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1,4-Bis(4-chlorophenylseleno)-2,5-dimethoxybenzene

Henning Osholm Sørensen^{a*} and Nicolai Stuhr-Hansen^b

^aCenter for Fundamental Research: Metal Structures in Four Dimensions, Risø National Laboratory for Sustainable Energy, Technical University of Denmark, Frederiksborgvej 399, PO 49, DK-4000 Roskilde, Denmark, and ^bDepartment of Medicinal Chemistry, University of Copenhagen, Universitetsparken 2, DK-2100 Copenhagen, Denmark

Correspondence e-mail: henning.sorensen@risoe.dk

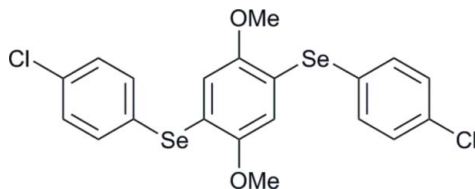
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Key indicators: single-crystal X-ray study; $T = 122$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.028; wR factor = 0.076; data-to-parameter ratio = 16.6.

The title compound, $\text{C}_{20}\text{H}_{16}\text{Cl}_2\text{O}_2\text{Se}_2$, utilizes the symmetry of the crystallographic inversion center. Molecular chains are formed through symmetric $\text{C}-\text{H}\cdots\text{Cl}$ interactions around inversion centers, mimicking the commonly observed symmetric hydrogen-bonded dimer pattern often found in carboxylic acids.

Related literature

For background to the electrophilic arylselenylation of reactive arenes, see: Santi *et al.* (2008); Nicolaou *et al.* (1979); Gassman *et al.* (1982); Yoshida *et al.* (1991); Tiecco *et al.* (1994); Engman & Eriksson (1996); Henriksen (1994); Henriksen & Stuhr-Hansen (1998). For related structures, see: Oddershede *et al.* (2003). For related supramolecular patterns, see: Gavezzotti & Filippini (1994); Allen *et al.* (1999); Sørensen & Larsen (2003); Sørensen *et al.* (1999).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{16}\text{Cl}_2\text{O}_2\text{Se}_2$
 $M_r = 517.15$
 Monoclinic, $P2_1/n$
 $a = 11.7737$ (17) Å
 $b = 6.6535$ (6) Å
 $c = 13.438$ (5) Å
 $\beta = 114.136$ (16)°

$V = 960.7$ (4) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 7.47$ mm⁻¹
 $T = 122$ (1) K
 $0.44 \times 0.15 \times 0.13$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: gaussian (DeTitta, 1985).
 $T_{\min} = 0.242$, $T_{\max} = 0.796$
 2641 measured reflections

1976 independent reflections
 1919 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 5 standard reflections
 frequency: 166.7 min
 intensity decay: 5.7%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.075$
 $S = 1.10$
 1976 reflections

119 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.59$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.20$ e Å⁻³

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *DREAR* (Blessing, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2281).

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supplementary materials

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1,4-Bis(4-chlorophenylseleno)-2,5-dimethoxybenzene

H. O. Sørensen and N. Stuhr-Hansen

Comment

The structure of the title compound, shown in Fig. 1, crystallized in space group $P 2_1/n$ utilizing the crystallography inversion center in the molecular symmetry. Generally the molecular geometry of 1 is in close agreement with the related compound 1,3-dimethoxy-4,6-bis(phenylseleno)benzene, hereafter DMPSB. All bond distances and angles are the same within the experimental uncertainty. The molecular conformation of 1 is also very similar to the chloro-unsubstituted compound DMPSB having the planes of phenylseleno groups arranged perpendicular to the plane of the central benzene moiety, but rotated in opposite directions forming a *Z* like conformation (Fig. 1). Leading to the formation of intramolecular $C_{ar}-H\cdots\pi$ interactions.

The molecular packing arrangement is dominated by molecular chains (see Fig. 2) formed by cyclic $C_{ar}-H\cdots Cl$ interactions [$H7\cdots Cl_i = 2.96 \text{ \AA}$, $C7-H7\cdots Cl_i = 166.0^\circ$; symmetry code: (i) $2 - x, 1 - y, 1 - z$] around an inversion center leading to a pattern, which highly resembles the cyclic hydrogen-bonded dimers frequently observed in carboxylic acids. The $C_{ar}-H\cdots Cl$ type of cyclic interaction found in 1 has also been observed in other compounds having a *p*-chlorosubstituted phenyl group, *e.g.* in the structure of racemic *p*-chlorophenoxypropionic acid, where the distance $H\cdots Cl$ is 2.92 \AA [$C-H\cdots Cl$ 175°]. The chains are stacked such that the $\pi-\pi$ interactions between the phenylseleno groups and between the benzene rings along the diagonal of the *b* and *c*-axes, respectively. Due to the chain formation in 1 the packing arrangement is rather different from the pattern found in DMPSB, where interactions with chlorine cannot be formed.

Experimental

Crystals suitable for an X-ray diffraction experiment were obtained by slow crystallization from hot toluene.

Refinement

Hydrogen atoms of (1) were found in the difference Fourier map. All hydrogen atoms were treated as riding atoms with C—H distances of 0.95 for C_{ar} and 0.98 for the C_{Me} . Isotropic displacement parameters for all H atoms were constrained to $1.2U_{eq}$ of the connected non-hydrogen atom ($1.5U_{eq}$ for Me groups).

Figures

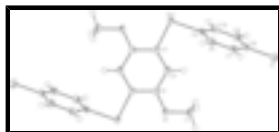


Fig. 1. Thermal ellipsoid plot of (1) including labelling of the atoms. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres with an arbitrary radii.

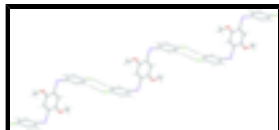


Fig. 2. A view of the cyclic C—H...Cl interactions linking the molecules into a chain.

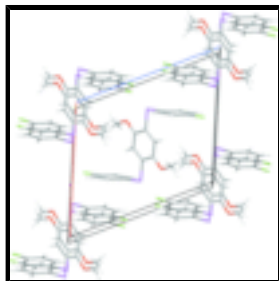


Fig. 3. Packing diagram of viewed down the *b* axis.

1,4-Bis(4-chlorophenylseleno)-2,5-dimethoxybenzene

Crystal data

$C_{20}H_{16}Cl_2O_2Se_2$

$M_r = 517.15$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 11.7737$ (17) Å

$b = 6.6535$ (6) Å

$c = 13.438$ (5) Å

$\beta = 114.136$ (16)°

$V = 960.7$ (4) Å³

$Z = 2$

$F_{000} = 508$

$D_x = 1.788$ Mg m⁻³

Melting point: 193 K

Cu $K\alpha$ radiation

$\lambda = 1.54184$ Å

Cell parameters from 20 reflections

$\theta = 39.3$ – 40.7 °

$\mu = 7.47$ mm⁻¹

$T = 122$ (1) K

Block, white

$0.44 \times 0.15 \times 0.13$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 122$ (1) K

ω - 2θ scans

Absorption correction: Gaussian
(DeTitta, 1985).

$T_{\min} = 0.242$, $T_{\max} = 0.796$

2641 measured reflections

1976 independent reflections

1919 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 74.9$ °

$\theta_{\min} = 4.2$ °

$h = -14 \rightarrow 14$

$k = -7 \rightarrow 8$

$l = 0 \rightarrow 16$

5 standard reflections

every 166.7 min

intensity decay: 5.7%

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.028$	$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.9774P]$
$wR(F^2) = 0.076$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{\max} < 0.001$
1976 reflections	$\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
119 parameters	$\Delta\rho_{\min} = -1.20 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0029 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.745362 (19)	0.19839 (3)	0.007848 (17)	0.01812 (12)
Cl	1.00097 (5)	0.82041 (8)	0.40315 (5)	0.02427 (15)
O1	1.23891 (13)	0.1213 (2)	0.13084 (12)	0.0192 (3)
C1	1.2567 (2)	0.2869 (3)	0.20396 (19)	0.0232 (5)
H1A	1.3459	0.3140	0.2431	0.035*
H1B	1.2213	0.2535	0.2564	0.035*
H1C	1.2150	0.4063	0.1623	0.035*
C2	1.11846 (19)	0.0668 (3)	0.06767 (16)	0.0160 (4)
C3	1.0144 (2)	0.1577 (3)	0.07253 (17)	0.0168 (4)
H3	1.0245	0.2653	0.1219	0.020*
C4	0.89557 (19)	0.0910 (3)	0.00514 (16)	0.0159 (4)
C5	0.82080 (19)	0.3830 (3)	0.12694 (17)	0.0172 (4)
C6	0.8614 (2)	0.3181 (3)	0.23448 (19)	0.0194 (4)
H6	0.8506	0.1815	0.2493	0.023*
C7	0.9173 (2)	0.4513 (3)	0.32023 (17)	0.0197 (4)
H7	0.9457	0.4067	0.3936	0.024*
C8	0.93109 (19)	0.6504 (3)	0.29699 (17)	0.0174 (4)
C9	0.8891 (2)	0.7196 (3)	0.19057 (19)	0.0211 (5)
H9	0.8985	0.8570	0.1762	0.025*
C10	0.8331 (2)	0.5850 (3)	0.10537 (17)	0.0201 (4)
H10	0.8030	0.6308	0.0321	0.024*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.01674 (16)	0.01956 (17)	0.01817 (16)	0.00021 (7)	0.00723 (11)	-0.00363 (7)
Cl	0.0267 (3)	0.0243 (3)	0.0206 (3)	-0.0041 (2)	0.0084 (2)	-0.00601 (18)
O1	0.0174 (7)	0.0196 (7)	0.0194 (7)	-0.0023 (6)	0.0063 (6)	-0.0059 (6)
C1	0.0242 (11)	0.0227 (11)	0.0217 (11)	-0.0045 (9)	0.0084 (9)	-0.0084 (8)
C2	0.0178 (9)	0.0154 (9)	0.0145 (9)	-0.0025 (8)	0.0064 (7)	0.0012 (7)
C3	0.0215 (10)	0.0136 (9)	0.0164 (9)	-0.0016 (8)	0.0089 (8)	-0.0014 (7)
C4	0.0192 (9)	0.0143 (9)	0.0156 (9)	0.0004 (7)	0.0084 (8)	0.0013 (7)
C5	0.0169 (9)	0.0178 (10)	0.0196 (10)	0.0013 (8)	0.0103 (8)	-0.0016 (8)
C6	0.0211 (10)	0.0160 (10)	0.0216 (11)	0.0022 (8)	0.0091 (9)	0.0024 (8)
C7	0.0208 (10)	0.0215 (11)	0.0170 (9)	0.0033 (8)	0.0080 (8)	0.0037 (8)
C8	0.0169 (9)	0.0173 (10)	0.0189 (10)	0.0001 (8)	0.0080 (8)	-0.0038 (8)
C9	0.0255 (11)	0.0163 (10)	0.0213 (11)	-0.0001 (8)	0.0093 (9)	0.0018 (8)
C10	0.0245 (11)	0.0196 (10)	0.0170 (10)	0.0017 (8)	0.0093 (8)	0.0031 (8)

Geometric parameters (\AA , $^\circ$)

Se1—C4	1.921 (2)	C4—C2 ⁱ	1.398 (3)
Se1—C5	1.923 (2)	C5—C6	1.393 (3)
Cl—C8	1.741 (2)	C5—C10	1.395 (3)
O1—C2	1.371 (2)	C6—C7	1.388 (3)
O1—C1	1.433 (2)	C6—H6	0.9500
C1—H1A	0.9800	C7—C8	1.386 (3)
C1—H1B	0.9800	C7—H7	0.9500
C1—H1C	0.9800	C8—C9	1.387 (3)
C2—C3	1.391 (3)	C9—C10	1.389 (3)
C2—C4 ⁱ	1.398 (3)	C9—H9	0.9500
C3—C4	1.392 (3)	C10—H10	0.9500
C3—H3	0.9500		
C4—Se1—C5	97.92 (9)	C6—C5—Se1	120.74 (16)
C2—O1—C1	116.88 (17)	C10—C5—Se1	119.64 (16)
O1—C1—H1A	109.5	C7—C6—C5	120.6 (2)
O1—C1—H1B	109.5	C7—C6—H6	119.7
H1A—C1—H1B	109.5	C5—C6—H6	119.7
O1—C1—H1C	109.5	C8—C7—C6	118.9 (2)
H1A—C1—H1C	109.5	C8—C7—H7	120.6
H1B—C1—H1C	109.5	C6—C7—H7	120.6
O1—C2—C3	124.29 (19)	C7—C8—C9	121.7 (2)
O1—C2—C4 ⁱ	115.37 (18)	C7—C8—Cl	119.75 (17)
C3—C2—C4 ⁱ	120.34 (19)	C9—C8—Cl	118.60 (17)
C2—C3—C4	120.07 (19)	C8—C9—C10	119.0 (2)
C2—C3—H3	120.0	C8—C9—H9	120.5
C4—C3—H3	120.0	C10—C9—H9	120.5
C3—C4—C2 ⁱ	119.60 (19)	C9—C10—C5	120.3 (2)
C3—C4—Se1	123.88 (16)	C9—C10—H10	119.9

C2 ⁱ —C4—Se1	116.50 (15)	C5—C10—H10	119.9
C6—C5—C10	119.6 (2)		
C1—O1—C2—C3	-1.8 (3)	C10—C5—C6—C7	-2.0 (3)
C1—O1—C2—C4 ⁱ	178.06 (18)	Se1—C5—C6—C7	179.10 (16)
O1—C2—C3—C4	-179.78 (19)	C5—C6—C7—C8	0.6 (3)
C4 ⁱ —C2—C3—C4	0.4 (3)	C6—C7—C8—C9	0.8 (3)
C2—C3—C4—C2 ⁱ	-0.4 (3)	C6—C7—C8—C1	179.98 (16)
C2—C3—C4—Se1	177.83 (15)	C7—C8—C9—C10	-0.7 (3)
C5—Se1—C4—C3	-3.27 (19)	C1—C8—C9—C10	-179.91 (17)
C5—Se1—C4—C2 ⁱ	174.97 (16)	C8—C9—C10—C5	-0.8 (3)
C4—Se1—C5—C6	-83.67 (18)	C6—C5—C10—C9	2.1 (3)
C4—Se1—C5—C10	97.46 (18)	Se1—C5—C10—C9	-179.02 (17)

Symmetry codes: (i) $-x+2, -y, -z$.

Fig. 1

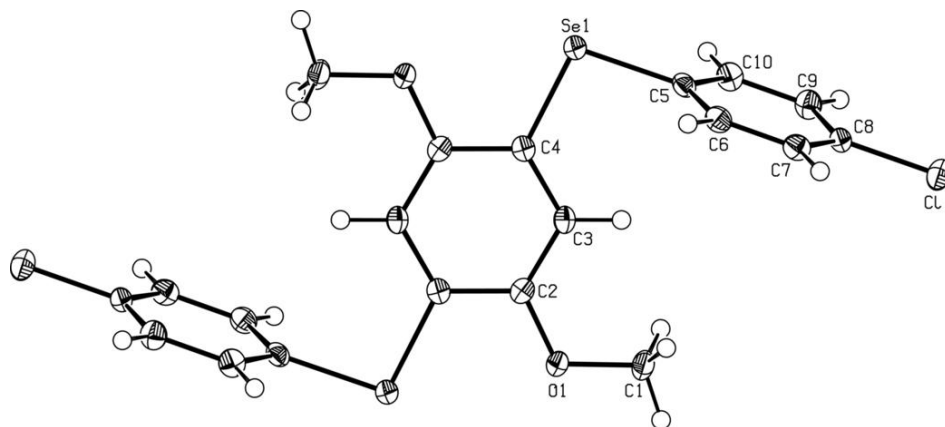


Fig. 2

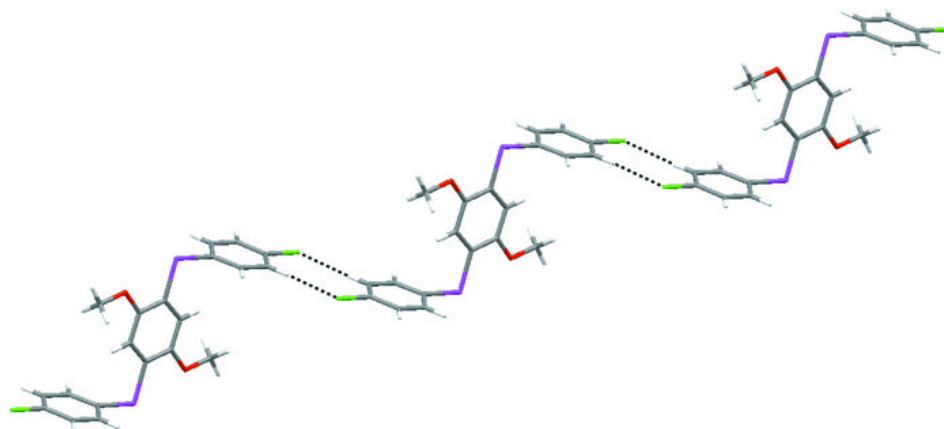


Fig. 3

