50	64	16*	6	89	20	-20*
7	365	12	-7	17	160	27	18	1	116	34	36*	H,L=		4,	2	7	346	11	9
8	117	9	4	18	0	62	-15*	2	0	66	-8*	0	370	12	-4	8	102	13	-9
9	626	19	15	19	60	89	35*	3	0	67	-65*	1	607	19	-24	9	91	14	2
10	67	31	10*	20	0	65	-25*	4	0	67	-22*	2	0	37	-39*	10	78	49	-2*
11	0	58	-32*	H,L=		3,	6	5	89	30	-24*	3	669	21	14	11	322	12	9
12	0	49	-26*	0	96	20	34*	6	0	93	-9*	4	327	11	9	12	0	54	-64*
13	425	14	-5	1	387	13	9	7	0	68	-21*	5	397	12	1	13	0	53	-14*
14	82	18	25*	2	0	53	-22*	H,L=		4,	0	6	44	58	-26*	14	67	31	-13*
15	33	52	2*	3	444	14	9	0	416	13	-9	7	668	21	-9	15	254	11	5
16	0	57	-15*	4	0	60	-52*	1	675	21	-56	8	183	9	9	16	19	71	-11*
17	340	12	0	5	209	10	-1	2	303	10	-1	9	130	11	-7	17	0	60	-24*
18	27	83	-28*	6	89	17	75*	3	730	22	-6	10	163	8	4	18	83	63	2*
19	77	35	-20*	7	458	15	2	4	432	14	-11	11	546	17	7	19	183	12	17
20	0	65	-20*	8	55	49	-7*	5	563	17	-3	12	119	13	-1	20	0	82	-8*
21	199	13	8	9	118	21	5	6	302	11	15	13	0	49	-5*	21	0	97	-51*
22	0234		-37*	10	82	22	43*	7	959	29	16	14	156	10	-9	H,L=		4,	5
H,L=		3,	4	11	380	15	-7	8	317	10	26	15	425	14	3	1	371	15	0
0	83	13	-12	12	33	58	13*	9	237	8	-1	16	0	83	-54*	2	231	13	14
1	521	16	22	13	0	59	-14*	10	226	10	-7	17	94	20	21*	3	261	11	8
2	0	44	-32*	14	0	60	-31*	11	741	23	32	18	102	26	-38*	4	19	50	-16*
3	521	16	20	15	341	12	12	12	183	9	0	19	305	11	14	5	387	13	0
4	86	12	2	16	52	64	-4*	13	0	47	-16*	20	0	67	-1*	6	152	10	9
5	225	9	1	17	120	17	26	14	213	16	1	21	100	21	7*	7	156	10	-1
6	0	54	-42*	18	0	66	-5*	15	521	16	-4	22	91	26	1*	8	119	13	-8
7	568	18	10	H,L=		3,	7	16	17	69	-53*	23	184	14	2	9	360	12	5
8	169	11	5	1	41	60	21*	17	90	28	-2*	H,L=		4,	3	10	136	13	-3
9	124	10	9	2	0	58	-2*	18	157	13	-21	1	488	15	-9	11	0	63	-46*
10	0	49	-5*	3	0	59	-15*	19	363	13	0	2	263	9	12	12	95	35	31*
11	436	14	-6	4	0	59	-18*	20	0	97	-7*	3	300	10	0	13	328	12	8
12	0	52	-10*	5	38	59	-1*	21	105	39	-9*	4	0	42	-39*	14	0	73	-78*
13	58	30	39*	6	0	53	-3*	22	62	81	-54*	5	474	15	8	15	0	61	-68*
14	45	54	12*	7	0	62	-39*	23	209	21	5	6	137	8	14	16	139	14	15
15	360	13	1	8	60	45	49*	H,L=		4,	1	7	156	9	-5	17	282	11	5
16	0	58	-26*	9	69	33	35*	1	223	7	-8	8	163	13	0	18	48	61	35*
17	124	16	-8	10	0	59	-26*	2	77	17	14*	9	438	16	-2	19	85	29	4*
18	0	62	-4*	11	0	85	-36*	3	52	70	-7*	10	70	75	5*	20	105	23	21*
19	249	11	2	12	54	61	52*	4	108	9	-11	11	74	25	7*	H,L=		4,	6
20	0	97	-35*	13	78	58	71*	5	194	9	8	12	70	22	-13*	0	31	55	16*
21	94	28	-24*	14	0	63	-16*	6	91	8	8	13	337	12	-3	1	38	56	-14*
H,L=		3,	5	15	115	81	42*	7	43	23	20*	14	38	64	-7*	2	68	27	61*
1	185	9	8	16	0	66	-41*	8	148	7	4	15	70	27	-12*	3	46	56	-13*
2	28	57	-9*	H,L=		3,	8	9	151	11	10	16	116	37	-8*	4	57	39	14*
3	115	11	18	0	0	83	-44*	10	84	15	11	17	282	11	6	5	76	23	14*
4	0	49	-6*	1	229	12	6	11	40	43	-9*	18	0	60	-9*	6	0	62	-19*
5	191	9	-6	2	0	61	-9*	12	88	12	23	19	46	61	-35*	7	52	56	1*
6	13	49	-5*	3	273	12	-2	13	99	14	2	20	84	26	13*	8	0	57	-13*
7	117	12	0	4	88	39	61*	14	49	37	18*	21	159	15	20	9	67	30	15*
8	0	60	-27*	5	121	24	-11*	15	34	60	12*	22	0	80	-26*	10	56	58	29*
9	209	9	17	6	66	39	54*	16	51	53	-30*	H,L=		4,	4	11	98	19	16*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
12	85	22	59*	12	294	11	-2	20	161	14	-20	9	70	73	-14*	11	105	12	-9
13	70	31	0*	13	338	11	-1	21	149	23	18	10	159	13	-21	12	386	14	-3
14	0	60	-26*	14	247	13	1	22	45	65	28*	11	241	11	20	13	128	11	6
15	107	19	48	15	0	58	-3*	H,L=	5,	4		12	181	12	-1	14	407	14	-6
16	63	68	14*	16	318	17	-14	0	491	15	-8	13	0	61	-23*	15	136	12	-3
17	63	61	18*	17	309	11	-1	1	203	9	-7	14	215	11	8	16	242	10	5
18	61	65	41*	18	72	32	-30*	2	56	34	34*	15	200	13	-16	17	0	60	-72*
H,L=	4,	7		19	0	65	-39*	3	295	13	-8	16	94	24	12*	18	373	13	-16
1	354	13	7	20	225	11	2	4	441	14	3	17	0	77	-12*	19	96	11	-15*
2	175	12	9	21	178	24	-6	5	173	11	0	H,L=	5,	7		20	70	38	-16*
3	237	11	2	22	0	63	-12*	6	193	8	10	1	48	61	39*	21	70	82	23*
4	90	22	34*	23	36	72	-26*	7	264	10	8	2	48	80	15*	22	230	12	-5
5	361	13	-10	H,L=	5,	2		8	298	11	2	3	0	61	-9*	H,L=	6,	1	
6	102	22	-26*	0	286	9	-1	9	129	23	-5	4	70	72	57*	1	80	11	-11
7	113	25	-23*	1	159	12	-8	10	217	13	-1	5	0	61	-49*	2	132	9	-18
8	76	55	-48*	2	77	12	-10	11	249	10	1	6	0	67	-19*	3	0	38	-5*
9	344	12	6	3	306	10	-6	12	199	10	4	7	0	60	-4*	4	212	8	8
10	112	10	26	4	298	10	-7	13	0	55	-18*	8	83	24	68*	5	34	38	-18*
11	70	83	11*	5	14	40	-23*	14	245	11	-6	9	0	60	-30*	6	185	7	12
12	92	32	-9*	6	177	9	-3	15	238	14	3	10	0	61	-11*	7	0	42	-22*
13	308	17	9	7	178	8	2	16	133	22	40	11	57	61	26*	8	92	12	-7
14	93	25	61*	8	234	9	14	17	0	61	-9*	12	6	63	-16*	9	0	44	-19*
15	0	67	-10*	9	67	38	12*	18	184	13	-20	13	60	83	48*	10	99	12	-7
H,L=	4,	8		10	120	10	0	19	165	14	12	14	0	65	-44*	11	17	46	7*
0	82	29	34*	11	152	9	-1	20	70	77	38*	15	40	67	-12*	12	87	16	-15
1	32	64	-13*	12	106	13	-18	21	67	49	22*	H,L=	5,	8		13	11	49	4*
2	57	71	55*	13	0	59	-29*	H,L=	5,	5		0	234	14	13	14	61	27	11*
3	92	34	25*	14	164	10	4	1	138	17	-3	1	145	16	7	15	69	24	65*
4	0	78	-22*	15	160	10	18	2	173	10	-4	2	35	64	16*	16	90	21	-20*
5	49	61	8*	16	25	56	-20*	3	80	21	7*	3	180	13	9	17	28	56	18*
6	57	61	44*	17	60	38	23*	4	66	23	36*	4	198	12	-10	18	0	57	-30*
7	105	21	9*	18	136	13	2	5	109	14	2	5	110	24	18*	19	0	104	-2*
8	87	25	52*	19	89	23	-11*	6	139	12	-15	6	100	23	-7*	20	76	30	0*
9	20	63	12*	20	0	61	-9*	7	102	28	19*	7	153	16	-4	21	51	61	34*
10	0	76	-48*	21	0	82	-42*	8	65	27	1*	8	153	18	-3	22	0	64	-1*
11	85	29	21*	22	0	66	-74*	9	147	12	14	9	88	32	46*	H,L=	6,	2	
12	0	67	-6*	H,L=	5,	3		10	128	14	13	10	78	39	-37*	0	754	23	-44
H,L=	4,	9		1	383	13	-19	11	58	69	24*	11	155	16	2	1	88	10	2
1	142	19	-11	2	545	17	14	12	51	56	15*	H,L=	5,	9		2	32	39	-12*
2	90	29	7*	3	218	9	-7	13	118	16	1	1	57	70	33*	3	127	8	-2
3	130	19	18	4	112	9	13	14	97	20	-5*	2	0	112	-81*	4	700	22	0
4	0	67	-20*	5	360	12	-4	15	0	60	-30*	3	0	69	-41*	5	24	42	-30*
5	170	30	9	6	365	12	1	16	79	27	14*	4	77	38	50*	6	262	9	0
6	67	68	-13*	7	149	9	1	17	114	18	34	5	96	38	35*	7	146	15	8
H,L=	5,	1		8	257	10	-11	18	50	62	13*	H,L=	6,	0		8	547	17	16
1	418	14	-39	9	330	12	2	19	27	64	14*	0	622	19	-24	9	20	60	-23*
2	710	22	-15	10	260	10	4	H,L=	5,	6		1	137	11	-16	10	287	10	-10
3	393	12	-12	11	48	49	-11*	0	402	13	0	2	79	9	-7	11	90	15	-4
4	139	7	-13	12	248	10	-4	1	180	12	-9	3	84	30	-17*	12	342	12	2
5	501	15	-12	13	256	10	5	2	43	64	10*	4	791	24	1	13	0	73	-39*
6	629	19	-5	14	186	11	0	3	283	11	-4	5	197	8	-1	14	326	11	-7
7	261	9	0	15	25	67	-1*	4	361	14	-9	6	367	12	-12	15	101	28	7*
8	322	11	17	16	264	11	-4	5	126	15	-8	7	153	8	3	16	223	11	2
9	516	16	11	17	199	12	-5	6	185	12	-9	8	728	22	26	17	0	59	-42*
10	422	13	17	18	77	41	12*	7	256	11	8	9	170	8	-8	18	296	12	-12
11	98	12	-1	19	0	61	-12*	8	280	11	-1	10	377	16	2	19	95	78	30*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
20	100	20	17*	9	33	57	-33*	10	89	28	34*	4	122	14	6	16	74	34	-2*
21	0	64	-20*	10	308	11	13	11	105	21	96*	5	116	11	-1	17	0	64	-16*
22	197	13	3	11	0	59	-52*	H,L=		6,	9	6	457	14	-3	18	89	47	40*
H,L=		6,	3	12	255	11	2	1	0	71	-41*	7	0	51	-5*	H,L=		7,	6
1	113	9	9	13	78	28	-12*	2	196	15	5	8	230	9	0	0	400	15	-20
2	523	16	-9	14	187	21	11	3	47	78	19*	9	129	12	7	1	108	16	26
3	30	42	13*	15	0	84	-57*	H,L=		7,	1	10	356	12	26	2	66	68	12*
4	158	8	7	16	261	14	10	1	180	7	4	11	31	53	19*	3	99	20	5*
5	10	45	-31*	17	53	64	-24*	2	619	19	-46	12	217	10	4	4	418	17	-2
6	484	15	13	18	77	50	-11*	3	33	40	-14*	13	64	34	-25*	5	0	60	-62*
7	61	22	21*	19	0	65	-9*	4	135	8	-5	14	263	11	-4	6	152	18	3
8	173	9	3	H,L=		6,	6	5	254	9	0	15	64	37	-12*	7	151	23	21
9	43	49	-15*	0	84	23	-16*	6	714	22	0	16	229	11	-5	8	354	13	16
10	291	10	9	1	0	57	-19*	7	43	35	25*	17	90	22	1*	9	0	77	-11*
11	84	17	35*	2	0	59	-21*	8	309	10	-3	18	130	16	-2	10	183	12	0
12	267	10	11	3	69	30	41*	9	213	9	14	19	0	63	-75*	11	63	61	-36*
13	18	56	-60*	4	103	18	9	10	440	15	4	20	190	13	26	12	227	12	1
14	164	11	8	5	0	59	-22*	11	26	49	-35*	21	37	79	13*	13	87	25	30*
15	84	27	42*	6	0	62	-2*	12	304	11	7	H,L=		7,	4	14	193	12	2
16	250	11	0	7	40	57	16*	13	108	25	-2*	0	412	13	-30	15	65	53	6*
17	96	20	28*	8	100	19	12*	14	325	11	5	1	40	64	20*	16	152	16	19
18	106	18	19	9	0	62	-41*	15	103	17	-18	2	66	21	12*	H,L=		7,	7
19	50	61	44*	10	77	25	54*	16	363	13	3	3	117	12	1	1	0	61	-28*
20	173	13	12	11	0	82	-10*	17	106	51	2*	4	423	14	-12	2	32	62	13*
21	25	80	-26*	12	82	28	0*	18	146	14	-9	5	41	50	-13*	3	0	72	-5*
H,L=		6,	4	13	0	74	-34*	19	0	94	-36*	6	144	11	-4	4	0	61	-28*
0	361	12	-18	14	76	30	55*	20	245	11	7	7	160	11	3	5	0	62	-15*
1	73	15	18*	15	0	64	-4*	21	0	64	-6*	8	376	13	11	6	47	69	40*
2	26	46	2*	16	114	19	50	22	47	72	-21*	9	0	54	-39*	7	57	62	52*
3	109	24	-19*	17	87	29	66*	H,L=		7,	2	10	244	11	1	8	181	32	81*
4	304	11	-7	H,L=		6,	7	0	325	11	-4	11	105	16	4	9	36	62	27*
5	67	20	10*	1	85	26	-1*	1	31	39	-1*	12	253	11	0	10	0	63	-14*
6	148	10	2	2	381	13	-12	2	90	11	-5	13	116	16	-5	11	29	63	25*
7	60	29	-28*	3	24	85	-21*	3	80	12	-3	14	236	11	-9	12	0	66	-19*
8	293	11	6	4	113	47	12*	4	320	11	-6	15	91	25	28*	13	34	64	29*
9	61	67	0*	5	0	63	-73*	5	69	21	-2*	16	176	11	26	H,L=		7,	8
10	165	11	-1	6	342	13	-4	6	105	10	-3	17	78	30	-13*	0	236	12	-6
11	79	21	13*	7	57	62	-3*	7	80	15	11	18	201	12	9	1	67	44	34*
12	201	11	-15	8	156	15	-19	8	259	9	4	19	45	63	9*	2	39	65	20*
13	71	27	37*	9	42	75	6*	9	0	61	-44*	20	63	65	-4*	3	70	74	34*
14	184	10	17	10	251	11	13	10	122	20	-15	H,L=		7,	5	4	263	13	13
15	72	80	10*	11	0	63	-43*	11	44	50	-6*	1	57	33	18*	5	37	65	24*
16	121	16	-3	12	189	13	-4	12	165	10	15	2	199	10	6	6	92	30	14*
17	61	46	28*	13	0	66	-54*	13	52	52	24*	3	30	54	-21*	7	61	66	0*
18	146	15	-14	14	158	15	-6	14	151	12	-17	4	0	54	-13*	8	216	13	25
19	0	64	-45*	H,L=		6,	8	15	0	58	-54*	5	54	59	31*	9	0	67	-6*
20	67	71	20*	0	92	26	14*	16	0	60	-82*	6	210	10	-10	10	76	43	-37*
H,L=		6,	5	1	0	83	-2*	17	0	58	-23*	7	0	56	-5*	H,L=		8,	0
1	121	12	28	2	0	64	-22*	18	126	16	-12	8	0	76	-48*	0	445	14	-34
2	465	17	7	3	26	64	19*	19	25	59	5*	9	66	34	-1*	1	305	12	-19
3	53	63	32*	4	88	113	13*	20	36	66	-6*	10	171	18	11	2	2	39	-21*
4	144	11	7	5	0	70	-35*	21	0	64	-24*	11	0	59	-4*	3	452	14	-7
5	96	16	15	6	104	28	63*	H,L=		7,	3	12	51	74	-5*	4	540	17	-16
6	439	16	6	7	0	79	-22*	1	130	9	0	13	0	59	-34*	5	149	11	-5
7	73	25	15*	8	42	66	-23*	2	497	16	-3	14	109	19	1	6	139	9	-1
8	229	18	-1	9	0	65	-17*	3	85	16	24*	15	0	62	-13*	7	521	16	3

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
8	397	13	2	19	153	14	2	11	0	73	-41*	8	59	67	18*	H,L=	9,	4	
9	38	81	27*	20	0	65	-85*	12	142	15	0	9	458	15	0	0	121	12	6
10	197	10	-2	21	0	77	-98*	13	188	17	-10	10	163	10	7	1	231	13	-19
11	368	14	12	H,L=	8,	3		14	137	17	-23	11	38	52	21*	2	0	53	-31*
12	336	12	1	1	193	9	-4	15	67	44	-27*	12	0	55	-34*	3	288	11	-11
13	119	13	-5	2	268	11	-12	16	127	18	0	13	417	14	11	4	122	13	14
14	210	11	-4	3	110	11	-11	17	143	16	17	14	187	11	13	5	136	12	-4
15	341	12	18	4	0	48	-33*	H,L=	8,	6		15	58	64	-4*	6	0	72	-3*
16	229	11	13	5	246	10	-4	0	92	20	55*	16	29	74	-17*	7	338	13	-12
17	118	30	-50*	6	215	9	-5	1	46	84	-3*	17	294	12	-1	8	93	19	-8*
18	222	11	17	7	101	29	7*	2	62	83	57*	18	106	29	27*	9	54	56	23*
19	169	13	-4	8	92	16	4	3	48	59	19*	19	134	19	-7	10	6	58	-32*
20	77	35	-28*	9	241	17	13	4	67	83	-8*	20	93	26	34*	11	338	12	15
21	125	20	-21	10	198	10	5	5	73	101	40*	21	180	16	20	12	111	18	9
H,L=	8,	1		11	48	54	22*	6	0	60	-8*	H,L=	9,	2		13	0	62	-59*
1	48	25	-44*	12	130	14	7	7	0	61	-51*	0	0	46	-44*	14	68	34	53*
2	166	8	-14	13	169	12	-16	8	64	42	11*	1	193	8	-9	15	211	13	-3
3	0	42	-22*	14	146	28	17	9	24	59	11*	2	0	46	-43*	16	42	65	-47*
4	0	43	-15*	15	110	33	25*	10	0	62	-4*	3	182	9	-15	17	81	41	-11*
5	70	15	-24*	16	107	19	-4	11	60	68	10*	4	88	14	-6	18	75	36	44*
6	160	8	-7	17	95	25	-25*	12	43	62	10*	5	138	9	13	H,L=	9,	5	
7	0	46	-14*	18	57	62	-17*	13	0	63	-6*	6	0	48	-18*	1	140	12	0
8	54	27	2*	19	117	22	55*	14	0	64	-18*	7	166	10	-14	2	37	56	2*
9	65	23	-5*	20	116	20	13	15	0	67	-32*	8	100	14	-2	3	134	12	33
10	116	12	-9	H,L=	8,	4		H,L=	8,	7		9	51	66	12*	4	41	56	39*
11	7	50	-17*	0	188	10	-1	1	149	15	-8	10	0	53	-25*	5	122	15	-1
12	55	55	5*	1	155	10	6	2	232	12	1	11	175	11	-4	6	84	23	12*
13	46	55	-35*	2	23	50	12*	3	117	25	-12*	12	75	26	8*	7	77	26	8*
14	103	19	37	3	169	9	11	4	0	63	-23*	13	29	56	-3*	8	36	57	35*
15	74	25	33*	4	246	10	-3	5	174	14	-16	14	0	58	-7*	9	145	26	13
16	0	62	-47*	5	33	53	-26*	6	236	12	11	15	133	15	5	10	0	60	-36*
17	0	61	-60*	6	70	22	5*	7	61	62	17*	16	0	61	-52*	11	25	60	-4*
18	63	79	17*	7	198	10	10	8	90	27	16*	17	24	61	-23*	12	0	61	-8*
19	0	85	-35*	8	230	16	8	9	192	13	1	18	19	61	-6*	13	99	24	-13*
20	92	23	41*	9	0	65	-40*	10	170	15	8	19	136	20	38	14	65	51	17*
21	80	30	42*	10	100	18	15	11	0	66	-7*	20	81	40	47*	15	0	65	-2*
H,L=	8,	2		11	165	12	5	12	84	33	-5*	H,L=	9,	3		16	0	64	-18*
0	442	15	-34	12	159	14	-22	13	160	15	4	1	316	12	-4	17	111	23	29*
1	322	11	2	13	41	58	35*	H,L=	8,	8		2	160	10	9	H,L=	9,	6	
2	0	43	-9*	14	72	77	-15*	0	0	76	-58*	3	188	9	6	0	63	50	-33*
3	392	13	-13	15	125	25	-9*	1	0	65	-37*	4	0	58	-41*	1	241	11	-11
4	474	15	-17	16	112	20	-6	2	0	66	-14*	5	333	11	-6	2	65	79	55*
5	162	9	4	17	48	63	1*	3	85	30	33*	6	161	11	-1	3	274	12	-2
6	101	12	-1	18	72	42	-13*	4	0	68	-28*	7	74	23	-14*	4	114	19	-2
7	391	14	-7	19	80	36	-2*	5	15	66	12*	8	0	70	-21*	5	145	15	0
8	332	11	8	H,L=	8,	5		6	0	69	-18*	9	330	12	5	6	43	67	35*
9	83	25	9*	1	204	17	3	7	87	31	36*	10	127	13	0	7	292	12	-11
10	186	11	7	2	305	13	-7	8	80	37	56*	11	42	74	10*	8	135	15	21
11	273	10	-7	3	149	23	-7	H,L=	9,	1		12	73	53	51*	9	0	64	-42*
12	282	11	9	4	0	87	-56*	1	503	16	-33	13	297	11	13	10	48	62	23*
13	0	55	-27*	5	270	11	-6	2	226	10	-22	14	167	12	21	11	274	24	4
14	187	11	13	6	263	15	3	3	352	11	-16	15	0	62	-21*	12	104	22	25*
15	256	12	-6	7	65	36	4*	4	40	46	12*	16	43	61	17*	13	0	65	-20*
16	181	20	-2	8	106	12	14	5	513	17	-16	17	228	12	12	14	0	84	-17*
17	87	88	-9*	9	273	11	-4	6	193	9	-3	18	89	27	10*	H,L=	9,	7	
18	160	24	7	10	242	11	4	7	192	9	13	19	82	32	1*	1	23	60	12*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
2	0	62	-6*	16	0	88	-37*	9	68	34	2*	3	51	68	7*	15	0	62	-20*
3	0	61	-17*	17	53	62	-23*	10	0	60	-74*	H,L=	11,	1		16	90	28	-21*
4	0	63	0*	18	0	62	-26*	11	190	12	-2	1	294	10	-7	17	180	20	9
5	75	32	51*	19	40	64	17*	12	66	35	55*	2	244	10	-20	18	20	66	17*
6	82	28	59*	20	93	23	69*	13	0	62	-39*	3	181	9	-12	H,L=	11,	4	
7	86	27	76*	H,L=	10,	2		14	69	36	14*	4	37	49	-19*	0	190	11	-16
8	27	64	19*	0	60	23	6*	15	182	13	20	5	307	12	-13	1	179	11	0
9	65	61	43*	1	317	12	0	16	0	65	-14*	6	264	12	11	2	62	52	11*
10	86	30	79*	2	66	21	16*	17	0	72	-11*	7	135	14	3	3	233	10	-6
11	0	98	-11*	3	395	13	-19	H,L=	10,	5		8	157	11	-13	4	146	22	-8
H,L=	9,	8		4	59	25	32*	1	286	11	-11	9	323	15	19	5	150	12	25
0	67	53	18*	5	211	10	-1	2	61	70	-6*	10	157	13	-3	6	74	84	-21*
1	146	26	14	6	53	67	-24*	3	201	11	-7	11	130	20	43	7	213	12	-7
2	88	34	79*	7	386	13	5	4	46	60	2*	12	186	16	-29	8	162	12	21
3	132	38	-25*	8	42	53	-2*	5	322	12	4	13	295	11	19	9	69	70	7*
4	84	50	7*	9	112	19	32	6	45	81	31*	14	83	24	15*	10	101	20	-13*
5	60	69	-30*	10	102	19	-9*	7	100	21	3*	15	0	59	-26*	11	197	22	6
6	0	77	-2*	11	351	12	-2	8	0	61	-52*	16	157	13	-8	12	0	62	-92*
H,L=	10,	0		12	0	76	-35*	9	325	12	11	17	232	18	23	13	53	61	20*
0	129	10	-7	13	51	58	19*	10	54	60	32*	18	0	79	-1*	14	101	24	-35*
1	385	13	-16	14	63	58	-11*	11	60	61	37*	19	68	89	43*	15	189	13	24
2	35	45	1*	15	308	12	19	12	106	31	36*	H,L=	11,	2		16	0	66	-15*
3	503	17	-23	16	40	61	2*	13	281	12	30	0	194	12	-6	H,L=	11,	5	
4	108	13	15	17	26	72	-37*	14	57	63	38*	1	75	18	8*	1	97	32	-5*
5	269	10	2	18	64	52	8*	15	9107		-26*	2	21	52	-9*	2	24	59	-56*
6	0	50	-19*	19	215	13	17	16	60	67	-5*	3	106	14	-15	3	75	28	-8*
7	471	15	17	H,L=	10,	3		H,L=	10,	6		4	123	12	-1	4	60	44	50*
8	82	18	17*	1	227	10	-3	0	0	81	-7*	5	90	34	19*	5	117	17	19
9	96	19	-9*	2	116	23	12*	1	31	63	-16*	6	33	55	-38*	6	64	67	-14*
10	121	13	-3	3	173	10	6	2	0	71	-8*	7	160	11	12	7	73	89	24*
11	440	15	1	4	84	17	26*	3	49	74	-24*	8	112	14	30	8	0	72	-22*
12	89	26	49*	5	237	13	-10	4	0	62	-23*	9	0	56	-21*	9	39	62	-41*
13	0	59	-49*	6	40	62	4*	5	54	61	24*	10	99	40	4*	10	74	68	14*
14	114	18	-1	7	120	31	27*	6	34	60	20*	11	147	13	12	11	75	31	40*
15	376	13	8	8	86	19	37*	7	72	35	20*	12	6	97	-38*	12	0	65	-68*
16	0	61	-38*	9	261	11	9	8	0	63	-10*	13	16	58	4*	13	108	45	33*
17	29	61	-44*	10	0	80	-47*	9	0	65	-20*	14	185	18	17	14	32	65	0*
18	0	64	-89*	11	0	61	-12*	10	0	64	-13*	15	136	15	28	15	0	67	-22*
19	227	13	-9	12	45	58	-14*	11	67	46	23*	16	71	31	68*	H,L=	11,	6	
20	101	23	30*	13	204	12	2	12	0	66	-3*	17	22	61	16*	0	180	13	-16
H,L=	10,	1		14	89	21	70*	13	2	65	-9*	18	81	44	14*	1	178	12	17
1	109	11	1	15	0	61	-30*	H,L=	10,	7		H,L=	11,	3		2	54	69	13*
2	89	13	-13	16	86	44	33*	1	170	15	-9	1	177	11	-7	3	170	37	-8*
3	62	39	-23*	17	153	15	9	2	27	83	-28*	2	185	10	-4	4	139	16	-3
4	0	47	-24*	18	49	65	25*	3	151	22	7	3	168	12	2	5	111	21	-7*
5	130	10	15	19	63	66	2*	4	40	64	-4*	4	59	37	-26*	6	63	63	-19*
6	0	50	-18*	H,L=	10,	4		5	192	24	-9	5	203	11	1	7	184	14	10
7	47	49	2*	0	19	56	-51*	6	56	65	48*	6	192	17	4	8	95	26	-8*
8	35	50	7*	1	144	19	-22	7	114	26	33*	7	125	14	2	9	56	64	-4*
9	135	12	12	2	70	30	40*	8	0	68	-45*	8	116	29	-22*	10	108	22	9*
10	80	19	48*	3	209	10	8	9	210	14	3	9	213	13	8	11	165	20	-8
11	0	55	-4*	4	27	55	8*	10	114	55	103*	10	130	15	19	12	41	71	-29*
12	46	70	3*	5	117	15	-5	H,L=	10,	8		11	0	60	-70*	H,L=	11,	7	
13	103	18	3	6	0	65	-28*	0	20	66	14*	12	127	16	-33	1	48	67	45*
14	0	70	-38*	7	210	13	11	1	75	38	27*	13	180	13	1	2	0	88	-25*
15	57	72	47*	8	82	22	51*	2	0	68	-9*	14	76	28	43*	3	47	64	36*

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
4	73	46	71*	11	150	14	27	H,L=	12,	6		1	12	57	-29*	1	153	11	10
5	0	75	-15*	12	158	13	8	0	0	62	-17*	2	277	14	4	2	0	67	-44*
6	0	66	-15*	13	0	60	-60*	1	27	62	-12*	3	83	59	81*	3	152	12	5
7	70	44	61*	14	154	14	-28	2	39	69	11*	4	0	96	-36*	4	272	11	8
8	0	67	-35*	15	129	16	15	3	0	63	-29*	5	0	59	-28*	5	56	47	48*
H,L=	12,	0		16	91	46	29*	4	0	63	-17*	6	248	12	1	6	22	57	8*
0	381	14	-12	17	25	65	-10*	5	0	68	-22*	7	86	26	64*	7	140	17	-13
1	112	12	-2	H,L=	12,	3		6	0	66	-2*	8	106	18	3	8	235	11	3
2	64	23	19*	1	103	15	17	7	39	65	-6*	9	0	77	-9*	9	65	67	60*
3	153	11	8	2	167	13	-11	8	64	65	25*	10	221	11	32	10	95	21	9*
4	328	12	-4	3	68	28	-3*	9	0	66	-13*	11	0	63	-53*	11	116	19	-13
5	84	20	-11*	4	79	90	-3*	10	0	67	-14*	12	112	23	-26*	12	157	14	8
6	140	18	12	5	87	21	-11*	H,L=	12,	7		13	47	74	43*	13	61	61	28*
7	112	24	-24*	6	166	12	-5	1	76	35	20*	14	112	56	14*	14	86	31	-21*
8	329	12	14	7	71	29	5*	2	175	15	-2	15	47	65	-10*	15	70	47	-23*
9	48	57	-48*	8	140	13	23	3	67	51	13*	H,L=	13,	4		16	128	30	-3*
10	251	20	3	9	54	59	-6*	4	33	68	-29*	0	240	11	-4	H,L=	14,	1	
11	153	15	25	10	148	13	-3	5	108	23	62*	1	52	69	29*	1	0	76	-28*
12	217	11	1	11	0	61	-39*	6	154	17	0	2	82	23	44*	2	97	25	15*
13	16	60	-54*	12	124	19	-3	H,L=	13,	1		3	74	27	66*	3	68	28	42*
14	199	14	-16	13	69	76	2*	1	0	55	-31*	4	192	12	-27	4	0	56	-2*
15	117	19	4	14	90	24	8*	2	352	12	-14	5	25	59	8*	5	78	24	36*
16	132	17	18	15	16	63	-22*	3	34	72	7*	6	87	23	17*	6	73	28	5*
17	81	29	19*	16	118	43	1*	4	64	31	-10*	7	81	25	61*	7	0	58	-4*
18	198	28	-4	17	110	27	44*	5	43	55	22*	8	169	13	2	8	0	76	-12*
H,L=	12,	1		H,L=	12,	4		6	321	12	3	9	74	30	39*	9	82	25	23*
1	0	50	-5*	0	209	21	-2	7	85	23	1*	10	64	55	-36*	10	0	61	-35*
2	71	24	-41*	1	93	36	20*	8	148	14	-1	11	41	70	15*	11	0	60	-13*
3	0	59	-11*	2	0	59	-20*	9	96	18	55*	12	114	21	-17	12	0	66	-35*
4	49	72	19*	3	85	34	40*	10	234	12	12	13	65	54	9*	13	20	83	6*
5	0	53	-30*	4	198	12	3	11	63	36	11*	14	114	21	-1*	14	68	75	44*
6	74	36	-5*	5	35	60	-17*	12	158	14	-30	H,L=	13,	5		15	0	64	-25*
7	0	55	-34*	6	0	82	-23*	13	53	60	40*	1	69	50	63*	H,L=	14,	2	
8	85	62	5*	7	78	45	16*	14	179	13	20	2	110	19	8	0	235	11	-6
9	32	56	-5*	8	184	12	15	15	87	58	17*	3	82	75	43*	1	87	39	-29*
10	0	59	-55*	9	77	31	40*	16	146	17	-22	4	0	66	-15*	2	60	37	11*
11	9	57	-14*	10	84	54	-17*	17	80	32	76*	5	59	61	57*	3	81	27	-43*
12	103	17	18	11	64	55	9*	H,L=	13,	2		6	105	21	24*	4	239	11	11
13	0	61	-47*	12	92	24	8*	0	153	12	-5	7	72	34	68*	5	29	58	15*
14	74	58	45*	13	86	25	42*	1	5	62	-2*	8	0	65	-36*	6	0	60	-52*
15	13	62	-11*	14	75	59	-19*	2	0	55	-21*	9	20	64	6*	7	133	14	6
16	58	61	1*	15	0	67	-48*	3	66	81	60*	10	104	112	28*	8	222	12	-10
17	0	93	-35*	H,L=	12,	5		4	122	16	-35	11	0	91	-22*	9	0	60	-26*
18	24	65	16*	1	57	61	-39*	5	56	57	18*	12	54	66	9*	10	69	43	-33*
H,L=	12,	2		2	256	11	-7	6	0	60	-63*	H,L=	13,	6		11	98	22	-14*
0	320	14	-16	3	78	43	-7*	7	0	58	-37*	0	205	13	-12	12	158	15	1
1	116	13	-2	4	27	76	-40*	8	143	13	17	1	77	30	72*	13	0	64	-64*
2	91	15	30	5	120	21	31	9	42	58	0*	2	55	61	27*	14	103	30	-6*
3	117	25	13*	6	179	19	-10	10	67	39	-27*	3	5	65	-29*	15	67	55	-8*
4	303	12	-11	7	26	63	-41*	11	77	28	39*	4	205	13	13	H,L=	14,	3	
5	131	13	9	8	61	64	-35*	12	93	22	6*	5	0	64	-18*	1	76	26	-2*
6	132	13	-4	9	76	35	-8*	13	44	70	8*	6	54	75	-5*	2	146	16	-3
7	133	14	15	10	143	25	-2	14	75	95	-8*	7	0	66	-34*	3	60	44	0*
8	307	12	17	11	55	79	6*	15	53	73	42*	8	179	14	25	4	57	78	6*
9	84	24	-20*	12	102	26	-39*	16	61	64	20*	H,L=	14,	0		5	71	32	-10*
10	212	11	-14	13	131	23	41	H,L=	13,	3		0	288	13	-13	6	160	13	10

STRUCTURE FACTORS CONTINUED FOR
UC(22)H(24)

K	FOB	SG	DEL
7	0	73	-3*
8	0	82	-47*
9	80	56	-25*
10	89	27	-13*
11	29	62	9*
12	18	64	-35*
13	12	65	-57*
14	120	19	40
H,L= 14, 4			
0	144	14	-9
1	0	60	-45*
2	0	72	-17*
3	60	54	-6*
4	139	22	28
5	55	68	31*
6	34	63	7*
7	78	35	22*
8	145	15	45
9	59	62	53*
10	70	71	13*
11	18	64	-37*
12	107	22	24*
H,L= 14, 5			
1	110	19	11
2	134	18	-24
3	60	62	-1*
4	0	66	-30*
5	99	23	-7*
6	181	13	35
7	60	64	57*
8	18	67	-36*
9	96	26	4*
10	108	26	-9*
H,L= 14, 6			
0	72	37	29*
1	0	65	-4*
2	27	65	26*
3	0	78	-23*
4	44	66	24*
5	103	22	91*
H,L= 15, 1			
1	185	11	-18
2	153	17	29
3	92	26	-9*
4	0	68	-24*
5	182	12	-13
6	131	16	-3
7	18	60	-29*
8	0	60	-12*
9	182	13	-8
10	129	17	17
11	0	64	-11*
12	0	64	-40*
13	149	16	-1
14	118	20	15
H,L= 15, 2			

K	FOB	SG	DEL
0	0	68	-67*
1	62	41	-13*
2	0	59	-19*
3	74	28	-1*
4	86	36	15*
5	58	72	10*
6	14	65	3*
7	89	25	-12*
8	0	62	-47*
9	66	66	51*
10	36	63	26*
11	61	64	-15*
12	15	65	-21*
13	0	67	-19*
H,L= 15, 3			
1	129	21	7
2	116	44	17*
3	63	44	-2*
4	0	62	-37*
5	129	18	0
6	78	34	-11*
7	78	30	35*
8	73	33	62*
9	128	18	-9
10	66	66	-24*
11	0	79	-15*
12	0	67	-26*
H,L= 15, 4			
0	65	42	11*
1	109	19	3
2	0	63	-20*
3	122	17	-10
4	104	21	27*
5	71	83	18*
6	70	84	54*
7	142	16	2
8	102	23	21*
9	38	77	34*
10	62	65	44*
H,L= 15, 5			
1	0	66	-51*
2	99	22	39*
3	0	66	-17*
4	0	65	-1*
5	75	61	18*
6	0	66	-35*
7	0	65	-18*
H,L= 16, 0			
0	0	75	-7*
1	155	13	3
2	0	68	-4*
3	203	12	-2
4	67	75	61*
5	107	68	16*
6	0	72	-51*
7	187	13	-6

K	FOB	SG	DEL
8	61	53	44*
9	51	63	12*
10	70	47	38*
11	161	16	-14
12	102	41	59*
H,L= 16, 1			
1	0	61	-31*
2	66	90	51*
3	9	59	-24*
4	0	60	-8*
5	0	61	-35*
6	30	75	29*
7	0	62	-18*
8	0	62	-4*
9	0	64	-40*
H,L= 16, 2			
0	58	59	37*
1	115	27	1*
2	40	53	26*
3	165	13	-3
4	64	66	48*
5	105	20	26*
6	11	78	-14*
7	171	14	1
8	73	37	46*
9	42	64	1*
10	0	65	-36*
11	149	24	-24
H,L= 16, 3			
1	118	17	28
2	57	60	56*
3	99	21	30*
4	31	62	26*
5	123	17	10
6	56	73	39*
7	109	20	77
8	64	59	40*
9	90	65	-23*
10	0	74	-23*
H,L= 16, 4			
0	21	87	-27*
1	102	54	43*
2	60	86	53*
3	93	26	-4*
4	55	62	40*
5	101	23	44*
6	43	65	26*
7	21	69	-64*
H,L= 16, 5			
1	118	35	14*
H,L= 17, 1			
1	150	14	18
2	60	81	-14*

K	FOB	SG	DEL
3	100	22	1*
4	0	63	-41*
5	166	17	30
6	51	80	-13*
7	65	65	-7*
8	86	95	15*
9	177	14	22
H,L= 17, 2			
0	0	64	-24*
1	78	30	32*
2	31	62	24*
3	78	30	30*
4	30	63	9*
5	81	28	53*
6	61	64	34*
7	52	79	-6*
8	75	34	58*
H,L= 17, 3			
1	133	16	40
2	65	46	32*
3	99	23	29*
4	75	38	55*
5	113	28	27*
6	79	32	31*
H,L= 17, 4			
0	94	24	39*
1	70	46	-5*
2	37	66	28*
H,L= 18, 0			
0	125	17	-9
1	84	40	4*
2	0	63	-20*
3	98	25	32*
4	136	16	22
5	0	67	-70*
6	84	30	13*
9	56	68	4*
H,L= 18, 1			
1	14	63	-2*
2	0	65	-17*
3	0	64	-13*
4	0	90	-10*
5	0	73	-11*
6	45	66	28*
9	0	89	-15*
H,L= 18, 2			
0	93	44	-18*
1	73	46	11*
2	0	70	-10*
3	57	67	1*
4	131	16	34
H,L= 19, 1			
1	67	46	56*
2	119	29	-7*
3	0	67	-23*
4	0	67	-25*

K	FOB	SG	DEL
5	0	81	-8*
H,L= 19, 2			
0	112	21	45*
1	0	76	-4*
2	0	68	-4*
3	0	68	-8*
H,L= 20, 0			
0	119	29	19*
1	104	23	85*
2	60	68	57*
3	108	22	79*
H,L= 20, 1			
1	0	69	-11*
2	0	70	-23*
3	80	35	77*
H,L= 20, 2			
0	96	35	13*
1	70	88	52*
2	114	21	108*
3	25	69	0*

TECHNICAL INFORMATION DEPARTMENT
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720