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Octakis(phenylseleno)naphthalene

DAVID D. MACNICOL, PAUL R. MALLINSON AND COLIN D. ROBERTSON

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland

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Abstract

The title molecule possesses crystallographic $\bar{1}$ symmetry. It has a side-chain conformational distribution, *abaababb* [MacNicol, Mallinson & Robertson (1985). *J. Chem. Soc. Chem. Commun.* pp. 1649–1651], analogous to that found in a red unsolvated form of octakis(phenylthio)naphthalene. The central naphthalene core is not planar but is comprised of two fused centrosymmetrically related shallow boats.

Comment

Fig. 1 shows a view illustrating the conformation of octakis(phenylseleno)naphthalene (1) in its molecular crystal. The centrosymmetric conformation corresponds to the classification *abaababb* which was previously found in a red unsolvated form of octakis(phenylthio)naphthalene (Barbour, Freer & MacNicol, 1983). As can be seen, the central naphthalene core of the octaseleno ether (1) is non-planar; the six-membered rings are fused shallow boats which are centrosymmetrically related. C1 and C4 have displacements of −0.17(1) and −0.13(1) Å respectively from the mean plane defined by the atoms C2, C3, C9* and C9. Corresponding displacements for the octathio ether are −0.19 and −0.15 Å. As in octakis(phenylthio)naphthalene, the six central naphthalene C atoms are roughly coplanar; the C1—C9—C9*—C4 torsion angle is 4.7(8)° for the seleno ether compared with 2.6° for the corresponding angle in its sulfur counterpart. The Se atoms Se1–Se4 are displaced by −0.826(1), +0.438(1), +0.252(1) and −0.744(1) Å respectively from the mean plane of the naphthalene core. The mean lengths of the four inner and outer C—Se bonds are 1.932(5) and 1.913(5) Å respectively (the latter is presumably affected by thermal motion) compared with corresponding (unique) values of 1.921(8) and 1.909(8) Å for the inner and outer C—Se bonds in hexakis(phenylseleno)benzene (CBr_4 clathrate) (Gilmore, MacNicol, Mallinson, Murphy & Russell, 1984). The magnitudes of the displacements of the atoms Se1–Se4 from the planes of their attached phenyl groups are 0.277(1), 0.196(1), 0.038(1) and 0.063(1) Å respectively.

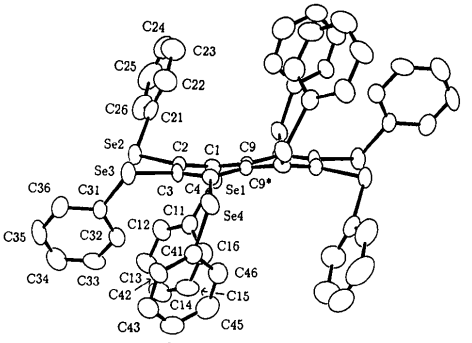


Fig. 1. The conformation of octakis(phenylseleno)naphthalene (1) in its molecular crystal showing the atom labels. Thermal ellipsoids indicate the 50% probability level.

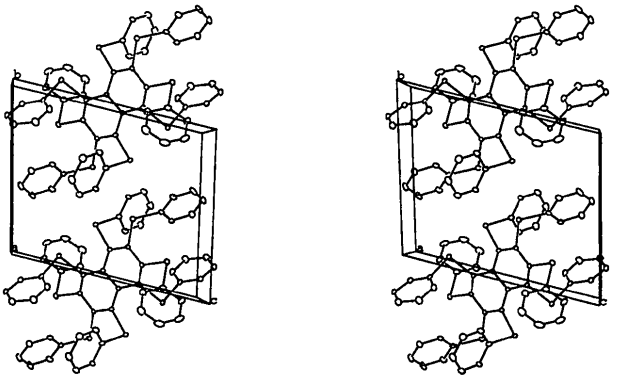
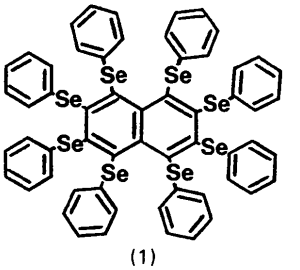


Fig. 2. A stereoview of the unit cell looking along the *a* axis.



(1)

Experimental

Crystal data

$C_{58}H_{40}Se_8$
M_r = 1368.64
Triclinic
$P\bar{1}$
a = 9.288 (1) Å
b = 11.301 (2) Å
c = 12.737 (2) Å
α = 101.10 (1)°
β = 95.39 (1)°
γ = 109.46 (1)°
V = 1219 (1) Å^3
Z = 1
D_x = 1.86 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11–15°
μ = 59.8 cm^{-1}
T = 293 K
Plate
0.2 × 0.2 × 0.07 mm
Dark orange

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Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
5635 measured reflections
5302 independent reflections
2451 observed reflections [$I>2\sigma(I)$]
R_{int} = 0.025
θ_{max} = 27°
h = 0 → 11
k = −14 → 13
l = −16 → 15
2 standard reflections
frequency: 120 min
intensity variation: none

Refinement

Refinement on F
Final R = 0.044
wR = 0.047
S = 1.9
2451 reflections
362 parameters
All H-atom parameters refined except those of H25, H42, H43 and H45
$w = \sigma^{-2}(F)$
$(\Delta/\sigma)_{max}$ = 0.33
$\Delta\rho_{max}$ = 2.0 e Å^{-3}
$\Delta\rho_{min}$ = −2.2 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	−0.0552 (10)	0.0166 (8)	−0.3618 (6)	0.030
C2	−0.0797 (10)	0.1314 (8)	−0.3474 (7)	0.033
C3	−0.0227 (10)	0.2141 (7)	−0.4184 (7)	0.034
C4	0.0360 (10)	0.1722 (7)	−0.5060 (7)	0.032
C9	−0.0207 (10)	−0.0402 (7)	−0.4618 (6)	0.027
C11	0.1269 (11)	0.0831 (9)	−0.1473 (7)	0.042
C12	0.1119 (14)	0.1323 (9)	−0.0433 (7)	0.051
C13	0.2355 (15)	0.2202 (10)	0.0306 (8)	0.056
C14	0.3771 (16)	0.2630 (11)	0.0005 (10)	0.070
C15	0.3946 (14)	0.2213 (12)	−0.1055 (11)	0.069
C16	0.2703 (13)	0.1295 (11)	−0.1760 (9)	0.062
C21	−0.3621 (12)	0.0331 (9)	−0.2586 (9)	0.051
C22	−0.4541 (15)	−0.0198 (12)	−0.3587 (11)	0.071
C23	−0.5897 (19)	−0.1218 (16)	−0.3714 (18)	0.094
C24	−0.6422 (19)	−0.1654 (13)	−0.2856 (18)	0.091
C25	−0.5550 (18)	−0.1141 (15)	−0.1865 (14)	0.092
C26	−0.4159 (16)	−0.0138 (12)	−0.1690 (10)	0.071
C31	0.0522 (11)	0.4611 (8)	−0.2522 (7)	0.039
C32	0.1607 (12)	0.4271 (9)	−0.1967 (8)	0.046
C33	0.2249 (13)	0.4927 (11)	−0.0899 (9)	0.056
C34	0.1853 (17)	0.5904 (12)	−0.0398 (11)	0.070
C35	0.0793 (18)	0.6260 (14)	−0.0958 (12)	0.080
C36	0.0109 (13)	0.5623 (10)	−0.1997 (10)	0.058
C41	0.3294 (11)	0.3744 (8)	−0.4520 (8)	0.043
C42	0.3739 (12)	0.5041 (8)	−0.4035 (8)	0.045
C43	0.4928 (14)	0.5598 (10)	−0.3190 (10)	0.060
C44	0.5705 (15)	0.4896 (13)	−0.2808 (11)	0.070
C45	0.5288 (14)	0.3591 (12)	−0.3317 (10)	0.074
C46	0.4103 (14)	0.3027 (11)	−0.4159 (10)	0.057
Se1	−0.04145 (12)	−0.05939 (9)	−0.23912 (7)	0.041
Se2	−0.17859 (12)	0.18551 (9)	−0.23080 (8)	0.043
Se3	−0.04317 (13)	0.38087 (9)	−0.39957 (8)	0.047
Se4	0.16730 (12)	0.29549 (9)	−0.57532 (8)	0.041

Table 2. *Geometric parameters* (Å)

C1—C2	1.372 (12)	C21—Se2	1.922 (10)
C1—C9	1.427 (12)	C22—C23	1.37 (3)
C1—Se1	1.936 (9)	C23—C24	1.35 (4)
C2—C3	1.434 (12)	C24—C25	1.34 (3)
C2—Se2	1.924 (9)	C25—C26	1.37 (3)
C3—C4	1.360 (13)	C31—C32	1.375 (14)
C3—Se3	1.929 (8)	C31—C36	1.397 (14)
C4—C9*	1.424 (11)	C31—Se3	1.904 (9)
C4—Se4	1.937 (9)	C32—C33	1.386 (15)
C9—C9*	1.447 (12)	C33—C34	1.344 (18)
C11—C12	1.372 (13)	C34—C35	1.37 (3)
C11—C16	1.372 (16)	C35—C36	1.358 (19)
C11—Se1	1.913 (9)	C41—C42	1.379 (13)
C12—C13	1.367 (15)	C41—C46	1.384 (16)
C13—C14	1.36 (2)	C41—Se4	1.911 (10)
C14—C15	1.385 (19)	C42—C43	1.352 (15)
C15—C16	1.365 (17)	C43—C44	1.361 (18)
C21—C22	1.368 (17)	C44—C45	1.396 (19)
C21—C26	1.413 (18)	C45—C46	1.348 (17)

Symmetry code: (*) $-x, -y, -1-z$.

The title compound was initially prepared by persubstitution of octafluoronaphthalene with phenylselenide anion (Robertson, 1984), and was identified spectroscopically in the first instance.

The structure was solved by a manual Patterson solution. Calculations were performed with the *GX* package (Mallinson & Muir, 1985), which includes local modifications of *ORTEP* (Johnson 1971).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55217 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1012]

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Structure of δ-(BEDT-TTF)PF_6

XIANHUI BU, IVANA CISAROVA AND PHILIP COPPENS

Department of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214, USA

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Abstract

The structure consists of two-dimensional sheets of BEDT-TTF [BEDT-TTF, or ET = 3,4;3′,4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene] separated by PF_6^- along the crystallographic b axis. Intermolecular S—S

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