Crystal structure of samarium tris[bis(dimethylsilyl)amide]-bis(tetrahydrofuran), $Sm[N(SiHMe_2)_2]_3(THF)_2$

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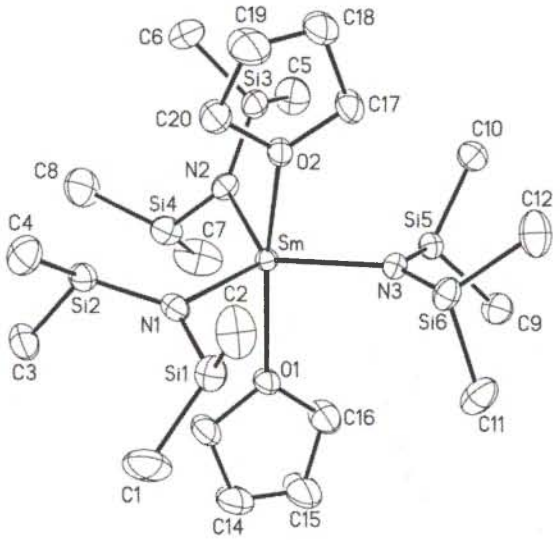

Abstract

$C_{20}H_{58}N_3O_2Si_6Sm$, monoclinic, $P12_1/c1$ (No. 14), $a = 13.146(2)$ Å, $b = 16.377(2)$ Å, $c = 16.853(2)$ Å, $\beta = 91.191(2)°$, $V = 3627.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.034$, $wR_{ref}(F^2) = 0.085$, $T = 203$ K.

Source of material

Addition of a solution of potassium bis(dimethylsilyl)amid (0.21 g, 1.2 mmol) in 20 mL toluene to a yellow suspension of $SmI_3(THF)_{3.5}$ (0.31 g, 0.4 mmol) in 20 mL tetrahydrofuran under inert atmosphere resulted in formation of a pale yellow suspension. The reaction mixture was stirred for 30 minutes, the volatiles were removed, and the residue was extracted with hexanes. Centrifugation followed by cooling of the obtained pale yellow solution to 243 K resulted in almost complete crystallization of $Sm[N(SiHMe_2)_2]_3(THF)_2$. Removal of the mother liquor followed by drying under vacuum gave $Sm[N(SiHMe_2)_2]_3(THF)_2$ as a colourless material (0.21 g, 75% yield). Analytically pure complex $Sm[N(SiHMe_2)_2]_3(THF)_2$ is well soluble in hexanes, in aromatic solvents, and in tetrahydrofuran.

Experimental details

Data colection is performed with three batch runs at $\varphi = 0°$ (606 frames), at $\varphi = 90°$ (435 frames) and $\varphi = 180°$ (230 frames). A fourth batch run is collected at $\varphi = 0°$ (50 frames) to monitor crystal and diffractometer stability. Frame width = 0.3° in ω.

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Discussion

$Sm[N(SiHMe_2)_2]_3(THF)_2$ crystallizes in an approximately trigonal bipyramidal coordination geometry with the nitrogen donor atoms in a trigonal plane (sum of N–Sm–N angles is 359.7°) and with the oxygen donor atoms bent away from the axis (O1–Sm–O1 angle is 162.27(9)°).

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.2 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	18.36 cm^{-1}
Diffractometer, scan mode:	Bruker CCD, φ and ω scans
$2\theta_{max}$.	57.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28279, 8627
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 7205
$N(param)_{refined}$:	289
Program:	SHELXTL [1]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.3094	0.3139	0.3326	0.065
H(2)	4e	0.1725	0.3627	0.0696	0.054
H(3)	4e	0.0997	0.6298	0.1847	0.060
H(4)	4e	0.3708	0.4683	0.0832	0.070
H(5)	4e	0.3422	0.6490	0.2796	0.049
H(6)	4e	0.1810	0.4229	0.4122	0.056
H(1A)	4e	0.4030	0.1948	0.2367	0.136
H(1B)	4e	0.3038	0.1403	0.2279	0.136
H(1C)	4e	0.3573	0.1518	0.3123	0.136
H(2A)	4e	0.0982	0.2812	0.3389	0.122
H(2B)	4e	0.1590	0.2078	0.3785	0.122
H(2C)	4e	0.1059	0.1965	0.2940	0.122
H(3A)	4e	0.3765	0.3130	0.0512	0.096
H(3B)	4e	0.3054	0.2619	–0.0073	0.096
H(3C)	4e	0.3540	0.2199	0.0692	0.096
H(4A)	4e	0.0580	0.2294	0.1270	0.099
H(4B)	4e	0.1479	0.1656	0.1200	0.099
H(4C)	4e	0.0999	0.2061	0.0425	0.099
H(5A)	4e	0.2536	0.7465	0.1541	0.128
H(5B)	4e	0.1499	0.7775	0.1151	0.128
H(5C)	4e	0.2373	0.7437	0.0607	0.128
H(6A)	4e	0.0282	0.5642	0.0386	0.134
H(6B)	4e	0.0902	0.6264	–0.0136	0.134
H(6C)	4e	0.0025	0.6586	0.0413	0.134
H(7A)	4e	0.4488	0.6257	0.0016	0.159
H(7B)	4e	0.4959	0.5882	0.0808	0.159
H(7C)	4e	0.4162	0.6605	0.0850	0.159
H(8A)	4e	0.3242	0.5482	–0.0814	0.150
H(8B)	4e	0.2130	0.5324	–0.0504	0.150
H(8C)	4e	0.2907	0.4589	–0.0568	0.150

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(9A)	4e	0.4552	0.6275	0.4232	0.095
H(9B)	4e	0.3694	0.6651	0.4768	0.095
H(9C)	4e	0.4246	0.7206	0.4138	0.095
H(10A)	4e	0.1436	0.7024	0.3176	0.090
H(10B)	4e	0.2238	0.7687	0.3466	0.090
H(10C)	4e	0.1693	0.7124	0.4093	0.090
H(11A)	4e	0.3896	0.4247	0.4622	0.110
H(11B)	4e	0.3197	0.4173	0.5373	0.110
H(11C)	4e	0.3760	0.5005	0.5193	0.110
H(12A)	4e	0.0857	0.5644	0.4729	0.115
H(12B)	4e	0.1819	0.5924	0.5241	0.115
H(12C)	4e	0.1248	0.5103	0.5452	0.115
H(13A)	4e	0.4562	0.3333	0.2004	0.090
H(13B)	4e	0.5153	0.4025	0.1536	0.090

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14A)	4e	0.5787	0.3170	0.2848	0.125
H(14B)	4e	0.6461	0.3608	0.2201	0.125
H(15A)	4e	0.6596	0.4657	0.2875	0.215
H(15B)	4e	0.6163	0.4134	0.3587	0.215
H(16A)	4e	0.4778	0.4845	0.3558	0.094
H(16B)	4e	0.5229	0.5389	0.2866	0.094
H(17A)	4e	0.0286	0.5646	0.3047	0.069
H(17B)	4e	0.0093	0.4838	0.3548	0.069
H(18A)	4e	–0.1502	0.5051	0.3093	0.092
H(18B)	4e	–0.1128	0.5470	0.2297	0.092
H(19A)	4e	–0.1532	0.4266	0.1844	0.124
H(19B)	4e	–0.1219	0.3824	0.2655	0.124
H(20A)	4e	0.0205	0.3549	0.2026	0.092
H(20B)	4e	0.0054	0.4293	0.1428	0.092

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sm	4e	0.25369(1)	0.468922(8)	0.230364(8)	0.02884(8)	0.03049(8)	0.02810(8)	0.00020(5)	0.00096(5)	0.00022(5)
Si(1)	4e	0.26026(8)	0.26994(7)	0.28028(6)	0.0604(6)	0.0498(6)	0.0513(6)	–0.0077(4)	0.0022(5)	–0.0006(4)
Si(2)	4e	0.21307(7)	0.30015(6)	0.10570(6)	0.0421(5)	0.0393(5)	0.0529(5)	0.0006(4)	–0.0027(4)	–0.0065(4)
Si(3)	4e	0.15016(8)	0.63236(6)	0.11922(5)	0.0629(6)	0.0498(5)	0.0370(4)	0.0061(4)	–0.0017(4)	0.0040(4)
Si(4)	4e	0.32959(9)	0.53624(7)	0.05909(7)	0.0622(7)	0.0616(7)	0.0514(6)	0.0099(5)	0.0132(5)	0.0188(5)
Si(5)	4e	0.30129(7)	0.63769(5)	0.34829(5)	0.0426(5)	0.0395(4)	0.0406(4)	–0.0020(4)	0.0023(3)	–0.0032(3)
Si(6)	4e	0.23018(8)	0.48810(6)	0.43445(6)	0.0590(6)	0.0410(5)	0.0394(5)	–0.0102(4)	–0.0047(4)	–0.0006(4)
N(1)	4e	0.2409(2)	0.3303(2)	0.2004(2)	0.035(1)	0.037(1)	0.056(2)	0.001(1)	0.004(1)	–0.006(1)
N(2)	4e	0.2390(2)	0.5579(2)	0.1250(2)	0.047(2)	0.043(1)	0.038(1)	–0.003(1)	–0.001(1)	0.008(1)
N(3)	4e	0.2620(2)	0.5388(2)	0.3505(2)	0.040(1)	0.040(1)	0.035(1)	0.000(1)	–0.002(1)	–0.003(1)
O(1)	4e	0.4397(2)	0.4454(2)	0.2476(1)	0.033(1)	0.054(1)	0.056(1)	0.004(1)	0.001(1)	–0.013(1)
O(2)	4e	0.0676(2)	0.4615(1)	0.2470(1)	0.029(1)	0.054(1)	0.044(1)	–0.0026(9)	0.0034(9)	–0.0138(9)
C(1)	4e	0.3407(4)	0.1782(3)	0.2621(3)	0.100(4)	0.069(3)	0.102(4)	0.033(3)	–0.004(3)	0.026(3)
C(2)	4e	0.1416(4)	0.2346(3)	0.3288(3)	0.099(3)	0.072(3)	0.074(3)	–0.030(3)	0.029(3)	–0.001(2)
C(3)	4e	0.3257(3)	0.2701(3)	0.0478(2)	0.066(2)	0.063(2)	0.063(2)	0.002(2)	0.011(2)	–0.019(2)
C(4)	4e	0.1183(3)	0.2151(2)	0.0978(3)	0.060(2)	0.064(2)	0.074(3)	–0.017(2)	–0.009(2)	–0.014(2)
C(5)	4e	0.2043(4)	0.7377(2)	0.1113(3)	0.126(4)	0.044(2)	0.086(3)	0.006(2)	–0.004(3)	0.014(2)
C(6)	4e	0.0566(4)	0.6188(3)	0.0365(3)	0.069(3)	0.124(4)	0.075(3)	0.025(3)	–0.024(2)	–0.012(3)
C(7)	4e	0.4352(4)	0.6117(3)	0.0563(4)	0.073(3)	0.102(4)	0.145(5)	–0.008(3)	0.037(3)	0.042(4)
C(8)	4e	0.2839(5)	0.5166(4)	–0.0449(3)	0.123(5)	0.131(5)	0.048(2)	0.033(4)	0.019(3)	0.004(3)
C(9)	4e	0.3993(3)	0.6661(2)	0.4247(3)	0.056(2)	0.054(2)	0.079(3)	–0.008(2)	–0.014(2)	–0.017(2)
C(10)	4e	0.1970(3)	0.7145(2)	0.3564(2)	0.066(2)	0.047(2)	0.065(2)	0.009(2)	–0.003(2)	–0.005(2)
C(11)	4e	0.3424(4)	0.4535(3)	0.4957(3)	0.098(4)	0.060(3)	0.062(3)	–0.007(2)	–0.027(2)	0.013(2)
C(12)	4e	0.1455(4)	0.5458(3)	0.5023(3)	0.092(3)	0.084(3)	0.055(2)	–0.018(2)	0.028(2)	–0.003(2)
C(13)	4e	0.4968(3)	0.3831(3)	0.2064(3)	0.047(2)	0.083(3)	0.094(3)	0.019(2)	–0.005(2)	–0.041(3)
C(14)	4e	0.5871(4)	0.3673(4)	0.2541(4)	0.067(3)	0.112(4)	0.133(5)	0.045(3)	–0.032(3)	–0.042(4)
C(15)	4e	0.6018(5)	0.4329(6)	0.3047(6)	0.091(4)	0.222(8)	0.221(9)	0.093(5)	–0.090(5)	–0.150(8)
C(16)	4e	0.5078(3)	0.4829(3)	0.3030(3)	0.037(2)	0.088(3)	0.109(4)	–0.000(2)	–0.009(2)	–0.046(3)
C(17)	4e	0.0058(3)	0.5077(3)	0.3016(2)	0.040(2)	0.076(2)	0.058(2)	–0.003(2)	0.012(2)	–0.023(2)
C(18)	4e	–0.0998(3)	0.5025(3)	0.2673(3)	0.040(2)	0.100(3)	0.089(3)	0.008(2)	0.003(2)	–0.035(3)
C(19)	4e	–0.1034(3)	0.4257(4)	0.2284(4)	0.042(2)	0.116(5)	0.152(5)	0.001(3)	–0.013(3)	–0.060(4)
C(20)	4e	0.0015(3)	0.4126(3)	0.1985(3)	0.043(2)	0.091(3)	0.095(3)	0.002(2)	–0.016(2)	–0.047(3)

Reference

1. Bruker. SHELXTL. Structure Determination Programs. Version 5.1, Bruker AXS, Inc., Madison, Wisconsin, USA 1997.