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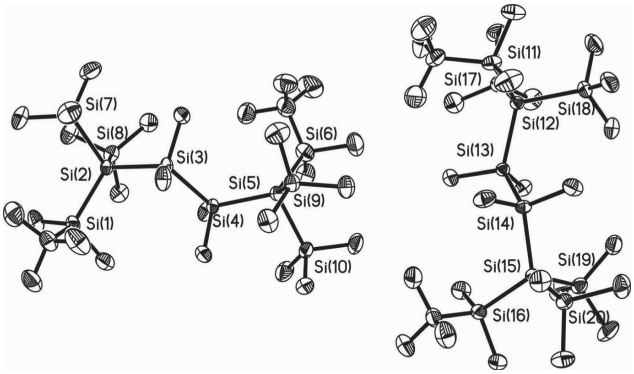
The crystal structure of 1,6-di-*tert*-butyl-1,1,3,3,4,4,6,6-octamethyl-2,2,5,5-tetrakis(trimethylsilyl)hexasilane, $C_{28}H_{78}Si_{10}$



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Abstract

$C_{28}H_{78}Si_{10}$, monoclinic, *Pn* (no. 7), $a = 21.413(4)$ Å, $b = 9.8568(17)$ Å, $c = 22.074(4)$ Å, $\beta = 93.426(4)°$, $V = 4650.6(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0613$, $wR_{ref}(F^2) = 0.1750$, $T = 296(2)$ K.

CCDC no.: 1913963

The molecular structure (asymmetric unit) is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.25 × 0.20 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.30 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	29235, 15502, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 9548
$N(param)_{refined}$:	737
Programs:	Bruker [1], SHELX [2–4]

Source of material

In a representative experiment, the mixture of 2,2,5,5-tetrakis(trimethylsilyl)-1,1,1,3,3,4,4,6,6,6-decamethylhexasilane [5] (18.30 g, 30 mmol) and *t*-BuOK (7.00 g, 63 mmol) was stirred in 100 mL THF at 60 °C for 10 hours. After removing the solvent, 40 mL toluene was added and then 20 mL toluene solution of *tert*-butyldimethyl chlorosilane was added dropwise at −20 °C. The mixture was slowly warmed to room temperature and stirred for 2 hours. After aqueous workup and recrystallization from ethanol, the title compound were obtained in the yield of 92%. Crystals were obtained by slow evaporating the hexane solution at 5 °C in 2 days. **^{1}H NMR** ($CDCl_3$, 400 MHz): δ [ppm] 0.95 (s, 18H, *t*-Bu), 0.45 (s, 12H, $SiMe_2$), 0.27 (s, 36H, $SiMe_3$), 0.19 (s, 12H, $SiMe_2$(*t*-Bu)); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ [ppm] 28.44 (*t*-Bu), 19.02 (*t*-Bu), 4.42 ($SiMe_2$), 2.47 ($SiMe_2$(*t*-Bu)), −0.07 ($SiMe_3$); **^{29}Si NMR** ($CDCl_3$, 80 MHz): δ [ppm] 5.15 ($SiMe_2$(*t*-Bu)), −9.04($SiMe_3$), −27.44($SiMe_2$), −127.43($Si(SiMe_3)_4$).

Experimental details

A suitable crystal was selected and was mounted onto the tip of glass fibres. An empirical (multi-scan) absorption correction was applied with the program SADABS [3]. The structures were solved with the ShelXS structure solution program using Direct Methods and subsequently refined on F^2 (ShelXL) [4]. The figure of the solid-state molecular structure was generated using XP as implemented in Shelxtl program [1].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Si1	0.99894(11)	1.0164(3)	0.01981(10)	0.0554(6)
Si2	0.90737(9)	1.0505(2)	0.07317(8)	0.0394(4)
Si3	0.89817(9)	0.9222(2)	0.16375(8)	0.0416(5)
Si4	0.97431(9)	0.9587(2)	0.24484(9)	0.0429(5)
Si5	0.95029(9)	0.8876(2)	0.34393(8)	0.0394(4)
Si6	0.89429(13)	1.0388(3)	0.40548(13)	0.0702(7)
Si7	0.81432(11)	1.0116(3)	0.01157(11)	0.0634(7)
Si8	0.90308(11)	1.2883(2)	0.09181(10)	0.0538(6)
Si9	0.91322(10)	0.6616(2)	0.34537(10)	0.0523(5)
Si10	1.05020(11)	0.8755(3)	0.39857(11)	0.0637(7)
Si11	0.71885(10)	0.4928(2)	0.63324(11)	0.0569(6)
Si12	0.79200(8)	0.6052(2)	0.70117(9)	0.0399(4)
Si13	0.90057(8)	0.58940(19)	0.68316(8)	0.0375(4)
Si14	0.94275(8)	0.3685(2)	0.68237(9)	0.0406(4)
Si15	1.05274(8)	0.3474(2)	0.70306(9)	0.0376(4)
Si16	1.12214(9)	0.4557(2)	0.63823(10)	0.0532(6)
Si17	0.77355(11)	0.8412(2)	0.70840(11)	0.0564(6)
Si18	0.76834(12)	0.5221(3)	0.79878(12)	0.0694(7)
Si19	1.08630(10)	0.4308(3)	0.80123(10)	0.0548(6)
Si20	1.06689(10)	0.1082(2)	0.71137(10)	0.0509(5)
C1	1.0073(5)	0.8506(10)	−0.0239(4)	0.073(2)
C2	1.0039(5)	1.1575(11)	−0.0357(4)	0.082(3)
H2A	1.0415	1.1483	−0.0569	0.122*
H2B	1.0044	1.2427	−0.0146	0.122*
H2C	0.9683	1.1543	−0.0642	0.122*
C3	1.0680(4)	1.0351(12)	0.0750(5)	0.087(3)
H3A	1.0624	1.1134	0.1000	0.130*
H3B	1.1052	1.0457	0.0533	0.130*
H3C	1.0718	0.9557	0.1001	0.130*
C4	0.9030(5)	0.7336(9)	0.1464(4)	0.077(3)
H4A	0.8824	0.7156	0.1074	0.115*
H4B	0.8830	0.6831	0.1770	0.115*
H4C	0.9461	0.7069	0.1461	0.115*
C5	0.8163(4)	0.9459(11)	0.1868(4)	0.070(3)
H5A	0.8149	0.9306	0.2297	0.105*
H5B	0.7891	0.8826	0.1652	0.105*
H5C	0.8028	1.0368	0.1774	0.105*
C6	1.0448(4)	0.8560(10)	0.2253(4)	0.069(2)
H6A	1.0765	0.8626	0.2577	0.104*
H6B	1.0607	0.8904	0.1885	0.104*
H6C	1.0328	0.7628	0.2196	0.104*
C7	1.0000(5)	1.1409(10)	0.2482(5)	0.076(3)
H7A	1.0239	1.1609	0.2139	0.114*
H7B	1.0254	1.1562	0.2849	0.114*
H7C	0.9639	1.1988	0.2476	0.114*
C8	0.8766(7)	0.9493(13)	0.4813(5)	0.105(4)
H8A	0.8548	0.8657	0.4725	0.157*
H8B	0.8511	1.0074	0.5044	0.157*
H8C	0.9152	0.9306	0.5041	0.157*
C9	0.9468(6)	1.1888(12)	0.4241(5)	0.095(4)
H9A	0.9233	1.2583	0.4430	0.143*
H9B	0.9627	1.2235	0.3874	0.143*
H9C	0.9810	1.1607	0.4513	0.143*
C10	0.9986(7)	0.7291(11)	0.0171(6)	0.116(5)
H10A	1.0274	0.7351	0.0520	0.174*
H10B	1.0063	0.6471	−0.0048	0.174*
H10C	0.9565	0.7282	0.0299	0.174*
C11	1.0723(5)	0.8471(13)	−0.0484(5)	0.099(4)
H11A	1.0884	0.7563	−0.0457	0.148*
H11B	1.0995	0.9069	−0.0249	0.148*
H11C	1.0698	0.8759	−0.0900	0.148*
C12	0.9606(6)	0.8463(13)	−0.0783(5)	0.103(4)
H12A	0.9193	0.8634	−0.0653	0.154*
H12B	0.9616	0.7584	−0.0970	0.154*
H12C	0.9712	0.9142	−0.1071	0.154*
C16	0.7979(5)	0.8257(11)	0.0021(6)	0.098(4)
H16A	0.8346	0.7806	−0.0106	0.147*
H16B	0.7641	0.8129	−0.0279	0.147*
H16C	0.7865	0.7884	0.0401	0.147*
C17	0.8208(6)	1.0858(13)	−0.0665(5)	0.099(4)
H17A	0.8435	1.1698	−0.0634	0.149*
H17B	0.7797	1.1020	−0.0848	0.149*
H17C	0.8426	1.0233	−0.0911	0.149*
C18	0.7457(5)	1.0950(14)	0.0443(6)	0.100(4)
H18A	0.7082	1.0708	0.0208	0.150*
H18B	0.7511	1.1917	0.0436	0.150*
H18C	0.7426	1.0654	0.0855	0.150*
C19	0.8711(6)	1.3829(10)	0.0233(5)	0.089(3)
H19A	0.8793	1.4780	0.0286	0.133*
H19B	0.8267	1.3684	0.0182	0.133*
H19C	0.8907	1.3507	−0.0119	0.133*
C20	0.9812(4)	1.3677(9)	0.1116(5)	0.072(3)
H20A	0.9780	1.4643	0.1067	0.109*
H20B	1.0114	1.3328	0.0852	0.109*
H20C	0.9941	1.3467	0.1529	0.109*
C21	0.8524(5)	1.3236(10)	0.1564(4)	0.076(3)
H21A	0.8131	1.2774	0.1496	0.113*
H21B	0.8451	1.4195	0.1592	0.113*
H21C	0.8730	1.2921	0.1936	0.113*
C22	0.9177(5)	0.5964(10)	0.4258(4)	0.077(3)
H22A	0.9174	0.4990	0.4255	0.115*
H22B	0.8824	0.6291	0.4464	0.115*
H22C	0.9557	0.6279	0.4466	0.115*
C23	0.9621(6)	0.5469(11)	0.2990(5)	0.089(3)
H23A	0.9646	0.5841	0.2591	0.134*
H23B	0.9431	0.4587	0.2962	0.134*
H23C	1.0034	0.5395	0.3182	0.134*
C24	0.8297(5)	0.6435(10)	0.3149(6)	0.094(4)
H24A	0.8029	0.6927	0.3404	0.141*
H24B	0.8183	0.5493	0.3144	0.141*
H24C	0.8255	0.6792	0.2745	0.141*
C25	1.0441(5)	0.8778(14)	0.4827(4)	0.093(4)
H25A	1.0245	0.9607	0.4944	0.140*
H25B	1.0851	0.8719	0.5025	0.140*
H25C	1.0194	0.8020	0.4946	0.140*
C26	1.0934(4)	0.7159(13)	0.3817(5)	0.090(4)
H26A	1.0672	0.6389	0.3885	0.136*
H26B	1.1308	0.7103	0.4077	0.136*
H26C	1.1042	0.7167	0.3401	0.136*
C27	1.1031(5)	1.0205(14)	0.3796(6)	0.106(5)
H27A	1.1391	1.0214	0.4078	0.158*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H27B	1.0809	1.1046	0.3823	0.158*
H27C	1.1164	1.0092	0.3392	0.158*
C28	0.7116(4)	0.5566(10)	0.5513(4)	0.066(2)
C29	1.1185(4)	0.4022(10)	0.5556(4)	0.071(3)
C30	0.7373(5)	0.3034(10)	0.6292(7)	0.105(4)
H30A	0.7039	0.2577	0.6066	0.157*
H30B	0.7416	0.2667	0.6695	0.157*
H30C	0.7756	0.2907	0.6095	0.157*
C31	0.6398(4)	0.5079(14)	0.6647(6)	0.103(4)
H31A	0.6080	0.4932	0.6328	0.154*
H31B	0.6351	0.5969	0.6814	0.154*
H31C	0.6358	0.4412	0.6959	0.154*
C32	0.9439(4)	0.6985(8)	0.7415(4)	0.059(2)
H32A	0.9286	0.7899	0.7383	0.089*
H32B	0.9878	0.6975	0.7347	0.089*
H32C	0.9376	0.6639	0.7813	0.089*
C33	0.9170(4)	0.6646(9)	0.6081(4)	0.061(2)
H33A	0.9614	0.6652	0.6036	0.091*
H33B	0.9014	0.7559	0.6059	0.091*
H33C	0.8968	0.6114	0.5762	0.091*
C34	0.9042(4)	0.2581(9)	0.7394(5)	0.070(2)
H34A	0.9137	0.1646	0.7320	0.104*
H34B	0.8597	0.2712	0.7355	0.104*
H34C	0.9196	0.2825	0.7797	0.104*
C35	0.9191(4)	0.2930(10)	0.6051(4)	0.073(3)
H35A	0.8778	0.2555	0.6058	0.110*
H35B	0.9481	0.2227	0.5959	0.110*
H35C	0.9196	0.3627	0.5748	0.110*
C36	1.1094(5)	0.6460(9)	0.6376(5)	0.076(3)
H36A	1.0703	0.6665	0.6161	0.113*
H36B	1.1428	0.6894	0.6179	0.113*
H36C	1.1086	0.6784	0.6786	0.113*
C37	1.2043(4)	0.4182(12)	0.6710(5)	0.078(3)
H37A	1.2338	0.4723	0.6506	0.116*
H37B	1.2135	0.3238	0.6655	0.116*
H37C	1.2071	0.4395	0.7135	0.116*
C39	0.6608(7)	0.4737(18)	0.5166(6)	0.139(6)
H39A	0.6548	0.5079	0.4759	0.208*
H39B	0.6224	0.4809	0.5366	0.208*
H39C	0.6734	0.3803	0.5154	0.208*
C40	0.6913(10)	0.7009(15)	0.5486(6)	0.160(8)
H40A	0.6690	0.7181	0.5104	0.240*
H40B	0.7273	0.7588	0.5528	0.240*
H40C	0.6644	0.7188	0.5810	0.240*
C41	0.7714(6)	0.5375(18)	0.5200(6)	0.124(5)
H41A	0.8045	0.5867	0.5415	0.186*
H41B	0.7661	0.5710	0.4792	0.186*
H41C	0.7818	0.4428	0.5193	0.186*
C42	1.1259(7)	0.2459(10)	0.5520(5)	0.100(4)
H42A	1.1214	0.2175	0.5104	0.149*
H42B	1.0943	0.2032	0.5745	0.149*
H42C	1.1666	0.2204	0.5689	0.149*
C43	1.0566(5)	0.4452(17)	0.5236(5)	0.117(5)
H43A	1.0540	0.4101	0.4829	0.175*
H43B	1.0542	0.5424	0.5224	0.175*
H43C	1.0226	0.4101	0.5453	0.175*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C44	1.1727(6)	0.4715(16)	0.5232(5)	0.109(4)
H44A	1.1720	0.5675	0.5306	0.164*
H44B	1.1676	0.4550	0.4803	0.164*
H44C	1.2121	0.4349	0.5387	0.164*
C45	0.6879(5)	0.8852(12)	0.7054(5)	0.089(3)
H45A	0.6703	0.8735	0.6647	0.134*
H45B	0.6830	0.9779	0.7176	0.134*
H45C	0.6668	0.8268	0.7323	0.134*
C46	0.8068(6)	0.9078(11)	0.7826(5)	0.091(3)
H46A	0.7898	0.9962	0.7896	0.137*
H46B	0.8515	0.9138	0.7818	0.137*
H46C	0.7961	0.8476	0.8146	0.137*
C47	0.8095(6)	0.9387(11)	0.6480(5)	0.093(3)
H47A	0.8542	0.9349	0.6541	0.139*
H47B	0.7960	1.0314	0.6493	0.139*
H47C	0.7970	0.9003	0.6092	0.139*
C48	0.7468(5)	0.3410(13)	0.8004(6)	0.101(4)
H48A	0.7349	0.3174	0.8403	0.152*
H48B	0.7819	0.2868	0.7903	0.152*
H48C	0.7123	0.3246	0.7715	0.152*
C49	0.7003(6)	0.6205(13)	0.8271(5)	0.105(4)
H49A	0.6918	0.5898	0.8670	0.158*
H49B	0.6639	0.6062	0.8001	0.158*
H49C	0.7104	0.7153	0.8284	0.158*
C50	0.8355(6)	0.5466(15)	0.8569(5)	0.106(4)
H50A	0.8673	0.4805	0.8506	0.158*
H50B	0.8208	0.5357	0.8969	0.158*
H50C	0.8525	0.6360	0.8529	0.158*
C51	1.0191(5)	0.0100(9)	0.6510(4)	0.069(2)
H51A	1.0241	−0.0856	0.6582	0.104*
H51B	1.0332	0.0319	0.6117	0.104*
H51C	0.9757	0.0338	0.6525	0.104*
C52	1.0408(5)	0.0439(10)	0.7870(4)	0.079(3)
H52A	1.0485	0.1123	0.8175	0.119*
H52B	1.0637	−0.0368	0.7983	0.119*
H52C	0.9968	0.0236	0.7833	0.119*
C53	1.1509(4)	0.0567(12)	0.7075(5)	0.088(3)
H53A	1.1625	0.0614	0.6662	0.131*
H53B	1.1562	−0.0345	0.7221	0.131*
H53C	1.1770	0.1168	0.7321	0.131*
C54	1.1519(5)	0.3233(13)	0.8347(5)	0.096(4)
H54A	1.1679	0.3624	0.8723	0.143*
H54B	1.1847	0.3189	0.8069	0.143*
H54C	1.1367	0.2335	0.8419	0.143*
C55	1.0209(5)	0.4287(11)	0.8544(4)	0.077(3)
H55A	0.9877	0.4865	0.8388	0.116*
H55B	1.0362	0.4607	0.8936	0.116*
H55C	1.0055	0.3377	0.8580	0.116*
C56	1.1166(5)	0.6065(12)	0.8021(5)	0.087(3)
H56A	1.1534	0.6107	0.7794	0.130*
H56B	1.1269	0.6338	0.8432	0.130*
H56C	1.0853	0.6662	0.7842	0.130*
C57	0.8177(6)	1.1094(13)	0.3750(5)	0.093(3)
C58	0.8328(6)	1.1927(13)	0.3156(5)	0.098(4)
H58A	0.7955	1.2375	0.2997	0.147*
H58B	0.8474	1.1317	0.2857	0.147*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H58C	0.8645	1.2591	0.3258	0.147*
C60	0.7707(5)	0.9910(15)	0.3597(7)	0.112(4)
H60A	0.7318	1.0277	0.3431	0.168*
H60B	0.7636	0.9411	0.3960	0.168*
H60C	0.7879	0.9315	0.3305	0.168*
C59	0.7895(7)	1.1985(17)	0.4256(7)	0.146(6)
H59A	0.7894	1.1475	0.4627	0.218*
H59B	0.7474	1.2238	0.4130	0.218*
H59C	0.8144	1.2788	0.4321	0.218*

Comment

The chemistry of carbenes and their heavier analogues (tetrylenes) has attracted more and more attention because of the occurrence of orthogonal donor and acceptor orbitals within a small energetic separation, which made them good candidates to mimic transition-metal behavior, for example, in oxidative addition or reductive elimination reactions [6–8]. Many examples of these tetrylenes are stabilized by the presence of nitrogen directly adjacent to the divalent centres, if electronegative groups are replaced by alkyl groups, the energy difference between singlet and triplet states (ΔE_{st}) will be reduced. The even more electropositive silyl or oligosilyl substituents are of particular interest as for these tetrylenes even smaller ΔE_{st} values are expected [9]. During the synthesis of silyl-substituted tetrylenes, [10, 11] we prepared the 2,2,5,5-tetrakis(*tert*-butyldimethylsilyl)-1,1,1,3,3,4,4,6,6,6-decamethylhexasilane as the pre-ligands. However, the yield is quite low [12]. Another route for its syntheses was adopted, during which the title compound was prepared.

In the asymmetric unit of the title compound, two crystallographically independent molecules are shown. The silicon atoms in the main chain are of zig-zag type. The two *tert*-butyldimethylsilyl groups are arranged in an *anti*-fashion. The dihedral angle of Si2-Si3-Si4-Si5 is 161.5°. The 28 Si-Si-Si bond angles average 110.4°. The average Si-Si distance of these 18 Si-Si bonds is 2.379 Å, which is larger than that of 2.340 Å found in hexamethyldisilane. The average bond length of these 44 Si-C bonds is 1.882 Å. But all geometric parameters are in the expected ranges [12].

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