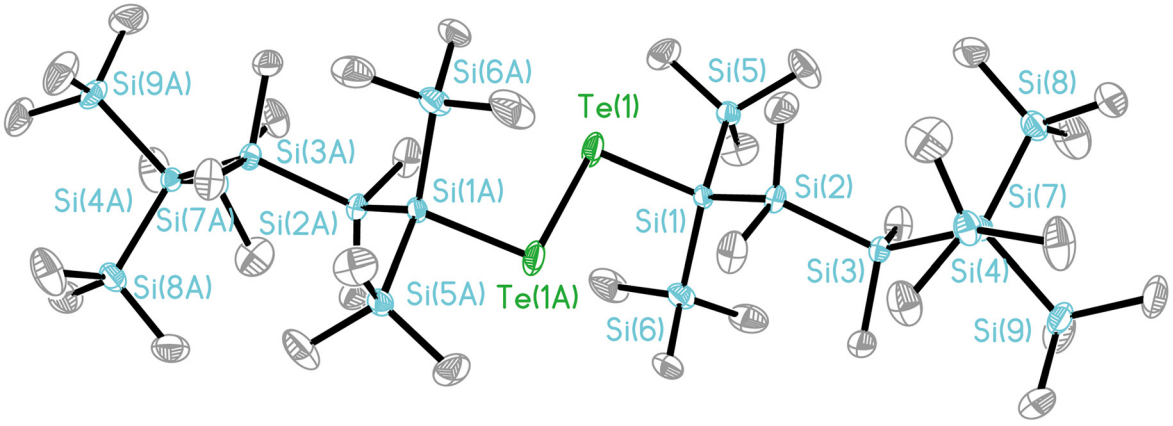
Fan Qi and Xu-Qiong Xiao*

Crystal structure of 1,2-bis(2,2,3,3,5,5,5-heptamethyl-1,1,4,4-tetrakis(trimethylsilyl)pentasilan-1-yl)ditellane, $C_{38}H_{114}Si_{18}Te_2$



https://doi.org/10.1515/ncrs-2022-0216
Received April 26, 2022; accepted May 12, 2022;
published online May 25, 2022

Abstract

$C_{38}H_{114}Si_{18}Te_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.0857(18) Å, b = 9.7412(19) Å, c = 24.213(5) Å, α = 94.25(3)°, β = 95.26(3)°, γ = 117.58(3)°, V = 1874.9(6) Å^3, Z = 1, $R_{gt}(F)$ = 0.0425, $wR_{ref}(F^2)$ = 0.1580, T = 296(2) K.

CCDC no.: 2168716

The molecular structure 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:	Green prism
Size:	0.20 × 0.15 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω
θ_{max}, completeness:	27.6°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23686, 8586, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5474
$N(param)_{refined}$:	281
Programs:	Bruker [1, 2], SHELX [3–5]

Source of material

In a representative experiment, the mixture of 1,1,1,3,3,4,4,6,6,6-decamethyl -2,2,5,5-tetrakis(trimethylsilyl) hexasilane [6–9] (2.00 g, 3.3 mmol) and potassium *tert*-butoxide (0.37 g, 3.3 mmol) was refluxed in 10 mL THF for 6 h to afford the anions of the aforementioned hexasilane starting material. After removal of the solvent in a vacuum, 10 mL toluene and tellurium (0.50 g, 7.9 mmol) were added, stirred for 3 h and refluxed overnight. Then the solvent was removed and *n*-hexane was added to extract the residue, which was purified by column chromatography. Green crystals were obtained by cooling the hexane solution of the title compound.

***Corresponding author: Xu-Qiong Xiao**, College of Material, Chemistry and Chemical Engineering, Key Laboratory of Organosilicon Chemistry and Material Technology of Ministry of Education, Hangzhou Normal University, No. 2318 Yuhangtang Rd., Hangzhou 311121, Zhejiang, China, E-mail: xqxiao@hznu.edu.cn. https://orcid.org/0000-0001-8424-9898
Fan Qi, College of Material, Chemistry and Chemical Engineering, Key Laboratory of Organosilicon Chemistry and Material Technology of Ministry of Education, Hangzhou Normal University, No. 2318 Yuhangtang Rd., Hangzhou 311121, Zhejiang, China

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

Atom	x	y	z	U_{iso}*/U_{eq}
Te1	0.51989 (6)	−0.11837 (4)	0.52132 (2)	0.07786 (18)
Si1	0.39333 (14)	−0.13595 (12)	0.61149 (4)	0.0361 (2)
Si2	0.59653 (14)	0.07771 (12)	0.67563 (4)	0.0396 (3)
Si3	0.49110 (14)	0.09054 (12)	0.76060 (4)	0.0384 (3)
Si4	0.68479 (13)	0.23052 (12)	0.84232 (4)	0.0362 (2)
Si5	0.37083 (16)	−0.38183 (13)	0.62935 (5)	0.0442 (3)
Si6	0.12892 (18)	−0.14708 (15)	0.58945 (6)	0.0565 (3)
Si7	0.91337 (17)	0.46644 (14)	0.82817 (5)	0.0517 (3)
Si8	0.78255 (19)	0.07550 (16)	0.88599 (5)	0.0573 (3)
Si9	0.53729 (18)	0.2943 (2)	0.90554 (6)	0.0652 (4)
C1	0.7925 (6)	0.0573 (7)	0.6821 (2)	0.0727 (15)
H1A	0.825417	0.052243	0.645788	0.109*
H1B	0.771760	−0.036541	0.697833	0.109*
H1C	0.880533	0.145816	0.706063	0.109*
C2	0.6448 (8)	0.2606 (5)	0.6435 (2)	0.0748 (17)
H2A	0.753791	0.341776	0.660051	0.112*
H2B	0.562043	0.292848	0.650018	0.112*
H2C	0.643246	0.240918	0.603944	0.112*
C3	0.3539 (7)	−0.1130 (6)	0.7766 (2)	0.0683 (15)
H3A	0.304018	−0.107441	0.809395	0.102*
H3B	0.420607	−0.164650	0.783044	0.102*
H3C	0.267231	−0.170559	0.745492	0.102*
C4	0.3505 (7)	0.1826 (7)	0.7460 (2)	0.0665 (14)
H4A	0.267362	0.122793	0.714197	0.100*
H4B	0.416804	0.287569	0.738270	0.100*
H4C	0.296206	0.184100	0.778023	0.100*
C5	1.0755 (8)	0.4317 (8)	0.7984 (3)	0.100 (2)
H5A	1.177473	0.528675	0.801640	0.150*
H5B	1.037316	0.389258	0.759607	0.150*
H5C	1.095802	0.359275	0.818352	0.150*
C6	1.0144 (9)	0.6005 (7)	0.8949 (2)	0.096 (2)
H6A	1.114225	0.690250	0.888578	0.144*
H6B	1.043030	0.547036	0.922217	0.144*
H6C	0.937987	0.633366	0.908321	0.144*
C7	0.8445 (9)	0.5725 (6)	0.7798 (3)	0.0855 (18)
H7A	0.761553	0.593065	0.794919	0.128*
H7B	0.797198	0.509562	0.743956	0.128*
H7C	0.939193	0.669511	0.775533	0.128*
C8	0.9601 (8)	0.1991 (8)	0.9430 (3)	0.0892 (19)
H8A	0.938118	0.276712	0.961651	0.134*
H8B	1.062317	0.249602	0.927138	0.134*
H8C	0.971006	0.134414	0.969387	0.134*
C9	0.8613 (9)	−0.0221 (8)	0.8353 (3)	0.0899 (19)
H9A	0.861671	−0.110909	0.849873	0.135*
H9B	0.973253	0.050583	0.830039	0.135*
H9C	0.789228	−0.055653	0.800040	0.135*
C10	0.6193 (11)	−0.0779 (9)	0.9202 (3)	0.118 (3)
H10A	0.667830	−0.132141	0.940140	0.177*
H10B	0.529245	−0.150606	0.892177	0.177*
H10C	0.576833	−0.029901	0.945844	0.177*
C11	0.5002 (11)	0.4587 (10)	0.8850 (3)	0.116 (3)
H11A	0.433103	0.477259	0.909918	0.174*
H11B	0.442616	0.431659	0.847379	0.174*
H11C	0.605853	0.551531	0.887137	0.174*
C12	0.6573 (8)	0.3577 (9)	0.9783 (2)	0.095 (2)
H12A	0.763685	0.447950	0.978226	0.143*
H12B	0.674990	0.274373	0.990675	0.143*
H12C	0.594395	0.382969	1.003193	0.143*
C13	0.3300 (8)	0.1205 (10)	0.9096 (3)	0.106 (3)
H13A	0.276144	0.146826	0.937798	0.158*
H13B	0.347937	0.034920	0.919128	0.158*
H13C	0.260017	0.090949	0.874087	0.158*
C14	0.5039 (10)	−0.3652 (7)	0.6963 (3)	0.094 (2)
H14A	0.487486	−0.467049	0.702815	0.141*
H14B	0.472324	−0.320933	0.726875	0.141*
H14C	0.619942	−0.299082	0.693490	0.141*
C15	0.4401 (9)	−0.4663 (6)	0.5728 (3)	0.0810 (18)
H15A	0.559001	−0.404749	0.573654	0.121*
H15B	0.383428	−0.466417	0.537283	0.121*
H15C	0.413708	−0.571479	0.578038	0.121*
C16	0.1495 (8)	−0.5224 (6)	0.6348 (3)	0.0848 (18)
H16A	0.077452	−0.525150	0.602727	0.127*
H16B	0.117872	−0.489995	0.668146	0.127*
H16C	0.139051	−0.624680	0.636488	0.127*
C17	−0.0054 (7)	−0.2254 (8)	0.6452 (3)	0.0879 (19)
H17A	−0.116874	−0.242975	0.632706	0.132*
H17B	0.040710	−0.151288	0.678561	0.132*
H17C	−0.009128	−0.322095	0.652870	0.132*
C18	0.0234 (10)	−0.2761 (8)	0.5224 (3)	0.110 (3)
H18A	−0.017830	−0.383180	0.528627	0.164*
H18B	0.102046	−0.251896	0.496126	0.164*
H18C	−0.068577	−0.260256	0.507848	0.164*
C19	0.1500 (8)	0.0513 (6)	0.5818 (2)	0.0709 (15)
H19A	0.226487	0.098778	0.555776	0.106*
H19B	0.191965	0.115409	0.617531	0.106*
H19C	0.042237	0.041054	0.568315	0.106*

Experimental details

A suitable crystal was selected and mounted onto the tip of a glass fibre. The diffraction data were collected on a Bruker Apex II CCD diffractometer at 296 K. An empirical (multi-scan) absorption correction was applied with the program SADABS [4]. The structure was solved with the ShelXT structure solution program and subsequently refined on F^2 (SHELXL-2018) [5]. Coordinates Hydrogen atoms were refined with constraints or restraints. Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms. Figures of the solid-state molecular structures were generated using XP as implemented in Shelxtl program [2].

Comment

Silyl anions are important reactive intermediates not only in organic synthesis but also in organosilicon chemistry [6–8].

only a few studies have concentrated on its reaction with chalcogenic elements. On the other hand, disilylchal cogenides such as $(Me_3Si)_2E$ (E = S, Se, Te) have been used as chalcogen transfer reagents in the preparation of nanosized copper chalcogenide clusters. The synthetic routes towards disilylchalcogenides have been developed. However, these routes utilize the unpleasant H_2S or need *in situ* synthesis of Na_2Se or Li_2Te. We have reported the reaction of disilyl dianions with chalcogenide elements and obtained a series of cyclic disilyl-chal cogenides. In the course of our investigations, the titled compound was obtained.

The title compound crystallizes in the triclinic space group $P\bar{1}$. The main chain of the molecules is of zig-zag type. The Te–Te bond distance is 2.7518(9) Å. The Te–Si bond length is 2.5400(12) Å. The bond lengths of Si–Si bonds in the title compound range from 2.344 to 2.384 Å, with the average bond length of 2.362 Å, which are comparable with those of the oligosilanes or polysilanes [9–11]. The average Si–C distance of the Si–C bonds is 1.871 Å. The bond angle of Si(1)–Te(1)–Te(1A) is 102.04(4)°. The Si–Si–Si bond angle range from 97.4° to 118.5°, with the average value of 109.99°. The title compound might be a good chalcogen transfer reagents.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was funded by Natural Science Foundation of Zhejiang Province (Grant No. LY22B010002).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Bruker. SHELXTL Version 6.22 Program for Solution and Refinement of Crystal Structures; Bruker AXS Inc.: Madison, WI, USA, 2001.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. *Program for Empirical Absorption Correction of Area Detector Data (SADABS)*; University of Göttingen: Göttingen, Germany, 1994.
5. Sheldrick G. M. A short history of *SHELX*. *Acta Crystallogr.* 2008, *A64*, 112–122.
6. Xiao X. Q., Zhao H., Xu Z., Lai G. Q., He X. L., Li Z. F. Reduction of the dihalocyclopentasilanes. A quest for a homocyclic silylene. *Chem. Commun.* 2013, *49*, 2706–2708.
7. Luo J., Yan E. H., Zhao H., Xiao X. Q., Li Z. F. Synthesis, structure and reactivity of sterically demanding oligosilanylmagnesium compounds. *Polyhedron* 2015, *102*, 233–238.
8. Fischer R., Frank D., Gaderbauer W., Kayser C., Mechtler C., Baumgartner J., Marschner C. α,ω–Oligosilyl dianions and their application in the synthesis of homo- and heterocyclosilanes. *Organometallics* 2003, *22*, 3723–3731.
9. Zhao H., Li J. X., Xiao X. Q., Kira M., Li Z. F., Müller T. Cation-triggered stannate(II)/stannylenoid/stannylene conversion. *Chem. Eur J.* 2018, *24*, 5967–5973.
10. Xiao X. Q., Xu J. T., Wu Y., Wang Q. Y. 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, $C_{28}H_{78}Si_{10}$. *Z. Kristallogr. N. Cryst. Struct.* 2019, *234*, 1195–1198.
11. Xiao X.-Q., Zhao H., Luo J., Xu Z., Lai G.-Q., Li Z. Synthesis and characterization of heterocyclic disilylchalcogenides. *Dalton Trans.* 2013, *42*, 3994–4001.