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DIVALENT LANTHANIDE CHEMISTRY;
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SODIUM TRIS[BIS(TRIMETHYLSILYL)AMIDO]-EUROPATE(II)
AND -YTTERBATE(II), $NaM[N(SiMe_3)_2]_3$

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and Allan Zalkin

March 1983

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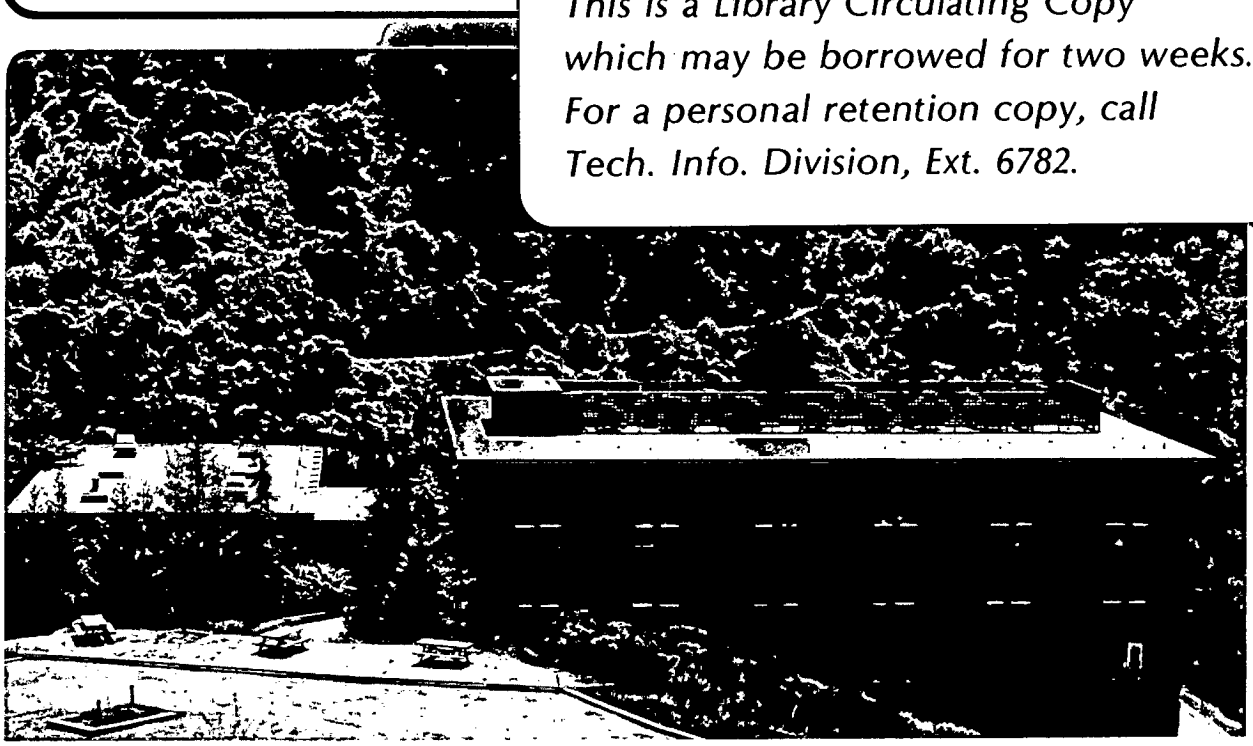

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DIVALENT LANTHANIDE CHEMISTRY; PREPARATION AND CRYSTAL STRUCTURES OF SODIUM TRIS[BIS(TRIMETHYLSILYL)AMIDO]-EUROPATE(II) AND -YTTERBATE(II), $NaM[N(SiMe_3)_2]_3$

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ABSTRACT

A new synthetic method for high yield preparations of the divalent silylamides $M[N(SiMe_3)_2]_2[MeOCH_2CH_2OMe]_2$, where M is Eu or Yb, has been developed that involves reaction of MI_2 and $NaN(SiMe_3)_2$ in $MeOCH_2CH_2OMe$. Reaction of EuI_2 and $NaN(SiMe_3)_2$ in Et_2O yields $NaEu[N(SiMe_3)_2]_3$. Reaction of YbI_2 with $NaN(SiMe_3)_2$ in Et_2O yields two products depending upon the crystallization conditions. If Et_2O is used, $Yb[N(SiMe_3)_2]_2(OEt_2)_2$ is isolated. In contrast, if toluene is used, $NaYb[N(SiMe_3)_2]_3$ is isolated. The crystal structures of $NaEu[N(SiMe_3)_2]_3$ and $NaYb[N(SiMe_3)_2]_3$ were determined by X-ray diffraction methods. Crystals of $NaEu[N(SiMe_3)_2]_3$ are monoclinic, $P2_1/n$, with cell dimensions, $\underline{a}$ = 17.586(6) Å, $\underline{b}$ = 19.170(6) Å, $\underline{c}$ = 21.808(6) Å, β = 107.90(4)°; for Z = 8, the calculated density is 1.246 g/cm^3. Crystals of $NaYb[N(SiMe_3)_2]_3$ are orthorhombic, Pbca, with cell dimensions, $\underline{a}$ = 19.334(6) Å, $\underline{b}$ = 18.534(6) Å, $\underline{c}$ = 20.020(6) Å; for Z = 8, the calculated density is 1.254 g/cm^3. The structures were refined to conventional R factors of 0.043 (6757 data, $F^2 > 3\sigma(F^2)$ for the Eu

complex, and 0.056 (2546 data, $F^2 > 3\sigma(F^2)$) for the Yb complex. In the europium complex there are two crystallographically distinct, but chemically equivalent molecules. The molecular structures of the Eu and Yb complexes are similar. The lanthanide atom bonds to one of the silylamide ligands exclusively, and shares the two others with the sodium atom. The Eu-N and Yb-N (unshared) distances are 2.46(1) Å and 2.38(2) Å respectively; the average Eu-N and Yb-N (shared) distance are 2.55(2) Å and 2.46(2) Å respectively; the average Na-N distance is 2.46(1) Å. Several noteworthy close contacts between the metal ions and methyl carbon atoms have been noted; i.e. Na-C 2.70 Å, Eu-C 2.97 Å and Yb-C 2.86 Å.

Our synthetic studies in f-metal chemistry have centered on the preparation of complexes with low coordination numbers so that the large, electropositive, metal centers are accessible to external reagents.[1] In developing this theme we have studied the bis(trimethylsilyl)amido ligand, $(Me_3Si)_2N^-$, since its size prevents coordination numbers from exceeding three in binary compounds.[2] We have been particularly interested in the redox chemistry of monomeric divalent lanthanide complexes.[3]

We have described a synthetic method for $Eu[N(SiMe_3)_2]_2L_2$, where L is tetrahydrofuran or 1,2-dimethyoxyethane, that involves sodium naphthalene reduction of $Eu[N(SiMe_3)_2]_2Cl$ in tetrahydrofuran.[4] This is a poor synthetic method since the yields are low (ca. 15 percent) and the method cannot be extended to ytterbium. Further, sodium naphthalene must be used in the relatively basic solvent, tetrahydrofuran. This latter deficiency is particularly troublesome since tetrahydrofuran tenaciously coordinates to the metal center. Accordingly, we wished to find a synthetic method that would allow us to prepare solvent-free, divalent silylamides or at the very least, diethyl ether complexes. Since diethyl ether is a weaker base than tetrahydrofuran it can be displaced from $Yb(Me_5C_5)_2(OEt_2)$[5a] or $Yb[N(SiMe_3)_2]_2(OEt_2)_2$,[5b] whereas tetrahydrofuran cannot be displaced, by various phosphines. Thus, a synthetic method that yields diethyl ether complexes, rather than tetrahydrofuran ones, was desired.

Synthetic Studies. Europium diiodide reacts with sodium bis(trimethylsilyl)amide in 1,2-dimethyoxyethane to give the six-coordinate, yellow complex, $Eu[N(SiMe_3)_2]_2(dme)_2$, previously characterized by X-ray crystallography,[4] in 79 percent yield. The yield is significantly improved over that previously reported. If diethyl ether is used as a reaction solvent, the reaction takes a different course. Stirring sodium bis(trimethylsilyl)amide with europium diiodide in diethyl ether affords a yellow solution from which the anionic complex, $NaEu[N(SiMe_3)_2]_3$, is isolated after crystallization from toluene, as shown by X-ray crystallography (see below).

Ytterbium diiodide and sodium silylamide in 1,2- dimethyoxyethane give the blue, diamagnetic, six-coordinate complex, $Yb[N(SiMe_3)_2]_2(dme)_2$, presumably isostructural with its europium analogue.[4] Thus, the new synthetic method is more general than the one previously described.[4] Curiously, the blue, 1:2 dimethoxyethane complex dissolves in pentane to give a red solution, and the red 1:1 complex, $Yb[N(SiMe_3)_2](dme)$, can be crystallized from the pentane solution. The ready loss of the diether from the ytterbium complex is presumably due to steric effects, Yb(II) being ca. 0.15 Å smaller than Eu(II).[6]

As observed for europium, use of diethyl ether as a solvent rather than dimethoxyethane changes the reaction product. Ytterbium diiodide and sodium silylamide yield an orange-red solution in diethyl ether from which orange $Yb[N(SiMe_3)_2]_2(OEt_2)_2$ may be crystallized from pentane. In contrast, if the orange-red diethyl ether solution formed by reaction of ytterbium diiodide and sodium silylamide is taken to dryness and the residue

extracted with toluene, the red $NaYb[N(SiMe_3)_2]_3$ is isolated, as shown by X-ray crystallography (see below). The behavior of these complexes as a function of the metal and solvent is summarized in the Scheme. The rather diverse behavior of the complexes with ethereal solvents can be described in terms of ligand displacement reactions. 1,2-Dimethoxyethane and diethyl ether are better ligands toward ytterbium than $NaN(SiMe_3)_2$. Though the chelating ether is a better ligand than $[N(SiMe_3)_2]^-$ towards europium, diethyl ether is not. The difference in behavior of diethyl ether towards the two lanthanides may be accounted for by noting that the size of Eu(II), in a given coordination number, is ca. 0.15 Å larger than Yb(II).[6] The $[N(SiMe_3)_2]^-$ presumably is larger than two diethyl ether molecules and the former can fit more comfortably into the coordination sphere of $Eu[N(SiMe_3)_2]_2$.

Crystallograpic Studies. ORTEP diagrams of the two independent $NaEu[N(SiMe_3)_2]_3$ molecules in the same unit cell are shown in Figures 1 and 2. Figure 3 shows an ORTEP diagram of $NaYb[N(SiMe_3)_2]_3$, with a numbering scheme identical to that of $NaEu[N(SiMe_3)_2]_3$ shown in Figure 1 to facilitate discussion. Table I gives the positional parameters and Tables II and III list some selected bond lengths and angles.

The Eu and Yb complexes crystallize in different space groups. The europium complex crystallizes in a monoclinic space group and contains two crystallographically independent molecules that are chemically identical and geometrically very similar. The $NaYb[N(SiMe_3)_2]_3$ complex is in an

$$EuI_2 + 2\,NaN(SiMe_3)_2 \xrightarrow[2.)\,C_5H_{12}]{1.)\,dme} Eu\left[N(SiMe_3)_2\right]_2(dme)_2$$

$$EuI_2 + 2\,NaN(SiMe_3)_2 \xrightarrow[2.)\,PhMe]{1.)\,Et_2O} NaEu\left[N(SiMe_3)_2\right]_3$$

$$YbI_2 + 2\,NaN(SiMe_3)_2 \xrightarrow[2.)\,PhMe]{1.)\,dme} Yb\left[N(SiMe_3)_2\right]_2(dme)_2$$

$$YbI_2 + 2\,NaN(SiMe_3)_2 \xrightarrow{Et_2O} Yb\left[N(SiMe_3)_2\right]_2(OEt_2)_2$$

$$YbI_2 + 2\,NaN(SiMe_3)_2 \xrightarrow[2.)\,PhMe]{1.)\,Et_2O} NaYb\left[N(SiMe_3)_2\right]_3$$

orthorhombic space group, and its molecular geometry is very similar to the Eu complexes. In each case the NaN(2)MN(3)N(1), (M = Eu(1), Eu(2) and Yb), atoms are coplanar. The coordination number of the lanthanide metal atom is three and the metal atom is located in the center of an isosceles triangle. The coordination number of the alkali-metal atom is two. In both cases the inter- and intra-molecular M...CH_3Si interactions (see latter) are ignored in counting the coordination numbers. The coordination number of N(1) and N(2) is three and the geometry is trigonal planar. The coordination number of N(2), N(3) and N(5), N(6) is four and the geometry is a distorted tetrahedron. These anionic complexes are further examples of the rather rare situation in which the nitrogen atom of a $N(SiMe_3)_2$ group bridges two metal atoms. The other examples are $Li_3[\mu\text{-}N(SiMe_3)_2]_3$,[7a] $[Na\mu\text{-}N(SiMe_3)_2]_x$ (x = large),[7b] $Cu_4[\mu\text{-}N(SiMe_3)_2]_4$,[7c] $Al_2Me_4(\mu\text{-}Me)[\mu\text{-}N(SiMe_3)_2]$,[7d] and $Mn[N(SiMe_3)_2]_2[\mu\text{-}N(SiMe_3)_2]_2$.[7e]

The averaged Eu-N (terminal) distance, in the two independent molecules, of 2.45 ± 0.01 Å is 0.07 Å longer than the Yb-N (terminal) distance of 2.38(2) Å. The averaged Eu-N (bridging) distance of 2.55 ± 0.02 Å is 0.09 Å longer than the Yb-N (bridging) distance of 2.46 ± 0.02 Å. The difference in the Eu-N and Yb-N distances is slightly smaller than the difference in metal radii of 0.12 Å suggested by Shannon.[6] The averaged Eu-N (terminal) distance in the three-coordinate anionic complex is 0.08 Å shorter than that found in the six coordinate europium(II) complex, $Eu[N(SiMe_3)_2]_2(dme)_2$, of 2.530(4) Å.[4] The averaged Eu-N (terminal) distance in the anionic, europium(II) complex is 0.019 Å longer than that found in the three-coordinate, europium(III) complex, $EuN(SiMe_3)_3$

[2.259(9) Å].[8] The Yb–N (terminal) distance in the anionic ytterbium(II) complex is 0.23 Å longer than that found in the three-coordinate ytterbium(III) complex, $Yb[N(SiMe_3)_2]_3$, of 2.16(2) Å.[8] These bond length changes are in accord with the changes in metal radii as a function of oxidation state and coordination number, as suggested previously.[6,9]

The terminal europium and ytterbium to nitrogen bond lengths are shorter than the bridging europium and ytterbium to nitrogen bond lengths by 0.10 Å and 0.07 Å, respectively. A similar trend was found in $Mn_2[N(SiMe_3)_2]_2[\mu\text{-}N(SiMe_3)_2]_2$; the manganese to nitrogen (terminal) bond length is 0.19 Å shorter than the bridging bond length.[7e]

The average sodium to nitrogen bond lengths in the two europium complexes of 2.47 ± 0.02 Å are equal within experimental error to that found in the ytterbium complex of 2.46 ± 0.02 Å. The Na–N (bridging) bond lengths are ca. 0.1 Å longer than that found in polymeric $NaN(SiMe_3)_2$ of 2.355(3) Å in which the coordination number of sodium is also two.[7b]

The anionic compounds are Lewis acid-base complexes, the lone pair of electrons on a nitrogen atom of two bis(trimethylsilyl)amido groups are acting as electron-pair donors towards either sodium or the lanthanide metal. The hydrocarbon solubility of the complexes is readily explained since the two electropositive atoms in each complex is effectively encapsulated by the very large $(Me_3Si)_2N$ groups and the complexes appear, from the viewpoint of the solvent, to be a saturated hydrocarbon molecule.

Close inspection of the interatomic distances (Table II) reveal some short methyl to sodium intermolecular distances and some short methyl to lanthanide intramolecular distances. The line drawings in Figures 4 and 5 illustrate the short contacts around the ytterbium and sodium ions. In the

following discussion we shall describe these interactions in some detail for the ytterbium complex since the europium complex is similar, as shown in Figure 5.

The Yb...C(2,4,8,18) contacts yield an averaged value of 3.15 ± 0.33 Å and the averaged intramolecular Na...C(11,14) contact is 3.27 ± 0.16 Å. The intermolecular Na...C(1) contact is 2.90 ± 0.03 Å. These distances are shorter than the sum of the van der Waals radius of a methyl group (2.0 Å)[10a] or a carbon atom (1.6 Å)[10b] and the metallic radius of Yb(II) of 1.7 Å[10c] or Na(I) of 1.6 Å.[10c]

The Eu...C contacts in the two independent molecules of $NaEu[N(SiMe_3)_2]_3$ are also shorter than the sum of the van der Waals radii. The averaged Eu(1)...C(2,4,8,13) and Eu(2)...C(19,24,25,31) are 3.22 ± 0.10 Å and 3.21 ± 0.21 Å, respectively. The averaged intramolecular Na(1)...C(10,14) and Na(2)...C(29,32) distances are 3.30 ± 0.02 Å and 3.24 ± 0.02 Å and the intermolecular Na(1)...C(3) and Na(2)...C(22) distances are 2.80 ± 0.01 Å and 2.70 ± 0.01 Å, respectively.

The shorter metal...carbon contacts also appear to distort the N-Si-C bond angles, though the differences in this angle, in which the carbon atom approaches the metal atoms and those in which the carbon atom is further away, are small. In the ytterbium complex the N(1)Si(1)C(2), N(1)Si(2)C(4), N(2)Si(3)C(8), N(3)Si(6)C(18), N(2)Si(4)C(11), N(3)Si(5)C(14), and N(1)Si(1)C(1) angles that are involved with the two metal atoms yield an averaged value of 112.6 ± 1.7°. The other N-Si-C angles average to 113.7 ± 3.6°. This difference is not statistically significant. Similar values are observed in the europium complex, 110.3 ± 1.3° and 110.0 ± 1.7° for those carbon aoms that closely approach the metal atoms relative to 114.3 ± 1.4° and 114.2 ± 2.1° for those that are further away.

The electropositive metal atoms in the anionic complexes clearly interact with some of the methyl groups of the ligand, causing them to become semi-bridging. This has been observed before,[5b] and attributed to an electrostatic interaction of the type Yb(δ+)...C(δ-) or an interaction of the metal with the pair of electrons in the C-H bond. A more interesting question, though one that is more difficult to answer, is why does this interaction occur? The interaction might be a general one for large, electropositive metal atoms when it is placed in a coordinatively unsaturated environment. Under these circumstances the metal atom can relieve itself of coordinative unsaturation by interacting with a Lewis base, by forming associated molecules, or when these two alternatives are not sterically feasible, the metal may undergo secondary interactions with ligands in its immediate neighborhood. In the latter case the metal...carbon interaction might be viewed as a hydrocarbon activation. On the other hand the interaction might be a manifestation of crystal packing forces since we have observed this interaction only in the solid state by X-ray crystallography.[5b] That is, the molecule packs in the crystal so that it minimizes all H-H repulsions and maximizes attractions between atoms of different electronegativity. There are no low energy absorptions in the C-H stretching frequency region of the infrared spectrum (solid state) for the anionic complex. This seems to indicate that the C-H bonds are not weakened appreciably by interaction with the metal atoms. No sign of the M...C interaction is observed in solution since the ^{1}H and $^{13}C\{^{1}H\}$NMR spectra yield a singlet at room temperature and at -80°C for $NaYb[N(SiMe_3)_2]_3$. Whatever the origin of the close contacts, the energy of the interaction is undoubtably small.

EXPERIMENTAL SECTION

All operations were preformed under nitrogen. The microanalyses were done by the microanalytical laboratory of this department. The nuclear magnetic resonace spectra were recorded on a JEOL-FX90Q instrument operating at 89.56 and 22.50 MHz for proton and carbon nuclei, respectively.

$Eu[N(SiMe_3)_2]_2[MeOCH_2CH_2OMe]_2$. Europium diiodide[11] (1.8 g, 0.0044 mol), sodium bis(trimethylsilyl)amide (1.6 g. 0.0088 mol), and 1,2-dimethyoxyethane (75 mL) were stirred together for 10h. The ether was removed under reduced pressure and the residue was extracted with and crystallized from pentane (ca. 30 mL, -10°C) in a yield of 79 percent (2.3 g). The infrared spectrum and melting point were identical to those previously reported.[4]

$Yb[N(SiMe_3)_2]_2[MeOCH_2CH_2OMe]_2$. Ytterbium diiodide[11] (3.9 g, 0.0092 mol), sodium bis(trimethylsilyl)amide (3.4 g, 0.018 mol), and 1.2-dimethoxyethane (75 mL) were stirred together for 10h. The ether was removed under reduced pressure and the residue was extracted into toluene (40 mL). The blue extract was filtered and the filtrate was concentrated to ca. 10 mL and cooled (-10°C). The blue prisms (mp 162-165°C) were collected and dried under reduced pressure. The yield was 3.9 g (63%). Anal. Calcd. for $C_{20}H_{56}N_2O_4Si_4Yb$: C, 35.6; H, 8.37; N, 4.16. Found: C, 34.5; H, 8.07; N, 4.11. The mass spectrum contained a molecular ion at 674 amu. IR (Nujol): 1248 s, 1194 w, 1127 m, 1086 s, 1062 wsh, 968 s, 864 m, 832 s, 776 m, 757 m, 662 s, 601 m, 587 w, 387 msh, and 372 m cm^{-1}.

$Yb[N(SiMe_3)_2]_2[MeOCH_2CH_2OMe]$. The blue bis-dimethoxyethane complex (1.8 g, 0.0026 mol) was dissolved in pentane (40 mL). The red solution was concentrated to ca. 10 mL. Cooling (-10°C) yielded red prisms in quantitative yield, mp 118-120°C. Anal. Calcd. for $C_{16}H_{46}N_2O_2Si_4Yb$: C, 32.9; H, 7.94; N, 4.80. Found: C, 33.1, H, 7.95, N, 4.60. IR (Nujol): 1249 s, 1238 wsh, 1112 wsh, 1092 wsh, 1054 s, 1024 wsh, 962 s, 887 wsh, 862 m, 828 s, 749 w, 662 m, 604 w, 589 w, 373 wsh, and 363 m cm^{-1}. A molecular ion was observed in the mass spectrum at 584 amu.

$Yb[N(SiMe_3)_2]_2[OEt_2]_2$. Diethyl ether (125 ml), cooled to 0°C, was added to an intimate mixture of ytterbium diiodide (4.8 g, 0.011 mol) and sodium bis(trimethylsilyl)amide (4.1 g, 0.022 mol). The blue mixture was stirred at 0°C for 1h, then warmed to room temperature. The red solution was stirred for 10h, filtered, and the filtrate was concentrated to ca. 20 mL and cooled (-70°C). The yellow prisms that were isolated (-70°C) became orange upon warming to room temperature. The orange prisms (mp 69-71°C) were isolated in 56 percent (4.0 g) yield. The compound gives a red solution in either pentane or benzene. Anal. Calcd. for $C_{20}H_{56}N_2O_2Si_4Yb$: C, 37.4; H, 8.79; N, 4.36. Found: C, 33.3; H, 8.06; N, 4.18. We cannot account for the discrepancy in the calculated and found analytical results. The spectroscopy, however, leaves no doubt as to the identity of the compound. The mass spectrum afforded a molecular ion at 570 amu. IR (Nujol): 1249 s, 1187 m, 1151 m, 1122 m, 1091 w, 1040 m, 968 s, 862 msh, 830 s, 774 m, 753 wsh, 732 wsh, 663 wsh, 662 m, 609 m, 592 w, 503 w, 391 m,

and 372 m cm^{-1}. NMR (PhH-d_6, 26°C): ^1H; δ 0.41 (s, 36H), 1.06 (t, J = 7 Hz, 12 H), 3.51 (q, J = 7 Hz, 8H). $^{13}C\{^1H\}$; δ 5.57, 14.1, and 65.2 due to the Me_3Si, Me, and CH_2 resonances, respectively.

$NaYb[N(SiMe_3)_2]_3$. Ytterbium diiodide (4.4 g, 0.010 mol) and sodium bis(trimethylsilyl)amide (3.8 g, 0.021 mol) were stirred together for 1h in diethyl ether (100 mL) at 0°C. The mixture was warmed to room temperature and stirred for an additional 10h. The diethyl ether was removed under reduced pressure and the residue was extracted with toluene (60 mL). The red solution was evaporated to dryness and the red residue was dissolved in pentane (40 mL) and filtered. The filtrate was concentrated to ca. 15 mL and cooling (-10°C) yielded red needles (mp 154-157°C) in 46% (3.2 g) yield. IR (Nujol): 1241 s, 1203 w, 1046 s, 964 s, 857 s, 818 s, 752 s, 653 m, 602 w, 579 w, 392 m, and 357 m cm^{-1}, NMR (PhH-d_6, 26°C): ^1H; 0.24 s, $^{13}C\{^1H\}$; 6.38. Anal. Calcd for $C_{18}H_{54}N_3Si_6NaYb$: C, 31.9; H, 8.04; N. 6.20. Found: C, 28.9; H, 7.29; N, 5.36.

$NaEu[N(SiMe_3)_2]_3$. Europium diiodide (5.4 g, 0.013 mol) and sodium bis(trimethylsilyl)amide (4.9 g, 0.027 mol) were stirred together for 14h in diethyl ether (100 mL). The diethyl ether was removed under reduced pressure and the residue was extracted with toluene (60 mL). The yellow solution was filtered and the volume of the filtrate was reduced to ca. 15 mL. Cooling (-10°C) afforded large, yellow prisms (mp 132-134°C) in 41% (3.6 g) yield. A portion of the complex was dissolved in PhH-d_6 and hydrolyzed with D_2O. Examination of the benzene solution by ^1H NMR

spectroscopy showed the presence of $DN(SiMe_3)_2$. IR (Nujol): 1240 s, 1206 w, 1069 s, 982 s, 858 s, 811 s, 753 m, 739 wsh, 720 wsh, 652 s, 602 w, 577 m, 389 m, and 359 m cm^{-1}. Anal. Calcd for $C_{18}H_{54}N_3Si_6NaEu$: C, 32.9; H, 8.30; N, 6.40. Found: C, 30.8; H, 8.11; N, 5.45. The elemental analyses for the europium and ytterbium anionic complexes are poor. The solution spectroscopy, however, are consistent with the X-ray crystallography results. We cannot account for the discrepancy, other than to suggest that the combustion gives rise to carbides and nitrides which do not burn completely, giving low C, H, and N data.

X-RAY DIFFRACTION. The crystals, because of their sensitivity to the atmosphere, were sealed inside thin walled quartz capillaries, and mounted on a CAD4 automatic diffractometer for a set of θ-2θ scan data. Details of the data collection are given in Table IV. Data were corrected for crystal decay, absorption[12] and Lorentz and polarization effects.

The lanthanide atomic positions were located with the use of 3-dimensional Patterson maps, and subsequent least-squares and electron density maps revealed the locations of all the non-hydrogen atoms. The structures were refined to convergence using anisotropic thermal parameters for all but the carbon atoms. Atomic scattering factors of Doyle and Turner[13] were used, and anomalous scattering[14] corrections were applied. Extinction corrections were applied to the observed structure factors.[15] The final R factors[16] are as follows: for the Eu complex $R_F = 0.043$ for 6751 data, $(F^2 > \sigma(F^2))$ and $R_{wF} = 0.060$ for the Yb complex $R_F = 0.056$ for 2546 data $(F^2 > 3\sigma(F^2))$ and $R_{wF} = 0.080$.

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SUPPLEMENTARY MATERIAL AVAILABLE

Listing of thermal parameters and listings of observed structure factors (41 pages). Ordering information is given on any current masthead.

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15. $(F_o)_{corr} = (1 + kI)F_o$ where $k = 1.17 \times 10^{-7}$ and 1.82×10^{-7} for the Eu and Yb complexes respectively, and I is the uncorrected intensity.

16. $R_F = \Sigma||F_o|-|F_c||/\Sigma|F_o|$;
$R_{wF} = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2}$

Table I. Positional Parameters[a]

$EuNa[N(SiMe_3)_2]_3$

ATOM	X	Y	Z
EU(1)	.20794(2)	.11221(2)	.20794(2)
EU(2)	.20449(2)	.74192(2)	.68631(2)
NA(1)	.21575(19)	-.02694(16)	.30277(14)
NA(2)	.19922(21)	.60248(17)	.77665(16)
N(1)	.2047(3)	.2091(3)	.13364(27)
N(2)	.1552(3)	-.0120(3)	.18432(26)
N(3)	.2711(3)	.0914(3)	.32790(26)
N(4)	.2057(3)	.8499(3)	.62703(26)
N(5)	.1459(4)	.6184(3)	.65969(29)
N(6)	.2561(3)	.7197(3)	.80628(26)
SI(1)	.29192(13)	.24276(11)	.13389(10)
SI(2)	.11130(13)	.22621(12)	.08616(10)
SI(3)	.21311(14)	-.04871(13)	.14395(11)
SI(4)	.06014(12)	-.04057(12)	.17337(10)
SI(5)	.21207(14)	.13240(13)	.36491(10)
SI(6)	.36947(13)	.07670(14)	.36816(11)
SI(7)	.11423(12)	.87344(11)	.58581(10)
SI(8)	.29502(12)	.88543(11)	.63427(10)
SI(9)	.21052(18)	.58109(14)	.62482(12)
SI(10)	.05055(15)	.58951(14)	.64350(15)
SI(11)	.19282(15)	.75790(13)	.84034(11)
SI(12)	.35281(13)	.70273(13)	.85124(10)
C(1)	.3189(6)	.2273(6)	.0591(5)
C(2)	.3730(6)	.2041(6)	.2011(5)
C(3)	.2967(5)	.3401(5)	.1479(4)
C(4)	.0397(6)	.1736(6)	.1154(5)
C(5)	.0906(7)	.1988(7)	-.0006(6)
C(6)	.0802(6)	.3199(6)	.0853(5)
C(7)	.2723(6)	-.1251(6)	.1878(5)
C(8)	.2911(7)	.0173(6)	.1350(6)
C(9)	.1607(7)	-.0806(7)	.0597(6)
C(10)	.0226(7)	-.0034(6)	.2387(5)
C(11)	.0500(6)	-.1380(5)	.1775(5)
C(12)	-.0145(6)	-.0149(6)	.0952(5)
C(13)	.1258(6)	.1770(6)	.3025(5)
C(14)	.1636(6)	.0708(6)	.4084(5)
C(15)	.2596(7)	.2033(7)	.4246(6)
C(16)	.3829(7)	.0296(7)	.4472(6)
C(17)	.4329(7)	.1563(6)	.3860(5)
C(18)	.4147(7)	.0190(7)	.3205(6)
C(19)	.0427(6)	.8038(6)	.5796(5)
C(20)	.1000(6)	.9077(6)	.5016(5)

C(21)	.0773(6)	.9508(6)	.6259(5)
C(22)	.3050(6)	.9774(5)	.6678(5)
C(23)	.3215(6)	.8926(5)	.5577(5)
C(24)	.3747(6)	.8334(6)	.5918(5)
C(25)	.2890(6)	.6477(6)	.6206(5)
C(26)	.1691(9)	.5459(8)	.5410(7)
C(27)	.2717(7)	.5065(6)	.6748(6)
C(28)	.0414(7)	.4927(7)	.6463(6)
C(29)	.0080(9)	.6256(8)	.7085(8)
C(30)	-.0179(9)	.6152(8)	.5623(8)
C(31)	.1144(6)	.8093(6)	.7753(5)
C(32)	.1357(6)	.6918(6)	.8759(5)
C(33)	.2352(7)	.8226(7)	.9066(6)
C(34)	.4059(6)	.6514(6)	.8029(5)
C(35)	.3566(6)	.6475(6)	.9238(5)
C(36)	.4174(7)	.7798(7)	.8811(6)

$YbNa[N(SiMe_3)_2]_3$

ATOM	X	Y	Z
YB	.20729(3)	.13042(4)	.17408(3)
NA	.0589(4)	.1038(4)	.2457(4)
SI(1)	.32313(28)	.22001(29)	.07942(26)
SI(2)	.36914(26)	.07293(28)	.12165(25)
SI(3)	.21305(28)	.11706(28)	.33469(23)
SI(4)	.1588(4)	-.02371(29)	.2877(3)
SI(5)	.0757(3)	.1119(3)	.0752(3)
SI(6)	.04913(26)	.2447(3)	.1557(4)
N(1)	.3154(6)	.1402(7)	.1175(6)
N(2)	.1762(7)	.0640(7)	.2750(7)
N(3)	.0889(6)	.1652(8)	.1405(8)
C(1)	.4053(14)	.2704(14)	.1013(14)
C(2)	.2522(12)	.2844(12)	.1009(10)
C(3)	.3248(16)	.2128(15)	-.0099(13)
C(4)	.3283(22)	-.0048(21)	.1397(20)
C(5)	.4282(17)	.0851(17)	.1850(15)
C(6)	.4127(22)	.0535(23)	.1484(20)
C(7)	.1488(11)	.1552(12)	.3956(11)
C(8)	.2528(10)	.1985(10)	.2947(9)
C(9)	.2818(13)	.0762(15)	.3880(13)
C(10)	.0901(18)	-.0594(19)	.2260(17)
C(11)	.1100(14)	-.0437(15)	.3641(13)
C(12)	.2289(23)	-.0791(23)	.2974(20)
C(13)	.1475(11)	.0445(11)	.0670(9)
C(14)	-.0027(16)	.0621(16)	.0798(14)
C(15)	.0732(18)	.1595(18)	-.0048(17)
C(16)	-.0474(12)	.2323(12)	.1674(10)
C(17)	.0534(15)	.3127(15)	.1868(14)
C(18)	.0836(14)	.2864(14)	.2330(13)

a. Here and in the following tables the number in parenthesis is the estimated standard deviation in the least significant digit.

Table II. Selected Interatomic Distances

Yb	-N(1)	2.38(2)	Eu(1)-N(1)	2.46(1)	Eu(2)-N(4)	2.45(1)
	-N(2)	2.44(2)	-N(2)	2.55(1)	-N(5)	2.58(1)
	-N(3)	2.47(2)	-N(3)	2.54(1)	-N(6)	2.53(1)
Na	-N(2)	2.46(2)	Na(1)-N(2)	2.49(1)	Na(2)-N(5)	2.45(1)
	-N(3)	2.46(2)	-N(3)	2.46(1)	-N(6)	2.46(1)
N(1)-Si(1)		1.63(2)	N(1) -Si(1)	1.66(1)	N(4) -Si(7)	1.68(1)
	-Si(2)	1.67(2)	-Si(2)	1.68(1)	-Si(8)	1.67(1)
N(2)-Si(3)		1.70(2)	N(2) -Si(3)	1.69(1)	N(5) -Si(9)	1.71(1)
	-Si(4)	1.68(2)	-Si(4)	1.71(1)	-Si(10)	1.70(1)
N(3)-Si(5)		1.69(2)	N(3) -Si(5)	1.69(1)	N(6) -Si(11)	1.68(1)
	-Si(6)	1.66(2)	-Si(6)	1.71(1)	-Si(12)	1.71(1)
Yb	-C(8)	2.86(2)	Eu(1)-C(8)	3.07(2)	Eu(2)-C(25)	2.97(1)
	-C(18)	2.91(2)	-C(13)	3.12(1)	-C(31)	3.14(1)
	-C(4)	3.32(3)	-C(4)	3.25(1)	-C(19)	3.29(1)
	-C(2)	3.50(4)	-C(2)	3.44(1)	-C(24)	3.44(1)
Na	-C(1)[a]	2.90(3)	Na(1)-C(3)[b]	2.80(1)	Na(2)-C(22)[b]	2.70(1)
	-C(11)	3.11(4)	-C(10)	3.28(1)	-C(32)	3.22(1)
	-C(14)	3.43(3)	-C(14)	3.31(1)	-C(29)	3.26(2)

a. Atom in position 1/2 + x, y, 1/2 - z.
b. Atom in position 1/2 - x, 1/2 + y, 1/2 - z.

Table III. Selected Angles (deg).

N(1) -Yb -N(4)	130.4(4)	N(1) -Eu(1)-N(2)	129.8(2)	N(4) -Eu(2)-N(6)	129.2(2)
N(1) -Yb -N(3)	131.5(6)	N(1) -Eu(1)-N(3)	135.5(5)	N(4) -Eu(2)-N(5)	136.7(2)
N(2) -Yb -N(3)	97.4(5)	N(2) -Eu(1)-N(3)	94.5(2)	N(5) -Eu(2)-N(6)	94.0(2)
N(2) -Na -N(3)	97.2(5)	N(2) -Na(1)-N(3)	98.0(2)	N(5) -Na(2)-N(6)	98.9(2)
Yb -N(2)-Na	83.0(5)	Eu(1)-N(2) -Na(1)	83.4(2)	Eu(2) -N(5) -Na(2)	83.2(2)
Yb -N(3)-Na	82.2(5)	Eu(1)-N(3) -Na(1)	84.1(2)	Eu(2) -N(6) -Na(2)	84.0(2)
Yb -N(1)-Si(1)	118.5(7)	Eu(1)-N(1) -Si(1)	117.2(3)	Eu(2) -N(4) -Si(7)	113.4(3)
Yb -N(1)-Si(2)	111.3(6)	Eu(1)-N(1) -Si(2)	111.9(3)	Eu(2) -N(4) -Si(8)	117.1(3)
Yb -N(2)-Si(3)	100.7(6)	Eu(1)-N(2) -Si(3)	104.7(3)	Eu(2) -N(5) -Si(9)	102.2(3)
Yb -N(2)-Si(4)	131.5(7)	Eu(1)-N(2) -Si(4)	127.4(3)	Eu(2) -N(5) -Si(10)	130.4(4)
Yb -N(3)-Si(5)	126.8(8)	Eu(1)-N(3) -Si(6)	127.6(3)	Eu(2) -N(6) -Si(12)	127.0(3)
Yb -N(3)-Si(6)	101.6(7)	Eu(1)-N(3) -Si(5)	106.2(3)	Eu(2) -N(6) -Si(11)	107.9(3)
Si(1)-N(1)-Si(2)	130.2(8)	Si(1)-N(1) -Si(2)	130.7(4)	Si(7) -N(4) -Si(8)	129.6(4)
Si(3)-N(2)-Si(4)	122.5(9)	Si(3)-N(2) -Si(4)	121.0(3)	Si(9) -N(5) -Si(10)	121.4(4)
Si(5)-N(3)-Si(6)	126.4(9)	Si(5)-N(3) -Si(6)	120.3(3)	Si(11)-N(6) -Si(12)	120.3(4)

Table IV. Data Collection Details for $NaEu(N(SiMe_3)_2)_3$ and $NaYb(N(SiMe_3)_2)_3$.

	Crystal Parameters[a]	
	Eu	Yb
Space Group	$P2_1/n$	Pbca
a	17.586(6) Å	19.334(6) Å
b	19.170(6) Å	18.534(6) Å
c	21.808(6) Å	20.020(6) Å
β	107.90(4)°	---
V	6996 $Å^3$	7174 $Å^3$
Z	8	8
Mol wt	656.2	677.3
Density (calcd)	1.246 g/cm^3	1.254 g/cm^3
μ (calcd, MoKα)	20.2 cm^{-1}	28.2 cm^{-1}

Table IV. Data Collection .... (Continued).

	Intensity Data Measurement
Radiation:	MoKα (λ = 0.71073 Å)
Monochromator:	highly oriented graphite, $2\theta_m = 12.2^\circ$
scan type:	θ(crystal)-2θ(counter)
reflections measured	$3^\circ < 2\theta < 45^\circ$
	+h +K ±l for Eu, +h +k +l for Yb
scan speed:	variable from 0.63 to 6.7 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
scans collected:	for Eu 7643 (inc standards) yield 7397 unique reflections. For Yb 3199 (inc standards) yield 2904 unique reflections.
std reflections:	3 measured after every 250 scans. A 5% decay in intensity was observed for both complexes.

a. Unit cell parameters were derived by a least-squares fit to the setting angles of the unresolved MoKα components of 24 reflections.

FIGURE CAPTIONS

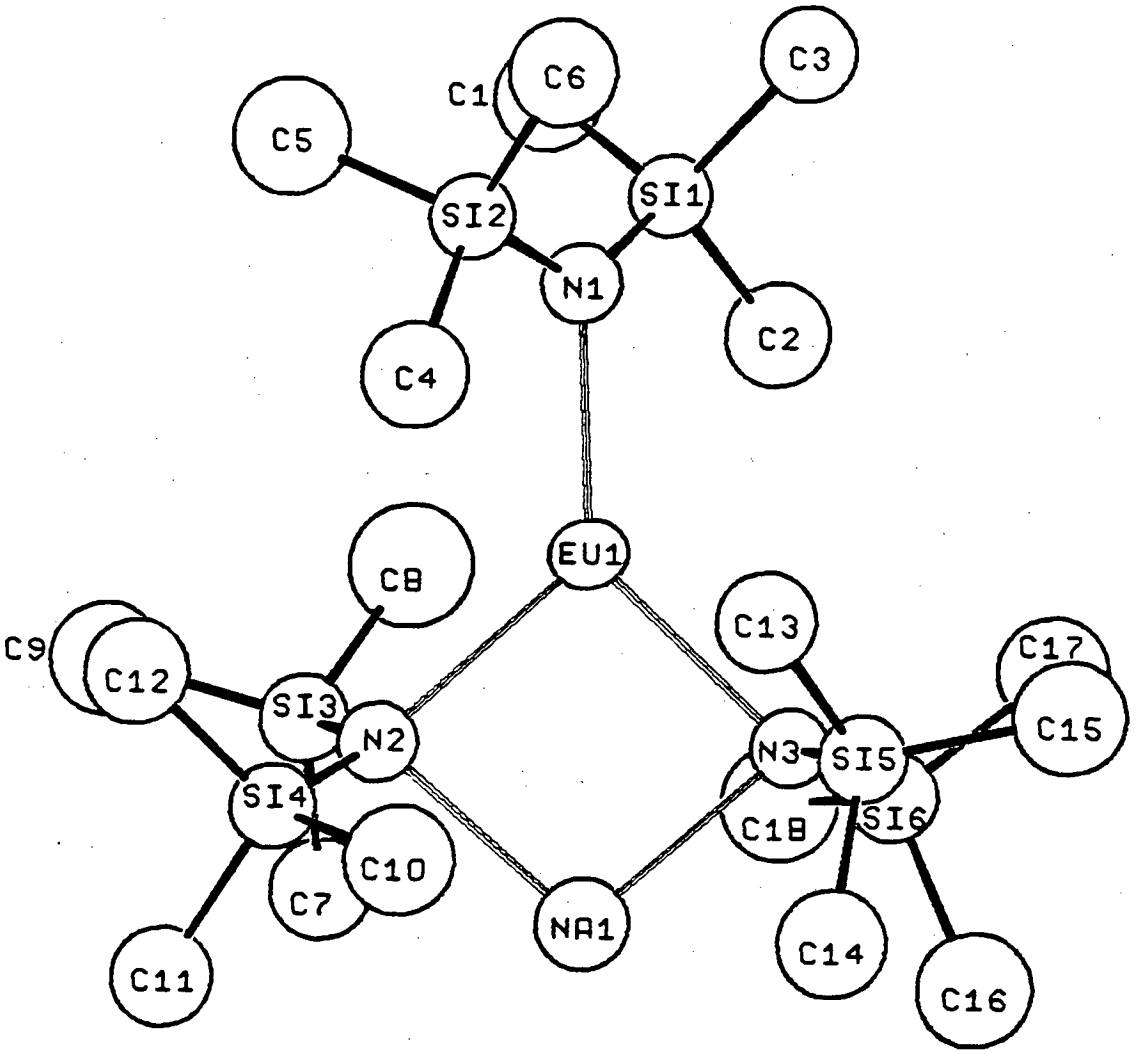
Fig. 1. ORTEP drawing of $NaEu[(Me_3Si)_2N]_3$, molecule 1.

Fig. 2. ORTEP drawing of $NaEu[(Me_3Si)_2N]_3$, molecule 2.

Fig. 3. ORTEP drawing of $NaYb[(Me_3Si)_2N]_3$.

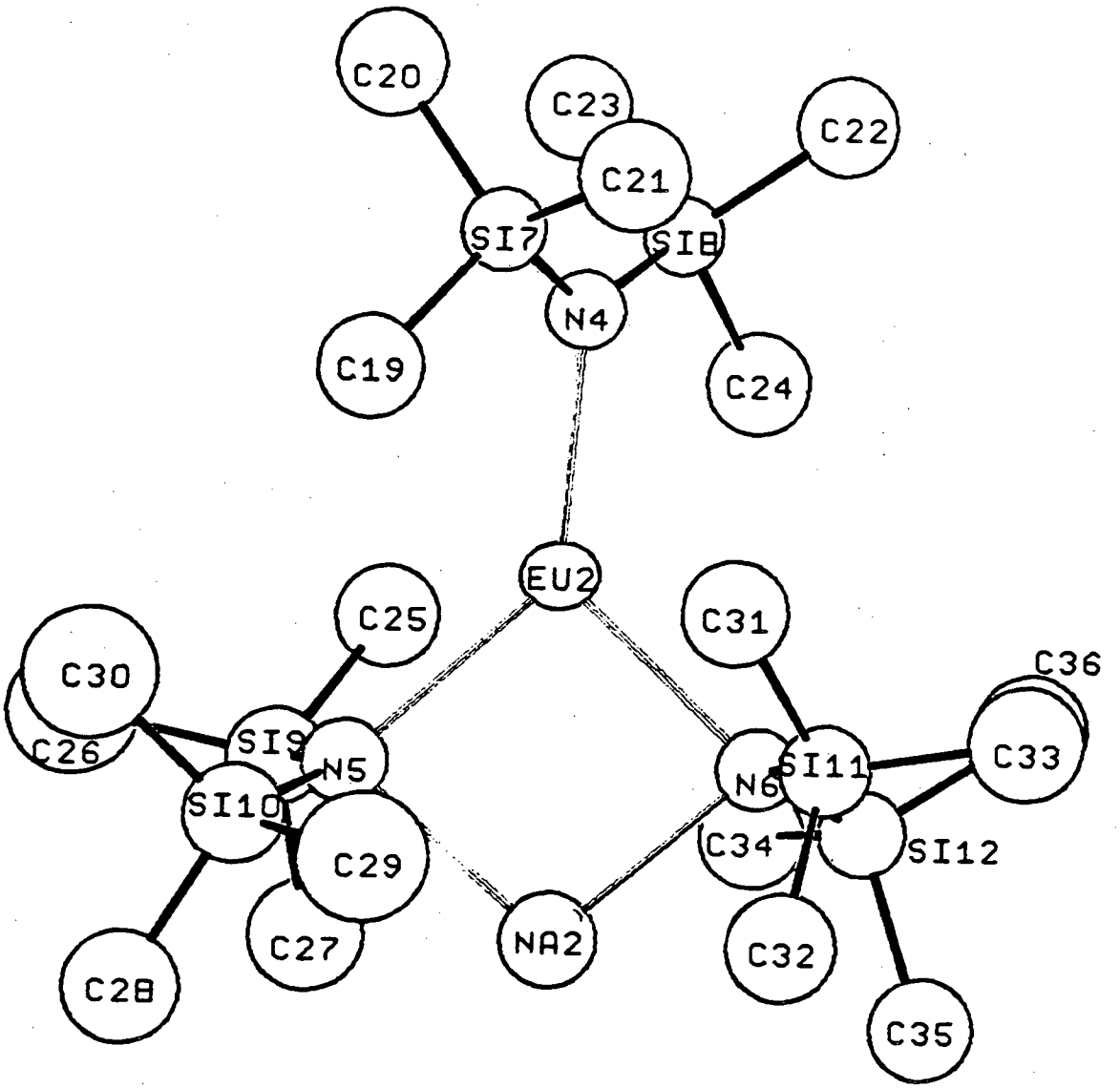
Fig. 4. Line drawing of $NaEu[N(SiMe_3)_2]_3$ showing $M...CH_3$ interactions.

Fig. 5. Line drawing of $NaYb[N(SiMe_3)_2]_3$ showing $M...CH_3$ interactions.



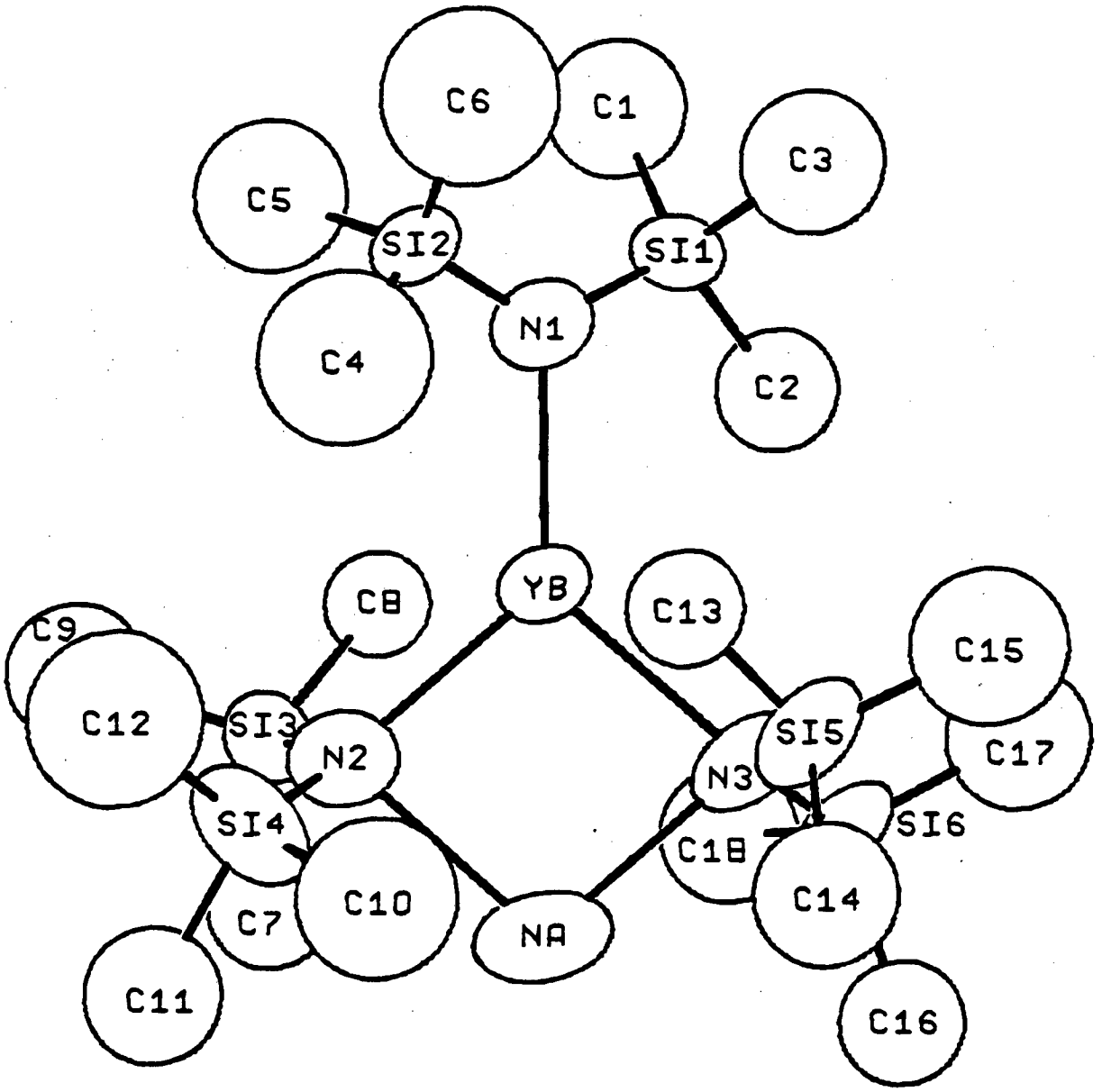
XBL 8110-7069

Fig. 1



XBL 818-11433

Fig. 2



XBL 823-8228

Fig. 3

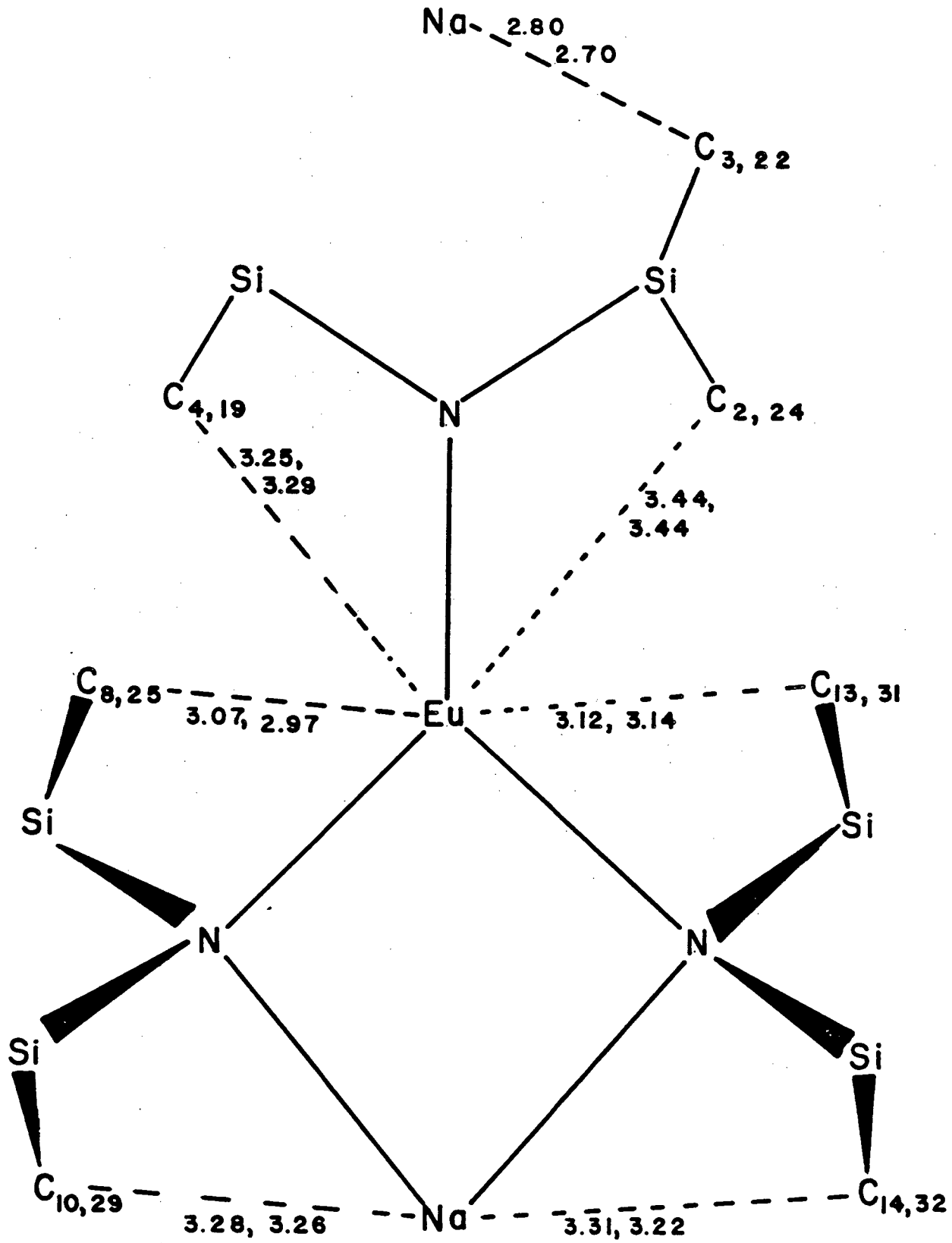

Na
2.80
2.70
C3, 22
Si
Si
C4, 19
N
C2, 24
3.25,
3.29
3.44,
3.44
C8, 25
3.07, 2.97
Eu
3.12, 3.14
C13, 31
Si
Si
N
N
Si
Si
C10, 29
3.28, 3.26
Na
3.31, 3.22
C14, 32

Fig. 4

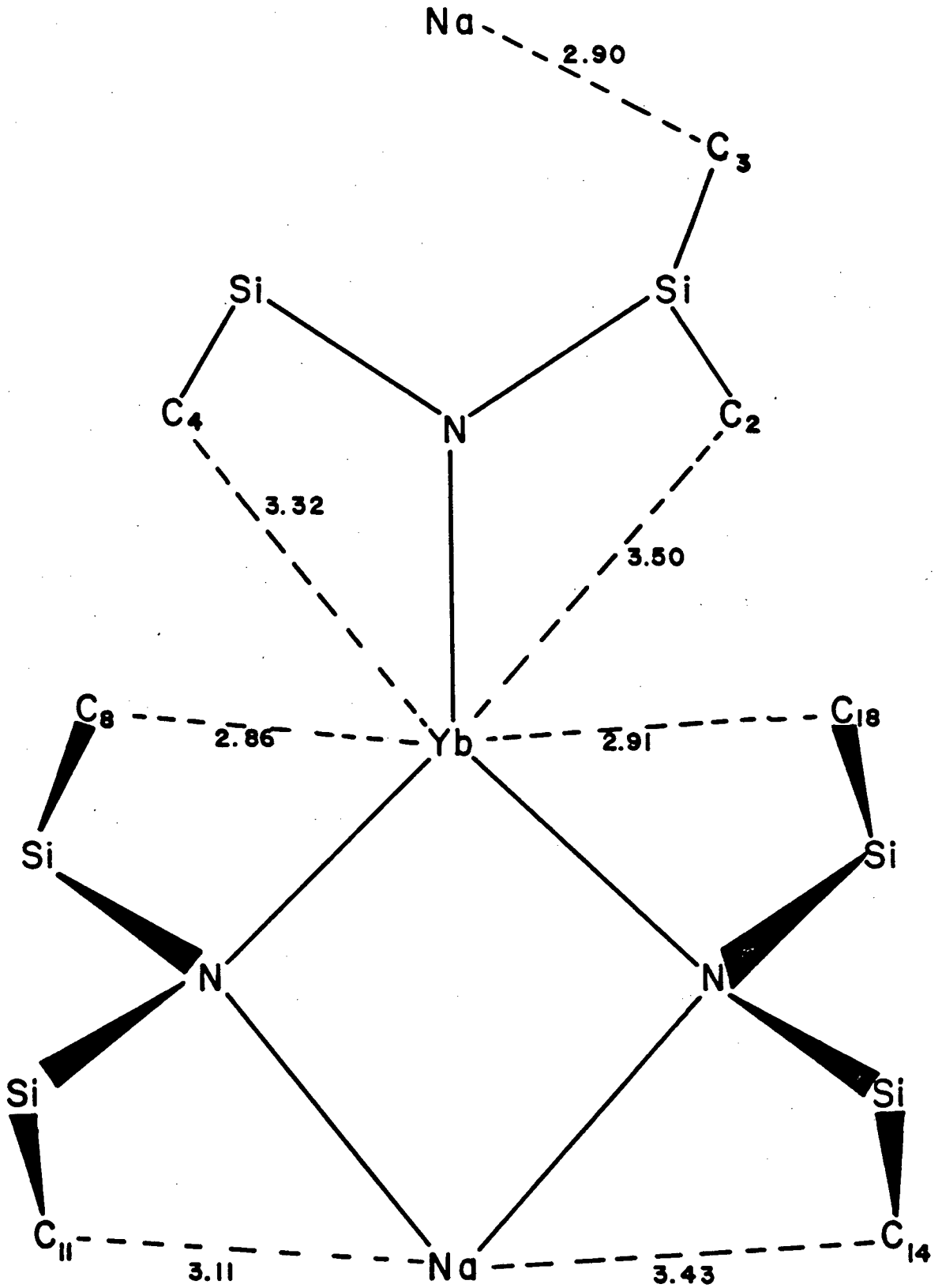

Na
2.90
C3
Si
Si
C4
N
C2
3.32
3.50
C8
2.86
Yb
2.91
C18
Si
Si
N
N
Si
Si
C11
3.11
Na
3.43
C14

Fig. 5

SUPPLEMENTARY MATERIAL FOR:

DIVALENT LANTHANIDE CHEMISTRY; PREPARATION AND CRYSTAL STRUCTURES OF SODIUM TRIS[BIS(TRIMETHYLSILYL)AMIDO]-EUROPATE(II) AND -YTTERBATE(II), $NaM[N(SiMe_3)_2]_3$

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MARCH 1983

NOTE: Table of observed structure factors available upon request from the authors.

THERMAL PARAMETERS FOR $YbNa(((CH_3)_3Si)_2N)_3$.[a]

ATOM	B11	B22	B33	B12	B13	B23
YB	3.89(3)	7.02(4)	6.09(4)	.15(3)	.376(28)	.56(4)
NA	6.4(4)	8.8(4)	14.0(6)	-.0(3)	2.9(4)	.2(4)
SI(1)	4.77(25)	5.68(28)	5.54(27)	.02(22)	1.44(21)	.65(23)
SI(2)	3.86(22)	6.1(3)	5.11(27)	1.02(22)	.34(19)	-.55(21)
SI(3)	6.36(29)	5.80(28)	4.71(24)	.44(24)	.47(22)	.35(22)
SI(4)	13.5(6)	3.56(27)	7.9(4)	-.1(3)	2.9(4)	.31(27)
SI(5)	4.89(28)	7.8(4)	9.3(4)	.02(26)	-2.69(27)	-1.9(3)
SI(6)	3.81(23)	4.92(26)	11.5(5)	1.21(21)	-.90(27)	.24(28)
N(1)	5.2(6)	7.5(8)	6.3(7)	.0(5)	.1(5)	-.7(6)
N(2)	6.9(7)	6.0(7)	7.7(8)	.3(6)	1.2(6)	-.9(6)
N(3)	4.3(6)	7.8(8)	11.4(10)	.8(5)	-.6(7)	.1(8)
C(1)	13.2(8)					
C(2)	10.1(6)					
C(3)	14.1(8)					
C(4)	20.9(14)					
C(5)	15.8(10)					
C(6)	22.0(15)					
C(7)	10.4(6)					
C(8)	7.6(4)					
C(9)	13.2(8)					
C(10)	17.8(11)					
C(11)	12.7(7)					
C(12)	21.0(14)					
C(13)	9.3(5)					
C(14)	14.4(8)					
C(15)	16.9(10)					
C(16)	10.4(6)					
C(17)	14.3(8)					
C(18)	12.8(7)					

a. The anisotropic temperature factor has the form $\exp[-0.25(B_{11}h^2a^{*2} + 2B_{12}hka^*b^* + \ldots)]$. The isotropic thermal parameter has the form $\exp(-B\sin^2\theta/\lambda^2)$.

THERMAL PARAMETERS FOR $EuNa(((CH_3)_3Si)_2N)_3$[a]

ATOM	B11	B22	B33	B12	B13	B23
EU(1)	5.499(22)	3.844(21)	3.693(19)	-.546(15)	.985(14)	.543(13)
EU(2)	5.488(22)	3.662(21)	3.759(19)	-.606(15)	.769(14)	.648(13)
NA(1)	7.25(17)	4.20(15)	4.96(15)	-.80(13)	.75(13)	.93(11)
NA(2)	7.58(19)	4.84(16)	5.53(16)	-.63(13)	1.03(14)	1.76(13)
N(1)	4.51(28)	3.80(29)	4.21(27)	.31(22)	1.22(22)	.23(22)
N(2)	4.51(28)	3.81(29)	3.91(26)	-.24(22)	.99(22)	-.26(21)
N(3)	4.28(27)	4.2(3)	3.63(26)	-.51(22)	.84(21)	.22(21)
N(4)	4.63(28)	3.57(29)	4.18(27)	-.14(22)	1.66(22)	.30(21)
N(5)	5.5(3)	3.9(3)	4.8(3)	-1.07(25)	.74(24)	.22(24)
N(6)	4.54(28)	4.5(3)	3.62(26)	.53(23)	.78(21)	.51(22)
SI(1)	5.29(10)	3.60(10)	4.15(9)	-.46(8)	1.40(8)	-.09(7)
SI(2)	5.15(11)	4.07(10)	4.67(10)	.19(8)	1.23(8)	.54(8)
SI(3)	5.49(11)	5.25(12)	4.62(10)	.16(9)	1.70(9)	-.57(9)
SI(4)	4.25(10)	4.59(11)	5.06(11)	-.54(8)	.62(8)	.08(8)
SI(5)	5.64(11)	5.26(12)	4.16(10)	.04(9)	1.49(8)	-.30(8)
SI(6)	4.59(11)	6.06(13)	4.91(11)	.39(9)	.34(8)	.72(9)
SI(7)	4.56(10)	3.98(10)	4.96(10)	.34(8)	1.28(8)	1.10(8)
SI(8)	4.52(9)	3.51(10)	4.37(9)	-.20(8)	1.68(7)	-.15(7)
SI(9)	8.86(16)	5.38(13)	5.13(12)	-1.32(12)	2.82(11)	-.91(10)
SI(10)	5.54(13)	5.28(13)	8.21(16)	-1.46(10)	-.72(11)	1.13(11)
SI(11)	6.50(13)	6.00(13)	4.70(11)	1.69(10)	2.42(9)	1.02(9)
SI(12)	4.47(10)	6.03(13)	4.10(10)	.31(9)	.50(8)	.19(9)

Atom	B	Atom	B
C(1)	7.42(23)	C(19)	7.21(22)
C(2)	7.29(22)	C(20)	8.12(25)
C(3)	5.99(19)	C(21)	8.20(26)
C(4)	7.62(24)	C(22)	6.75(21)
C(5)	9.5(3)	C(23)	7.26(23)
C(6)	7.90(24)	C(24)	7.04(22)
C(7)	7.03(22)	C(25)	7.15(22)
C(8)	22.7(3)	C(26)	11.1(4)
C(9)	8.68(27)	C(27)	8.54(27)
C(10)	8.26(25)	C(28)	8.96(28)
C(11)	6.91(22)	C(29)	11.2(4)
C(12)	7.58(23)	C(30)	11.6(4)
C(13)	7.11(22)	C(31)	7.80(24)
C(14)	7.92(25)	C(32)	8.23(26)
C(15)	8.76(27)	C(33)	8.93(28)
C(16)	9.3(3)	C(34)	7.70(24)
C(17)	8.26(26)	C(35)	7.10(22)
C(18)	9.4(3)	C(36)	8.79(28)

a. See reference in previous table.

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