

Ungaro, R., Pochini, A., Andreotti, G. D. & Sangermano, V. (1984). *J. Chem. Soc. Perkin Trans. 2*, pp. 1979–1985.
Zsolnai, L. (1996). *ZORTEP. An Interactive ORTEP Program*. University of Heidelberg, Germany.

Acta Cryst. (1998). **C54**, 1010–1011

Tris(phenylseleno)methane

ERNESTO SCHULZ LANG,^a JULIANA PRADELLA ZIANI^a AND ULRICH ABRAM^b

^aUniversidade Federal de Santa Maria, Departamento de Química, 97.111 Santa Maria-RS, Brazil, and ^bTechnische Universität Dresden, Institut für Analytische Chemie, D-01062 Dresden, Germany. E-mail: abram@fz-rossendorf.de

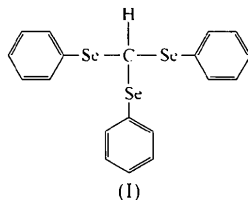
(Received 10 December 1997; accepted 16 January 1998)

Abstract

Tris(phenylselenanyl)methane, $\text{C}_{19}\text{H}_{16}\text{Se}_3$, crystallizes in the rhombohedral space group $R\bar{3}$, with the CH group situated on a threefold axis. The selenium–carbon bond to the aromatic ring is slightly shorter than that to the aliphatic C atom. The tetrahedral angles at the methane C atom range between $107.4(1)$ and $111.5(1)^\circ$.

Comment

As part of our systematic work on selenium and tellurium compounds with the potential to coordinate to metal ions, we studied the structure of tris(phenylseleno)methane, (I), which may act as a tridentate bridging selenoether ligand. The title compound is the first representative of tris(alkylseleno) or tris(arylseleno) compounds to have been studied crystallographically.



The molecular structure of tris(phenylseleno)methane is illustrated in Fig. 1. Atoms C1 and H1 are situated on a threefold axis, producing three symmetry-related phenylseleno units. C—Se bond lengths of $1.928(3)$ and $1.958(1)$ Å have been found. The shorter one belongs to the bond to the aromatic C11 atom. This is in agreement with the bonding situation in other asymmetric selenoethers which have both an aliphatic and an aromatic substituent (Dupont *et al.*, 1990; Fong

et al., 1996). The C—Se—C angles of $101.4(1)^\circ$ have the expected magnitude, due to the repulsion of the electron pairs. The torsion angle H1—C1—Se—C11 is $-47.67(8)^\circ$, and the angle between the phenyl ring and the plane which is formed by H1, C1 and Se has been found to be $80.53(7)^\circ$. This obviously results in slight sterical hindrance, and has consequences for the tetrahedral angles at C1. The Se—C—Se angles are slightly smaller than, and the H—C—Se angles slightly greater than, 109.5° . This is in contrast to the situation in the comparable sulfur compounds tris(ethylsulfonyl)methane (McGregor & Speakman, 1969) and methylidynetrisioacetic acid (Cannone *et al.*, 1980), where the S—C—S angles almost fit the ideal tetrahedral angle. S—C—S angles greater than 109.5° have been found for tripotassium methanetrissulfonate hydrate (Hall *et al.*, 1980).

At 3.73 Å, the Se···Se distances are too long to suggest bonding interactions.

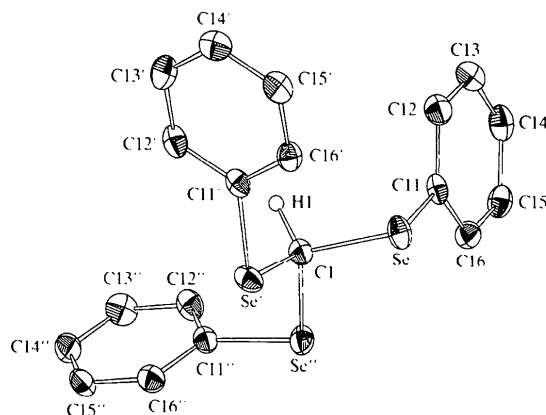


Fig. 1. ZORTEP (Zsolnai, 1997) diagram of $(\text{PhSe})_3\text{CH}$ showing 30% probability displacement ellipsoids. Phenyl H atoms have been omitted for clarity.

Experimental

Tris(phenylseleno)methane was prepared according to the literature procedure of Syper & Młochowski (1984) and was characterized by elemental analysis (found: C 48.12, H 3.28%; $\text{C}_{19}\text{H}_{16}\text{Se}_3$ requires: C 47.38, H 3.33%), IR, ^1H and ^{13}C NMR spectroscopy, and FAB mass spectrometry. The spectroscopic data obtained are in agreement with values published previously.

Crystal data

$\text{C}_{19}\text{H}_{16}\text{Se}_3$
 $M_r = 481.20$
Trigonal
 $R\bar{3}$
 $a = 12.5841(8)$ Å
 $c = 18.993(2)$ Å
 $V = 2604.7(4)$ Å³
 $Z = 6$
 $D_x = 1.841$ Mg m⁻³
 D_m , not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
 $\theta = 6.47$ – 15.20°
 $\mu = 6.348$ mm⁻¹
 $T = 203(2)$ K
Block
 $0.20 \times 0.20 \times 0.15$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: semi-empirical ψ scans (Spek, 1995b)
 $T_{\min} = 0.349$, $T_{\max} = 0.386$
 3030 measured reflections
 1534 independent reflections

1199 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\text{max}} = 28.95^\circ$
 $h = -12 \rightarrow 17$
 $k = -17 \rightarrow 13$
 $l = -13 \rightarrow 25$
 3 standard reflections every 200 reflections
 intensity decay: 1.05%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.067$
 $S = 1.073$
 1534 reflections
 89 parameters
 H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0318P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.334 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.836 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^*$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Se	0.16480 (2)	0.05801 (3)	0.162402 (15)	0.02664 (10)
C1	0	0	0.1246 (2)	0.0224 (9)
C11	0.2174 (2)	-0.0361 (2)	0.10738 (13)	0.0224 (5)
C12	0.2865 (3)	0.0158 (3)	0.04703 (15)	0.0280 (6)
C13	0.3339 (3)	-0.0461 (3)	0.00977 (16)	0.0316 (6)
C14	0.3112 (3)	-0.1600 (3)	0.03236 (15)	0.0299 (6)
C15	0.2413 (3)	-0.2124 (3)	0.09203 (14)	0.0272 (6)
C16	0.1938 (3)	-0.1511 (3)	0.12988 (14)	0.0253 (5)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Se—C11	1.928 (3)	C1—H1	1.10 (6)
Se—C1	1.9581 (16)		
C11—Se—C1	101.40 (11)	Se—C1—H1	111.49 (12)
Se—C1—Se'	107.38 (12)		

Symmetry code: (i) $-x + y, -x, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SET4* in *CAD-4 Software*. Data reduction: *HELENA* (Spek, 1995a). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL97*.

This work was supported by the Deutsche Forschungsgemeinschaft, the DAAD and the Fonds der Chemischen Industrie. We thank Professor Joachim Strähle, University of Tübingen, Germany, for his kind hospitality and for providing facilities.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1344). Services for accessing these data are described at the back of the journal.

References

- Cannone, J., Nowogrocki, G., Boivin, J.-C. & Thomas, D. (1980). *Acta Cryst.* **B36**, 2664–2667.
 Dupont, L., Dideberg, O. & Jacquemin, P. (1990). *Acta Cryst.* **C46**, 484–486.
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Fong, M. C., Gable, R. W. & Schiesser, C. H. (1996). *Acta Cryst.* **C52**, 1886–1888.
 Hall, J. R., Johnson, R. A., Kennard, C. H. L. & Smith, G. (1980). *J. Chem. Soc. Dalton Trans.* pp. 149–155.
 McGregor, D. R. & Speakman, J. C. (1969). *Acta Cryst.* **B25**, 540–546.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Spek, A. L. (1995a). *HELENA. Program for Reduction of CAD-4 Data*. Version of July 1995. University of Utrecht, The Netherlands.
 Spek, A. L. (1995b). *PLATON. Program for Calculations on X-ray Data*. Version of July 1995. University of Utrecht, The Netherlands.
 Syper, L. & Młochowski, J. (1984). *Synthesis*, pp. 439–442.
 Zsolnai, L. (1997). *ZORTEP. Program for the Presentation of Thermal Ellipsoids*. University of Heidelberg, Germany.

Acta Cryst. (1998). **C54**, 1011–1013

9-Methyl-8,11,12-trioxatricyclo[7.2.1.0^{2,7}]-dodeca-2,4,6-trien-10-one

FRANK EILERS,^a THORSTEN BACH^a AND ROLAND FRÖHLICH^b

^aFachbereich Chemie der Universität Marburg, Hans-Meerwein-Straße, D-35032 Marburg, Germany, and
^bOrganisch-Chemisches Institut, Universität Münster, Corrensstraße 40, D-48149 Münster, Germany. E-mail: frohlic@nwz.uni-muenster.de

(Received 27 November 1997; accepted 23 January 1998)

Abstract

9-Methyl-8,11,12-trioxatricyclo[7.2.1.0^{2,7}]dodeca-2,4,6-trien-10-one, $\text{C}_{10}\text{H}_8\text{O}_4$, was formed as an unexpected product of the reaction between salicylaldehyde and pyruvic acid. The main structural feature is the tricyclic system, which contains an O atom in each of the bridges.

Comment

The esterification of phenol and pyruvic acid is simple, according to the procedure of Hassner & Alexanian (1978), shown in reaction scheme (1).

