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PREPARATION OF $Yb[N(SiMe_3)_2]_2[AlMe_3]_2$; A COMPLEX WITH FOUR Yb–Me–Al INTERACTIONS*

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*This paper is dedicated to the memory of the late Professor E.L. Muetterties with thanks for his wisdom and guidance.

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Summary: Trimethylaluminum reacts with base-free ytterbium bis (trimethylsilylamide) to give $Yb[N(SiMe_3)_2]_2[AlMe_3]_2$. The x-ray crystal structure provides evidence for four Yb-Me-Al and two Yb-Me-Si bridging interactions.

Bridging alkyl compounds formed by interaction of aluminum alkyls with early d- and f- element organometallic compounds are of interest relative to structure and bonding[1] as well as to the mechanism of Ziegler-Natta polymerization processes.[2] Several compounds have been isolated in which two methyl groups of an aluminum alkyl forms bridges between the main group and f-block metal,[3] as in $Me_4Al_2(\mu\text{-}Me)_2$,[4] viz., the methyl group contributes one sigma-type molecular orbital and a single electron to the bridge bonding.

Base-free $Yb_2[N(SiMe_3)_2]_4$[5] is a dimer with two terminal and two bridging silylamide groups and both ytterbium atoms are three coordinate. The overall geometry is similar to that found for $Mn_2[N(SiMe_3)_2]_4$[6a,b] and $Co_2[N(SiMe_3)_2]_4$.[6b] The Yb-N-Yb bridge in $Yb_2[N(SiMe_3)_2]_4$ can be cleaved by Lewis bases, such as phosphines, to give $Yb[N(SiMe_3)_2]_2(Me_2PCH_2CH_2PMe_2)$,[7a] and by molecules that have Lewis acidic and basic sites present in the same molecule, such as $NaN(SiMe_3)_2$ to give $NaYb[N(SiMe_3)_2]_3$.[7b] The latter complex contains two silylamide groups that bridge the metal atoms with the one-pairs of electrons on the nitrogen atoms acting as donors towards the alkali and lanthanide metal atoms. In this communication we describe a complex formed between $Yb_2[N(SiMe_3)_2]_4$ and the Lewis acid Me_6Al_2 that contains two bridging and two semi-bridging Yb-Me-Al interactions.

Two molar eqivalents of Me_6Al_2 reacts with $Yb_2[N(SiMe_3)_2]_4$ in pentane to give a bright yellow solution from which yellow plates of diamagnetic $Yb[N(SiMe_3)_2]_2(Me_3Al)_2$ (I) were obtained by crystallization from pentane at -20°C in essentially quantitative yield.[8] Triethylaluminum behaves similarly giving giving the yellow, low melting $Yb[N(SiMe_3)_2]_2(Et_3Al)_2$.[9] Figure I shows an ORTEP of I. The complex can be thought of as being derived from a

monomeric $Yb[N(SiMe_3)_2]_2$ fragment in which each lone-pair of electrons on the nitrogen atoms is coordinated to aluminum atoms so that the coordination number of the nitrogen and aluminum atoms is four. The averaged Al-N distance of 1.963(5) Å and the averaged Yb-N-Al angle of 80.5(7)° are similar to those found in $Me_4Al_2(\mu\text{-}NPh_2)$[10a] of 2.003(3) Å and 85.6(1)°, $Me_4Al_2(\mu\text{-}NMe_2)_2$[10c] of 1.96(1) Å and 91.6(2)°, respectively. The Yb-N(1) and Yb-N(2) bond lengths of 2.510(2) and 2.573(2) Å are longer than the equivalent bond length of 2.46(2) Å in $NaYb[N(SiMe_3)_2]_3$.[7b] The N(1)-Yb-N(2) angle of 131.56(5)° is similar to that found in $Yb[N(SiMe_3)_2]_2(Me_2PCH_2CH_2PMe_2)$[7a] of 123.6(6)°.

The methyl groups C(1,2) and C(5,6) bridge the aluminum and ytterbium atoms with Al(1)-C(1,2) and Al(2)-C(5,6) distances of 2.005(2), 2.030(2), 2.012(2), and 2.027(2) Å, respectively, with an average of 2.019(11) Å. The averaged bridge distance is significantly shorter than that found for the Al-C bridging distance in $Me_4Al_2(\mu\text{-}Me)_2$ and $Me_4Al_2(\mu\text{-}Me)(\mu\text{-}NPh_2)$ of 2.125(2) Å[4] and 2.142(2) Å[10a], respectively. The averaged Al-C(2,6)-Yb and Al-C(1,5)-Yb angles of 73.8(1)° and 65.9(7)°, respectively, are similar to the equivalent angles in $Cp_2Yb(\mu\text{-}Me)_2AlMe_2$ and $Cp_2Y(\mu\text{-}Me)_2AlMe_2$ of 78.9(6)°[3a] and 80.8(4)°,[3b] respectively. The averaged N(1)-Al(1)-C(1,2) and N(2)-Al(2)-C(5,6) angle is 106.1(1.6)° and the averaged N(1)-Al(1)-C(3) and N(2)-Al(2)-C(4) angle is 116.6(4)°, similar to the equivalent angles in $Me_4Al_2(\mu\text{-}Me)(\mu\text{-}NPh_2)$ of 108.9(1.0)° and 113.1(1.1)°, [10a] respectively. The averaged terminal Al-C distance in (I) of 1.959(2) Å is identical to that found for the equivalent distance in $Me_4Al_2(\mu\text{-}Me)_2$[4] of 1.953(2) Å, and in other related compounds as shown in the Table. Inspection of the Table shows that the bridging Al-C distances in (I) are ca. 0.1 Å shorter than this distance in related compounds. This concept may be expressed quantitatively by defining Δ, the averaged terminal Al-C distance minus the averaged bridging Al-C distance, for

the series of related complexes in the Table. The Δ-values show that the bridge bonds in I are approximately midway between bridging and terminal Al-C bonds in length and presumably in strength. As a consequence of the shorter bridging Al-C lengths in I, the bridging Yb-C bonds might be proportionately longer and therefore weaker.

The Yb-μ-C bond lengths fall into three ranges, two short distances, Yb-C(2,6) of 2.788(2) and 2.756(2) Å, respectively [ave. = 2.767(6) Å] with Yb-C-Al angles of 74.03(7) and 73.65(7)°, respectively, one intermediate distance, Yb-C(5) of 3.042(2) and the Yb-C(5)-Al angle of 67.21(7)°, and one long distance, Yb-C(1) of 3.202(3)Å [ave. = 2.95(17)Å]. Further, the shortest Al-C distances are pair-wise related to the longest Yb-C distances. The two short Yb-C distances are close to those found in $Cp_2Yb(\mu\text{-}Me)_2AlMe_2$ and $Cp_4Y_2(\mu\text{-}Me)_2$ of 2.59(2) and 2.54(1) Å, respectively,[3a] since the radius of Yb(II) is ca. 0.1 Å larger than that of Yb(III) or Y(III).[11] The two longer Yb-C distances are still shorter than the sum (3.3 Å) of the van der Waals radius of a carbon atom[12a] and the metallic radius of divalent ytterbium (1.7 Å).[12b] The relative weakness of the Yb-Me-Al interaction in I in the solid state is apparently true in solution since the line shape in the ^{1}HNMR resonance experiment does not change to -80°C. Assuming a chemical shift difference for the bridging and termial methyl groups of 1Hz at -80°C, the upper limit for $\Delta G^{\ddagger}$(Tc) is ca. 10 kcal mol^{-1}. This may be compared with $\Delta G^{\ddagger}$(Tc) for bridge-terminal exchange in $Me_4Al_2(\mu\text{-}Me)_2$[13] and in $Cp_2Y(\mu\text{-}Me)_2AlMe_2$[3a] of ca. 11 and 16 kcal mol^{-1}, respectively. As a consequence of the Yb-C(2,6)-Al bridge bond, two hydrogen atoms on each carbon atom, H(2,3) on C(2) and H(4,6) on C(6), approach the ytterbium atom. The Yb•••H (2,3,4,6) distances are 2.63, 2.72, 3.13 and 2.53 Å, respectively,[8c] suggesting that the Yb-Me-Al interaction is not just by way of the carbon atom, but the hydrogen

atoms on the bridging methyl groups also are involved.

In addition to the short Yb-C-Al distances, two of the methyl groups on the silicon atoms have short Yb-C contacts. The Yb•••C (12,21) distances are 3.067(2) and 3.039(2) Å, respectively, and all other intramolecular contacts are > 3.5 Å. A similar phenomenon was noted in $Yb[N(SiMe_3)_2]_2(Me_2PCH_2CH_2Me_2)$,[7a] in which the ytterbium to carbon contact distance is 3.04 Å, and discussed in some detail in ref. 7b.

In summary, the coordination at ytterbium in (I) may be described as either four or eight coordinate. However, the geometry is so irregular that it is impossible to describe the stereochemistry in a precise fashion using the dihedral angle formalism advocated by Muetterties.[14] Even though the bridging interaction in I is weak, the chemistry of I is substantially different than trimethylaluminum or its coordination complexes. In particular, I polymerizes ethylene at 20°C and 12 atm whereas trimethylamluminum does not polymerize ethylene under such mild conditions.[15] These and other reactions will be described later.

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(8) (a) NMR: ^{1}H, δ 0.28(s,36H), -0.25(s,18H), temperature independent to -85°C. $^{13}C\{^{1}H\}$, δ 5.36 and 0.85 due to Me_3Si and Me_3Al, respectively. (b) Crystal Data (-95°C): Triclinic, $P\bar{1}$, a= 9.8707(17), b = 12.9348(17), c = 13.1081(20), α = 68.12(11), β = 83.19(15), γ = 84.39(14), V = 1539.5 $Å^3$, Z = 2, d (calc.) = 1.39 $g\,cm^{-3}$, μ = 32.456 cm^{-1}, crystal size, 0.33 X 0.36 X 0.22 mm, M_oK_α (λ = 0.71073 Å). The 4012 raw intensity data were reduced to structure factor amplitudes and their esd's by correction for background, scan speed, and Lorentz and Polarization effects.[8d] A 2.75% linear decay in three standard reflections was observed and the data were corrected. A three-dimensional Patterson synthesis revealed the position of the Yb, Al, and Si atoms and the remaining atoms were located by Fourier techniques. An empirical absorption correction using azimuthal scan data was applied.[8d] The final residuals for 245 variables refined against 3814 data for which $F^2 > 3\sigma(F^2)$ were R = 0.0169, R_w = 0.0258 and G_{of} = 1.857. The R for all 4012 data was 0.019. (c) The hydrogen atoms on C(2) and C(6) were located in the Fourier difference maps but they were not refined since we did not collect high angle data ($2\theta > 45°$). (d) Boncella, J.M.; Andersen, R.A. Inorg. Chem. **1984** 23, 432.

(9) NMR: ^{1}H (PhMe-d_8, +30°C), δ 1.38 (t, J = 7.8 Hz, 18H), 0.34 (q, J = 7.8 Hz, 12H), 0.27 (s, 36H). Lowering the temperature resulted in severe line-broadening by -40°C. Further cooling did not result in better resolution.

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Table

Compound	Al-C_b[a] (Å)	Al-C_t[b] (Å)	Δ[c] (Å)	Me M Al (deg.)	Al X Me(b) (deg.)	Reference
$Me_4Al_2(\mu\text{-}Me)_2$	2.125(2)	1.953(2)	0.17	75.7(1)	104.3(1)	4
$Me_4Al_2(\mu\text{-}Me)(\mu\text{-}NPh_2)$	2.142(2)	1.948(5)	0.19	78.9(2)	94.7(2)	10a
$Me_4Al_2(\mu\text{-}Me)_4Mg$	2.13(2)	1.96(1)	0.17	77.7(3)	105.7(1)	10b
$Me_2Al_2(\mu\text{-}Me)_2YCp_2$	2.10(1)	1.94(1)	0.16	80.8(4)	112(1)	3b
$Me_2Al_2(\mu\text{-}Me)_2YbCp_2$	2.13(2)	2.00(1)	0.13	78.9(6)	113.3(8)	3a
(I)	2.009(2)	1.959(2)	0.05	73.8(1)	106.1(1.6)	This
	2.029(1)		0.07	65.9(7)		work

[a] b = bridging carbon atom
[b] t = terminal carbon atom
[c] Δ is $[Al\text{-}C_b] - [Al\text{-}C_t]$

Figure

An ORTEP drawing of (I), $Yb[N(SiMe_3)_2]_2(AlMe_3)_2$, showing the divalent ytterbium atom in eight coordination.

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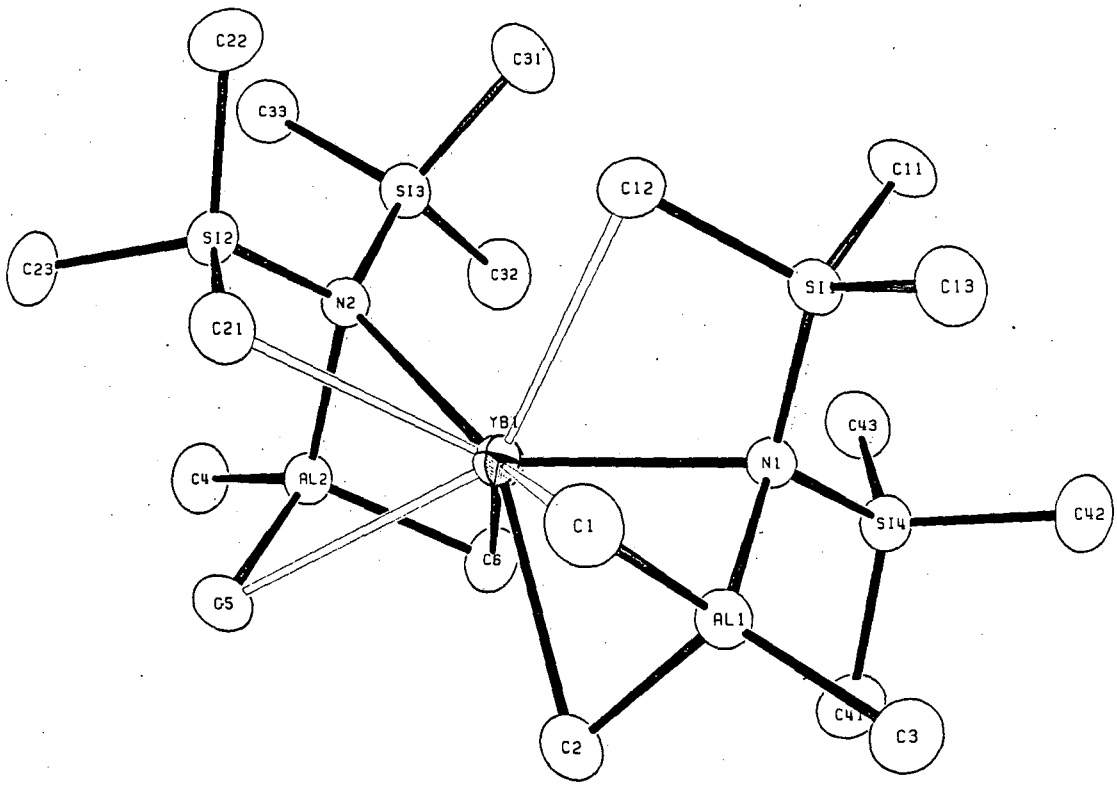
Supplementary Material. Bond length and angles and positional and thermal parameters (4 pages). Ordering information is given in any current masthead page.


C22
C31
C33
SI3
C12
C11
SI2
C23
C32
SI1
C13
N2
C21
YB1
C43
N1
C4
AL2
C1
SI4
C42
C6
G5
AL1
C41
C3
C2

XBL 847-3160

Supplementary Material for Preparation of $Yb[N(SiMe_3)_2]_2[AlMe_3]_2$; A Complex With Four Yb-Me-Al Interactions

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Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	$B(A^2)$
YB1	Ø.19426(1)	Ø.31799(1)	Ø.21184(1)	2.Ø22(3)
SI1	Ø.48996(8)	Ø.31Ø51(6)	Ø.32464(6)	2.Ø7(2)
SI2	Ø.24218(8)	Ø.2228Ø(6)	Ø.ØØØ1Ø(6)	2.13(2)
SI3	Ø.25322(8)	Ø.Ø1849(6)	Ø.2Ø474(6)	2.21(2)
SI4	Ø.26263(8)	Ø.27Ø76(6)	Ø.5Ø3Ø4(6)	2.13(2)
AL1	Ø.24712(9)	Ø.49111(6)	Ø.29736(7)	2.2Ø(2)
AL2	-Ø.ØØ36Ø(8)	Ø.18Ø55(6)	Ø.18Ø44(6)	2.Ø2(2)
N1	Ø.3164(2)	Ø.3342(2)	Ø.3621(2)	1.72(5)
N2	Ø.191Ø(2)	Ø.1577(2)	Ø.14Ø9(2)	1.82(5)
C1	Ø.3268(4)	Ø.5539(3)	Ø.14Ø1(3)	3.59(8)
C2	Ø.Ø489(3)	Ø.4839(2)	Ø.2746(3)	3.12(7)
C3	Ø.2727(4)	Ø.5861(3)	Ø.3794(3)	3.5Ø(7)
C4	-Ø.1276(3)	Ø.Ø8ØØ(3)	Ø.1631(3)	3.48(8)
C5	-Ø.Ø596(3)	Ø.34Ø1(3)	Ø.Ø927(2)	2.94(7)
C6	-Ø.Ø1Ø8(3)	Ø.1822(2)	Ø.335Ø(2)	2.73(7)
C11	Ø.5683(3)	Ø.1735(3)	Ø.4121(3)	3.11(7)
C12	Ø.5Ø76(3)	Ø.3Ø53(2)	Ø.1823(2)	2.82(7)
C13	Ø.5996(3)	Ø.4192(3)	Ø.3258(3)	3.2Ø(7)
C21	Ø.2557(3)	Ø.3768(2)	-Ø.Ø354(2)	2.89(7)
C22	Ø.4171(4)	Ø.1767(3)	-Ø.Ø441(3)	3.55(8)
C23	Ø.122Ø(4)	Ø.2Ø48(3)	-Ø.Ø9Ø8(2)	3.21(7)
C31	Ø.4372(3)	Ø.ØØ7Ø(3)	Ø.2299(3)	3.47(8)
C32	Ø.1637(4)	-Ø.Ø525(3)	Ø.3435(3)	3.5Ø(8)
C33	Ø.2285(3)	-Ø.Ø676(2)	Ø.1228(3)	3.21(7)
C41	Ø.Ø86Ø(3)	Ø.32Ø1(3)	Ø.5381(2)	3.41(8)
C42	Ø.3744(4)	Ø.3Ø17(3)	Ø.5926(2)	3.34(7)
C43	Ø.26Ø2(3)	Ø.1162(3)	Ø.5461(3)	3.13(7)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

$$(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos \text{gamma})*B(1,2)$$

$$+ ac(\cos \text{beta})*B(1,3) + bc(\cos \text{alpha})*B(2,3)]$$

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	B(Å^2)
H1	Ø.ØØØØ	Ø.543Ø	Ø.293Ø	4.Ø**
H2	Ø.ØØØØ	Ø.416Ø	Ø.3125	4.Ø**
H3	Ø.Ø293	Ø.5ØØØ	Ø.2Ø9Ø	4.Ø**
H4	-Ø.Ø82Ø	Ø.25ØØ	Ø.3555	3.7**
H5	-Ø.Ø547	Ø.125Ø	Ø.375Ø	3.7**
H6	Ø.Ø547	Ø.1875	Ø.375Ø	3.7**
H11	Ø.2947	Ø.6294	Ø.1Ø75	4.6**
H12	Ø.4239	Ø.5497	Ø.1383	4.6**
H13	Ø.3ØØ4	Ø.5118	Ø.1ØØ7	4.6**
H31	Ø.2363	Ø.6595	Ø.3417	4.5**
H32	Ø.2272	Ø.5568	Ø.4512	4.5**
H33	Ø.3677	Ø.588Ø	Ø.3847	4.5**
H41	-Ø.2188	Ø.Ø996	Ø.1848	4.5**
H42	-Ø.12Ø8	Ø.Ø869	Ø.Ø88Ø	4.5**
H43	-Ø.1Ø34	Ø.ØØ54	Ø.2Ø79	4.5**
H51	-Ø.1542	Ø.3538	Ø.11Ø2	3.9**
H52	-Ø.ØØ81	Ø.3884	Ø.11Ø5	3.9**
H53	-Ø.Ø431	Ø.3532	Ø.Ø162	3.9**
H111	Ø.6615	Ø.167Ø	Ø.3868	4.1**
H112	Ø.5613	Ø.169Ø	Ø.4867	4.1**
H113	Ø.5211	Ø.1148	Ø.4Ø79	4.1**
H121	Ø.6Ø17	Ø.2924	Ø.1613	3.8**
H122	Ø.4575	Ø.2465	Ø.1822	3.8**
H123	Ø.4741	Ø.3742	Ø.1316	3.8**
H131	Ø.6919	Ø.4Ø17	Ø.3Ø48	4.2**
H132	Ø.57Ø2	Ø.4898	Ø.2752	4.2**
H133	Ø.5928	Ø.4212	Ø.3979	4.2**
H211	Ø.2835	Ø.411Ø	-Ø.1117	3.9**
H212	Ø.1695	Ø.4Ø91	-Ø.ØØ189	3.9**
H213	Ø.3214	Ø.3875	Ø.ØØ67	3.9**
H221	Ø.4369	Ø.2159	-Ø.1211	4.6**
H222	Ø.4823	Ø.1928	-Ø.ØØ49	4.6**
H223	Ø.4213	Ø.Ø991	-Ø.Ø291	4.6**
H231	Ø.1542	Ø.2414	-Ø.1657	4.2**
H232	Ø.1164	Ø.1275	-Ø.Ø758	4.2**
H233	Ø.Ø339	Ø.2362	-Ø.Ø766	4.2**
H311	Ø.4657	-Ø.Ø694	Ø.2644	4.4**
H312	Ø.49Ø4	Ø.Ø384	Ø.1616	4.4**
H313	Ø.4489	Ø.Ø46Ø	Ø.2766	4.4**
H321	Ø.2Ø05	-Ø.1269	Ø.3742	4.5**
H322	Ø.1751	-Ø.Ø134	Ø.39Ø2	4.5**
H323	Ø.Ø688	-Ø.Ø53Ø	Ø.3365	4.5**

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	$B(A^2)$
H331	Ø.2629	-Ø.142Ø	Ø.1595	4.2**
H332	Ø.1338	-Ø.Ø673	Ø.1156	4.2**
H333	Ø.276Ø	-Ø.Ø374	Ø.Ø518	4.2**
H411	Ø.Ø613	Ø.2837	Ø.6145	4.4**
H412	Ø.Ø827	Ø.3984	Ø.52Ø2	4.4**
H413	Ø.Ø244	Ø.3Ø32	Ø.4967	4.4**
H421	Ø.3423	Ø.2664	Ø.6677	4.3**
H422	Ø.4656	Ø.2749	Ø.58Ø2	4.3**
H423	Ø.3722	Ø.38Ø3	Ø.5745	4.3**
H431	Ø.2773	Ø.Ø82Ø	Ø.5ØØØ	4.1**
H432	Ø.3249	Ø.Ø847	Ø.5994	4.1**
H433	Ø.171Ø	Ø.Ø967	Ø.58Ø5	4.1**

** -- Atoms included but not refined.

Intramolecular Angles

ATOM 1	ATOM 2	ATOM 3	ANGLE
YB1	N1	SI1	104.47(8)
YB1	N1	SI4	122.97(8)
YB1	N1	AL1	81.83(6)
SI1	N1	SI4	115.79(10)
SI1	N1	AL1	114.28(9)
SI4	N1	AL1	113.08(9)
YB1	N2	SI2	101.95(7)
YB1	N2	SI3	126.93(8)
YB1	N2	AL2	79.24(6)
SI2	N2	SI3	116.37(10)
SI2	N2	AL2	114.34(9)
SI3	N2	AL2	112.77(9)
N1	SI1	C11	114.66(10)
N1	SI1	C12	108.66(9)
N1	SI1	C13	113.45(10)
C11	SI1	C12	104.26(11)
C11	SI1	C13	106.37(11)
C12	SI1	C13	108.95(11)
N2	SI2	C21	110.18(9)
N2	SI2	C22	114.70(10)
N2	SI2	C23	112.82(10)
C21	SI2	C22	102.97(11)
C21	SI2	C23	108.23(11)
C22	SI2	C23	107.32(12)
N2	SI3	C31	112.69(10)
N2	SI3	C32	112.79(10)
N2	SI3	C33	111.55(10)
C31	SI3	C32	104.60(12)
C31	SI3	C33	109.25(11)
C32	SI3	C33	105.49(11)
N1	YB1	N2	131.56(5)
N1	YB1	C2	73.52(6)
N1	YB1	C5	152.56(6)
N1	YB1	C6	100.68(6)
N1	YB1	C12	63.01(6)
N1	YB1	C21	135.16(6)
N2	YB1	C2	148.35(6)
N2	YB1	C5	68.77(6)
N2	YB1	C6	71.94(6)
N2	YB1	C12	90.06(6)
N2	YB1	C21	63.59(6)
C2	YB1	C5	81.72(7)
C2	YB1	C6	85.76(7)
C2	YB1	C12	121.20(7)
C2	YB1	C21	115.39(7)
C5	YB1	C6	65.24(7)
C5	YB1	C12	143.19(6)
C5	YB1	C21	66.73(6)
C6	YB1	C12	137.74(7)
C6	YB1	C21	123.13(6)
C12	YB1	C21	77.00(6)

Intramolecular Distances

ATOM 1	ATOM 2	DISTANCE
N1	SI1	1.755(2)
N1	SI4	1.758(2)
N1	AL1	1.973(2)
N2	SI2	1.753(2)
N2	SI3	1.761(2)
N2	AL2	1.953(2)
SI1	N1	1.755(2)
SI1	C11	1.865(2)
SI1	C12	1.879(2)
SI1	C13	1.862(2)
SI2	N2	1.753(2)
SI2	C21	1.883(2)
SI2	C22	1.868(2)
SI2	C23	1.864(2)
SI3	N2	1.761(2)
SI3	C31	1.866(3)
SI3	C32	1.861(3)
SI3	C33	1.860(2)
SI4	N1	1.758(2)
SI4	C41	1.862(2)
SI4	C42	1.870(2)
SI4	C43	1.866(2)
YB1	AL1	2.963(1)
YB1	AL2	2.925(1)
YB1	N1	2.510(2)
YB1	N2	2.573(2)
YB1	C2	2.788(2)
YB1	C5	3.042(2)
YB1	C6	2.756(2)
YB1	C12	3.067(2)
YB1	C21	3.039(2)

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