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DIVALENT LANTHANIDE CHEMISTRY: PREPARATION OF SOME FOUR AND SIX COORDINATE BIS (TRIMETHYLSILYLAMIDO) COMPLEXES OF EUROPIUM (II). THE CRYSTAL STRUCTURE OF BIS(DI (TRIMETHYLSILYL) AMIDO) BIS(1,2-DIMETHOXYETHANE) EUROPIUM(II)

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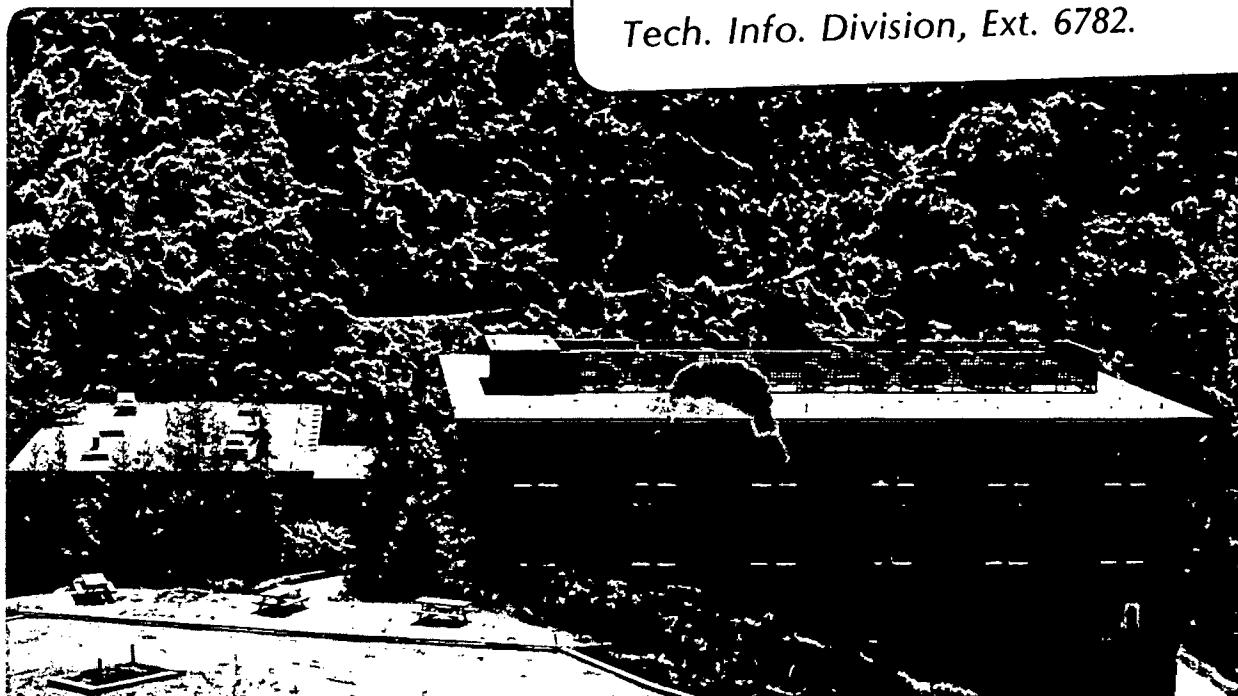
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T. Don Tilley, Allan Zalkin, Richard A. Andersen,  
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DIVALENT LANTHANIDE CHEMISTRY; PREPARATION OF SOME FOUR AND SIX COORDINATE BIS(TRIMETHYLSILYLAMIDO) COMPLEXES OF EUROPIUM(II).

THE CRYSTAL STRUCTURE OF  
BIS[DI(TRIMETHYLSILYL)AMIDO]BIS(1,2-DIMETHOXYETHANE)EUROPIUM(II).

T. Don Tilley, Allan Zalkin,\* Richard A. Andersen  
and David H. Templeton

ABSTRACT

The yellow, hydrocarbon-soluble, divalent derivatives of europium(II) of the type  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}(\text{L})_2$ , where L is tetrahydrofuran or 1,2-dimethoxyethane, and  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}(\text{bipyridine})$  have been prepared. The crystal structure of  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}[\text{MeOCH}_2\text{CH}_2\text{OMe}]_2$  has been determined by single crystal X-ray diffraction methods. The crystals are monoclinic, C2/c, with cell dimensions  $a = 13.398(4)\text{\AA}$ ,  $b = 17.454(6)\text{\AA}$ ,  $c = 15.013(5)\text{\AA}$ ,  $\beta = 92.91(3)\text{\AA}$  and  $V = 3506\text{\AA}^3$ . For  $Z = 4$  the calculated density is  $1.24 \text{ g/cm}^3$ . The structure was refined to a conventional R factor of 0.039 using 2259 data where  $F^2 > \sigma(F^2)$ . The Eu(II) atom is hexacoordinate and bonded to two nitrogen atoms of the two bis(trimethylsilyl)amido groups, and to four oxygen atoms of the 1,2-dimethoxyethane ligands. The molecule has  $C_2$  symmetry. The Eu(II)-N bond distance is  $2.530(4)\text{\AA}$  and the Eu(II)-O distances are  $2.634(4)\text{\AA}$  and  $2.756(4)\text{\AA}$ . The ethane carbon atoms in the 1,2-dimethoxyethane ligand are disordered.

## INTRODUCTION

Compounds of europium in its divalent oxidation state are rather less well-characterized than are those of the more common trivalent oxidation state. Some simple halides, chalcogenides, and organometallic compounds have been prepared and their physical properties have been studied.<sup>1,2</sup> These compounds are generally insoluble and their solid state structures are coordination polymers with high coordination number metal atoms. One way to prevent polymer formation is by use of sterically voluminous ligands, one such ligand being the bis(trimethylsilyl)amido ligand,  $(\text{Me}_3\text{Si})_2\text{N}$ . This ligand has been used to prepare low coordination number complexes of most elements in the Periodic Table.<sup>3,4</sup> Even the trivalent uranium derivative,  $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{U}$ , is monomeric and three coordinate, a most unusual constitution for this large actinide ion.<sup>5</sup>

In this paper we describe the synthesis of some coordination complexes of bis[di(trimethylsilyl)amido]europium(II) and the crystal structure of one of them, viz.,  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}(\text{dme})_2$ , where dme is the chelating ether, 1,2-dimethoxyethane. This is the only monomeric, molecular compound of Eu(II) whose crystal structure has been determined.

## EXPERIMENTAL SECTION

All analyses were by the microanalytical laboratory of this department. Magnetic susceptibility measurements were carried out as previously described.<sup>6</sup> All operations were performed under nitrogen.

Bis[di(trimethylsilyl)amido]europium(II)bis(tetrahydrofuran) Complex. Tris[di(trimethylsilyl)amido] europium<sup>7</sup> (2.2 g, 0.0034 mol) in tetrahydrofuran (75 mL) was added to europium trichloride (0.44 g, 0.0017 mol) suspended in tetrahydrofuran (25 mL). After stirring for 8 h sodium naphthalene [prepared from sodium metal (0.12 g, 0.0052 mol) and naphthalene (0.65 g, 0.0051 mol) in tetrahydrofuran (75 mL)] was added to the homogeneous solution and the brown-red solution was stirred for 12 h. The tetrahydrofuran was removed in vacuum, naphthalene was sublimed (40°C, 10<sup>-2</sup> torr) and the yellow residue was extracted with pentane (40 mL). The pentane extract was filtered and the filtrate was concentrated in vacuum to ca. 10 mL and cooled (-10°C). The orange-yellow crystals (0.45 g, 14%) were collected and dried in vacuum, mp 128-130°C. Anal. for C<sub>20</sub>H<sub>52</sub>EuN<sub>2</sub>O<sub>2</sub>Si<sub>4</sub>: C, 38.9; H, 8.50; N, 4.54. Found: C, 39.4; H, 7.73; N, 4.63.

Bis[di(trimethylsilyl)amido]europium(II)bis(bipyridine) Complex. 2,2'-Bipyridine (0.064 g, 0.00041 mol) in benzene (30 mL) was added to bis[di(trimethylsilyl)amido] europium bis(tetrahydrofuran) (0.25 g, 0.00041 mol) in benzene (20 mL). The dark brown solution was stirred for 1 h and benzene was removed in vacuum. The residue was extracted with toluene (10 mL) and the extract was filtered. The filtrate was concentrated to ca. 5 mL and cooled (0°C). The yellow needles

(0.16 g, 62%) were collected and dried in vacuum, mp 85–87°C (dec).

Anal. Calcd for  $C_{22}H_{44}EuN_4Si_4$ : C, 42.0; H, 7.05; N, 8.91.

Found: C, 42.0; H, 6.66; N, 8.85.

Bis[di(trimethylsilyl)amido]europium(II)bis(1,2-dimethoxyethane).

Tris[di(trimethylsilyl)amido]europium<sup>6</sup> (1.5 g, 0.0024 mol) in 1,2-dimethoxyethane (40 mL) was added to a suspension of europium trichloride (0.31 g, 0.0012 mol) in 1,2-dimethoxyethane (50 mL) and the orange solution was stirred for 8 h. A solution of sodium naphthalene [prepared from sodium (0.10 g, 0.0044 mol) and naphthalene (0.46 g, 0.0036 mol) in 1,2-dimethoxyethane (40 mL)] was added to the chlorosilylamide and the brown-red solution was stirred for 12 h. The 1,2-dimethoxyethane was evaporated and naphthalene was removed by sublimation at 40°C/10<sup>-2</sup> torr. Pentane (40 mL) was added to the residue and the yellow solution was filtered. The filtrate was concentrated to ca. 25 mL and cooled (-10°C, 3 weeks). The needles were collected and dried in vacuum, mp 83–86°C. Yield was 0.25 g (11%).

Anal. Calcd for  $C_{20}H_{56}EuN_2O_4Si_4$ : C, 36.8; H, 8.64; N,

4.29. Found: C, 35.7; H, 8.40; N, 4.28.

Some of the air sensitive yellow crystals were inserted into thin walled quartz capillaries in an argon filled dry box. A crystal was subjected to Weissenberg photography to study the crystal symmetry and to evaluate its suitability for a structure determination. A second crystal was examined with a Picker FACS-I automated diffractometer equipped with a graphite monochromator and a Mo X-ray tube ( $\lambda(K\alpha_1)$  0.70930 Å). Omega scans of several low angle reflections showed peaks

with half-widths of  $0.14^\circ$  to  $0.18^\circ$ . The space group is C2/c. The setting angles of twelve manually centered reflections ( $40 < 2\theta < 45^\circ$ ) were used to determine by least-squares the following cell parameters:  $a = 13.398(4)\text{\AA}$ ,  $b = 17.454(6)\text{\AA}$ ,  $c = 15.013(5)\text{\AA}$ ,  $\beta = 92.91(3)^\circ$  and  $V = 3506\text{\AA}^3$ . For  $Z = 4$  and a molecular weight of 652.98, the calculated density is  $1.24 \text{ g/cm}^3$ .

Intensity data were collected using a  $\theta-2\theta$  scan technique with a scan speed of  $2^\circ/\text{min}$  on  $2\theta$ . Each peak was scanned  $0.9^\circ$  before the  $K\alpha_1$  peak to  $0.9^\circ$  after the  $K\alpha_2$  peak, and backgrounds were counted for 10 s at each end of the scan range. The temperature during the data collection was  $20 \pm 1^\circ\text{C}$ . Three standard reflections were measured every 200th scan. A total of 6531 scans ( $4^\circ < 2\theta < 45^\circ$ ) yielded 2308 unique data of which 2259 had  $F^2 > 1\sigma$ . An absorption correction ( $\mu = 19.5 \text{ cm}^{-1}$ ) was applied<sup>8</sup> which ranged from 1.21 to 1.32. The intensities of the three standard reflections decayed about 16% during the data collection period and the data were corrected for this effect.

A three-dimensional Patterson calculation showed the Eu atom position, and a subsequent least-squares refinement and Fourier calculation revealed all the non-hydrogen atoms in the structure. After a few cycles of least-squares refinements of the structure in which only the Eu atom was assigned anisotropic thermal parameters the R factor,  $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ , was 0.062.

The methylene carbon atoms of the dimethoxyethane ligand were ill behaved in the refinements as indicated by large thermal parameters and chemically unsatisfactory bond distances to adjacent atoms. A

difference Fourier map showed additional electron density in the region of the methylene carbon atoms indicating disorder. The two methylene carbon atoms were subsequently described as four half-atoms. Additional refinements of the structure converged and gave reasonable thermal parameters on the disordered atoms but with bond distances that were still chemically unacceptable. Restraints on the interatomic distances to the disordered atoms were included in a manner suggested by Waser,<sup>9</sup> and described in a previous paper.<sup>10</sup> The distance restraints were applied as follows: C-C,  $1.54 \pm 0.01\text{\AA}$ , C-O,  $1.43 \pm 0.01\text{\AA}$ ; C-C (methyl),  $2.37 \pm 0.08\text{\AA}$ ; C-O (nearest non-bonded neighbor),  $2.36 \pm 0.05\text{\AA}$ .

Some of the hydrogen atoms were observed in regions where they were expected to be, but were scattered among a larger number of "noise" peaks of comparable size. Isotropic hydrogen atoms at calculated positions,  $0.95\text{\AA}$  from the atoms to which they are bonded, were included in the least squares refinements, but not refined; hydrogen atoms to the disordered atoms were not included. The least-squares function used minimizes the function  $\Sigma w(|F_0| - |F_C|)^2 / \Sigma w F_0^2$ . The expressions used in processing the data and estimating weights are given in the supplementary material; the "ignorance factor" was set to 0.04. Scattering factors from Doyle and Turner<sup>11</sup> were used, and anomalous dispersion corrections<sup>12</sup> were applied. In the final least-squares refinement all of the non-hydrogen atoms, with the exception of the disordered atoms, were refined with anisotropic thermal parameters.

The discrepancy indices for 2259 data were  $F^2 > \sigma$  are

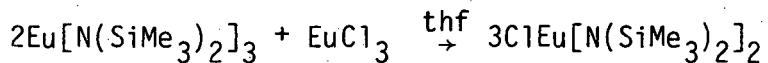
$$R = \Sigma |F_o| - |F_c| | / \Sigma |F_o| = 0.039$$

$$R_w = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma |F_o|^2]^{1/2} = 0.037$$

R for all 2362 data is 0.042. The error in an observation of unit weight is 1.10. In the last cycle, no parameter changed more than 0.16  $\sigma$ . In the final difference Fourier the two largest electron density peaks were  $\sim 0.8 \text{ e}/\text{\AA}^3$  and were  $\sim 0.8\text{\AA}$  from the disordered carbon atoms.

## RESULTS AND DISCUSSION

The best synthesis of the tetrahydrofuran complex of the europium(II)silylamide,  $\text{Eu}[\text{N}(\text{SiMe}_3)_2]_3(\text{thf})_2$ , is shown below.



Europium trichloride, which is only sparingly soluble in tetrahydrofuran, slowly dissolves in a tetrahydrofuran solution of the europium(III) silylamide,  $\text{Eu}[\text{N}(\text{SiMe}_3)_2]_3$ . All attempts to isolate an intermediate chloro-amide have failed. However, addition of sodium naphthalene to the chloro-amide, prepared in situ, yields a yellow solution from which yellow  $\text{Eu}[\text{N}(\text{SiMe}_3)_2]_2(\text{thf})_2$  may be isolated by crystallization from pentane. Though the yield is low (ca. 10%) the preparative method is the best that we have been able to develop. Further, we have been unable to prepare divalent silylamides of samarium or ytterbium, both of which have accessible divalent oxidation states.

The tetrahydrofuran in  $\text{Eu}[\text{N}(\text{SiMe}_3)_2]_2(\text{thf})_2$  can be displaced by 1,2-dimethoxyethane or bipyridine yielding yellow  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}(\text{MeOCH}_2\text{CH}_2\text{OMe})$  or  $[(\text{Me}_3\text{Si})_2\text{N}]_2\text{Eu}(\text{Bipy})$ , respectively. Both coordination complexes are soluble in hydrocarbon solvents from which they may be crystallized. The 1,2-dimethoxyethane complex

follows Curie behavior,  $X_M = C_M(T)^{-1}$ , from 5 to 45 K with  $C_M = 8.82$  and  $\mu_{eff} = 8.43$  B.M. The magnetic moment is similar to that found for the isoelectronic gadolinium(III) analogue (7.75 B.M. at 98 K)<sup>7</sup> which is consistent with a  $^8S_{7/2}$  ground state. In contrast the base-free europium(III) silylamine,  $Eu[N(SiMe_3)_2]_3$ , is a temperature independent paramagnet (T.I.P.); the temperature independent susceptibility being ca. 0.4 B.M. consistent with a  $^7F_0$  ground state. In addition, the ytterbium(III) derivative,  $Yb[N(SiMe_3)_2]_3$ , follows Curie behavior (5-45 K) with  $\mu_{eff} = 3.10$  B.M.

Isolation of well-developed crystals of  $Eu[N(SiMe_3)_2]_2(dme)_2$  allowed us to carry out this first single crystal x-ray analysis of a molecular complex of europium(II).

Positional parameters, distances and angles are listed in Tables I-III. The list of thermal parameters and observed structure factors have been submitted as supplementary data. Figure 1a is an ORTEP view providing the atom-labeling scheme. The view is along a crystallographic two-fold axis relating the two silylamine ligands and the two chelating ethers.

Figures 1a and 1b illustrate the two conformations of the 1,2-dimethoxyethane ligands that give rise to the disorder in the C(9) and C(10) atoms. It is not apparent whether this disorder is static or dynamic, but the two structures represent two different puckered conformations for the five-membered chelate rings. Disorder in the rest of the 1,2-dimethoxyethane ligand is probably present, but the effects are not as severe and are absorbed in the anisotropic thermal

parameters; because of this disorder, bond distances involving this ligand are probably not as accurate as indicated (see experimental section).

Figure 2 is a stereochemical view of the coordination geometry about the six-coordinate europium(II) ion. The coordination polyhedron cannot be described by a simple regular geometric figure. The two bulky silylamide groups are surprisingly close to one another, resulting in a N-Eu-N angle of only 134.5°. Kepert has shown, from points-on-a-sphere repulsion energy calculations, that for six-coordinate complexes of the type  $M(\text{bidentate})_2(\text{unidentate})_2$ , the unidentate ligands are pulled together when the bidentate ligands have small normalized bites.<sup>13</sup> Kepert defines the normalized bite of a chelating ligand as the distance between donor atoms in the chelate group, divided by the metal-donor atom distance. It is the bidentate nature of the 1,2-dimethoxyethane ligands that prevents the two silylamide groups from repelling each other to a greater extent. The normalized bite of the 1,2-dimethoxyethane ligands in this complex is among the smallest known, 0.98. Clearly, repulsive interactions between coordinated atoms are more important in defining this detail of the structure than the overall steric bulk of the ligands.

Bond angles and distances in the planar bis(trimethylsilyl)amido ligands are comparable to those found in other structures.<sup>14-18</sup> The Eu(II)-N bond distance is 2.53 Å, the first such bond length determined, whereas the Eu(III)-N bond length in  $\text{Eu}[\text{N}(\text{SiMe}_3)_2]_3$  is 2.26 Å. The difference of 0.27 Å, is due mostly to the change in bond length with

oxidation state, estimated as  $0.23\text{\AA}$  from the ionic radii listed by Shannon.<sup>19</sup> The Eu(II)-O bond lengths in this structure are  $2.638\text{\AA}$  and  $2.756\text{\AA}$ , and Kepert has shown that such a difference may accompany the bending distortion discussed above. These Eu(II)-O distances are in good agreement with those reported in the  $\text{EuCl}_2 \cdot 2\text{H}_2\text{O}$ <sup>20</sup> structure, which range from  $2.69\text{\AA}$  to  $2.74\text{\AA}$ .

**ACKNOWLEDGMENT**

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**SUPPLEMENTARY MATERIAL AVAILABLE**

Data processing formulas, the table of anisotropic thermal parameters and the listing of structure factor amplitudes (11 pages). Ordering information is given on any current masthead page.

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Table I. Positional Parameters<sup>a</sup>

Atom	x	y	z
Eu	0	.19015(2)	1/4
N	.1734(3)	.1341(2)	.2411(3)
Si(1)	.2594(1)	.1938(1)	.2010(1)
Si(2)	.2009(2)	.0436(1)	.2727(2)
O(1)	.0133(3)	.1674(3)	.4239(3)
O(2)	.0703(3)	.3065(3)	.3603(3)
C(1)	.1972(5)	.2815(4)	.1506(5)
C(2)	.3524(6)	.2350(6)	.2873(6)
C(3)	.3372(5)	.1520(5)	.1110(5)
C(4)	.326(1)	.0316(6)	.3334(9)
C(5)	.108(1)	.0042(6)	.3459(9)
C(6)	.2080(8)	-.0255(5)	.1775(7)
C(7)	-.0497(6)	.1275(5)	.4815(5)
C(8)	.0375(6)	.3821(5)	.3781(7)
C(9)	.067(1)	.2234(7)	.4809(8)
C(9')	.1078(9) <sup>b</sup>	.1962(8)	.457(1)
C(10)	.1376(8)	.2657(7)	.4212(8)
C(10')	.092(1) <sup>b</sup>	.2813(8)	.4533(8)
H(1)	.1623	.3076	.1950
H(2)	.1517	.2667	.1033
H(3)	.2466	.3145	.1284
H(4)	.3885	.1944	.3162

Table I (Continued)

H(5)	.3179	.2634	.3300
H(6)	.3976	.2677	.2588
H(7)	.2944	.1373	.0613
H(8)	.3726	.1087	.1336
H(9)	.3830	.1897	.0923
H(10)	.3288	.0619	.3859
H(11)	.3773	.0476	.2957
H(12)	.3359	-.0208	.3486
H(13)	.0442	.0040	.3153
H(14)	.1057	.0348	.3980
H(15)	.1261	-.0467	.3621
H(16)	.2588	-.0092	.1396
H(17)	.1458	-.0268	.1448
H(18)	.2239	-.0750	.2002
H(19)	-.0941	.1628	.5074
H(20)	-.0103	.1029	.5273
H(21)	-.0877	.0902	.4483
H(22)	-.0119	.3887	.4196
H(23)	.0260	.4155	.3288
H(24)	.1055	.3756	.4003

<sup>a</sup>Here and in the following tables the number in parentheses is the estimated standard deviation in the least significant digit.

<sup>b</sup>The disordered atom pair has the same number, and the two positions are distinguished with a prime on one of them.

Table II. Interatomic Distances ( $\text{\AA}$ )

Eu	-2N	2.530(4)
	-20(1)	2.638(4)
	-20(2)	2.756(4)
N	-Si(1)	1.686(5)
	-Si(2)	1.685(5)
	Si(1)-C(1)	1.88(1)
	-C(2)	1.89(1)
	-C(3)	1.90(1)
	Si(2)-C(4)	1.88(1)
	-C(5)	1.83(1)
	-C(6)	1.88(1)
O(1)	-C(7)	1.42(1)
	-C(9)	1.47(1)
	-C(9')	1.43(1)
O(2)	-C(8)	1.42(1)
	-C(10)	1.44(1)
	-C(10')	1.48(1)
C(9)	-C(10)	1.52(1)
	C(9')-C(10')	1.50(1)

Table III. Selected Angles (deg.)<sup>a</sup>

N	-Eu	-N'	134.5(2)
N	-Eu	-O(1)	88.7(2)
N	-Eu	-O(1)'	84.6(2)
N	-Eu	-O(2)	91.7(2)
N	-Eu	-O(2)'	122.7(2)
O(1)-Eu		-O(1)'	162.7(2)
O(1)-Eu		-O(2)	60.8(2)
O(1)-Eu		-O(2)'	135.2(2)
O(2)-Eu		-O(2)'	85.1(2)
Eu	-N	-Si(1)	115.2(2)
Eu	-N	-Si(2)	122.4(2)
N	-Si(1)-C(1)		110.5(3)
N	-Si(1)-C(2)		115.3(3)
N	-Si(1)-C(3)		115.3(3)
N	-Si(2)-C(4)		114.7(4)
N	-Si(2)-C(5)		112.0(4)
N	-Si(2)-C(6)		114.1(4)
C(1)-Si(1)-C(2)			103.2(4)
C(1)-Si(1)-C(3)			105.9(4)
C(2)-Si(1)-C(3)			105.5(4)
C(4)-Si(2)-C(5)			106.5(6)
C(4)-Si(2)-C(6)			102.6(5)
C(5)-Si(2)-C(6)			106.1(6)

Table III (Continued)

C(7)-O(1) -C(9)	105(1)
C(7)-O(1) -C(9')	120(1)
C(8)-O(2) -C(10)	122(1)
C(8)-O(2) -C(10')	98(1)
O(1)-C(9) -C(10)	106(1)
O(2)-C(10)-C(9)	103(1)
O(1)-C(9')-C(10')	116(1)
O(2)-C(10')-C(9')	124(2)

<sup>a</sup>Primed atoms inside the parentheses, i.e. C(9), C(9'), refers to a disordered pair. Primed atoms outside the parentheses, i.e. O(1)', refer to the symmetrically equivalent atom at -x, y, 1/2-z.

## FIGURE CAPTIONS

- Fig. 1a. ORTEP drawing as viewed down the 2-fold axis. The primed atoms are related to the unprimed atoms by the 2-fold axis perpendicular to the drawing on the Eu atom.
- Fig. 1b. ORTEP drawing showing the molecule in the other configuration due to the disorder in C(9) and C(10).
- Fig. 2. ORTEP view of the coordination sphere about Eu(II).

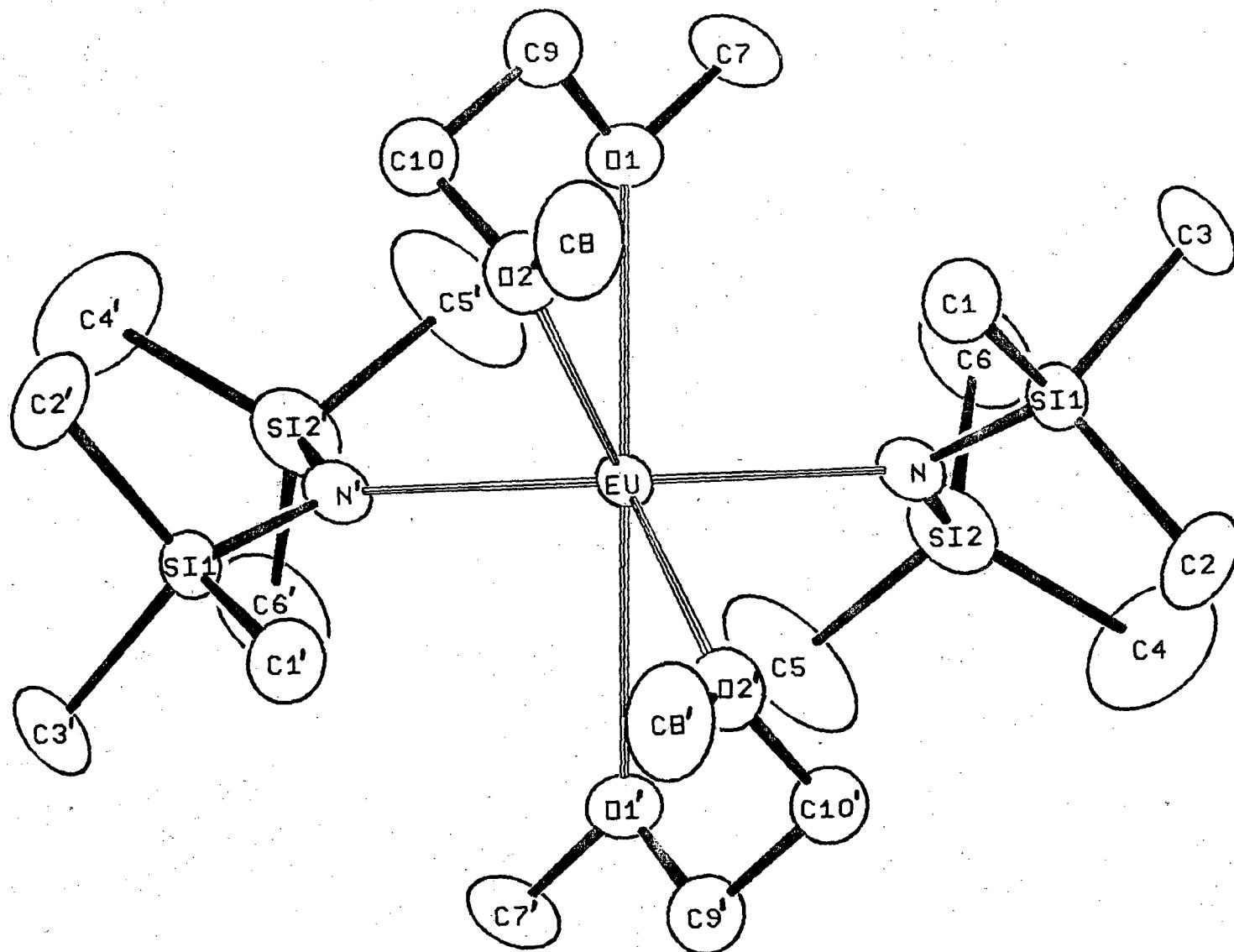


Fig. 1a

XBL 7911-12675

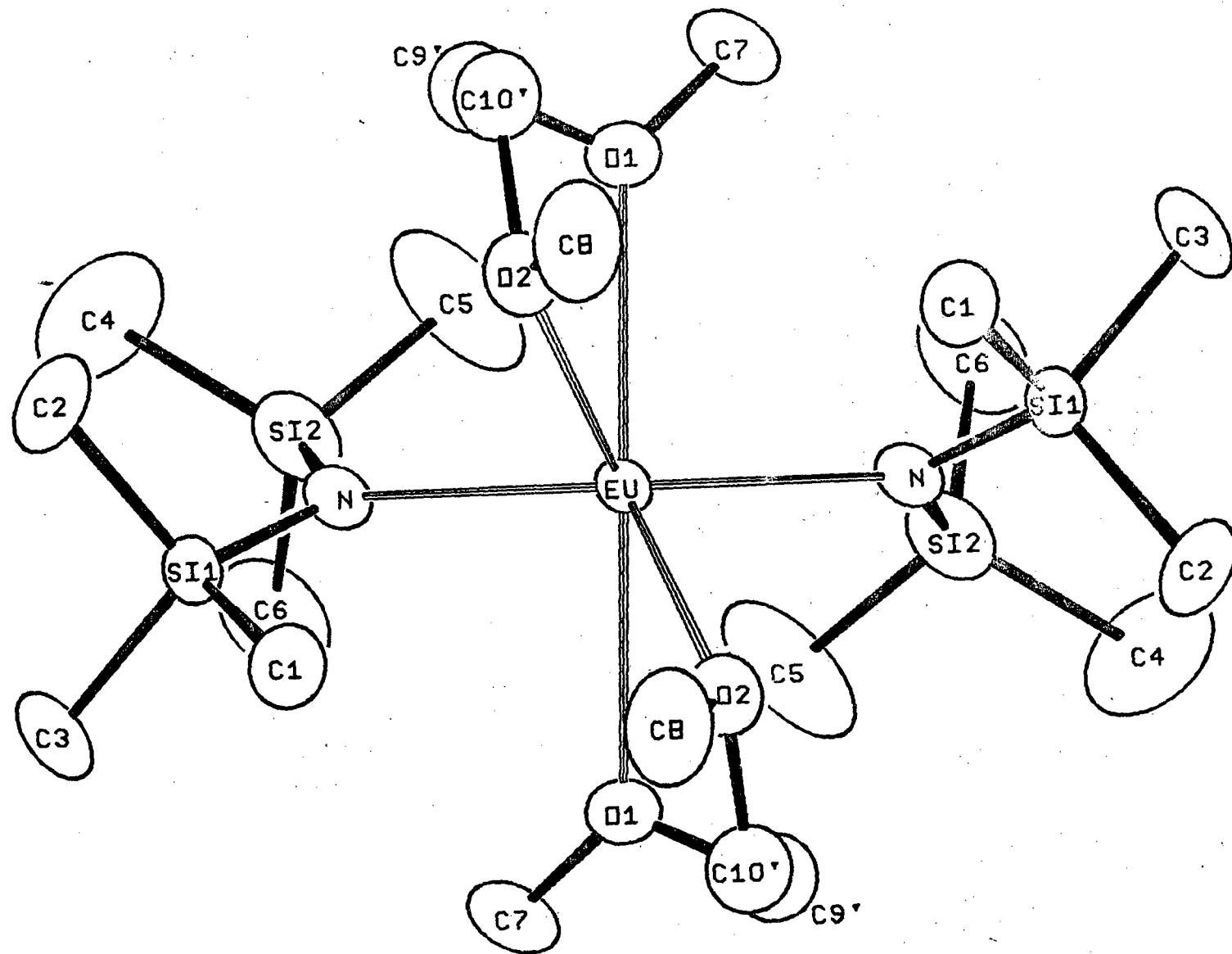


Fig. 1b

XBL 7911-12676

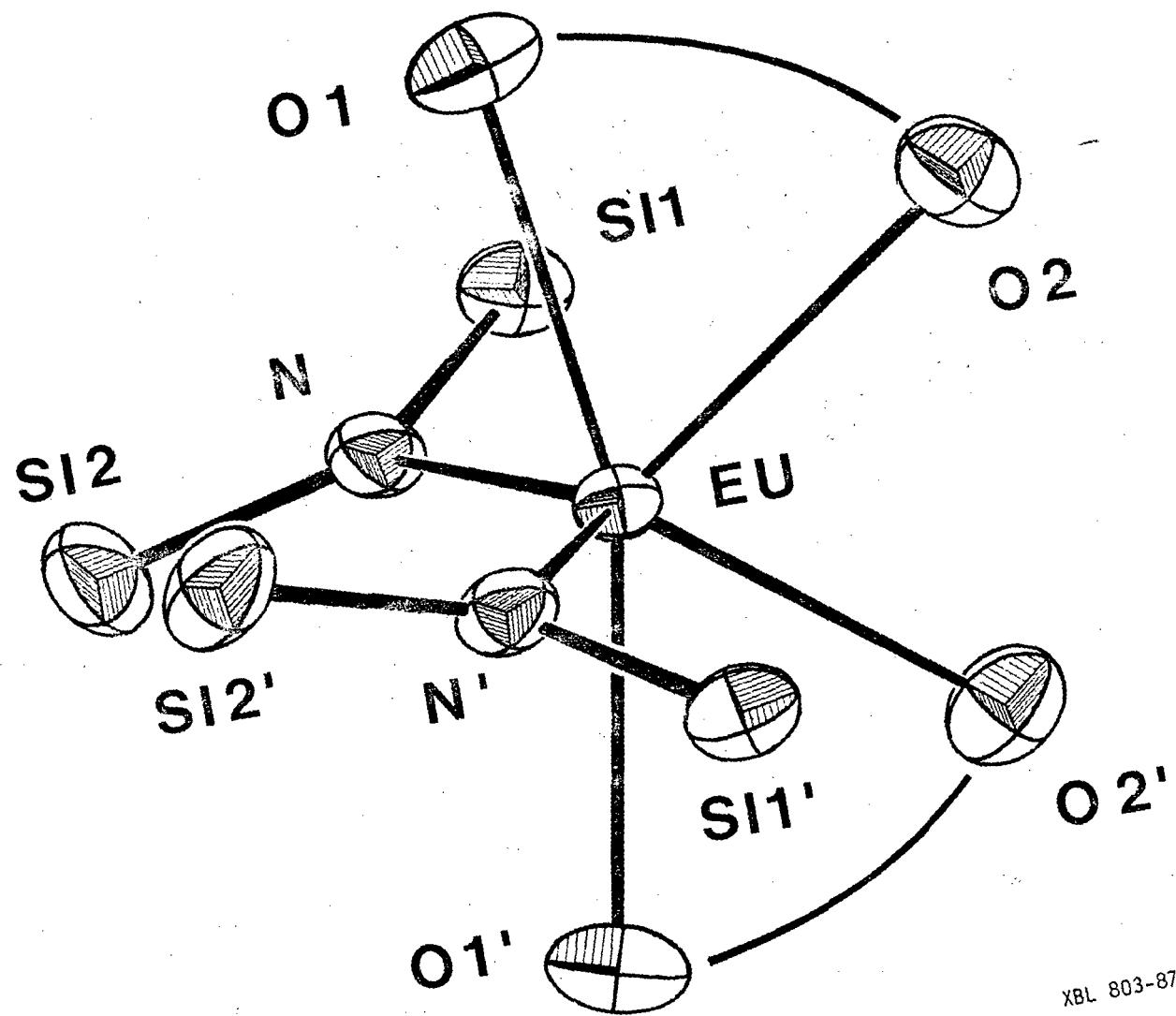


Fig. 2

Supplementary Materials for

DIVALENT LANTHANIDE CHEMISTRY;  
PREPARATION OF SOME FOUR AND SIX COORDINATE  
BIS(TRIMETHYLSILYLAMIDO)COMPLEXES OF EUROPiUM(II).  
THE CRYSTAL STRUCTURE OF BIS[DI(TRIMETHYL-  
SILYL)AMIDO]BIS(1,2-DIMETHOXYETHANE)EUROPiUM(II).

By T. Don Tilley, Allan Zalkin\*, Richard A. Andersen and  
David H. Templeton

DATA PROCESSING FORMULAE

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

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

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

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

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

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

$$\sigma(F_a^2) = [\sum \sigma^2(F^2)/n]^{\frac{1}{2}} \quad \text{When } S(F_a^2) > 4\sigma(F_a^2), \sigma(F_a^2) \text{ is replaced by } S(F_a^2).$$

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

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

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

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

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

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

C = counts recorded during a scan

$\theta_m$  = monochromater angle

I = individual raw intensity,  
background removed.

$\theta$  = crystal diffraction angle

$t_c$  = scan count time

S = scatter

$t_b$  = background count time

a = average

$B_1$  = individual background count

q = additional uncertainty that  
affects the weak intensities

$\sigma(B)$  = estimated standard dev-  
iation of the total back-  
ground count

p = estimate of non-statistical  
errors

F = structure factor

wtg = weighting factors in least  
squares

D = decay correction; an empir-  
ically applied correction  
obtained from the fluctuations  
of the standard reflections.

A = absorption correction

Lp = Lorentz and polarization  
corrections

Table of Thermal Parameters

Atom	$B_{11}$	$B_{22}$	$B_{33}$	$B_{12}$	$B_{13}$	$B_{23}$
Eu	3.64(2)	4.08(2)	3.16(2)	0	.56(1)	0
N	5.3(2)	5.2(2)	4.2(2)	1.2(2)	1.2(2)	.6(2)
Si(1)	4.07(6)	6.91(9)	4.98(8)	.46(7)	.70(5)	.40(8)
Si(2)	9.2(1)	6.3(1)	8.4(1)	3.14(9)	3.1(1)	2.49(9)
O(1)	6.4(2)	10.1(3)	4.6(2)	-.6(2)	.1(2)	1.6(2)
O(2)	6.5(2)	7.6(3)	7.4(3)	-.9(2)	.9(2)	-1.7(2)
C(1)	6.6(3)	6.0(3)	7.8(4)	-.7(3)	.3(3)	1.7(3)
C(2)	6.2(4)	12.8(6)	10.1(6)	-.4(4)	-2.6(4)	-1.1(5)
C(3)	7.0(4)	11.4(5)	8.3(5)	.5(4)	4.3(3)	.6(4)
C(4)	17.8(9)	11.9(7)	17.9(10)	7.0(7)	-5.9(8)	4.1(7)
C(5)	22.0(11)	8.9(6)	20.3(11)	4.5(6)	14.0(10)	6.7(7)
C(6)	15.3(7)	6.1(4)	14.0(7)	2.7(4)	5.8(6)	-.3(4)
C(7)	9.7(5)	10.9(5)	5.5(4)	1.1(4)	2.9(3)	2.7(4)
C(8)	8.8(5)	6.6(4)	15.7(8)	-.8(4)	.6(5)	-2.7(5)

Table of Thermal Parameters (Continued)

Atom	B <sup>b</sup>
C(9)	6.6(3)
C(9')	8.5(4)
C(10)	6.6(3)
C(10')	8.4(4)
H	10.0 <sup>c</sup>

<sup>a</sup>The anisotropic temperature factor has the form  $\exp(-0.25(B_{11}h^2a^{*2}+2B_{12}hka^{*}b^{*}+\dots))$ .

<sup>b</sup>The isotropic temperature factor has the form  $\exp(-B(\sin\theta/\lambda)^2)$ .

<sup>c</sup>All 24 hydrogen atoms were assigned the same thermal parameter and not varied.

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL > 3.0)  
 EU(N(SI(CH3)3)3)2.2(CH3OCH2CH2OCH3) F(0,0,0) = 3706

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.

SG = ESTIMATED STANDARD DEVIATION OF FCB. DEL = |FOB| - |FCA|.

\* INDICATES ZERO WEIGHTED DATA.

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
H,K= 0, 0	7 293	6	2	11 148	8	-6	6 233	5	12	-7	15 29	-28*			
21 304 26 -21	8 203	6	3	12 17	49	6	7 489	10	13	-6 404	8	3			
4 656 14 15	9 127	6	-7	H,K= 0, 14	8 119	6	-4	-5 0	25	-19*					
6 590 12 20	10 174	7	-1	0 172	8	1	9 472	10	2	-4 368	8	-4			
8 469 10 17	11 119	8	-2	1 291	7	-5	10 82	9	-16	-3 62	24	-8			
10 323 8 -1	12 170	13	-2	2 162	7	-11	11 308	7	6	-2 510	10	27			
12 247 6 2	13 99	8	-9	3 293	7	-0	12 86	18	8	-1 82	11	-6			
14 167 6 -1	14 99	11	-14	4 148	7	10	13 193	12	7	0 573	12	4			
16 158 7 -6	15 116	9	-1	5 222	8	-8	14 46	21	1	1 538	11	28			
H,K= 0, 2	H,K= 0, 8	6 127	13	-3	15 108	19	-10	2 520	11	-14					
0 894 18 36	0 568	12	-1	7 127	7	-2	16 37	42	-6*	3 361	8	25			
1 929 19 -9	1 168	4	3	8 74	9	-10	H,K= 1, 3	4 601	12	-4					
2 187 5 -35	2 574	12	-10	9 109	7	1	-15 97	9	-1	5 358	7	7			
3 657 13 -3	3 124	4	1	10 76	14	-0	-14 198	8	-6	6 607	13	10			
4 436 10 42	4 425	9	-10	H,K= 0, 16	-13 84	16	-8	7 274	6	-2					
5 828 17 24	5 22	28	17*	0 196	8	1	-12 305	7	9	8 403	8	-6			
6 380 8 -7	6 341	7	-5	1 46	15	9	-11 141	8	5	9 182	6	-15			
7 383 8 17	7 84	8	-11	2 181	5	-10	-10 209	6	6	10 269	6	0			
8 231 5 8	8 206	6	-0	3 43	21	11	-9 132	6	9	11 148	6	0			
9 186 5 -7	9 32	35	10	4 166	6	1	-8 168	6	8	12 204	14	-3			
10 194 5 1	10 191	9	-12	5 38	30	5	-7 73	6	-5	13 90	10	-4			
11 162 6 1	11 47	54	13	6 130	10	-5	-6 431	9	10	14 105	8	-16			
12 144 7 -9	12 147	9	6	7 53	14	16	-5 20	36	4	15 23	47	-30*			
13 172 11 -5	13 0	37	-13*	8 106	11	-5	-4 644	13	53	H,K= 1,	7				
14 130 8 11	14 113	11	-6	H,K= 0, 18	-3 235	9	13 -15 169	6	2						
15 95 13 -19	H,K= 0, 10	0 152	7	10	-2 59	15	22 -14 142	6	7						
16 127 8 0	0 343	8	1	1 34	35	-7	-1 194	7	-2	-13 211	7	-4			
H,K= 0, 4	1 225	5	-7	2 143	6	-6	0 417	9	-38	-12 115	16	-1			
0 329 7 10	2 289	7	3	3 40	22	-9	1 210	6	-9	-11 327	8	-6			
1 654 13 10	3 80	11	2	4 135	6	5	2 641	13	-6	-10 157	6	-3			
2 358 8 2	4 178	5	-7	H,K= 1, 1	3 412	6	10 -9 278	6	-5						
3 593 12 39	5 116	5	-4	-16 54	19	-19	4 995	20	3	-8 138	6	-20			
4 25 46 2	6 120	6	9	-15 180	8	-2	5 284	6	-18	-7 333	7	9			
5 651 13 1	7 117	6	3	-14 94	9	-15	6 703	14	15	-6 192	5	7			
6 115 4 1	8 149	5	0	-13 285	9	-1	7 204	5	3	-5 408	8	-11			
7 353 8 17	9 58	23	12	-12 194	6	-3	8 458	10	-7	-4 238	5	3			
8 42 13 -15	10 156	10	-10	-11 376	8	-4	9 215	7	7	-3 467	10	0			
9 167 6 -3	11 88	8	-17	-10 188	5	2	10 378	8	10	-2 251	5	8			
10 35 44 0	12 91	8	-9	-9 399	9	1	11 156	7	-5	-1 538	11	5			
11 224 6 6	13 45	30	-15	-8 198	6	6	12 286	8	1	0 12	33	-39*			
12 41 27 22	H,K= 0, 12	-7 423	9	13	13 83	22	-1	1 437	9	-18					
13 226 9 2	0 247	6	-16	-6 288	7	-1	14 163	6	4	2 153	4	-9			
14 44 49 33	1 252	6	-8	-5 566	12	31	15 60	15	-2	3 405	8	7			
15 181 8 -3	2 188	5	-2	-4 570	12	39	H,K= 1, 5	4 255	6	4					
H,K= 0, 6	3 293	7	0	-3 57	58	-4	-15 12	39	-12	5 427	9	-1			
0 712 14 15	4 143	6	-6	-2 238	5	7	-14 243	6	-6	6 232	6	-0			
1 583 12 -24	5 275	7	-14	-1 966	20	42	-13 65	24	2	7 389	8	-7			
2 435 9 4	6 75	14	8	1 942	19	-6	-12 309	5	-2	8 150	5	-11			
3 648 13 2	7 204	6	5	2 232	5	-8	-11 85	11	14	9 234	7	-13			
4 234 5 3	8 78	15	-13	3 452	10	12	-10 312	7	-4	10 89	8	13			
5 495 10 -1	9 158	6	-3	4 435	10	32	-9 40	18	8	11 196	6	9			
6 289 6 9	10 29	44	-15	5 754	15	16	-8 262	6	4	12 76	16	18			

STRUCTURE FACTORS CONTINUED FOR  
EU(N(SI(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>.2(CH<sub>3</sub>OCH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>)

PAGE 2

L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	
13	103	14	10	10	189	9	-10	1	155	6	-5	H,K#	2,	4	8 209	5 -14
14	53	17	-4	11	77	12	-2	2	10	39	8	-15	145	9 -7	9 246	6 -6
	H,K#	1,	9	12	139	6	-1	3	155	8	-8	-14	63	13 -2	10 198	6 4
-14	19	49	-12*	13	84	9	17	4	50	21	44	-13	230	10 -1	11 248	8 -1
-13	150	6	-12	H,K#	1,	13	5	177	7	1	-12	29	63	-6	12 164	9 -2
-12	85	20	8	-11	59	13	23	6	0	41	-7*	-11	352	8 0	13 172	9 2
-11	217	6	-13	-10	175	6	-6	H,K#	2,	0	-10	97	8 9	14 83	11 -4	
-10	86	15	-7	-9	24	53	21	-16	169	9	9	-9	382	8 -13	15 109	9 -8
-9	226	6	-9	-8	202	7	-2	-14	212	6	8	-8	36	17 -5	H,K#	2, 8
-8	121	6	1	-7	45	26	26	-12	389	8	5	-7	539	11 3	-14 185	7 -2
-7	157	5	1	-6	235	6	-5	-10	494	10	9	-6	177	4 5	-13 38	32 -1
-6	241	6	-5	-5	21	39	-11	-8	562	11	18	-5	617	13 -8	-12 288	8 -5
-5	115	5	-3	-4	242	6	-0	-6	423	9	-27	-4	6 41	3 -11	22 50	-7*
-4	62	11	6	-3	46	15	-5	-4	188	8	21	-3	410	9 -2	-10 413	9 -11
-3	166	5	-1	-2	222	6	-7	-2	101	12	2	-2	58	33 -11	-9 71	13 -15
-2	98	5	1	-1	21	33	-20	0	282	6	-12	-1	608	12 21	-8 406	9 11
-1	376	8	1	0	253	6	1	2	509	11	32	0	351	8 13	-7 48	13 3
0	194	5	9	1	33	26	21	4	681	14	91	1	516	11 -8	-6 482	10 -14
1	462	10	-14	2	309	7	-7	6	578	12	-21	2	76	15 6	-5 134	8 3
2	101	4	-2	3	18	34	13	8	450	10	8	3	526	11 -6	-4 355	8 -9
3	503	10	-8	4	356	8	-5	10	383	8	-2	4	206	5 23	-3 108	4 1
4	56	14	5	5	35	48	-2	12	322	8	-2	5	685	14 10	-2 366	8 9
5	427	9	-5	6	264	6	-1	14	219	6	5	6	37	12 20	-1 40	9 -5
6	75	7	-9	7	22	48	-39	H,K#	2,	2	7	545	11 1	0 299	6 -7	
7	375	9	6	8	177	8	5	-16	110	8	10	8	54	11 -4	1 156	4 -2
8	34	37	5	9	53	15	23	-15	102	9	-2	9	445	10 -4	2 288	6 -6
9	282	7	-3	10	136	8	-14	-14	138	10	3	10	19	38 9*	3 193	5 -1
10	41	30	26	11	58	13	16	-13	193	11	-4	11	417	9 5	4 276	6 12
11	198	8	-2	H,K#	1,	15	-12	227	7	1	12	37	64 36	5 63	12 13	
12	37	40	11*	-9	142	10	10	-11	276	6	9	13	267	8 -1	6 340	7 7
13	132	9	6	-8	59	17	-1	-10	306	7	-5	14	13	39 -2*	7 146	5 -2
14	47	52	42	-7	138	6	-2	-9	287	8	1	15	151	7 4	8 327	7 -6
	H,K#	1,	11	-6	64	10	1	-8	328	7	0	H,K#	2,	6	9 155	9 4
-13	92	15	3	-5	144	6	0	-7	329	7	9	-15	167	6 -4	10 298	7 1
-12	128	7	4	-4	84	8	-4	-6	448	9	4	-14	87	14 5	11 73	16 1
-11	101	8	4	-3	169	6	-4	-5	373	8	-5	-13	233	7 0	12 232	6 1
-10	135	6	-0	-2	101	9	1	-4	280	7	-3	-12	196	7 3	13 40	29 -4
-9	168	9	2	-1	190	7	-3	-3	214	8	30	-11	335	7 4	14 161	10 5
-8	104	7	-6	0	116	11	2	-2	493	10	-14	-10	237	8 -4	H,K#	2, 10
-7	109	6	14	1	178	10	-6	-1	36	41	6	-9	375	8 1	-13 83	10 -11
-6	166	5	-10	2	149	8	4	0	670	14	11	-8	223	6 -5	-12 149	7 -6
-5	58	15	-12	3	152	10	3	1	76	20	-8	-7	405	6 6	-11 147	8 -7
-4	101	6	21	4	162	6	-1	2	277	6	-8	-6	289	6 1	-10 241	7 -4
-3	140	5	6	5	144	7	-5	3	170	11	20	-5	405	8 9	-9 168	8 3
-2	157	5	-12	6	123	6	-2	4	694	14	47	-4	293	6 4	-8 265	7 9
-1	245	6	-4	7	122	8	-8	5	450	9	0	-3	188	5 8	-7 294	7 -13
0	270	6	-3	8	127	6	7	6	512	11	18	-2	372	6 16	-6 278	6 -10
1	229	6	5	9	106	7	10	7	200	5	10	-1	301	6 20	-5 280	6 -2
2	310	7	1	H,K#	1,	17	8	477	10	13	0	315	7 -6	-4 321	7 -0	
3	184	5	-7	-6	35	33	17	9	247	7	-0	1	195	5 6	-3 163	5 -7
4	329	7	-1	-5	91	9	7	10	364	8	1	2	79	5 -7	-2 370	8 0
5	217	5	-4	-4	38	39	2*	11	279	7	2	3	229	5 7	-1 230	5 -6
6	314	7	-10	-3	138	6	10	12	275	7	9	4	197	5 -7	0 352	8 -3
7	223	6	1	-2	35	27	11	13	200	7	-4	5	159	4 -2	1 209	5 6
8	214	6	12	-1	145	6	-4	14	167	7	6	6	358	8 2	2 293	7 10
9	176	8	-18	0	0	34	-12*	15	107	10	4	7	279	6 -14	3 234	5 8

STRUCTURE FACTORS CONTINUED FOR  
EU(N(SI(CH<sub>3</sub>)<sub>3</sub>)<sub>3</sub>)<sub>2</sub>.2(CH<sub>3</sub>OCH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>)

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L	FOB	SG	DEL	L	FCB	SG	DEL	L	FOB	SG	DEL	L	FCB	SG	DEL	L	FOB	SG	DEL
4	270	6	0	9	164	6	7	10	121	7	-7	2	491	10	20	-2	27	28	-7
5	260	6	-7	10	65	18	-10	11	216	6	8	3	208	5	24	-1	564	12	-5
6	314	7	-5	H,K=	2,	16	12	119	20	-5	4	309	6	10	0	70	6	4	
7	205	6	2	-8	145	6	5	13	228	7	10	5	163	4	3	1	359	8	-5
8	331	7	-6	-7	25	42	-9	14	76	17	-8	6	214	5	-4	2	16	32	6*
9	150	12	7	-6	139	7	6	15	179	6	1	7	87	6	-10	3	339	7	1
10	234	6	-2	-5	41	20	12	H,K=	3,	3	8	218	6	6	4	74	7	2	
11	151	6	-8	-4	168	6	9	-15	71	12	4	9	32	41	-9	5	296	6	3
12	158	6	9	-3	58	13	-1	-14	127	10	0	10	249	7	-1	6	144	5	-6
13	98	20	-1	-2	181	5	-2	-13	77	16	-4	11	48	20	-10	7	317	7	-0
	H,K=	2,	12	-1	53	13	-6	-12	229	11	3	12	280	8	4	8	101	7	8
-12	2	46	-30*	0	180	7	4	-11	169	6	5	13	55	24	11	9	247	6	2
-11	185	7	4	1	46	15	-3	-10	259	6	1	14	180	6	1	10	75	20	-4
-10	64	11	2	2	134	7	-3	-9	223	5	1	15	37	40	14*	11	202	7	-5
-9	213	6	2	3	73	9	10	-8	507	11	-0	H,K=	3,	7	12	72	10	2	
-8	101	10	5	4	126	7	8	-7	348	8	6	-14	89	10	10	13	165	9	2
-7	282	7	-4	5	66	13	9	-6	782	16	11	-13	137	8	6	H,K=	3,	11	
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12	132	7	-3	-9	192	9	-6	-8	162	5	0	-9	35	37	10	-7	270	€	1
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-13	29	44	27*	-6	75	11	4	-5	537	11	-7	-6	401	8	0	-4	38	21	-7
-12	229	7	1	-5	218	6	-1	-4	127	7	5	-5	154	5	-3	-3	355	€	-15
-11	16	50	3*	-4	114	7	10	-3	521	11	-8	-4	402	9	2	-2	33	27	27
-10	286	8	2	-3	197	8	7	-2	88	5	1	-3	202	5	-2	-1	330	7	1
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STRUCTURE FACTORS CONTINUED FOR  
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-6	225	6	0	0	354	8	1	8	37	59	-3	-10	122	7	2	-10	43	24	3
-5	166	6	6	2	364	8	-11	9	170	9	3	-9	99	11	-7	-9	179	7	4
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-5	65	15	-2	5	235	7	-7	-5	257	6	5	5	170	11	-0
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-3	0	36	-9*	7	208	7	5	-3	209	7	-1	7	162	6	1
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3	26	34	-11	-6	127	11	1	3	109	7	-3	-8	240	6	5
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5	81	9	5	-4	138	6	1	5	145	10	1	-6	286	7	5
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7	58	13	9	-2	147	6	2	7	138	8	3	-4	245	7	-8
8	240	6	14	-1	0	45	-26*	8	106	7	-15	-3	33	46	28
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	H,K=	11,	11	7	146	7	-1	-4	121	6	3	3	79	35	-7				
-5	103	9	-11	H,K=	12,	6	-3	52	14	-1	4	15	62	9					
-4	125	6	7	-8	96	8	8	-2	128	6	2	5	94	16	-1				
-3	120	7	5	-7	56	22	-5	-1	56	17	4	6	40	46	27				
-2	132	6	7	-6	118	7	5	0	136	9	10	7	76	17	-2				
-1	121	6	10	-5	86	10	-3	1	71	9	8	H,K=	14,	6					
0	130	6	6	-4	92	7	8	2	140	7	3	0	67	18	13				
1	93	13	-16	-3	136	6	-5	3	62	13	6	1	63	27	-22				
2	96	12	1	-2	128	7	-8	4	172	7	6	2	73	17	29				
3	83	9	-1	-1	146	7	7	5	75	20	5	3	90	14	8				
	H,K=	12,	0	0	166	7	2	H,K=	13,	5	4	31	46	-18					
-8	148	6	-5	1	131	6	-5	-6	117	7	1	5	89	14	12				
-6	185	6	-14	2	145	6	6	-5	64	16	5	6	0	51	-48*				
-4	156	8	-2	3	127	6	-8	-4	104	8	-7	H,K=	14,	8					
-2	213	8	-5	4	119	7	-10	-3	42	21	-11	0	120	23	11				

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