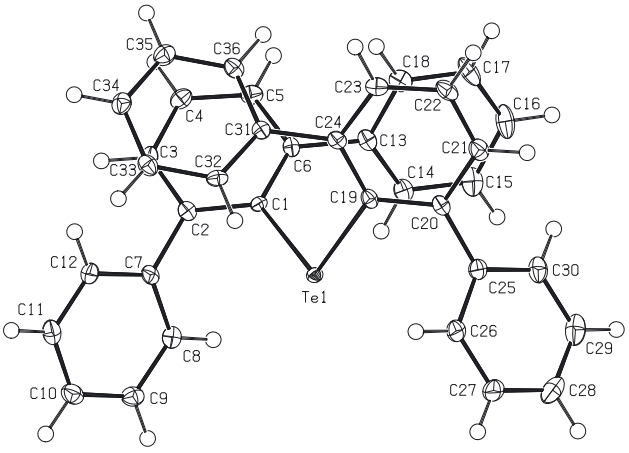
Yuga Shibuya, Shinichi Koguchi and Makoto Oba*

Crystal structure of di([1,1′:3′,1″-terphenyl]-2′-yl) tellane, $C_{36}H_{26}Te$



https://doi.org/10.1515/ncrs-2022-0367
Received July 19, 2022; accepted August 19, 2022;
published online September 2, 2022

Abstract

$C_{36}H_{26}Te$, orthorhombic, $Pca2_1$ (no. 29), a = 13.5004(4) Å, b = 13.5677(3) Å, c = 14.2006(4) Å, V = 2601.12(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0247, $wR_{ref}(F^2)$ = 0.0530, T = 93 K.

CCDC no.: 2202383

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.

Source of materials

Tellurium powder (7.20 g, 56.4 mmol) was added to a solution of 2,6-diphenylphenyl magnesium iodide prepared from Mg (1.36 g, 55.9 mmol) and the corresponding iodide (19.8 g, 55.6 mmol) in THF (70 ml) under an argon atmosphere. The mixture was heated under reflux for 22 h. After cooling, the reaction mixture was poured over crushed ice containing concentrated HCl, stirred for 4 h, and extracted using benzene. The organic layer was washed with brine, dried over $MgSO_4$, and concentrated. The residue was triturated with CH_2Cl_2—EtOH to afford crude bis(2,6-diphenylphenyl) ditelluride (5.17 g) as a dark red solid. The resulting ditelluride was dissolved in toluene (20 ml) and heated under reflux for two days in the presence of copper powder (1.03 g, 16.2 mmol). The insoluble materials were removed by filtration, and the solution was concentrated. Recrystallization of the residue from hexane gave the title compound (3.95 g, 6.74 mmol, 24%) as a pale-yellow solid.

Table 1: Data collection and handling.

Crystal:	Light yellow block
Size:	0.43 × 0.28 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.17 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω-scans
θ_{max}, completeness:	29.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	28145, 6283, 0.047
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 5622
$N(param)_{refined}$:	334
Programs:	CyrsAlis^{PRO} [1], SHELX [2, 3], OLEX2 [4]

Experimental details

The H atoms were placed at calculated positions and refined using a riding model with C—H bond distances of aromatic 0.950 Å. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

Comment

Diorganotellurides are a principal class of organotellurium compounds that can be converted to various functionalized tellurium compounds [5]. Particularly, tellurides with bulky substituents are useful as precursors for kinetically stabilized tellurium oxides [6–8]. The 2,6-diphenylphenyl (*m*-terphenyl) group is considered a promising substituent that can cover highly reactive tellurium atoms [9]. However, no synthetic examples of tellurides with a *m*-terphenyl group have been reported, except for a previous study [10]. This

***Corresponding author: Makoto Oba,** Department of Bioengineering, Tokai University, 4-1-1 Kitakaname, Hiratsuka, Kanagawa 259-1292, Japan, E-mail: moba@tokai-u.jp. https://orcid.org/0000-0001-8261-9234
Yuga Shibuya and Shinichi Koguchi, Department of Chemistry, Tokai University, 4-1-1 Kitakaname, Hiratsuka, Kanagawa 259-1292, Japan

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Te1	0.58226 (2)	0.64359 (2)	0.51097 (2)	0.01086 (6)
C24	0.4483 (3)	0.7893 (3)	0.6244 (3)	0.0121 (8)
C5	0.3818 (3)	0.8108 (3)	0.3265 (2)	0.0148 (7)
H6	0.372813	0.878028	0.309718	0.018*
C32	0.3509 (3)	0.6293 (2)	0.6179 (2)	0.0137 (7)
H32	0.404776	0.595864	0.646837	0.016*
C20	0.6259 (3)	0.8200 (3)	0.6412 (3)	0.0136 (8)
C27	0.8566 (3)	0.6665 (3)	0.6739 (3)	0.0215 (8)
H27	0.873670	0.602098	0.694440	0.026*
C36	0.2787 (3)	0.7770 (3)	0.5553 (2)	0.0155 (7)
H36	0.282855	0.845314	0.541157	0.019*
C3	0.3286 (3)	0.6426 (3)	0.3182 (2)	0.0151 (7)
H4	0.282708	0.594712	0.296195	0.018*
C14	0.6360 (4)	0.8510 (3)	0.3712 (3)	0.0161 (9)
H19	0.656499	0.789633	0.345630	0.019*
C35	0.1924 (3)	0.7256 (3)	0.5341 (2)	0.0184 (8)
H35	0.138239	0.758613	0.505271	0.022*
C30	0.8060 (3)	0.8532 (3)	0.6114 (3)	0.0201 (8)
H30	0.789516	0.917259	0.589478	0.024*
C2	0.4104 (3)	0.6123 (3)	0.3707 (2)	0.0120 (7)
C7	0.4281 (3)	0.5045 (3)	0.3801 (2)	0.0130 (7)
C28	0.9299 (3)	0.7310 (4)	0.6483 (3)	0.0263 (11)
H28	0.997371	0.711602	0.652074	0.032*
C8	0.5195 (3)	0.4638 (3)	0.3544 (2)	0.0154 (7)
H13	0.572932	0.505912	0.337934	0.019*
C4	0.3129 (3)	0.7413 (3)	0.2977 (2)	0.0164 (7)
H5	0.255452	0.761174	0.264040	0.020*
C15	0.7040 (3)	0.9275 (3)	0.3794 (2)	0.0215 (8)
H18	0.770846	0.918085	0.360384	0.026*
C11	0.3653 (3)	0.3391 (3)	0.4057 (3)	0.0189 (8)
H10	0.312830	0.296892	0.424687	0.023*
C12	0.3515 (3)	0.4405 (3)	0.4060 (2)	0.0160 (7)
H9	0.289114	0.466773	0.423973	0.019*
C31	0.3594 (3)	0.7294 (2)	0.5970 (2)	0.0118 (7)
C34	0.1855 (3)	0.6263 (3)	0.5552 (3)	0.0171 (7)
H34	0.126447	0.591169	0.541024	0.021*
C25	0.7306 (3)	0.7884 (3)	0.6378 (2)	0.0153 (7)
C21	0.6031 (3)	0.9086 (3)	0.6875 (2)	0.0172 (8)
H21	0.655427	0.949580	0.709174	0.021*
C18	0.5098 (3)	0.9530 (3)	0.4380 (2)	0.0169 (8)
H15	0.443673	0.961635	0.459209	0.020*
C16	0.6737 (3)	1.0183 (3)	0.4156 (3)	0.0249 (9)
H17	0.719269	1.071417	0.419830	0.030*
C23	0.4296 (3)	0.8773 (3)	0.6732 (3)	0.0151 (7)
H23	0.363159	0.895634	0.686421	0.018*
C22	0.5056 (3)	0.9377 (3)	0.7022 (2)	0.0173 (8)
H22	0.491593	0.998786	0.732115	0.021*
C26	0.7573 (3)	0.6941 (3)	0.6702 (2)	0.0169 (7)
H26	0.707424	0.649068	0.689538	0.020*
C1	0.4760 (2)	0.6846 (2)	0.4062 (2)	0.0103 (6)
C9	0.5328 (3)	0.3612 (3)	0.3529 (3)	0.0174 (8)
H12	0.594974	0.334464	0.334722	0.021*
C33	0.2648 (3)	0.5780 (3)	0.5971 (2)	0.0163 (7)
H33	0.260087	0.509730	0.611394	0.020*
C29	0.9053 (3)	0.8250 (3)	0.6167 (3)	0.0250 (9)
H29	0.955895	0.869915	0.598804	0.030*
C13	0.5389 (3)	0.8632 (2)	0.3999 (2)	0.0137 (7)
C19	0.5471 (3)	0.7624 (2)	0.6054 (2)	0.0128 (7)
C10	0.4558 (3)	0.2986 (3)	0.3778 (3)	0.0194 (9)
H11	0.464517	0.229198	0.375810	0.023*
C17	0.5770 (3)	1.0304 (3)	0.4453 (3)	0.0222 (9)
H16	0.556377	1.091756	0.470860	0.027*
C6	0.4643 (3)	0.7835 (3)	0.3801 (3)	0.0115 (8)

paper reports the crystal structure of di([1,1′:3′,1″-terphenyl]-2′-yl)tellane for the first time.

The asymmetric unit of the title structure contains one molecule (see the figure). The Te1—C1 (2.140(3) Å) and Te1—C19 (2.150(3) Å) bonds are elongated slightly compared to those of the related bis(1-naphthyl) (2.119(2) Å) [11] and bis(2-biphenyl) (average 2.115(5) Å) [12] tellurides. They are comparable to those of the sterically more demanding mesityl (2.140(3) Å) [13] and supermesityl (average 2.157(5) Å) [14] derivatives. The *m*-terphenyl groups are not coplanar. The phenyl rings of both wings are twisted by an average of 51° in the same direction relative to the central ring, thereby avoiding steric hindrance with the tellurium atom.

Weak intramolecular π–π stacking interactions can be observed between the *m*-terphenyl central ring and the side ring of another terphenyl group. The centroid distances between C1–6 and C31–36 rings and between C13–18 and C19–24 rings are 3.651 Å and 3.879 Å, respectively. Owing to these through-space interactions, the C—Te—C angle (95.2(1)°) is much smaller than that observed in similarly crowded tellanes bearing mesityl (101.0(1)°) [13] and supermesityl (107.3(2)°) [14] substituents. Furthermore, the angle is slightly smaller than that of naphthyl (96.32(9)°) [11] and biphenyl (96.2(2)°) [12] derivatives. In the crystal lattice, the molecules form a two-dimensional layer parallel to the *ab*-plane with short intermolecular H—H and H—C contacts (H33···C27^{i} = 2.864 Å; H33···C26^{i} = 2.889 Å; H36···H17^{ii} = 2.232 Å; H35···H16^{ii} = 2.363 Å; H16···H29^{ii} = 2.326 Å; *i* = –1/2 + *x*, 1 – *y*, *z*; and *ii* = –1/2 + *x*, 2 – *y*, *z*). The two-dimensional layers stack along the *c* axis with a short intermolecular contact of 2.866 Å (H5···C31^{iii}; *iii* = 1/2 – *x*, *y*, –1/2 + *z*) to construct the three-dimensional crystal structure.

Acknowledgements: The authors wish to thank Enago (www.enago.jp) for the English language review.

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

Research funding: This research was supported in part by a doctoral dissertation grant from Tokai University.

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

References

1. Rigaku Oxford Diffraction. *CrysAlis^{PRO}*; Rigaku Corporation: Tokyo, Japan, 2015.
2. Sheldrick G. M. SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
5. Sadekov I. D., Rivkin B. B., Maksimenko A. A., Sadekova E. I. Synthesis and reactions of diorganyl tellurides. *Sulfur Rep.* 1995, *17*, 1–88.
6. Oba M., Okada Y., Nishiyama K., Shimada S., Ando W. Synthesis, characterization and oxidizing properties of a diorgano tellurone carrying bulky aromatic substituents. *Chem. Commun.* 2008, *42*, 5378–5380.
7. Shibuya Y., Koguchi S., Oba M. Bis(2,6-diisopropylphenyl) tellurone: a well-defined monomeric diorganotellurone without cocrystallized solvents and without stabilizing intramolecular contacts. *Acta Crystallogr.* 2022, *C78*, 88–93.
8. Taka H., Yamazaki Y., Shimizu T., Kamigata N. Isolation of stable enantiomerically pure telluroxides and their stereochemistry. *J. Org. Chem.* 2000, *65*, 2127–2133.
9. Beckmann J., Finke P., Hesse M., Wettig B. Well-defined stibonic and tellurinic acids. *Angew. Chem. Int. Ed.* 2008, *47*, 9982–9984.
10. Oba M., Okada Y., Endo M., Tanaka K., Nishiyama K., Shimada S., Ando W. Formation of diaryl telluroxides and tellurones by photosensitized oxygenation of diaryl tellurides. *Inorg. Chem.* 2010, *49*, 10680–10686.
11. Chauhan A. K. S., Anamika, Srivastava R. C., Duthie A. Bis(1-naphthyl) telluride. *Acta Crystallogr.* 2007, *E63*, o4386.
12. Chen H., Hamor T. A., Singh K., McWhinnie W. R. Bis(2-biphenyl) telluride. *Acta Crystallogr.* 1996, *C52*, 711–713.
13. Klapötke T. M., Krumm B., Mayer P., Polborn K., Schwab I. Some investigations on organotellurium(IV) cyanides. *Z. Anorg. Allg. Chem.* 2005, *631*, 2677–2682.
14. Wieber M., Habersack R. Ortho- and para-methyl-substituted triphenyltelluronium halides. Crystal structure of trimesityltelluronium iodide and bis(2,4,6-tri-tert-butylphenyl) tellane. *Phosphorus, Sulfur, Silicon Relat. Elem.* 1995, *106*, 233–241.