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Publication Date

1982-04-01



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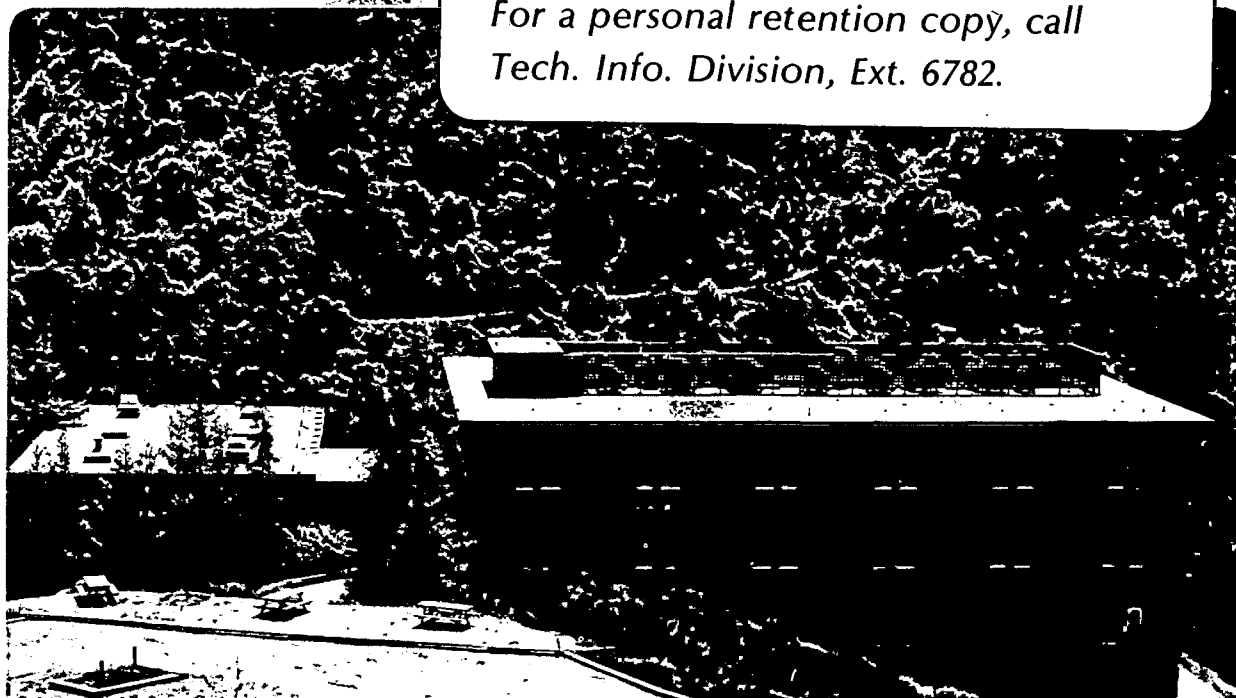
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April 1982

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APRIL 1982

ABSTRACT

The diphosphine, $\text{Me}_2\text{PCH}_2\text{CH}_2\text{PMe}_2$, reacts with $\text{M}(\text{Me}_5\text{C}_5)_2(\text{OEt}_2)$ to give insoluble $\text{M}(\text{Me}_5\text{C}_5)_2(\text{Me}_2\text{PCH}_2\text{CH}_2\text{PMe}_2)$, where M is Eu or Yb. In contrast, $\text{Me}_2\text{PCH}_2\text{PMe}_2$ gives the hydrocarbon soluble complexes $\text{M}(\text{Me}_5\text{C}_5)_2(\text{Me}_2\text{PCH}_2\text{PMe}_2)$, where M is Eu or Yb. The ytterbium complex reacts with YbCl_3 in toluene to give $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)$ and the crystal structure shows that the phosphine is acting as a monodentate ligand. $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)$ crystallizes in the monoclinic space group $\text{P}2_1/\text{c}$ with $\underline{a} = 16.358(6) \text{ \AA}$, $\underline{b} = 8.595(4) \text{ \AA}$, $\underline{c} = 20.712(7) \text{ \AA}$, $\beta = 104.75(4)^\circ$, $\underline{V} = 2816 \text{ \AA}^3$ and $d(\text{calcd}) = 1.45 \text{ g cm}^{-3}$ for $Z = 4$ and mol wt = 615.15. Diffraction data were collected with a CAD-4 automated diffractometer, and the structure refined to $R = 0.054$ for 3309 reflections with $4^\circ < 2\theta < 45^\circ$ ($\text{MoK}\alpha$ radiation). The Yb is coordinated to the two Me_5C_5 groups, the chlorine atom, and to one phosphorus atom of the bis(dimethylphosphino)methane ligand. The Yb-P and Yb-Cl distances are 2.94 \AA and 2.53 \AA , respectively. The centroids of the C_5Me_5 ligands, the Cl and P atoms are in an approximate tetrahedral arrangement about the Yb atom.

INTRODUCTION

The coordination chemistry of lanthanide metals with nitrogen or oxygen ligands is extensive.¹ Since lanthanide ions form their most stable coordination complexes with nitrogen or oxygen rather than with phosphorus or sulfur ligands, these metals are classified as "class a"^{2a} or "hard"^{2b} acceptors.³ Until recently the idea that f-block metals could form isolatable complexes with tertiary phosphines was viewed with considerable skepticism. The successful isolation of bis-1,2-dimethylphosphinoethane (dmpe) complexes of tetravalent thorium or uranium of the type $\text{MX}_4(\text{dmpe})_2$ ^{4a} and of the trivalent uranium species, $\text{U}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{dmpe})$ ^{4b} raises an obvious question about the "class a" classification of the f-block metals.

In order to examine the coordinative affinity of f-block metals for phosphine ligands we have begun a systematic study of their general coordination chemistry. In this paper we describe the phosphine complexes of the di- and tri-valent bis(pentamethylcyclopentadienyl)ytterbium and the divalent europium fragments. In a related study we described the preparation and crystal structure of $\text{Yb}[\text{N}(\text{SiMe}_3)_2]_2(\text{dmpe})$, and some related europium (II) derivatives.⁵

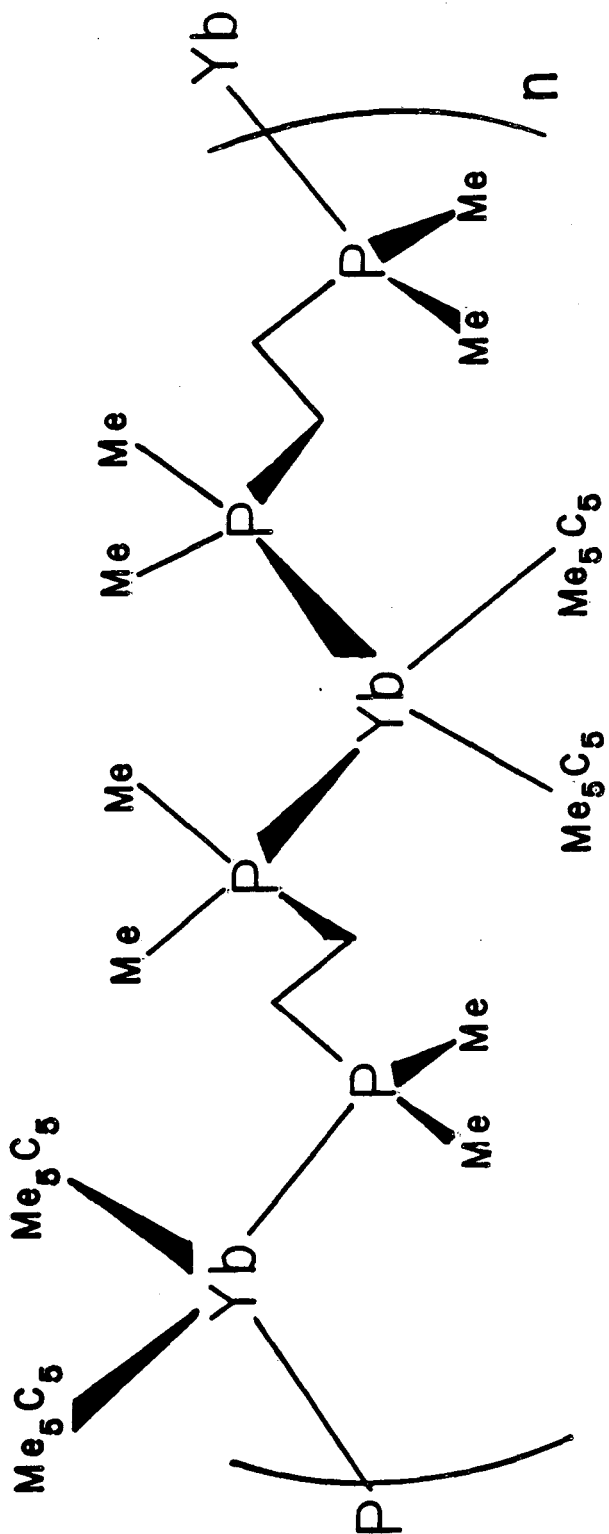
Synthetic Studies. Addition of one molar equivalent of bis-1,2-dimethylphosphinoethane to the diethyl ether complex of bis(pentamethylcyclopentadienyl)ytterbium (II) in benzene results in instantaneous precipitation of a green material whose elemental analysis suggests the formulation $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{dmpe})$. Support for this composition is obtained by aqueous hydrolysis of the complex suspended in

benzene- d_6 . Examination of the benzene solution by ^1H NMR spectroscopy shows resonances due to $\text{Me}_5\text{C}_5\text{H}$ and $\text{Me}_2\text{PCH}_2\text{CH}_2\text{PMe}_2$ in an area ratio of 2:1. In addition, the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of this solution shows the presence of $\text{Me}_2\text{PCH}_2\text{CH}_2\text{PMe}_2$ as the only phosphorus containing species. The compound is insoluble in hot toluene though sparingly soluble in diethyl ether. The complex dissolves in tetrahydrofuran giving $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{thf})^6$. The europium complex, $\text{Eu}(\text{Me}_5\text{C}_5)_2(\text{OEt}_2)$ behaves similarly, affording red, insoluble $\text{Eu}(\text{Me}_5\text{C}_5)_2(\text{dmpe})$.

The infrared spectra and melting points (see experimental section for details) of these two phosphine complexes are identical which suggests that the complexes have a similar structure. The insolubility in non-coordinating solvents indicates that the dmpe complexes are some type of coordination polymer. One such polymer is shown (I). The phosphine ligand is acting as a bridging rather than as a chelating ligand yielding an eight coordinate

See illustration, next page

3a



complex (defining the coordination number of the pentamethylcyclopentadienyl ligand as three). Eight coordination is not unusual since some $M(\text{Me}_5\text{C}_5)_2\text{L}_2$ complexes, where $M = \text{Yb}^{7a}$ or Sm^{7b} and L is a monodentate ligand, have been described. If it is accepted that (I) is a reasonable structural formulation then replacing the ethylene bridge in $\text{Me}_2\text{PCH}_2\text{CH}_2\text{PMe}_2$ by a methylene bridge should prevent polymerization, due to increased steric hindrance between the Me_5C_5 groups on adjacent metal atoms. Thus, $\text{Me}_2\text{PCH}_2\text{PMe}_2$ should yield hydrocarbon-soluble, monomeric complexes.

Addition of a slight excess of bis-dimethylphosphinomethane (dmpm) to a toluene solution of $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{OEt}_2)$ yields a green solution from which green, diamagnetic $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{dmpm})$ may be isolated by crystallization from that solvent. The NMR spectral data (see experimental section for details) support this formulation. In particular, the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of the complex contains a single resonance at δ -39.6, significantly deshielded from that of the free phosphine at δ -55.7. The red europium complex, $\text{Eu}(\text{Me}_5\text{C}_5)_2(\text{dmpm})$, was prepared similarly. As europium (II) is paramagnetic (a f^7 ion) no NMR spectral data are obtainable. However, hydrolysis of a benzene solution of the complex and examination of this solution by ^1HMR spectroscopy shows that $\text{Me}_5\text{C}_5\text{H}$ and $\text{Me}_2\text{PCH}_2\text{PMe}_2$ are present in a 2:1 ratio. Further, the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of the hydrolysate shows only the presence of $\text{Me}_2\text{PCH}_2\text{PMe}_2$. Not surprisingly the infrared spectra and melting points of the Yb(II) and Eu(II) complexes are identical. It is noteworthy that diethyl ether displaces the coordinated $\text{Me}_2\text{PCH}_2\text{PMe}_2$, giving $M(\text{Me}_5\text{C}_5)_2(\text{OEt}_2)$, $M = \text{Yb}$ or Eu . This is to

be contrasted with the observation that diethyl ether will not, though tetrahydrofuran will, displace dmpe from (I).

It is significant that we have been unable to isolate complexes with the monodentate phosphines, PMe_3 or PBu_3^n , nor with the bidentate phosphine, $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2$. In the monodentate phosphine case this is presumably due to the inability of two monodentate phosphines to fit comfortably around the sterically congested metal atom and the heat of formation of one metal-phosphorus bond is less than that of one metal-oxygen bond. In the bidentate phosphine case, the reason is less clear since $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2$ is a poorer σ -donor than its per-methyl analogue and it is also sterically larger.

Yb^{8a} and others^{8b} have shown that the divalent species, $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{L})$, where L is tetrahydrofuran or 1,2-dimethoxyethane, may be oxidized by ytterbium trichloride or dichloromethane to the trivalent metallocene, $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{L})$. The divalent phosphine complex behaves similarly. Mixing $\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{dmpm})$ and YbCl_3 in toluene gives the purple, hydrocarbon soluble $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{dmpm})$. The complex is paramagnetic, $\mu_B = 4.4$ B.M. at 30°C in benzene solution, and accordingly we have been unable to observe a $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of this complex.

The coordination geometry of this complex is of considerable interest as numerous possibilities exist. If both phosphorus atoms of the diphosphine are coordinated to the ytterbium atom, a nine-coordinate complex will result. Nine coordination in bis-pentamethylcyclopentadienyl lanthanide chemistry is unknown, though eight coordination is common.^{7,9} Two eight coordinate formulations are possible. In one isomer, $\text{Me}_2\text{PCH}_2\text{PMe}_2$ is acting as a monodentate phosphine and in the other

isomer an outer sphere complex, $[\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{dmpm})]\text{Cl}$ may be written. To answer this question, an X-ray crystal structure determination was undertaken, and the results are described below.

Structural Studies. ORTEP diagrams of $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)$ are shown in Figs. 1 and 2. Positional and thermal parameters are given in Table I and some selected bond lengths and angles are listed in Tables II and III. The key point to emerge from the X-ray study is that the diphosphine is acting as a monodentate ligand giving an eight-coordinate complex. The coordination geometry may be described as pseudo-tetrahedral if the mid-points of the two Me_5C_5 -centroids occupy two coordination sites. The other two coordination positions are occupied by a phosphorus atom and a chlorine atom. The angle defined by the intersection of the two planes that contain the centroid-Yb-centroid and P(1)-Yb-Cl atom is 88.1° . In a regular tetrahedron this angle is 90° . The centroid-Yb-centroid angle is 134.9° , and the C(1...5)centroid-Yb-P(1) and C(1...5)centroid-Yb-Cl angles are 108.0° and 106.3° , respectively. The C(6...10)centroid-Yb-P(1) and C(6...10)centroid-Yb-Cl angles are 105.7° and 108.6° , respectively. The P(1)-Yb-Cl angle is $79.69(9)^\circ$.

The ytterbium-chloride bond length of $2.532(3) \text{ \AA}$ is longer than that found in $\text{Li}(\text{thf})_4[(\text{Me}_3\text{Si})_2\text{CH}]_3\text{YbCl}$, $2.486(6) \text{ \AA}$,¹⁰ as expected since the latter is a four-coordinate Yb(III) ion whereas the former is eight coordinate. The latter distance is close to the value of 2.43 \AA suggested for three coordinate, monomeric YbCl_3 in the gas phase.¹¹

These are the only terminal Yb-Cl distances that are available, though several Yb-(μ -Cl) distances are known. These distances are 2.58 Å in Yb₂[(Me₃Si)₂C₅H₃]₄(μ -Cl)₂,^{9c} 2.756 ± 0.004 Å in Yb(Me₅C₅)₂(μ -Cl)₂AlCl₂,^{9a} and 2.637 ± 0.010 Å in Yb₂(MeC₅H₄)₄(μ -Cl)₂.¹²

The ytterbium-phosphorus(1) distance of 2.941(3) Å is unique and no comparisons are possible. The other ytterbium-phosphorus distance, Yb...P(2) is greater than 3.5 Å and is obviously non-bonding.

It is instructive to compare the bond lengths and angles of monodentate Me₂PCH₂PMe₂ in Yb(Me₅C₅)₂Cl(Me₂PCH₂PMe₂) with that of free Me₂PCH₂PMe₂ in the gas phase. The averaged P-C distance in the ytterbium complex is 1.849 ± 0.006 Å. This is identical to that found in the free phosphine, 1.849(2) Å.¹³ The averaged C-P-C and P-C-P angles in the complexed phosphine of 100.4 ± 2.2° and 118.0(5)° also are identical to those found in the free phosphine, 100° and 118°, respectively. Further the P...P distance in the coordination complex of 3.157(4) Å is very close to that found [3.139(9) Å] in the free phosphine. Thus, Me₂PCH₂PMe₂ is not perturbed very much upon coordination to Yb(Me₅C₅)₂Cl.

The averaged ytterbium-carbon bond length in the phosphine complex of 2.65 ± 0.03 Å is equal to that (2.63 ± 0.03 Å) found in Yb(Me₅C₅)₂S₂SNEt.¹⁴ This is expected since the coordination environment of ytterbium is similar in both complexes. The averaged ytterbium to carbon bond length is in the range found for related complexes (2.56 - 2.65 Å).⁹

EXPERIMENTAL SECTION

All reactions were performed under nitrogen. Analyses were carried out by the microanalytical laboratory of this department. Proton, carbon, and phosphorus NMR spectra were obtained on a JEOL-FX90Q instrument operating at 89.56, 22.50 and 36.25 MHz, respectively.

Eu(Me₅C₅)₂(Me₂PCH₂CH₂PMe₂). 1,2-Dimethylphosphinoethane (0.17 mL, 0.0010 mol) was added to the diethyl ether complex of bis(pentamethylcyclopentadienyl)europium (0.51 g, 0.0010 mol) in toluene (20 mL). The red suspension was stirred for 1 h and the solid was collected by filtration, washed with toluene (75 mL), and dried under reduced pressure. A small quantity (ca. 0.15 g) of the precipitate was crystallized from diethyl ether (ca. 20 mL, -10°C), mp. 288–292°C. Anal. Calcd for C₂₆H₄₆EuP₂: C, 54.5; H, 8.10; P, 10.8. Found: C, 53.7; H, 7.83; P, 10.5. IR(Nujol); 2721 w, 1421 m, 1302 m, 1284 w, 1150 w, 1091 w, 1015 w, 945 s, 926 m, 889 w, 829 w, 796 w, 720 s, 672 w, 624 m, 637 w, 589 w, 360 m, 349 m and 253 s cm⁻¹. A portion of the complex was hydrolyzed (H₂O) in benzene-d₆ and the benzene solution was shown to contain Me₅C₅H and Me₂PCH₂CH₂PMe₃ in a 2:1 area ratio by ¹H NMR spectroscopy. The phosphine was identified by its ³¹P{¹H} NMR spectrum.

Yb(Me₅C₅)₂(Me₂PCH₂CH₂PMe₂). 1,2-Dimethylphosphinoethane (0.17 mL, 0.0010 mol) was added to the diethyl ether complex of bis(pentamethylcyclopentadienyl)ytterbium (0.48 g, 0.00093 mol) in benzene (25 mL). After stirring for 1 h the green suspension was collected by filtration, washed

with toluene (75 mL) and dried under reduced pressure. A small portion (ca. 0.2 g) was crystallized from diethyl ether (ca. 20 mL, -10°C), mp: 283–285 $^{\circ}\text{C}$. Anal. Calcd for $\text{C}_{26}\text{H}_{46}\text{P}_2\text{Yb}$: C, 52.6; H, 7.81; P, 10.4. Found: C, 51.8; H, 7.69; P, 10.0. The infrared spectrum was identical to that of its europium analogue.

$\text{Eu}(\text{Me}_5\text{C}_5)_2(\text{Me}_2\text{PCH}_2\text{PMe}_2)$. 1,2-Dimethylphosphinomethane (0.30 mL, 0.0018 mol) was added to the diethylether complex of bis(pentamethylcyclopentadienyl)europium (0.87 g, 0.0018 mol) in toluene (40 mL) and the red solution was stirred for 30 min. The solution was cooled (-10°C) and the red needles, mp 251–253 $^{\circ}\text{C}$, were isolated in quantitative yield. Anal. Calcd for $\text{C}_{25}\text{H}_{44}\text{EuP}_2$: C, 53.8; H, 7.94; P, 11.1. Found: C, 53.4; H, 7.85; P, 10.8. IR(Nujol); 2720 w, 1420 m, 1380 m, 1285 m, 1161 w, 1109 m, 1055 w, 1015 m, 942 s, 925 m, 888 s, 830 m, 795 w, 749 s, 725 s, 705 w, 693 w, 647 w, 623 w, 589 w, 352 s, and 253 s cm^{-1} . A portion of the complex was hydrolyzed (H_2O) in benzene- d_6 and the benzene solution was shown to contain $\text{Me}_5\text{C}_5\text{H}$ and $\text{Me}_2\text{PCH}_2\text{PMe}_2$ in an area ratio of 2:1. The $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of the solution contained only $\text{Me}_2\text{PCH}_2\text{PMe}_2$.

$\text{Yb}(\text{Me}_5\text{C}_5)_2(\text{Me}_2\text{PCH}_2\text{PMe}_2)$. 1,2-Dimethylphosphinomethane (0.16 mL, 0.0011 mol) was added to bis(pentamethylcyclopentadienyl)-diethylether-ytterbium (0.57 g, 0.0011 mol) in toluene (25 mL). After stirring for 8 h, the green solution was filtered and the filtrate was cooled (-10°C). The green needles, mp 250–253 $^{\circ}\text{C}$, were isolated in quantitative yield. Anal. Calcd for $\text{C}_{25}\text{H}_{44}\text{P}_2\text{Yb}$: C, 51.8; H, 7.65; P, 10.7.

Found: C, 50.6; H, 7.41; P, 10.2. The infrared spectrum was identical to that of its europium analogue. ^1H NMR (PhH-d_6 , 26°C): δ 2.17 s (30 H), Me_5C_5 ; 1.78 d, $J = 4.1$ Hz (2 H), CH_2P ; and 0.95 s (12 H), Me_2P . $^{13}\text{C}\{^1\text{H}\}$ NMR (PhH-d_6 , 26°C): δ 112 s, Me_5C_5 ; 44.6 s; CH_2P , 15.8 s, Me_2P ; and 12.0 s, Me_5C_5 . $^{31}\text{P}\{^1\text{H}\}$ NMR (PhH-d_6 , 26°C): δ -39.6.

$\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)$. Bis(pentamethylcyclopentadienyl)(1,2-dimethylphosphinomethane)ytterbium (0.74 g, 0.0013 mol) in toluene (75 mL) was added to a suspension of ytterbium trichloride (0.36 g, 0.0013 mol) in toluene (10 mL). The suspension was stirred for 24 h, the solution was filtered and the filtrate was concentrated to ca. 10 mL and cooled (-10°C). The prisms (0.52 g, 66%) were collected and dried under reduced pressure, mp $208^\circ\text{C}(\text{dec})$. Anal. Calcd for $\text{C}_{25}\text{H}_{44}\text{ClP}_2\text{Yb}$: C, 48.8; H, 7.21; Cl, 5.77; P, 10.1. Found: C, 48.6, H, 7.06; Cl, 5.77; P, 9.88. IR(Nujol); 2718 w, 1292 m, 1276 w, 1149 m, 1087 w, 1014 m, 943 s, 908 s, 880 w, 830 w, 799 w, 759 m, 723 m, 709 w, 699 w, 683 w, 615 w, 590 w, 372 m, 298 s, and 248 s cm^{-1} . The effective magnetic moment (PhH , 30°C) was 4.4 B. M.

X-RAY CRYSTALLOGRAPHY

A crystal, approximately 0.4 x 0.4 x 0.25 mm in size, was sealed inside a quartz capillary and mounted on a CAD4 automatic diffractometer, and a set of θ - 2θ scan data were collected. Details are given in Table IV. Data were corrected for crystal decay, absorption¹⁵ and Lorentz and polarization effects.

The ytterbium atom position was located with the use of a 3-dimensional Patterson map, and subsequent least-squares and electron density maps resolved the location of all of the non-hydrogen atoms. The structure was refined to convergence using anisotropic thermal parameters for the Yb, Cl and P atoms and isotropic thermal parameters for the carbon atoms. Atomic scattering factors of Doyle and Turner¹⁶ were used, and anomalous scattering corrections¹⁷ were applied. The final R factors for 3202 unique data, ($F^2 > 3\sigma(F^2)$), are $R_F = 0.053$ and $R_{WF} = 0.073$.¹⁸ An extinction correction was applied to the observed structure factors.¹⁹

ACKNOWLEDGMENT

This work is 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 DE-AC03-76SF00098. We thank Dr. F. L. Hollander, staff crystallographer of the U.C. Berkeley X-ray facility (CHEXRAY) for collecting the X-ray data.

SUPPLEMENTARY MATERIAL AVAILABLE

Listing of anisotropic thermal parameters and listings of observed and calculated structure factors (14 pages). Ordering information is given on any current masthead.

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18. $R_F = \Sigma ||F_O| - |F_C|| / \Sigma |F_O|$;
 $R_{wf} = [\Sigma w(|F_O| - |F_C|)^2 / \Sigma w |F_O|^2]^{1/2}$.
19. $(F_O)_{corr} = (1 + kI)F_O$ where $R = 2.6 \times 10^{-7}$ and I is the uncorrected intensity.

Table I. Positional and isotropic thermal^a parameters in
 $(C_5Me_5)_2YbCl(Me_2PCH_2CHPMe_2)$.

Atom	x	y	z	B, Å
YB	.23280(2)	.08305(4)	.17649(2)	2.78*
CL	.31442(19)	.3313(3)	.17174(15)	5.37*
P(1)	.35006(15)	-.0247(3)	.09936(13)	3.50*
P(2)	.48632(21)	-.2765(4)	.00839(17)	5.59*
C(1)	.3512(6)	.0223(12)	.2862(5)	3.64(18)
C(2)	.2933(6)	.1204(12)	.3052(5)	4.08(28)
C(3)	.2179(6)	.0323(13)	.2983(5)	4.28(20)
C(4)	.2296(6)	-.1139(12)	.2733(5)	4.07(20)
C(5)	.3129(6)	-.1219(11)	.2653(5)	3.37(17)
C(6)	.0904(7)	-.0369(13)	.0976(5)	4.46(21)
C(7)	.0676(7)	.0651(12)	.1431(5)	4.42(21)
C(8)	.0876(6)	.2202(12)	.1281(5)	4.37(20)
C(9)	.1239(6)	.2121(12)	.0711(5)	4.39(21)
C(10)	.1250(7)	.0528(13)	.0533(5)	4.48(21)
C(11)	.4448(8)	.0695(14)	.2951(6)	5.56(26)
C(12)	.3112(9)	.2808(17)	.3373(7)	7.0(3)
C(13)	.1447(10)	.0904(16)	.3276(8)	7.0(3)
C(14)	.1719(9)	-.2524(17)	.2687(7)	6.49(28)
C(15)	.3529(7)	-.2691(14)	.2473(6)	5.46(24)
C(16)	.0732(9)	-.2080(17)	.0918(7)	7.1(3)
C(17)	.0094(9)	.0231(19)	.1852(7)	7.1(3)
C(18)	.0735(9)	.3720(18)	.1620(7)	7.2(3)
C(19)	.1451(8)	.3504(17)	.0356(7)	6.40(28)
C(20)	.1389(8)	-.0038(18)	-.0117(7)	6.62(29)
C(21)	.3477(8)	.1055(15)	.0273(7)	5.72(27)
C(22)	.4642(7)	-.0109(15)	.1403(6)	5.53(25)
C(23)	.3407(6)	-.2236(12)	.0653(5)	4.03(19)
C(24)	.3318(10)	-.2246(20)	-.0724(8)	8.6(4)
C(25)	.3886(10)	-.4896(22)	.0076(8)	8.3(4)

^a B values that were derived from anisotropic thermal parameters are marked with an asterisk.

Table II. Selected Interatomic Distances (Å).

Yb-C1	2.532(3)	P(1)-C(21)	1.858(13)
Yb-P(1)	2.941(3)	P(1)-C(22)	1.847(12)
Yb-C(1)	2.635(9)	P(1)-C(23)	1.841(10)
Yb-C(2)	2.617(10)	P(2)-C(23)	1.842(10)
Yb-C(3)	2.633(10)	P(2)-C(24)	1.855(17)
Yb-C(4)	2.634(10)	P(2)-C(25)	1.854(19)
Yb-C(5)	2.642(9)		
Yb-C(6)	2.688(11)		
Yb-C(7)	2.617(11)		
Yb-C(8)	2.612(10)		
Yb-C(9)	2.680(10)		
Yb-C(10)	2.718(11)		

Table III. Selected angles($^{\circ}$).^a

C1	-Yb	-P(1)	79.7(1)
C1	-Yb	-Cp(1) ^a	106.3
C1	-Yb	-Cp(2) ^a	108.6
P(1)	-Yb	-Cp(1) ^a	108.0
P(1)	-Yb	-Cp(2) ^a	105.7
Cp(1) ^a	-Yb	-Cp(2) ^a	134.9
Yb	-P(1)	-C(21)	111.3(4)
Yb	-P(1)	-C(22)	116.9(4)
Yb	-P(1)	-C(23)	119.9(3)
C(21)	-P(1)	-C(22)	98.2(6)
P(1)	-C(23)	-P(2)	118.0(5)
C(23)	-P(2)	-C(24)	99.2(6)
C(23)	-P(2)	-C(25)	97.7(6)
C(24)	-P(2)	-C(25)	99.5(7)

^aCp(1) is the centroid of the cyclopentadienyl ring represented by atoms C(1) through C(5). Cp(2) is the centroid of atoms, C(6) through C(10).

Table IV. Data collection details for $(C_5Me_5)_2YbCl(Me_2PCH_2PMe_2)$.

<u>Crystal Parameters (25°C)^a</u>	
Space Group $P2_1/c$	$V = 2816 \text{ \AA}^3$
$a = 16.358(4) \text{ \AA}$	$Z = 4$
$b = 8.595(2) \text{ \AA}$	mol wt = 615.1
$c = 20.712(6) \text{ \AA}$	density (calcd) = 1.45 g/cm^3
$\beta = 104.75(3)^\circ$	$\mu(\text{calcd, MoK}\alpha) = 35.3 \text{ cm}^{-1}$

Intensity Data Measurement

radiation:	MoK α ($\lambda = 0.71073 \text{ \AA}$).
monochromator:	highly oriented graphite, $2\theta_m = 12.2^\circ$
scan type:	$\theta(\text{crystal}) - 2\theta(\text{counter})$
reflections measured:	$+h, +k, \pm l$ from $3^\circ < 2\theta < 45^\circ$
scan speed:	variable from 0.77 to 0.67 deg(θ)/min
scan width:	$\Delta\theta = 0.5 + 0.347 \tan(\theta)$
background:	An additional $\Delta 2\theta/4$ at each end of the scan
scan collected:	4205 (inc standards) yielding 3309 unique reflections
std reflections:	3 measured every 2 hours. A 10% decay in intensity was observed

^aUnit cell parameters were derived by a least squares fit to the setting angles of the unresolved MoK α components of 24 reflections with 2θ between 27° and 30.2° .

FIGURE CAPTIONS.

Fig. 1. ORTEP drawing of the molecule showing the numbering scheme.

Fig. 2. ORTEP view looking down a line through the centroids of the cyclopentadienyl rings. The second methyl carbon of the P(2) atom is eclipsed by the adjacent methyl group.

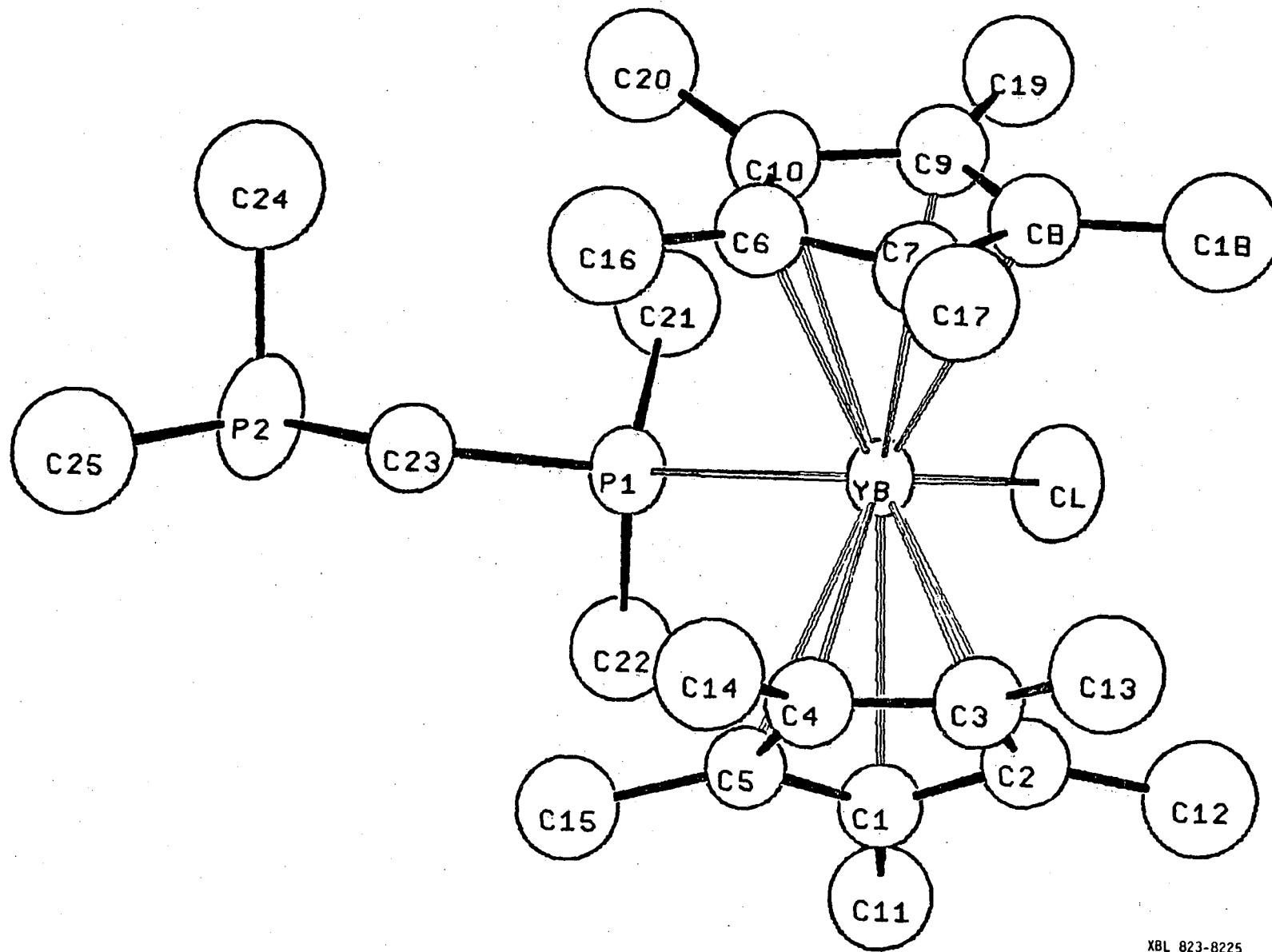


Fig. 1

XBL 823-8225

XBL 823-8224

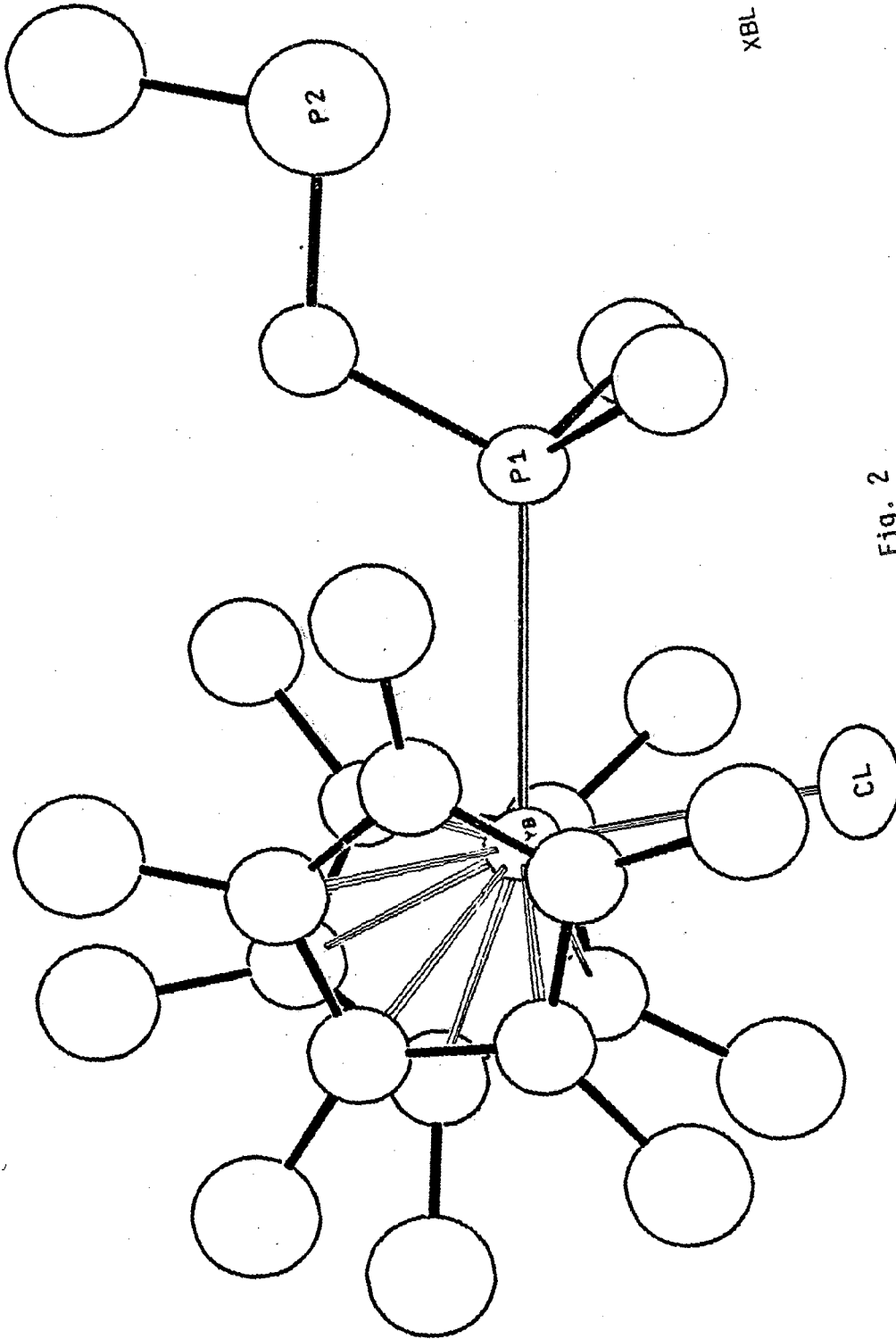


Fig. 2

Supplementary Material for

TERTIARY PHOSPHINE COMPLEXES OF THE f-BLOCK METALS:
PREPATION OF PENTAMETHYLCYCLOPENTADIENYL-TERTIARY
PHOSPHINE COMPLEXES OF YTTERBIUM (II AND III)
AND EUROPIUM (II). THE CRYSTAL STRUCTURE OF
 $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)$

T. Don Tilley, Richard A. Andersen and Allan Zalkin*

Anisotropic Thermal Parameters in $\text{Yb}(\text{Me}_5\text{C}_5)_2\text{Cl}(\text{Me}_2\text{PCH}_2\text{PMe}_2)^a$.

ATOM	B11	B22	B33	312	B13	B23
YB	2.773(24)	2.565(24)	3.071(24)	.017(13)	.888(15)	-.298(14)
CL	6.47(15)	3.52(12)	6.41(15)	-1.50(11)	2.19(12)	-.46(11)
P(1)	3.58(11)	3.39(11)	3.77(11)	.14(9)	1.36(9)	-.24(9)
P(2)	6.66(16)	5.14(16)	5.94(16)	-.57(13)	3.38(14)	-1.54(13)

^aThe anisotropic temperature factor has the form $\exp[-0.25(B_{11}h^2a^2 + 2B_{12}hka^*b^* + \dots)]$.

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 3.0)
 YTTERBIUM BIS(CYCLOPENTADIENYL) CHLORIDE DIMETHYLPHOSPHONETHA F(0,0,0) = 3199

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

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
	H,K=	0,	0	-21	142	3	7	-1	194	4	-32	18	55	3	3	-1	33	3	-12
2	497	11	-51	-20	43	5	-3	1	191	4	-35	19	143	3	1	0	377	9	-9
4	80	2	-32	-19	145	3	-1	2	490	10	19		H,K=	0,	5	1	34	4	-10
6	730	17	-69	-18	40	3	5	3	170	4	15	-18	126	3	9	2	218	5	-16
8	510	11	-39	-17	76	4	3	4	506	11	-30	-17	194	5	2	3	94	3	1
10	167	4	-5	-14	227	5	13	5	50	5	-16	-16	66	3	-6	5	50	3	-9
12	470	10	-25	-13	256	7	2	6	254	6	-1	-15	122	3	1	6	318	7	26
14	370	9	-29	-12	40	3	-5	7	86	2	-3	-14	59	3	2	7	49	8	-4
16	136	4	22	-9	258	6	18	8	421	9	-20	-13	73	3	2	8	227	5	6
18	151	3	-5	-6	341	7	49	10	387	8	-40	-12	64	3	16	12	181	4	-8
20	182	4	-11	-5	350	8	17	11	95	2	-8	-11	290	7	48	13	49	3	0
	H,K=	0,	1	-4	210	5	10	12	194	4	-31	-10	170	4	19	14	176	4	-16
-19	112	3	-2	-3	126	3	12	14	102	3	-6	-9	302	7	8	16	121	3	-6
-18	157	4	10	-2	267	6	-23	15	96	3	-4	-8	116	3	-8		H,K=	0,	7
-17	241	5	29	-1	498	10	17	16	258	6	0	-7	152	4	14	-14	61	4	-2
-16	216	5	20	0	352	7	-43	18	239	5	-13	-6	79	3	11	-13	47	5	-5
-15	229	6	26	1	453	9	-29		H,K=	0,	4	-5	474	10	39	-12	79	3	3
-14	57	2	10	2	258	6	-32	-19	150	3	9	-4	186	5	1	-11	177	4	10
-13	123	3	-6	3	126	3	12	-18	57	3	5	-3	305	7	-5	-10	61	3	0
-12	47	9	-10	4	199	4	-2	-16	37	4	8	-2	53	3	-10	-9	276	6	9
-11	460	11	18	5	335	7	2	-15	243	5	15	-1	141	3	13	-6	32	5	7
-10	284	7	14	6	326	7	33	-14	208	5	17	1	144	4	16	-5	202	5	13
-9	410	9	9	7	555	12	-44	-13	273	6	55	2	59	2	-5	-4	96	3	8
-8	233	6	-4	8	252	6	-8	-12	104	3	15	3	305	7	-5	-3	342	8	17
-7	148	3	14	9	240	5	-7	-11	180	4	14	4	180	4	-4	-2	154	4	11
-6	186	4	25	10	168	4	-5	-10	115	3	-2	5	457	10	22	-1	156	4	21
-4	414	9	-5	11	64	2	2	-9	251	6	5	6	75	3	8	1	157	4	22
-3	503	11	7	12	40	3	-5	-8	310	7	16	7	146	3	8	2	157	4	14
-2	461	9	-2	13	236	6	-18	-7	663	14	24	8	108	3	-17	3	335	7	11
-1	548	12	14	14	205	5	-9	-6	346	8	18	9	286	7	-7	4	95	3	8
1	489	10	-45	15	208	5	14	-5	426	10	23	10	157	4	6	5	202	5	13
2	434	9	-29	17	71	3	-2	-4	56	2	-12	11	271	6	29	8	28	7	12*
3	476	10	-20	18	38	4	2	-3	170	4	-6	12	58	4	10	9	263	6	-4
4	395	8	-24	19	135	3	-11	-2	350	8	14	13	70	3	0	10	62	3	1
5	65	2	3	20	43	3	-4	-1	577	12	-25	14	53	3	-3	11	167	4	0
6	175	4	14	21	131	3	-4	0	41	2	-11	15	113	3	-8	12	75	3	-1
7	138	3	4		H,K=	0,	3	1	542	12	-60	16	59	3	-13	13	55	4	2
8	216	5	-21	-18	257	6	5	2	343	7	7	17	182	5	-9	14	56	4	-7
9	381	9	-20	-17	28	7	1*	3	169	4	-7	18	116	3	-1		H,K=	0,	8
10	267	6	-3	-16	278	6	21	4	53	2	-15		H,K=	0,	6	-9	92	4	-1
11	417	10	-25	-15	103	3	3	5	409	9	6	-16	126	3	-1	-8	152	4	-8
12	47	9	-11*	-14	113	3	5	6	333	7	6	-14	188	4	-5	-7	208	5	-8
13	115	3	-14	-12	213	5	-12	7	613	13	-27	-13	52	3	3	-6	140	4	-5
14	48	4	1	-11	102	2	-2	8	301	7	7	-12	193	4	4	-5	180	4	-3
15	206	5	3	-10	418	9	-3	9	232	5	-13	-9	54	3	-4	-4	71	3	-2
16	201	5	4	-8	460	10	20	10	107	3	-9	-8	247	6	26	-3	26	8	4*
17	219	5	7	-7	92	2	3	11	167	4	2	-7	36	6	-17	-2	88	3	-2
18	144	3	-3	-6	272	6	17	12	91	2	3	-6	321	8	29	-1	261	6	-12
19	105	3	-9	-5	49	5	-16	13	244	6	26	-5	50	3	-10	0	185	3	-11
20	39	8	-4*	-4	533	11	-4	14	190	4	-1	-4	30	5	-1	1	260	6	-12
21	18	11	12*	-3	170	4	15	15	231	5	3	-3	94	3	1	2	90	4	0
	H,K=	0,	2	-2	519	11	48	16	30	6	1*	-2	222	5	-12	4	65	4	-8

STRUCTURE FACTORS CONTINUED FOR
 YTTERBIUM BISCYCLOPENTADIENYL CHLORIDE DIMETHYLPHOSPHOR METHA

PAGE 2

L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
5	175	4	-9	8	374	8	2	-18	117	3	-5	2	127	3	-6	-6	176	4	13
6	143	4	-2	9	322	8	-28	-17	53	3	5	3	438	10	-6	-5	94	3	3
7	207	5	-2	11	110	2	-18	-16	227	5	29	4	357	8	18	-4	417	10	4
8	146	4	-6	12	131	3	-2	-14	301	7	35	5	385	8	22	-3	24	7	5*
9	87	3	-7	13	297	7	-21	-13	75	2	7	6	67	2	-2	-2	183	4	2
	H,K=	0,	9	14	230	6	-3	-12	282	6	4	7	84	2	6	-1	45	2	-5
-4	192	4	-11	15	243	6	7	-10	124	3	2	8	209	5	8	0	74	2	-2
-3	43	9	5*	16	61	6	-8	-9	125	3	20	9	396	9	-17	2	300	7	-9
-2	209	5	-12	17	78	2	4	-8	539	12	32	10	235	6	-6	3	75	3	-9
-1	22	8	7*	18	81	2	-5	-7	34	3	-6	11	275	6	-8	4	246	6	23
2	206	5	-15	19	129	3	-11	-6	601	13	-6	12	47	3	9	6	104	3	4
4	190	4	-12	20	104	3	-3	-4	84	2	3	13	27	5	10*	7	65	3	-8
	H,K=	1,	0	21	127	4	-10	-3	123	4	-10	15	148	4	-19	8	203	5	12
-20	82	3	5		H,K=	1,	2	-2	453	9	-13	16	106	3	-1	9	55	3	-7
-18	243	6	14	-20	67	3	5	-1	221	5	38	17	218	5	-17	10	251	6	15
-16	338	8	49	-19	179	4	7	0	953	21	106	18	57	3	-5	12	140	3	3
-14	60	2	-3	-18	104	3	9	1	232	5	15	19	121	3	-2	13	20	9	-14*
-12	404	9	-14	-17	287	7	28	2	218	5	-29		H,K=	1,	5	14	45	4	-1
-10	729	15	-25	-16	124	3	14	3	32	7	29*	-16	82	3	-3	15	40	5	11
-8	346	7	-3	-15	126	3	30	4	146	3	-6	-15	211	5	8	16	122	3	-11
-6	77	2	14	-14	53	2	6	5	233	5	39	-14	107	3	2		H,K=	1,	7
-4	518	11	-75	-13	75	3	13	6	603	13	-14	-13	288	7	27	-14	119	3	-3
-2	699	15	-152	-12	266	6	-2	7	94	3	8	-11	140	4	9	-13	163	4	-7
2	937	20	-76	-11	349	8	-3	8	472	10	-14	-9	159	4	16	-12	83	3	-2
4	74	2	-19	-10	307	7	33	9	44	3	11	-8	195	4	-7	-11	67	3	-9
6	47	2	-17	-9	264	6	1	10	51	4	13	-7	448	10	17	-10	73	3	1
8	574	12	-52	-8	23	4	-3	11	59	2	4	-6	178	5	16	-9	130	3	1
10	489	11	-31	-7	33	4	8	12	290	7	-20	-5	337	8	0	-8	121	3	3
12	395	9	-29	-6	135	3	-33	13	86	3	4	-3	282	6	16	-7	329	7	1
14	144	3	-23	-5	521	11	-3	14	318	7	39	-2	381	7	-11	-6	111	3	9
16	319	7	19	-4	82	2	28	15	32	4	3	-1	377	8	-11	-5	203	5	13
18	200	5	-31	-3	362	8	-13	16	131	3	5	0	109	3	-12	-4	66	4	4
20	26	5	11*	-2	313	7	-29	18	70	3	-2	1	503	11	9	-3	55	5	9
	H,K=	1,	1	-1	97	2	9	20	185	4	-4	2	94	2	6	-2	39	5	12
-21	177	4	12	0	88	2	-14		H,K=	1,	4	3	94	3	15	-1	338	7	26
-20	154	3	5	1	281	6	-17	-19	149	3	14	4	96	2	14	0	126	3	22
-19	117	3	2	2	269	6	18	-18	118	3	3	5	235	5	19	1	195	4	27
-18	36	5	12	3	378	8	11	-17	251	6	8	6	189	4	-13	2	25	8	2*
-15	225	6	30	4	319	7	-3	-16	123	3	1	7	470	10	-29	4	154	3	5
-14	276	7	23	5	239	5	-4	-15	170	4	22	8	168	4	-5	5	201	5	-1
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STRUCTURE FACTORS CONTINUED FOR
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L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
-8	113	3	-3	-11	61	2	-5	10	94	2	-6	-2	340	7	31	-13	68	3	3
-7	193	4	-13	-10	161	4	-9	11	181	5	-12	-1	207	5	24	-12	299	7	-2
-6	143	4	-7	-9	363	8	-29	12	48	2	0	0	146	3	8	-10	248	6	6
-5	146	3	-14	-8	147	3	-21	13	83	2	7	1	334	7	-12	-9	46	4	-2
-4	54	4	-10	-5	32	2	11	14	59	3	10	2	266	6	-23	-7	65	3	6
-3	73	6	8	-4	45	2	1	15	271	6	4	3	438	9	7	-6	247	6	12
-2	101	3	-5	-1	471	10	-15	16	155	4	-8	4	114	3	-9	-5	67	3	-2
-1	193	4	-8	0	446	9	27	17	164	4	-13	5	317	7	-8	-4	415	9	1
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-12	263	7	-10	9	412	9	-19	-3	453	10	-5	-14	78	3	-5	-4	138	3	-12

STRUCTURE FACTORS CONTINUED FOR
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L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
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0	51	3	-8	0	51	1	-18	15	106	3	4	6	161	4	11	-6	238	5	28
1	91	3	-10	1	406	9	47	16	44	3	-9	7	306	7	11	-5	39	5	-11
2	115	3	-13	2	174	4	-28	17	102	3	-10	8	155	4	0	-4	73	2	6
3	259	6	-20	3	795	18	63	18	78	3	-12	9	140	3	10	-3	74	2	-8
4	146	3	-6	4	282	6	-1	19	122	3	-4	10	18	8	-11*	-2	389	8	23
5	129	3	-4	5	215	5	-17	H,K=	4,	3	11	151	3	21	-1	85	2	-4	
6	53	3	-5	6	235	5	-5	-20	120	3	-5	12	224	5	27	0	355	8	22
7	76	3	-3	7	454	18	10	-11	125	3	-6	13	310	7	14	1	39	3	5
8	63	3	-5	8	218	5	-4	-9	123	3	19	14	99	3	0	3	61	2	2
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STRUCTURE FACTORS CONTINUED FOR
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L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
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7	184	4	-3	9	104	3	-7	-12	69	3	-6	14	89	3	3	12	38	4	2
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	H,K=	5	8	14	114	3	9	-7	87	2	25	-17	66	4	-6	-11	21	10	-16
-22	57	3	4	15	58	3	-8	-6	403	9	5	-16	158	4	4	-10	46	4	4
-20	123	3	-16	17	67	3	-9	-5	95	2	1	-15	170	4	-6	-9	223	5	-3
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2	245	5	19	-20	137	3	-5	5	60	2	-8	1	68	3	-3	-14	271	6	-15
3	190	4	-10	-18	66	3	11	7	121	3	-11	2	423	9	43	-12	81	2	13
4	206	5	-10	-17	72	3	-4	8	134	3	10	4	259	6	33	-10	360	8	-35
5	619	14	32	-16	207	6	17	9	299	7	30	6	121	3	-5	-8	362	8	-6

STRUCTURE FACTORS CONTINUED FOR
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L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
-6	438	9	-29	-18	55	3	7	3	61	2	-11	-6	205	5	-12	8	105	3	3
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-2	496	10	-10	-16	187	4	16	5	87	2	14	-4	215	5	-11	2	129	3	-4
0	286	6	-37	-15	305	7	15	6	136	3	-4	-3	194	4	-7	3	274	6	4
2	128	3	-3	-14	205	5	2	7	65	2	-2	-2	65	2	1	4	91	3	1
4	455	10	3	-13	217	5	-8	8	319	8	6	0	121	3	-1	7	125	3	-9
6	525	11	-9	-12	37	3	3	9	33	3	-7	1	316	7	-3	8	153	3	6
8	90	2	2	-11	143	3	5	10	271	7	29	2	315	7	-7	9	165	4	-7
10	159	4	-1	-10	46	2	2	13	18	15	-3*	3	243	6	14	10	110	5	8
12	366	8	31	-9	410	9	-14	14	202	5	2	4	109	3	10		H,K=	6,	8
14	92	2	6	-8	355	8	-15	15	59	3	13	5	62	2	5	-9	113	3	1
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-20	92	3	2	-3	428	9	-19	-16	51	4	-1	10	133	4	1	-3	147	3	-7
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-17	273	6	18	1	222	5	-3	-13	164	4	24	14	74	3	-8	0	109	3	-4
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7	274	6	5	-13	27	6	-4*	13	170	4	-7	10	108	3	-5	16	137	3	-10
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L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
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-6	111	2	-4	-18	59	3	-2	9	237	5	27	6	133	3	4	14	95	2	-7
-5	149	3	-1	-16	257	6	-15	10	77	4	5	7	21	7	9*	16	97	3	2
-4	203	5	-5	-15	58	3	-0	11	72	2	-4	8	259	6	-2		H,K=	8,	1
-3	453	10	2	-14	278	7	25	13	48	3	-2	10	153	4	-5	-21	86	3	-1
-2	298	7	3	-13	37	3	15	14	47	3	-6		H,K=	7,	7	-20	87	3	-10
0	129	3	-11	-12	124	3	-6	15	140	3	-3	-13	99	3	11	-19	181	4	-5
1	32	2	-10	-11	75	3	0		H,K=	7,	5	-11	63	3	-3	-18	123	3	-3
3	329	7	22	-10	360	9	-18	-18	63	5	-6	-10	69	3	5	-17	219	5	-10
4	329	7	4	-9	47	2	-7	-17	125	3	-5	-9	202	5	0	-14	105	3	13
5	292	7	-27	-8	447	10	14	-16	86	3	-16	-8	115	3	-6	-13	357	8	-12
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7	258	6	-5	-6	261	6	-9	-14	152	3	8	-6	82	5	10	-11	280	7	-2
9	31	3	10	-5	81	2	11	-13	107	3	-12	-5	38	3	-3	-10	65	2	-1
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14	67	3	5	0	356	8	-1	-7	242	5	29	0	128	3	1	-5	464	10	4
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16	73	3	-2	6	166	4	5	2	390	9	28	8	86	2	13	-7	381	9	3

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L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
-6	233	6	15	-16	65	3	-3	10	125	3	-2	-2	109	3	11	-5	393	9	-11
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-4	29	2	4	-14	84	3	2	-14	193	4	8	2	523	11	15	-3	232	6	-15
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-2	319	7	5	-12	56	3	11	-10	196	4	-6	8	367	8	39	-1	158	4	-2
-1	446	10	-27	-11	175	4	2	-9	83	2	4	10	222	5	32	0	289	7	20
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1	183	5	-11	-9	342	8	14	-7	50	3	-1	14	191	4	-9	2	292	8	-3
2	103	3	-2	-8	247	6	11	-6	113	3	9		H,K=	9,	1	3	294	7	2
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6	138	3	-7	-4	172	4	2	0	215	5	9	-17	151	4	-14	8	150	4	27
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9	107	3	12	-1	261	6	-12	4	290	7	1	-14	106	3	18	11	81	3	-1
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-5	36	2	11	-12	130	3	5	4	44	4	4	10	105	3	9	-1	37	2	2
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-3	63	2	11	-10	127	3	1	7	136	3	-8	12	138	3	4	2	59	2	4
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2	447	10	9	-4	118	3	20	-2	106	3	-3	-18	112	3	-6	7	72	2	-7
3	90	2	4	-3	75	2	3	-1	168	4	-5	-17	126	3	-12	9	32	4	8
4	201	5	10	-2	35	3	4	0	48	3	-7	-15	51	3	-5	10	251	6	18
5	72	2	-2	-1	95	2	7		H,K=	9,	0	-15	72	2	0	12	185	4	-9
6	106	2	4	0	145	3	8	-20	259	6	1	-14	113	3	9	13	17	12	6*
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8	373	9	47	2	226	5	24	-16	98	2	3	-12	238	6	5		H,K=	9,	4
10	164	4	17	3	288	6	37	-14	331	7	22	-11	287	7	-18	-18	146	3	0
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14	144	3	-10	6	112	3	11	-10	190	5	-12	-9	85	2	-5	-15	136	3	5
	H,K=	8,	4	7	209	5	-9	-8	408	9	-2	-8	66	2	9	-14	93	3	3
-18	61	6	-3	8	159	4	5	-6	390	9	-7	-7	352	8	-2	-13	249	6	0
-17	206	5	2	9	171	4	-5	-4	323	7	-21	-6	174	5	7	-12	175	4	1

STRUCTURE FACTORS CONTINUED FOR

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L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
-11	186	5	10	-3	68	4	-5	-8	149	4	11	-13	45	3	-8	-10	62	4	-0
-10	29	4	14	-1	54	3	-4	-7	292	7	-4	-12	269	6	27	-9	45	4	7
-9	28	4	-2	0	218	5	5	-6	256	6	-13	-11	19	6	-2*	-8	42	3	1
-7	328	7	15	2	268	6	11	-5	249	6	-18	-10	107	2	10	-7	255	6	12
-6	268	6	-3	4	131	3	-1	-4	152	3	-8	-9	87	2	2	-6	128	3	13
-5	344	7	-8	6	92	3	-1	-3	60	2	-5	-8	191	4	11	-5	303	7	18
-4	53	2	6	7	30	5	8	-2	100	2	1	-7	61	2	-2	-4	123	3	11
-2	56	2	-0	8	237	5	-3	-1	158	3	1	-6	407	9	-7	-3	103	2	4
-1	177	4	-8		H,K=	9,	7	0	226	5	2	-5	68	2	-6	-2	74	2	8
0	115	3	4	-11	115	3	-4	1	465	11	-14	-4	256	6	-5	-1	238	6	-6
1	341	7	14	-10	78	3	-2	2	173	5	-12	-2	22	4	3*	0	162	4	13
2	123	3	24	-9	191	4	-10	3	250	6	-5	0	329	7	-2	1	264	6	12
3	188	4	19	-8	75	3	12	5	62	2	-1	1	45	3	-2	2	139	3	4
4	116	3	18	-7	150	4	-2	6	90	2	-1	2	283	7	34	3	184	4	7
6	75	2	-3	-6	58	3	-1	7	304	7	41	4	58	2	11	4	70	2	-11
7	301	7	14	-5	103	3	-1	8	219	6	18	5	89	2	-2	5	48	3	-9
8	106	3	4	-4	43	3	-3	9	217	5	15	6	238	5	36	6	55	4	0
9	212	5	1	-3	188	4	-8	10	52	3	1	7	56	3	0	7	181	4	-10
11	42	3	8	-2	132	3	-4	11	56	2	-1	8	314	7	5	8	151	3	-2
12	33	5	-4	-1	167	5	-8	12	42	3	1	10	152	4	4	9	157	4	1
13	82	3	-9	0	104	3	-2	13	121	3	-1	12	61	3	-7	10	73	3	-4
	H,K=	9,	5	2	56	3	2	14	112	3	-1		H,K=	10,	4		H,K=	10,	6
-17	140	3	-4	3	104	3	-12		H,K=	10,	2	-18	30	6	0*	-14	139	3	-1
-16	125	3	-1	4	111	3	-3	-19	31	4	14	-17	231	5	-9	-13	43	4	-11
-15	219	5	-8	5	154	3	-5	-18	28	5	3	-16	133	4	1	-12	52	3	3
-14	57	3	1		H,K=	10,	6	-17	179	4	-11	-15	196	5	-2	-10	167	4	-4
-11	131	3	-2	-20	73	3	4	-16	125	3	-7	-14	45	3	5	-8	254	6	-2
-10	153	3	-9	-18	194	4	-11	-15	249	7	3	-12	71	2	1	-6	55	2	1
-9	292	7	11	-16	229	5	-8	-14	53	3	7	-11	197	5	-6	-5	24	6	8*
-8	237	5	8	-14	238	5	31	-13	98	4	1	-10	145	4	-1	-4	170	4	-1
-7	189	4	15	-12	172	4	-3	-11	132	3	10	-9	295	7	21	-2	288	6	-10
-6	112	3	7	-10	428	9	-14	-10	271	7	-12	-8	153	4	21	0	120	3	-6
-5	90	2	-5	-8	423	10	-21	-9	293	8	1	-7	188	5	21	2	66	3	8
-4	117	3	25	-6	169	4	-16	-8	97	2	2	-6	134	3	-5	3	65	3	-7
-3	315	7	25	-4	239	5	-31	-7	191	5	-8	-5	141	4	4	4	197	5	-12
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3	211	5	20	6	231	5	34	-2	139	3	-1	0	45	3	14	-8	37	4	-5
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5	266	6	-10	10	271	6	-2	0	219	6	-8	3	247	6	27	-6	99	3	2
6	158	4	-8	12	203	5	-15	3	210	5	12	4	122	3	9	-5	194	4	-5
7	96	2	-3	14	73	4	-6	4	186	5	6	5	237	5	8	-4	79	5	-9
9	126	3	-2		H,K=	10,	1	5	312	8	24	6	123	3	-14	-1	119	3	-4
10	74	3	-7	-20	100	3	-8	6	149	3	23	7	105	3	-11	0	59	2	9
11	177	4	-1	-19	186	4	-12	8	44	4	4	8	41	3	2	1	191	4	2
	H,K=	9,	6	-18	113	3	-0	9	112	3	0	9	141	3	-1	2	118	3	5
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-10	173	4	-8	-13	277	6	41	13	111	3	-7	-15	71	3	-7	-18	197	4	5
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-7	56	3	4	-11	295	7	-13	-18	189	5	-4	-13	259	6	-10	-14	219	5	18
-6	309	7	5	-10	39	2	8	-17	38	5	-1	-12	97	3	-0	-12	458	10	31
-4	262	6	13	-9	96	2	-8	-14	276	6	6	-11	131	3	-5	-10	125	3	-3

STRUCTURE FACTORS CONTINUED FOR
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L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	
-8	297	7	-13	-6	112	3	-3	0	201	5	7	-12	77	2	15	-14	232	5	-4	
-6	507	11	-15	-5	335	8	-8	1	329	8	22	-10	388	8	33	-12	294	7	-3	
-4	98	2	-15	-4	69	2	6	2	168	4	1	-8	321	7	19	-11	85	2	3	
0	418	9	-30	-3	147	4	0	3	100	2	-7	-6	63	2	-9	-8	184	4	9	
2	348	8	-9	-2	67	2	-4	5	114	3	3	-4	386	9	14	-7	121	3	9	
4	90	3	5	-1	192	4	4	7	216	5	-1	-2	304	7	17	-6	399	9	29	
6	359	8	34	0	85	2	6	8	124	4	3	0	151	3	-1	-5	66	2	7	
8	276	7	-9	1	252	6	17	9	175	4	-2	2	206	5	18	-4	239	6	17	
10	84	2	-2	2	197	4	-4	H,K= 11,	5	4	388	9	26	-2	65	2	9			
12	93	2	1	3	124	3	16	-15	166	4	-0	6	185	4	10	-1	24	5	1*	
	H,K= 11,			4	27	4	2	-12	60	2	-6	8	158	4	-4	0	334	7	30	
-19	48	3	-6	5	68	2	3	-11	191	4	-6	10	193	4	-14	2	197	5	7	
-18	56	3	-6	6	125	3	6	-10	129	3	-3	H,K= 12,	1	4	43	3	6			
-17	162	4	-6	7	252	6	9	-9	209	5	4	-19	176	4	4	5	36	3	2	
-16	177	4	-0	8	101	2	-0	-8	66	2	0	-18	67	3	-1	6	204	5	-1	
-15	127	3	1	9	191	4	-3	-7	189	4	4	-15	144	3	-4	7	61	2	-2	
-14	87	3	6	10	17	19	-7*	-6	75	2	-7	-14	198	5	-11	8	211	5	-9	
-13	80	2	4	H,K= 11,				-5	92	2	-7	-13	235	5	20	10	100	3	1	
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-11	220	5	21	-16	248	6	-3	-3	295	6	1	-11	279	6	25	-16	113	3	-4	
-10	240	6	10	-14	153	4	-3	-2	162	4	-2	-10	74	2	1	-15	194	4	-5	
-9	363	8	-6	-13	46	4	9	-1	223	5	3	-9	61	2	-1	-14	52	4	-7	
-8	93	2	17	-12	103	3	2	0	42	4	11	-8	183	5	8	-13	29	5	7	
-7	110	3	3	-10	214	5	36	1	82	2	12	-7	344	8	29	-12	102	3	-8	
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-5	168	4	-9	-8	260	6	26	3	188	4	-9	-5	294	7	28	-10	149	3	-4	
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1	55	2	-8	0	166	4	14	-12	185	4	-6	2	162	4	1	-4	106	3	0	
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4	173	4	17	4	307	7	26	-7	42	3	-4	6	137	3	0	-1	212	5	14	
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6	168	4	4	6	195	4	3	-4	169	4	-15	8	151	4	-4	2	131	3	0	
7	34	3	0	7	44	6	-1	-2	45	3	2	9	92	2	-12	3	242	5	-7	
8	32	4	-4	10	189	4	-7	-1	60	2	-8	H,K= 12,				4	109	3	-1	
9	141	3	-7	H,K= 11,				0	231	5	0	-18	103	3	-3	5	178	4	-8	
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11	159	4	-3	-15	178	4	-6	3	24	6	0*	-16	54	3	-2	8	43	4	10	
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	H,K= 11,			-12	100	3	-4	H,K= 11,				7	-11	197	4	13	-13	222	5	-2
-19	190	5	5	-11	186	4	6	-7	70	3	9	-10	155	4	11	-12	87	2	6	
-17	55	3	-6	-10	45	5	2	-5	63	3	-4	-9	155	4	29	-11	132	3	-1	
-15	118	3	-6	-9	42	6	-2	-4	57	7	-9	-6	79	2	4	-9	66	2	0	
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-13	281	7	10	-7	247	6	10	-2	40	4	-5	-1	184	5	34	-7	195	4	4	
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-11	173	4	17	-5	322	7	29	0	61	3	-0	10	75	3	-5	-5	244	6	-7	
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-9	58	2	-1	-3	70	2	6	-18	177	4	2	-18	172	4	12	-3	36	6	3	
-8	161	4	5	-2	128	3	-3	-16	203	5	-7	-17	37	4	-3	-2	68	3	-4	
-7	246	6	13	-1	241	6	17	-14	131	3	5	-16	89	3	2	-1	212	5	-4	

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L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
0	134	3	-0	5	214	5	7	1	240	6	-7	1	199	6	1	-3	195	4	-14
1	266	7	-11	6	68	2	-8	2	99	2	-15	2	94	2	-2	-2	129	3	-7
2	82	3	-12	7	29	4	9	3	85	2	-3	3	104	2	0	-1	113	3	-7
3	67	2	-9	8	47	3	-2	5	103	3	-9	5	103	2	6	0	25	5	0
5	60	3	-1	9	158	4	-6	6	68	3	4	6	128	3	5	1	85	2	3
6	35	5	-6	10	94	3	-1		H,K= 13,	5	7	7	173	4	-2	2	86	3	-2
	H,K= 12,	6			H,K= 13,	2	-12	102	3	3	8	8	69	3	-3	3	178	4	-11
-11	61	3	2	-16	94	3	-1	-11	207	5	6		H,K= 14,	2	4	81	3	-11	
-10	201	5	6	-15	163	4	2	-10	91	2	1	-16	93	3	1		H,K= 14,	5	5
-9	54	4	9	-14	92	3	1	-9	199	5	-13	-15	130	3	-5	-10	58	3	9
-8	114	3	-2	-13	226	5	-2	-8	37	3	-0	-14	27	5	-6	-9	125	3	8
-4	173	4	-10	-12	105	3	1	-7	50	3	-4	-13	31	4	-3	-8	104	3	0
-3	28	4	5	-11	129	3	13	-6	33	4	9	-12	98	3	-0	-7	175	4	-0
-2	207	5	0	-10	54	2	1	-5	100	3	3	-11	199	5	-1	-6	102	3	2
-1	32	4	2	-9	158	4	2	-4	72	2	-4	-10	86	2	-1	-5	129	3	1
0	77	2	6	-8	163	4	9	-3	208	5	1	-9	182	4	-10	-2	55	3	4
1	46	3	1	-7	248	6	21	-2	120	4	1	-8	91	2	2	-1	151	3	-11
2	131	3	-2	4	35	3	-1	-1	142	3	0	-6	88	2	-2	0	123	3	3
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	H,K= 13,	0	-16	240	6	-3	2	71	2	-3	-4	146	3	-5		H,K= 15,	0	0	0
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-16	130	3	6	-13	49	5	4	4	90	3	-4	-2	96	2	-3	-14	295	7	-14
-14	253	6	-10	-12	100	2	-4		H,K= 13,	6	-1	67	2	-2	-12	108	3	-9	-9
-12	326	7	2	-11	39	3	3	-8	159	4	4	0	63	2	-1	-8	208	5	-7
-10	79	3	12	-10	313	7	1	-6	172	4	-6	1	104	2	-2	-6	252	6	-20
-8	246	6	29	-9	58	3	2	-4	96	3	6	2	95	2	2	-4	126	3	-1
-6	507	11	56	-8	132	3	-3	-2	98	3	-8	3	226	5	-13	-2	269	6	-7
-4	228	6	23	-7	33	5	3		H,K= 14,	0	4	4	112	3	5	0	225	5	-15
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0	356	8	31	-5	61	2	5	-12	135	3	-11	6	50	3	-11	4	30	4	6
2	315	7	11	-4	329	7	16	-10	263	6	-13	7	18	18	2*	6	143	3	-7
6	264	6	-9	-3	40	3	3	-8	188	4	-9		H,K= 14,	3		H,K= 15,	1	1	1
8	205	5	-9	-2	245	6	7	-4	370	8	25	-15	38	4	4	-15	121	3	-1
	H,K= 13,	1	0	26	4	-5	-2	220	5	1	-14	243	6	-4	-13	118	3	1	1
-18	108	3	-3	2	245	6	-5	0	31	13	-7*	-12	216	5	9	-12	40	6	-2
-17	188	4	1	3	28	12	8*	2	177	4	-9	-8	291	7	3	-11	196	4	-2
-16	111	3	-13	4	232	5	-14	4	258	6	-3	-7	39	3	-0	-10	122	3	1
-15	99	2	-7	5	60	2	-1	6	85	2	-5	-6	263	6	-3	-9	136	3	-3
-14	111	3	-6	6	107	3	4	8	120	3	-1	-5	58	3	-1	-8	79	2	1
-12	88	2	3	8	66	3	-2		H,K= 14,	1	-4	121	3	-8	-6	46	3	-6	-6
-11	167	4	0		H,K= 13,	4	-16	46	3	0	-2	184	4	9	-5	182	4	-11	-11
-10	118	3	12	-15	125	3	2	-15	193	5	-3	0	242	5	7	-4	126	3	-4
-9	255	6	26	-14	123	3	-4	-14	107	3	0	1	14	19	-8*	-3	227	5	-20
-8	132	3	7	-13	264	6	1	-13	208	6	-9	2	182	4	-3	-2	86	2	-8
-7	96	2	13	-12	134	3	2	-12	144	3	4	4	60	2	-2	-1	22	16	-8*
-6	51	3	9	-11	135	3	6	-11	46	3	-2	6	184	4	0	1	130	3	-7
-5	206	5	22	-9	95	2	2	-9	98	2	2		H,K= 14,	4	2	108	3	-1	-1
-4	120	3	10	-8	124	3	-8	-8	130	3	-11	-12	75	3	1	3	154	4	-8
-3	291	7	35	-7	280	6	0	-7	293	7	17	-11	214	5	6	4	107	3	-3
-2	218	5	15	-6	89	2	-2	-6	216	5	5	-10	174	4	0	5	96	2	-11
-1	83	2	22	-5	233	5	-2	-5	232	5	7	-9	180	4	-4	6	32	6	-0*
0	21	5	-3*	-4	70	3	3	-4	73	2	-5	-8	86	2	-2		H,K= 15,	2	2
1	68	2	7	-3	43	3	-7	-3	71	2	4	-7	38	7	-3*	-15	155	4	1
2	103	2	7	-2	81	2	-3	-2	40	3	0	-6	74	2	5	-14	118	3	-3
3	242	5	10	-1	247	6	9	-1	278	6	11	-5	155	4	-1	-13	182	4	-3
4	199	5	2	0	195	5	-3	0	183	4	-4	-4	81	3	-5	-12	61	2	-2

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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