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Citation (APA):
1,4-Bis(4-chlorophenylseleno)-2,5-dimethoxybenzene

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Key indicators: single-crystal X-ray study; T = 122 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.076; data-to-parameter ratio = 16.6.

The title compound, C_{20}H_{16}Cl_{2}O_{2}Se_{2}, utilizes the symmetry of the crystallographic inversion center. Molecular chains are formed through symmetric C–H···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); Nicolaou et al. (1979).

Experimental

Crystal data

C_{20}H_{16}Cl_{2}O_{2}Se_{2}  V = 960.7 (4) Å³
M_r = 517.15  Z = 2
Monoclinic, P2_1/n  Cu Kα radiation
a = 11.7737 (17) Å  μ = 7.47 mm⁻¹
b = 6.6555 (6) Å  T = 122 (1) K
C = 13.438 (5) Å  0.44 × 0.15 × 0.13 mm
β = 114.136 (16)°  2641 measured reflections

Data collection

Enraf–Nonius CAD-4 diffractometer  1797 independent reflections
Absorption correction: gaussian (DeTitta, 1985).  1919 reflections with I > 2σ(I)
(DeTitta, 1985).  R_{int} = 0.030
T_{min} = 0.242, T_{max} = 0.796  5 standard reflections
2641 measured reflections  frequency: 166.7 min
intensity decay: 5.7%  

Refinement

R[F^2 > 2σ(F^2)] = 0.028  119 parameters
wR(F^2) = 0.075  H-atom parameters constrained
S = 1.10  Δρ_{max} = 0.59 e Å⁻³
1976 reflections  Δρ_{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: SHELX97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

The authors thank Flemming Hansen, Centre of Crystallographic Studies, University of Copenhagen, for obtaining the crystallographic data. The Danish National Research Foundation is acknowledged for supporting the Center for Fundamental Research: Metal Structures in Four Dimensions.

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

References

supplementary materials
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 2₁/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=H···π interactions.

The molecular packing arrangement is dominated by molecular chains (see Fig. 2) formed by cyclic C=H···Cl interactions [H7···Cl₁ = 2.96 Å, C7—H7···Cl₁ = 166.0°; 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=H···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···Cl is 2.92 Å [C—H···Cl 175°]. The chains are stacked such that the π-π 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.
supplementary materials

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_{2}O_{2}Se_{2}  
F_{000} = 508

M_r = 517.15  
D_x = 1.788 Mg m^{-3}

Monoclinic, P2_1/n  
Melting point: 193 K

Hall symbol: -P 2yn  
Cu Kα radiation

a = 11.7737 (17) Å  
λ = 1.54184 Å

b = 6.6535 (6) Å  
θ = 39.3–40.7º

c = 13.438 (5) Å  
µ = 7.47 mm^{-1}

β = 114.136 (16)º  
T = 122 (1) K

V = 960.7 (4) Å^3  
Block, white

Z = 2  
0.44 × 0.15 × 0.13 mm

Data collection

Enraf–Nonius CAD-4  
R_{int} = 0.030

diffractometer  
Radiation source: fine-focus sealed tube

0_max = 74.9º  
0_min = 4.2º

T = 122(1) K  
h = −14→14

ω–20 scans  
k = −7→8

Absorption correction: Gaussian  
l = 0→16

(DeTitta, 1985).

T_{min} = 0.242, T_{max} = 0.796  
5 standard reflections

2641 measured reflections  
every 166.7 min

1976 independent reflections  
1919 reflections with I > 2σ(I)

1976 reflections with I > 2σ(I)

Refinement

Refinement on F^2  
Hydrogen site location: difference Fourier map

Least-squares matrix: full  
H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.028$
\[ w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.9774P] \]
where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.076$

$S = 1.10$

1976 reflections

119 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

**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 $F^2 > 2\sigma(F^2)$ 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 ($\angstrom^2$)**

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## Atomic displacement parameters (Å²)

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## Geometric parameters (Å, °)

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<th>C1—H1A</th>
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<th>C1—H1C</th>
<th>C2—C3</th>
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supplementary materials

Atomic displacement parameters ($\AA^2$)

Geometric parameters ($\AA$, °)

sup-4
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Symmetry codes: (i) −x+2, −y, −z.
Fig. 2
supplementary materials

Fig. 3