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2,13-Dithia[3]orthocyclo[3](2,3)-naphthalenophane[†]

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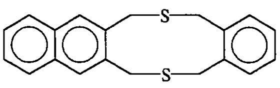
Abstract

The title compound, $C_{20}H_{18}S_2$, crystallizes with two independent but closely similar molecules in the asymmetric unit. The aromatic ring systems are parallel and adopt the *anti* conformation.

Comment

We are interested in structures, particularly conformational aspects, of cyclophane systems (see, for example, Bodwell, Ernst, Hopf & Jones, 1989; Jones, Hopf & Kuś, 1996). We present here the structure of the sulfur-containing cyclophane 2,13-dithia[3]orthocyclo[3](2,3)-naphthalenophane, (I).



(I)

The title compound crystallizes with two molecules in the asymmetric unit (Fig. 1), which are, however, closely similar; a least-squares fit (molecule 2 inverted) showed a mean deviation of 0.07 Å.

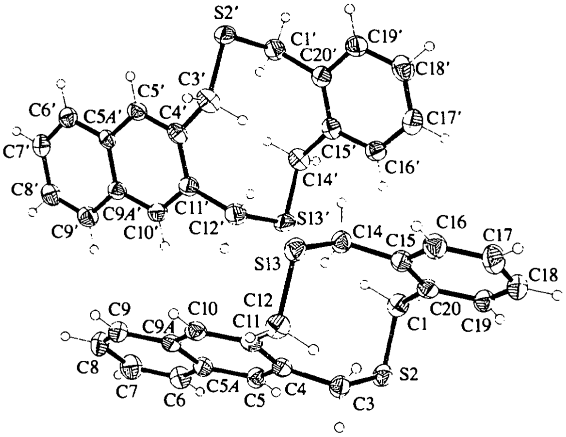


Fig. 1. Both independent molecules of the title compound in the crystal. Ellipsoids represent 50% probability levels and H-atom radii are arbitrary.

The aromatic ring systems in each molecule are almost parallel [interplanar angles of 1.4 (1) and 2.5 (1)°] and adopt the *anti* conformation, as previously observed by us in the closely related structure 2,11-dithia-[3.3](5,6)indanoorthocyclophane (Bodwell *et al.*, 1989) and by Okajima, Wang & Fukazawa (1989) in the parent compound 2,11-dithia[3.3]orthocyclophane, which displays inversion symmetry. The perpendicular distance between the ring systems (as given by the deviations of atoms C15–C20 and C15′–C20′ from the naphthalene best plane) is *ca* 2.4–2.5 Å.

Bond lengths, angles and torsion angles of the central ten-membered rings are given in Table 1. Starting with C1—S2—C3—C4 and moving to increasing atom number, the torsion angle sequence is +*sc*, −*sc*, *sp*, +*ac*, −*ac*, −*ac*, +*ac*, *sp*, −*sc*, +*sc* in molecule 1 (and in molecule 2 when inverted), as previously observed by Bodwell *et al.* (1989).

Experimental

The title compound was prepared as reported by Kuś (1987) and recrystallized from dichloromethane/*n*-butanol.

Crystal data

$C_{20}H_{18}S_2$
$M_r = 322.46$
Triclinic
$P\bar{1}$
$a = 10.646\,(3)$ Å
$b = 11.286\,(3)$ Å
$c = 14.205\,(4)$ Å
$\alpha = 78.20\,(2)°$
$\beta = 80.64\,(2)°$
$\gamma = 71.77\,(2)°$
$V = 1577.9\,(8)$ Å^3
$Z = 4$
$D_x = 1.357$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10.0–11.5°$
$\mu = 0.331$ mm^{-1}
$T = 143\,(2)$ K
Prism
0.80 × 0.20 × 0.20 mm
Colourless

[†] Alternative name: 3,12-dithiatetracyclo[12.8.0.0^{5,12}.0^{16,21}]docosane-5,7,9,14,16,18,20,22-octaene.

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
5868 measured reflections
5562 independent reflections
4215 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.0181$
$\theta_{\max} = 25.02°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0397$
$wR(F^2) = 0.1072$
$S = 1.053$
5562 reflections
397 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.9331P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.233$ e Å^{-3}
$\Delta\rho_{\min} = -0.218$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C20	1.512 (3)	C1′—C20′	1.511 (3)
C1—S2	1.820 (3)	C1′—S2′	1.814 (3)
S2—C3	1.809 (2)	S2′—C3′	1.804 (3)
C3—C4	1.517 (3)	C3′—C4′	1.512 (3)
C4—C11	1.426 (3)	C4′—C11′	1.429 (3)
C11—C12	1.513 (3)	C11′—C12′	1.512 (3)
C12—S13	1.818 (3)	C12′—S13′	1.809 (3)
S13—C14	1.807 (2)	S13′—C14′	1.805 (3)
C14—C15	1.516 (3)	C14′—C15′	1.512 (3)
C15—C20	1.402 (3)	C15′—C20′	1.401 (3)
C3—S2—C1	101.63 (11)	C3′—S2′—C1′	101.59 (12)
C14—S13—C12	102.37 (11)	C14′—S13′—C12′	102.00 (12)
C20—C1—S2—C3	59.09 (19)		
C1—S2—C3—C4	78.4 (2)		
C11—C12—S13—C14	−58.92 (19)		
C12—S13—C14—C15	−78.2 (2)		
C20′—C1′—S2′—C3′	−60.69 (19)		
C1′—S2′—C3′—C4′	−74.4 (2)		
C11′—C12′—S13′—C14′	60.7 (2)		
C12′—S13′—C14′—C15′	74.5 (2)		

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, least-squares-planes data, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1236). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Thieno[2,3-*c*]pyridine, the First Structure of a Thienopyridine

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Abstract

The title molecule, C_7H_5NS, is planar to within 0.009 Å. Bond lengths at the heteroatoms are S1—C2 1.7276 (14), S1—C7a 1.7308 (13), N6—C5 1.354 and N6—C7 1.332 (2) Å; the C—S—C angle is 90.76 (6)°. A libration analysis indicated corrections of 0.003–0.004 Å. Short intermolecular contacts [S1···N6 3.229 (1) Å] link the molecules into chains parallel to the *y* axis.

Comment

Thieno[2,3-*c*]pyridine, (1), is of pharmacological interest because it is an isostere of isoquinoline (Friedrichsen, 1984). It represents a fused 10π-ring system containing the π-rich thiophene and the π-deficient pyridine [for a review see Friedrichsen (1984)]. Recently, the *ab initio* geometries of all six isomers were discussed with regard to the mutual influence of the two heteroaromatics, but the absence of known structures necessarily prevented any correlation with structural data (Webber & Woolley, 1995). As a heteronaphthalene, (1) is an inherently polarized push–pull system with a ground-state dipole moment of 2.85 D (Klemm & Jacquot, 1975). The