Crystal structure of 1,1,2,2,6,6,7,7-octa-*tert*-butyl-5,10-dimethylene-1,2,6,7-tetrasilacyclodeca-3,8-diyne, $C_{40}H_{76}Si_4$

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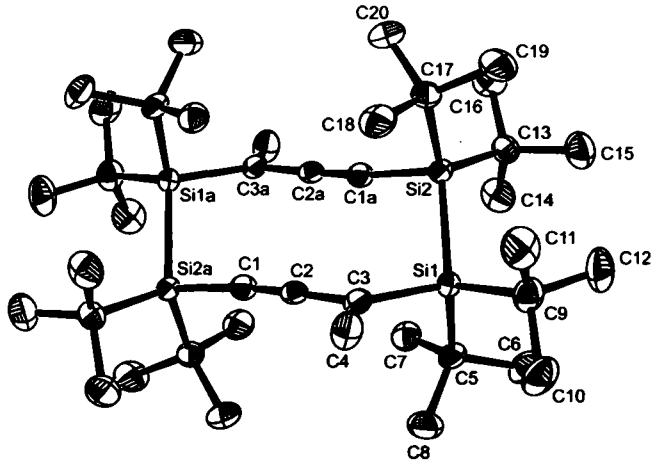

Abstract

$C_{40}H_{76}Si_4$, monoclinic, $P12_1/c1$ (No. 14), $a = 11.0844(3)$ Å, $b = 19.5735(7)$ Å, $c = 19.5787(5)$ Å, $\beta = 98.670(3)°$, $V = 4199.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.058$, $wR_{ref}(F^2) = 0.153$, $T = 193$ K.

Source of material

Reaction of tetra-*tert*-butyldisilene, generated by photolysis of hexa-*tert*-butylcyclotrisilane, with octa-2,4,6-triyne furnishes the title compound in low yield as colourless, rectangular crystals.

Experimental details

The asymmetric unit contains two half molecules. One of these (Si3 to C40) is disordered and was refined on two positions with occupancy factors of 0.75 and 0.25.

Discussion

The crystal structure analysis reveals a ten-membered ring with a centre of inversion in the middle of the ring. This novel ring system contains two C≡C triple bonds within the ring and two exocyclic methylene groups at the positions 5 and 10 and adopts a chair conformation. This type of reaction is very unusual since all other reactions between the disilene and di- or tetraynes proceed by [2+2] cycloadditions [1-3].

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Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.43 × 0.50 × 0.60 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.66 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{max}$:	51.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	31930, 7719
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6300
$N(param)_{refined}$:	464
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(4A)	4*e*		0.4469	0.9643	0.2061	0.047
H(4B)	4*e*		0.4093	0.8832	0.1966	0.047
H(6A)	4*e*		0.1684	0.8173	–0.0721	0.063
H(6B)	4*e*		0.0498	0.8595	–0.0601	0.063
H(6C)	4*e*		0.1077	0.8046	–0.0040	0.063
H(7A)	4*e*		0.2697	0.9331	–0.0867	0.053
H(7B)	4*e*		0.2729	0.9888	–0.0266	0.053
H(7C)	4*e*		0.1459	0.9686	–0.0724	0.053
H(8A)	4*e*		0.1241	0.8934	0.0891	0.066
H(8B)	4*e*		0.0596	0.9454	0.0318	0.066
H(8C)	4*e*		0.1866	0.9654	0.0777	0.066
H(10A)	4*e*		0.1673	0.7908	0.1056	0.074
H(10B)	4*e*		0.2596	0.8131	0.1728	0.074
H(10C)	4*e*		0.2321	0.7339	0.1573	0.074
H(11A)	4*e*		0.4838	0.7917	0.1782	0.073
H(11B)	4*e*		0.5314	0.7487	0.1181	0.073
H(11C)	4*e*		0.4478	0.7128	0.1676	0.073
H(12A)	4*e*		0.2504	0.7264	0.0096	0.079
H(12B)	4*e*		0.3096	0.6740	0.0679	0.079
H(12C)	4*e*		0.3931	0.7096	0.0182	0.079
H(14A)	4*e*		0.3810	0.8909	–0.1757	0.068
H(14B)	4*e*		0.2880	0.8389	–0.1481	0.068
H(14C)	4*e*		0.3469	0.8205	–0.2155	0.068
H(15A)	4*e*		0.4995	0.7086	–0.0890	0.072
H(15B)	4*e*		0.4179	0.7116	–0.1637	0.072
H(15C)	4*e*		0.3588	0.7300	–0.0963	0.072
H(16A)	4*e*		0.6013	0.8580	–0.1697	0.072
H(16B)	4*e*		0.5540	0.7899	–0.2105	0.072
H(16C)	4*e*		0.6458	0.7848	–0.1393	0.072
H(18A)	4*e*		0.6283	0.8452	0.1066	0.062
H(18B)	4*e*		0.6863	0.9122	0.0778	0.062
H(18C)	4*e*		0.7723	0.8494	0.1057	0.062
H(19A)	4*e*		0.6255	0.7341	0.0379	0.072
H(19B)	4*e*		0.7698	0.7429	0.0414	0.072
H(19C)	4*e*		0.6850	0.7330	–0.0317	0.072
H(20A)	4*e*		0.7676	0.9092	–0.0330	0.066
H(20B)	4*e*		0.7705	0.8391	–0.0748	0.066
H(20C)	4*e*		0.8555	0.8484	–0.0017	0.066
H(24A)	4*e*	0.75	1.0126	1.2009	0.4448	0.033
H(24B)	4*e*	0.75	1.0323	1.1885	0.3641	0.033
H(26A)	4*e*	0.75	0.9677	1.1551	0.2758	0.118
H(26B)	4*e*	0.75	0.9205	1.0888	0.2318	0.118

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(26C)	4e	0.75	1.0019	1.1445	0.2000	0.118
H(27A)	4e	0.75	1.2025	1.0110	0.2347	0.150
H(27B)	4e	0.75	1.1396	1.0611	0.1752	0.150
H(27C)	4e	0.75	1.0604	1.0031	0.2053	0.150
H(28A)	4e	0.75	1.2860	1.1136	0.2966	0.061
H(28B)	4e	0.75	1.1971	1.1762	0.3061	0.061
H(28C)	4e	0.75	1.2197	1.1541	0.2304	0.061
H(30A)	4e	0.75	1.2091	0.9844	0.4951	0.042
H(30B)	4e	0.75	1.3360	0.9503	0.4843	0.042
H(30C)	4e	0.75	1.2106	0.9211	0.4435	0.042
H(31A)	4e	0.75	1.3462	1.1076	0.3966	0.057
H(31B)	4e	0.75	1.4173	1.0605	0.4563	0.057
H(31C)	4e	0.75	1.2927	1.0971	0.4673	0.057
H(32A)	4e	0.75	1.3348	1.0077	0.3053	0.108
H(32B)	4e	0.75	1.2865	0.9347	0.3262	0.108
H(32C)	4e	0.75	1.4149	0.9630	0.3638	0.108
H(34A)	4e	0.75	0.7904	1.0565	0.3943	0.047
H(34B)	4e	0.75	0.6859	1.0682	0.3295	0.047
H(34C)	4e	0.75	0.8254	1.0836	0.3227	0.047
H(35A)	4e	0.75	0.8142	1.0018	0.2199	0.054
H(35B)	4e	0.75	0.6729	0.9924	0.2261	0.054
H(35C)	4e	0.75	0.7593	0.9269	0.2265	0.054
H(36A)	4e	0.75	0.7114	0.9374	0.4027	0.055
H(36B)	4e	0.75	0.6891	0.8905	0.3350	0.055
H(36C)	4e	0.75	0.6097	0.9582	0.3391	0.055
H(38A)	4e	0.75	0.8639	0.8076	0.3661	0.071
H(38B)	4e	0.75	0.8988	0.7669	0.3009	0.071
H(38C)	4e	0.75	0.8054	0.8297	0.2896	0.071
H(39A)	4e	0.75	1.0771	0.8962	0.2367	0.063
H(39B)	4e	0.75	0.9356	0.882	0.2102	0.063
H(39C)	4e	0.75	1.0291	0.8195	0.2238	0.063
H(40A)	4e	0.75	1.0862	0.8228	0.4048	0.059
H(40B)	4e	0.75	1.1695	0.8565	0.3538	0.059
H(40C)	4e	0.75	1.1108	0.7825	0.3372	0.059
C(21B)	4e	0.25	1.064(1)	1.0597(6)	0.4477(6)	0.0303(6)
C(22B)	4e	0.25	1.059(4)	1.085(2)	0.505(2)	0.0303
C(23B)	4e	0.25	1.041(1)	1.1216(6)	0.5651(6)	0.0303
C(24B)	4e	0.25	1.0367(9)	1.1913(5)	0.5598(6)	0.0303
H(24C)	4e	0.25	1.0458	1.2128	0.5174	0.036
H(24D)	4e	0.25	1.0245	1.2183	0.5986	0.036
Si(3B)	4e	0.25	1.0953(5)	1.0215(3)	0.3664(3)	0.0303
Si(4B)	4e	0.25	0.9495(5)	0.9263(2)	0.3487(3)	0.0303
C(25B)	4e	0.25	1.123(2)	1.087(1)	0.273(1)	0.0303
C(26B)	4e	0.25	0.987(2)	1.123(2)	0.238(2)	0.0303
H(26D)	4e	0.25	0.962	1.1563	0.2703	0.045
H(26E)	4e	0.25	0.9249	1.0879	0.2276	0.045
H(26F)	4e	0.25	0.9979	1.1469	0.1947	0.045

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
C(27B)	4e	0.25	1.130(2)	1.031(1)	0.212(2)	0.0303
H(27D)	4e	0.25	1.2045	1.0042	0.2233	0.045
H(27E)	4e	0.25	1.1295	1.0548	0.1683	0.045
H(27F)	4e	0.25	1.0587	1.0007	0.209	0.045
C(28B)	4e	0.25	1.192(1)	1.1457(5)	0.3192(7)	0.0303
H(28D)	4e	0.25	1.2711	1.1287	0.3418	0.045
H(28E)	4e	0.25	1.1427	1.1598	0.3544	0.045
H(28F)	4e	0.25	1.2045	1.1849	0.29	0.045
C(29B)	4e	0.25	1.263(2)	0.9890(7)	0.3906(8)	0.0303
C(30B)	4e	0.25	1.261(1)	0.9240(6)	0.4362(6)	0.0303
H(30D)	4e	0.25	1.2008	0.9302	0.4678	0.045
H(30E)	4e	0.25	1.3415	0.9165	0.4628	0.045
H(30F)	4e	0.25	1.2375	0.8843	0.4065	0.045
C(31B)	4e	0.25	1.343(1)	1.0436(7)	0.4344(7)	0.0303
H(31D)	4e	0.25	1.2987	1.0618	0.4701	0.045
H(31E)	4e	0.25	1.3617	1.0809	0.4043	0.045
H(31F)	4e	0.25	1.4194	1.0225	0.4564	0.045
C(32B)	4e	0.25	1.338(3)	0.974(2)	0.354(2)	0.0303
H(32D)	4e	0.25	1.3474	1.0125	0.3227	0.045
H(32E)	4e	0.25	1.3104	0.9337	0.3264	0.045
H(32F)	4e	0.25	1.4164	0.9646	0.3824	0.045
C(33B)	4e	0.25	0.779(2)	0.9585(8)	0.3334(9)	0.0303
C(34B)	4e	0.25	0.769(1)	1.0193(6)	0.3826(6)	0.0303
H(34D)	4e	0.25	0.7931	1.0045	0.4305	0.045
H(34E)	4e	0.25	0.6846	1.0358	0.3764	0.045
H(34F)	4e	0.25	0.823	1.0562	0.372	0.045
C(35B)	4e	0.25	0.742(2)	0.9814(9)	0.2607(9)	0.0303
H(35D)	4e	0.25	0.7973	1.0178	0.2501	0.045
H(35E)	4e	0.25	0.6584	0.9987	0.2549	0.045
H(35F)	4e	0.25	0.747	0.9428	0.2294	0.045
C(36B)	4e	0.25	0.692(1)	0.9032(7)	0.3509(7)	0.0303
H(36D)	4e	0.25	0.7172	0.8876	0.3985	0.045
H(36E)	4e	0.25	0.6932	0.8646	0.3191	0.045
H(36F)	4e	0.25	0.6088	0.9217	0.3464	0.045
C(37B)	4e	0.25	0.980(1)	0.8628(6)	0.280(1)	0.0303
C(38B)	4e	0.25	0.873(1)	0.8118(5)	0.2590(6)	0.0303
H(38D)	4e	0.25	0.7963	0.8373	0.2471	0.045
H(38E)	4e	0.25	0.866	0.7812	0.2979	0.045
H(38F)	4e	0.25	0.8879	0.7848	0.2191	0.045
C(39B)	4e	0.25	0.994(1)	0.8997(6)	0.2108(6)	0.0303
H(39D)	4e	0.25	0.9224	0.9287	0.1966	0.045
H(39E)	4e	0.25	1.0005	0.8657	0.1748	0.045
H(39F)	4e	0.25	1.0676	0.9281	0.2177	0.045
C(40B)	4e	0.25	1.096(1)	0.8190(5)	0.2966(6)	0.0303
H(40D)	4e	0.25	1.0928	0.7931	0.3391	0.045
H(40E)	4e	0.25	1.1681	0.8487	0.303	0.045
H(40F)	4e	0.25	1.1015	0.7872	0.2585	0.045

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	4e		0.36588(6)	0.86096(3)	0.04365(4)	0.0226(3)	0.0215(3)	0.0236(4)	-0.0020(3)	0.0027(3)	-0.0016(3)
Si(2)	4e		0.51279(6)	0.85497(3)	-0.03830(4)	0.0200(3)	0.0224(3)	0.0237(4)	-0.0013(2)	0.0025(3)	0.0012(3)
C(1)	4e		0.4696(2)	1.0550(1)	0.0647(1)	0.025(1)	0.029(1)	0.027(2)	-0.001(1)	0.005(1)	-0.003(1)
C(2)	4e		0.4492(2)	0.9990(1)	0.0838(1)	0.020(1)	0.032(1)	0.018(1)	-0.0001(9)	0.004(1)	-0.004(1)
C(3)	4e		0.4216(2)	0.9318(1)	0.1088(1)	0.022(1)	0.029(1)	0.028(2)	-0.003(1)	0.005(1)	0.000(1)
C(4)	4e		0.4264(3)	0.9259(2)	0.1771(2)	0.051(2)	0.040(2)	0.029(2)	-0.013(1)	0.008(1)	-0.001(1)
C(5)	4e		0.2078(2)	0.8948(1)	0.0005(2)	0.021(1)	0.034(1)	0.033(2)	-0.004(1)	0.002(1)	-0.003(1)
C(6)	4e		0.1260(3)	0.8390(2)	-0.0374(2)	0.031(1)	0.051(2)	0.043(2)	-0.016(1)	0.002(1)	-0.006(1)
C(7)	4e		0.2257(3)	0.9514(2)	-0.0510(2)	0.029(1)	0.036(2)	0.040(2)	0.002(1)	-0.002(1)	0.001(1)
C(8)	4e		0.1381(3)	0.9278(2)	0.0548(2)	0.030(1)	0.052(2)	0.051(2)	0.005(1)	0.009(1)	-0.009(2)
C(9)	4e		0.3496(3)	0.7754(2)	0.0944(2)	0.046(2)	0.033(2)	0.033(2)	-0.008(1)	0.011(1)	0.003(1)
C(10)	4e		0.2424(3)	0.7786(2)	0.1364(2)	0.054(2)	0.051(2)	0.045(2)	-0.018(2)	0.020(2)	0.006(2)
C(11)	4e		0.4633(3)	0.7554(2)	0.1440(2)	0.063(2)	0.040(2)	0.044(2)	0.001(2)	0.011(2)	0.017(1)
C(12)	4e		0.3233(4)	0.7160(2)	0.0429(2)	0.079(2)	0.027(2)	0.054(2)	-0.013(2)	0.016(2)	0.003(1)
C(13)	4e		0.4683(3)	0.8074(1)	-0.1248(2)	0.036(1)	0.034(1)	0.030(2)	-0.002(1)	0.007(1)	-0.009(1)
C(14)	4e		0.3614(3)	0.8426(2)	-0.1701(2)	0.051(2)	0.052(2)	0.030(2)	0.002(2)	-0.002(2)	-0.010(1)
C(15)	4e		0.4330(3)	0.7328(2)	-0.1179(2)	0.059(2)	0.038(2)	0.047(2)	-0.005(2)	0.008(2)	-0.011(1)
C(16)	4e		0.5772(3)	0.8103(2)	-0.1647(2)	0.050(2)	0.059(2)	0.038(2)	-0.006(2)	0.018(2)	-0.016(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	4e		0.6738(2)	0.8310(1)	0.0080(2)	0.023(1)	0.030(1)	0.038(2)	0.005(1)	-0.000(1)	0.001(1)
C(18)	4e		0.6918(3)	0.8623(2)	0.0812(2)	0.029(1)	0.051(2)	0.041(2)	0.001(1)	-0.005(1)	-0.003(1)
C(19)	4e		0.6900(3)	0.7532(2)	0.0145(2)	0.041(2)	0.038(2)	0.064(2)	0.015(1)	0.007(2)	0.010(2)
C(20)	4e		0.7761(3)	0.8595(2)	-0.0287(2)	0.024(1)	0.052(2)	0.056(2)	0.000(1)	0.008(1)	-0.001(2)
C(21)	4e	0.75	1.0504(3)	1.0707(2)	0.5534(2)	0.021(2)	0.019(2)	0.023(2)	-0.001(1)	0.005(1)	-0.005(1)
C(22)	4e	0.75	1.0499(9)	1.0850(5)	0.4932(4)	0.015(2)	0.012(1)	0.021(5)	0.001(2)	0.001(3)	-0.003(3)
C(23)	4e	0.75	1.0547(3)	1.1051(2)	0.4232(2)	0.015(1)	0.020(2)	0.023(2)	0.000(1)	0.003(1)	-0.001(1)
C(24)	4e	0.75	1.0309(3)	1.1713(2)	0.4094(2)	0.034(2)	0.022(2)	0.027(2)	0.004(1)	0.005(2)	0.001(1)
Si(3)	4e	0.75	1.1013(1)	1.03989(5)	0.35838(6)	0.0184(4)	0.0199(6)	0.0178(6)	0.0014(5)	0.0063(4)	-0.0017(5)
Si(4)	4e	0.75	0.9500(1)	0.94805(5)	0.35431(6)	0.0179(4)	0.0194(5)	0.0163(5)	0.0017(4)	0.0025(3)	-0.0023(4)
C(25)	4e	0.75	1.0965(5)	1.0853(4)	0.2713(6)	0.032(4)	0.029(2)	0.141(6)	-0.005(3)	0.003(4)	0.038(3)
C(26)	4e	0.75	0.988(2)	1.1213(9)	0.242(1)	0.097(5)	0.073(4)	0.061(5)	-0.006(3)	-0.001(4)	0.022(3)
C(27)	4e	0.75	1.128(1)	1.0357(7)	0.2168(7)	0.168(8)	0.097(6)	0.045(4)	0.002(5)	0.049(5)	0.011(4)
C(28)	4e	0.75	1.2112(4)	1.1374(2)	0.2766(2)	0.041(2)	0.040(2)	0.048(3)	-0.003(2)	0.026(2)	0.012(2)
C(29)	4e	0.75	1.2635(3)	1.0119(2)	0.4021(2)	0.017(2)	0.027(2)	0.035(2)	0.003(2)	0.008(2)	-0.002(2)
C(30)	4e	0.75	1.2540(3)	0.9625(2)	0.4616(2)	0.023(2)	0.029(2)	0.031(2)	0.007(1)	0.002(2)	-0.002(2)
C(31)	4e	0.75	1.3367(3)	1.0751(2)	0.4335(2)	0.024(2)	0.031(2)	0.056(3)	-0.004(2)	-0.000(2)	0.001(2)
C(32)	4e	0.75	1.332(1)	0.9757(7)	0.3435(8)	0.047(4)	0.065(3)	0.12(1)	0.028(3)	0.052(5)	0.022(5)
C(33)	4e	0.75	0.7862(4)	0.9791(2)	0.3205(2)	0.018(2)	0.030(2)	0.026(2)	-0.001(2)	0.001(2)	-0.002(2)
C(34)	4e	0.75	0.7705(3)	1.0537(2)	0.3439(2)	0.024(2)	0.034(2)	0.036(2)	0.010(1)	0.004(2)	-0.001(2)
C(35)	4e	0.75	0.7553(5)	0.9746(3)	0.2410(2)	0.032(2)	0.047(2)	0.026(3)	0.003(2)	-0.005(2)	-0.004(2)
C(36)	4e	0.75	0.6904(3)	0.9375(2)	0.3522(2)	0.022(2)	0.039(2)	0.049(3)	-0.004(2)	0.007(2)	-0.007(2)
C(37)	4e	0.75	0.9850(3)	0.8608(2)	0.3152(2)	0.035(2)	0.025(2)	0.024(2)	0.003(1)	0.003(2)	-0.007(2)
C(38)	4e	0.75	0.8788(4)	0.8119(2)	0.3182(3)	0.053(2)	0.024(2)	0.066(3)	-0.007(2)	0.012(2)	-0.018(2)
C(39)	4e	0.75	1.0089(4)	0.8650(2)	0.2396(2)	0.057(3)	0.038(2)	0.031(3)	0.012(2)	0.008(2)	-0.011(2)
C(40)	4e	0.75	1.0980(4)	0.8277(2)	0.3564(2)	0.048(2)	0.023(2)	0.045(3)	0.011(2)	0.000(2)	-0.007(2)

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