organic compounds

2,13-Dithia[3.3](1,3)(2,3)-naphthalenophane

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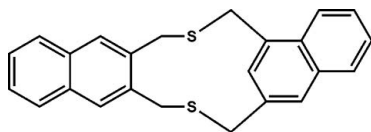Correspondence e-mail: p.jones@tu-bs.de

The title compound, $C_{24}H_{20}S_2$, adopts an *anti* conformation with approximately parallel ring systems; the interplanar angle is 5.57 (8)°. Apart from the outer ring of the *meta* naphthalene, the molecule displays approximate mirror symmetry.

Related literature

For related literature, see: Bodwell *et al.* (1990); Jones & Kus (1997); Jones *et al.* (1996); Kuś (1991).

Experimental

Crystal data

$C_{24}H_{20}S_2$
$M_r = 372.52$
Monoclinic, $P2_1/c$
$a = 10.6839$ (11) Å
$b = 8.4014$ (8) Å
$c = 20.285$ (2) Å
$\beta = 98.000$ (5)°

$V = 1803.0$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.30$ mm^{-1}
$T = 100$ (2) K
$0.26 \times 0.18 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
34380 measured reflections

4845 independent reflections
4078 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.120$
$S = 1.13$
4845 reflections

235 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Table 1
Selected torsion angles (°).

C21—C1—S2—C3	41.41 (16)	C12—S13—C14—C15	−40.79 (16)
C1—S2—C3—C4	64.53 (16)	S13—C14—C15—C22	98.75 (18)
S2—C3—C4—C11	−146.85 (15)	S2—C1—C21—C22	−99.84 (18)
C3—C4—C11—C12	0.6 (3)	C1—C21—C22—C15	170.29 (17)
C4—C11—C12—S13	142.21 (15)	C14—C15—C22—C21	−170.44 (17)
C11—C12—S13—C14	−63.60 (16)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$\cdots$S2^{i}	0.95	2.93	3.707 (2)	140
C9—H9$\cdots$S13^{ii}	0.95	3.00	3.6951 (19)	131

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 2, -y + 1, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

References

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supplementary materials

2,13-Dithia[3.3](1,3)(2,3)naphthalenophane

P. G. Jones and **P. Kus**

Comment

We have reported the structures of 2,13-dithia[3]metacyclo[3](2,3)-naphthalenophane [(IIa); Jones & Kus, 1997], and 17-methyl-2,13-dithia[3]metacyclo[3](2,3)naphthalenophane [(IIb); Jones *et al.*, 1996] as part of a series of *ortho-meta* dithia-phanes. Surprisingly, in the solid state both compounds exist in an anti conformation, in contrast to the simpler analogue 2,11-dithia[3.3]orthometacyclophane, which adopts a *syn* conformation (Bodwell *et al.*, 1990). Here we report the next compound in the *ortho-meta* series, the title compound (I).

Compound (I) (Fig. 1) displays approximate mirror symmetry except, necessarily, for the second ring of the *meta*-substituted naphthalene system. Torsion angles of the central 11-membered ring are given in Table 1. These torsion angles, and thus the overall molecular conformations, are closely similar in all three compounds (I), (IIa) and (IIb) (Fig. 3). The ring systems of I are approximately parallel [interplanar angle 5.57 (8)°] and adopt anti conformation. The perpendicular distance between the ring planes (as given by the distance of atoms of the *meta*-substituted ring from the best plane of the *ortho*-substituted naphthalene) is 2.8–3.1 Å.

The crystal packing of the three compounds is different; they crystallize in different space groups. In (I), two weak C—H···S interactions (Table 2) combine with a short C—H···π contact from H12B to the centroid of the ring C16A–C20A [2.55 Å, angle 160°, operator $1 - x$, $1 - y$, $-z$)] to form columns of molecules parallel to the y axis (Fig. 2).

Experimental

The title compound was prepared by cyclization of 1,3-di(bromomethyl)naphthalene and 2,3-di(mercaptomethyl)naphthalene as described by Kuś (1991). Single crystals were obtained from DMF solution by very slow evaporation (5 years).

Refinement

Hydrogen atoms were included using a riding model with C—H 0.95 (aromatic), 0.99 (methyene) Å; U(H) values were fixed at 1.2U(C) of the parent C atom.
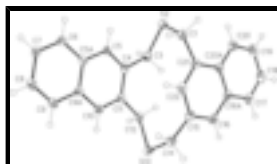
Figures

Fig. 1. The molecule of the title compound in the crystal. Ellipsoids represent 50% probability levels.

supplementary materials

Fig. 2. Packing diagram of I in the region $z \simeq 1/2$. View direction: perpendicular to the xy plane. Thin dashed bonds indicate C—H⋯S and thick dashed bonds C—H⋯π interactions. H atoms not involved in short contacts are omitted for clarity.
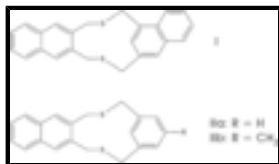

Fig. 3. Schematic representations of (I), (IIa) and (IIb).

2,13-Dithia[3.3](1,3)(2,3)naphthalenophane

Crystal data

$C_{24}H_{20}S_2$	$F_{000} = 784$
$M_r = 372.52$	$D_x = 1.372$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.6839 (11)$ Å	Cell parameters from 9929 reflections
$b = 8.4014 (8)$ Å	$\theta = 2.4–29.1°$
$c = 20.285 (2)$ Å	$\mu = 0.30$ mm^{-1}
$\beta = 98.000 (5)°$	$T = 100 (2)$ K
$V = 1803.0 (3)$ Å^3	Tablet, colourless
$Z = 4$	$0.26 \times 0.18 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	4078 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.042$
Monochromator: graphite	$\theta_{max} = 29.1°$
$T = 100(2)$ K	$\theta_{min} = 2.0°$
φ and ω scans	$h = -14 \rightarrow 14$
Absorption correction: none	$k = -11 \rightarrow 11$
34380 measured reflections	$l = -27 \rightarrow 27$
4845 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.120$	$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 1.9877P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{max} < 0.001$
4845 reflections	$\Delta\rho_{max} = 0.54$ e Å^{-3}

235 parameters

$\Delta\rho_{min} = -0.34$ e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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.

Least-squares planes (*x,y,z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

-0.6768 (0.0048) $x + 5.7973$ (0.0030) $y + 14.6617$ (0.0072) $z = 2.8381$ (0.0041)

* -0.0179 (0.0014) C4 * 0.0111 (0.0015) C5 * 0.0155 (0.0016) C5A * 0.0040 (0.0015) C6 * -0.0144 (0.0016) C7 * -0.0102 (0.0016) C8 * 0.0037 (0.0015) C9 * 0.0127 (0.0016) C9A * 0.0079 (0.0015) C10 * -0.0123 (0.0014) C11

Rms deviation of fitted atoms = 0.0118

-0.2034 (0.0040) $x + 5.2610$ (0.0039) $y + 15.7106$ (0.0078) $z = 5.7669$ (0.0029)

Angle to previous plane (with approximate e.s.d.) = 5.57 (0.08)

* -0.0328 (0.0015) C15 * -0.0107 (0.0016) C16 * 0.0253 (0.0017) C16A * 0.0224 (0.0017) C17 * -0.0097 (0.0017) C18 * -0.0196 (0.0017) C19 * -0.0062 (0.0016) C20 * 0.0115 (0.0017) C20A * 0.0052 (0.0015) C21 * 0.0146 (0.0015) C22

Rms deviation of fitted atoms = 0.0180

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 > 2sigma(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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.51333 (19)	0.4411 (2)	0.21556 (9)	0.0215 (4)
H1A	0.4539	0.4564	0.2483	0.026*
H1B	0.5978	0.4182	0.2404	0.026*
S2	0.46141 (4)	0.27093 (6)	0.16196 (3)	0.02078 (12)
C3	0.54841 (17)	0.3050 (2)	0.09254 (9)	0.0191 (4)
H3A	0.5197	0.2260	0.0575	0.023*
H3B	0.5253	0.4117	0.0740	0.023*
C4	0.69170 (17)	0.2959 (2)	0.10727 (9)	0.0161 (3)
C5	0.74674 (17)	0.1893 (2)	0.15396 (9)	0.0165 (3)
H5	0.6939	0.1264	0.1776	0.020*
C5A	0.87958 (17)	0.1700 (2)	0.16802 (9)	0.0159 (3)
C6	0.93695 (18)	0.0566 (2)	0.21471 (9)	0.0190 (4)
H6	0.8856	−0.0083	0.2384	0.023*
C7	1.06536 (19)	0.0402 (2)	0.22588 (10)	0.0210 (4)

H7	1.1026	−0.0367	0.2569	0.025*
C8	1.14336 (18)	0.1369 (2)	0.19153 (10)	0.0211 (4)
H8	1.2326	0.1250	0.1999	0.025*
C9	1.09126 (18)	0.2473 (2)	0.14642 (9)	0.0187 (4)
H9	1.1446	0.3116	0.1237	0.022*
C9A	0.95820 (17)	0.2666 (2)	0.13324 (9)	0.0156 (3)
C10	0.89987 (17)	0.3780 (2)	0.08618 (9)	0.0169 (3)
H10	0.9519	0.4439	0.0633	0.020*
C11	0.77070 (17)	0.3943 (2)	0.07241 (9)	0.0161 (3)
C12	0.71529 (18)	0.5127 (2)	0.01978 (9)	0.0187 (4)
H12A	0.6271	0.5351	0.0268	0.022*
H12B	0.7119	0.4610	−0.0243	0.022*
S13	0.79701 (5)	0.70201 (6)	0.01669 (2)	0.02253 (12)
C14	0.78066 (19)	0.7904 (2)	0.09742 (10)	0.0227 (4)
H14A	0.8450	0.7436	0.1318	0.027*
H14B	0.7972	0.9061	0.0956	0.027*
C15	0.65157 (19)	0.7646 (2)	0.11739 (9)	0.0198 (4)
C16	0.54634 (19)	0.8478 (2)	0.08957 (10)	0.0211 (4)
H16	0.5557	0.9339	0.0603	0.025*
C16A	0.42454 (19)	0.8071 (2)	0.10390 (9)	0.0201 (4)
C17	0.3147 (2)	0.8902 (3)	0.07447 (10)	0.0258 (4)
H17	0.3236	0.9786	0.0463	0.031*
C18	0.1969 (2)	0.8455 (3)	0.08588 (11)	0.0299 (5)
H18	0.1248	0.9016	0.0651	0.036*
C19	0.1817 (2)	0.7158 (3)	0.12849 (11)	0.0290 (5)
H19	0.0995	0.6856	0.1365	0.035*
C20	0.28544 (19)	0.6332 (3)	0.15835 (10)	0.0237 (4)
H20	0.2741	0.5465	0.1870	0.028*
C20A	0.40914 (18)	0.6755 (2)	0.14689 (9)	0.0192 (4)
C21	0.51962 (18)	0.5912 (2)	0.17616 (9)	0.0184 (4)
C22	0.63570 (18)	0.6398 (2)	0.16198 (9)	0.0186 (4)
H22	0.7089	0.5868	0.1833	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0248 (10)	0.0215 (9)	0.0179 (9)	0.0011 (7)	0.0020 (7)	0.0004 (7)
S2	0.0159 (2)	0.0197 (2)	0.0270 (3)	−0.00133 (17)	0.00408 (18)	0.00062 (18)
C3	0.0161 (8)	0.0214 (9)	0.0189 (9)	0.0019 (7)	−0.0005 (7)	−0.0016 (7)
C4	0.0156 (8)	0.0160 (8)	0.0162 (8)	0.0015 (7)	0.0002 (6)	−0.0034 (6)
C5	0.0143 (8)	0.0162 (8)	0.0191 (9)	0.0001 (6)	0.0024 (7)	−0.0007 (7)
C5A	0.0153 (8)	0.0167 (8)	0.0154 (8)	0.0011 (6)	0.0011 (6)	−0.0014 (6)
C6	0.0199 (9)	0.0185 (8)	0.0187 (9)	−0.0001 (7)	0.0028 (7)	0.0039 (7)
C7	0.0212 (9)	0.0220 (9)	0.0187 (9)	0.0044 (7)	−0.0011 (7)	0.0036 (7)
C8	0.0147 (8)	0.0278 (10)	0.0200 (9)	0.0024 (7)	−0.0004 (7)	−0.0008 (8)
C9	0.0178 (9)	0.0226 (9)	0.0159 (8)	−0.0006 (7)	0.0035 (7)	−0.0004 (7)
C9A	0.0159 (8)	0.0157 (8)	0.0153 (8)	−0.0006 (6)	0.0024 (6)	−0.0019 (6)
C10	0.0199 (9)	0.0166 (8)	0.0143 (8)	0.0001 (7)	0.0030 (7)	0.0004 (6)

C11	0.0197 (9)	0.0156 (8)	0.0125 (8)	0.0023 (7)	0.0008 (6)	−0.0015 (6)
C12	0.0215 (9)	0.0195 (9)	0.0146 (8)	0.0031 (7)	0.0007 (7)	0.0009 (7)
S13	0.0267 (3)	0.0208 (2)	0.0209 (2)	0.00169 (19)	0.00617 (19)	0.00531 (18)
C14	0.0226 (9)	0.0207 (9)	0.0247 (10)	−0.0032 (7)	0.0027 (8)	−0.0016 (8)
C15	0.0227 (9)	0.0181 (9)	0.0181 (9)	−0.0011 (7)	0.0012 (7)	−0.0051 (7)
C16	0.0275 (10)	0.0168 (8)	0.0192 (9)	0.0009 (7)	0.0034 (7)	−0.0006 (7)
C16A	0.0250 (10)	0.0178 (8)	0.0169 (9)	0.0032 (7)	0.0014 (7)	−0.0029 (7)
C17	0.0292 (11)	0.0218 (9)	0.0255 (10)	0.0082 (8)	0.0013 (8)	0.0000 (8)
C18	0.0253 (11)	0.0301 (11)	0.0330 (12)	0.0097 (9)	−0.0002 (9)	−0.0047 (9)
C19	0.0209 (10)	0.0326 (11)	0.0343 (12)	0.0026 (8)	0.0067 (8)	−0.0058 (9)
C20	0.0240 (10)	0.0243 (10)	0.0236 (10)	0.0012 (8)	0.0067 (8)	−0.0024 (8)
C20A	0.0217 (9)	0.0194 (9)	0.0167 (9)	0.0016 (7)	0.0031 (7)	−0.0041 (7)
C21	0.0244 (9)	0.0169 (8)	0.0137 (8)	0.0014 (7)	0.0018 (7)	−0.0023 (6)
C22	0.0209 (9)	0.0176 (8)	0.0158 (8)	0.0022 (7)	−0.0020 (7)	−0.0040 (7)

Geometric parameters (Å, °)

C1—C21	1.499 (3)	C18—C19	1.414 (3)
C1—S2	1.835 (2)	C19—C20	1.375 (3)
S2—C3	1.815 (2)	C20—C20A	1.419 (3)
C3—C4	1.520 (2)	C20A—C21	1.433 (3)
C4—C5	1.375 (3)	C21—C22	1.374 (3)
C4—C11	1.436 (3)	C1—H1A	0.9900
C5—C5A	1.418 (2)	C1—H1B	0.9900
C5A—C6	1.421 (2)	C3—H3A	0.9900
C5A—C9A	1.424 (3)	C3—H3B	0.9900
C6—C7	1.366 (3)	C5—H5	0.9500
C7—C8	1.415 (3)	C6—H6	0.9500
C8—C9	1.366 (3)	C7—H7	0.9500
C9—C9A	1.419 (3)	C8—H8	0.9500
C9A—C10	1.418 (2)	C9—H9	0.9500
C10—C11	1.376 (3)	C10—H10	0.9500
C11—C12	1.518 (2)	C12—H12A	0.9900
C12—S13	1.820 (2)	C12—H12B	0.9900
S13—C14	1.828 (2)	C14—H14A	0.9900
C14—C15	1.506 (3)	C14—H14B	0.9900
C15—C16	1.376 (3)	C16—H16	0.9500
C15—C22	1.410 (3)	C17—H17	0.9500
C16—C16A	1.414 (3)	C18—H18	0.9500
C16A—C17	1.423 (3)	C19—H19	0.9500
C16A—C20A	1.432 (3)	C20—H20	0.9500
C17—C18	1.364 (3)	C22—H22	0.9500
C21—C1—S2	111.82 (13)	S2—C1—H1A	109.3
C3—S2—C1	101.06 (9)	C21—C1—H1B	109.3
C4—C3—S2	116.75 (13)	S2—C1—H1B	109.3
C5—C4—C11	119.33 (16)	H1A—C1—H1B	107.9
C5—C4—C3	119.37 (17)	C4—C3—H3A	108.1
C11—C4—C3	121.28 (16)	S2—C3—H3A	108.1
C4—C5—C5A	122.23 (17)	C4—C3—H3B	108.1

C5—C5A—C6	122.54 (17)	S2—C3—H3B	108.1
C5—C5A—C9A	118.57 (16)	H3A—C3—H3B	107.3
C6—C5A—C9A	118.89 (16)	C4—C5—H5	118.9
C7—C6—C5A	120.56 (17)	C5A—C5—H5	118.9
C6—C7—C8	120.47 (18)	C7—C6—H6	119.7
C9—C8—C7	120.45 (17)	C5A—C6—H6	119.7
C8—C9—C9A	120.55 (17)	C6—C7—H7	119.8
C10—C9A—C9	122.52 (17)	C8—C7—H7	119.8
C10—C9A—C5A	118.41 (16)	C9—C8—H8	119.8
C9—C9A—C5A	119.07 (16)	C7—C8—H8	119.8
C11—C10—C9A	122.47 (17)	C8—C9—H9	119.7
C10—C11—C4	118.97 (16)	C9A—C9—H9	119.7
C10—C11—C12	119.34 (17)	C11—C10—H10	118.8
C4—C11—C12	121.68 (16)	C9A—C10—H10	118.8
C11—C12—S13	117.07 (13)	C11—C12—H12A	108.0
C12—S13—C14	102.58 (9)	S13—C12—H12A	108.0
C15—C14—S13	112.81 (14)	C11—C12—H12B	108.0
C16—C15—C22	118.44 (18)	S13—C12—H12B	108.0
C16—C15—C14	122.88 (18)	H12A—C12—H12B	107.3
C22—C15—C14	118.41 (17)	C15—C14—H14A	109.0
C15—C16—C16A	121.04 (18)	S13—C14—H14A	109.0
C16—C16A—C17	121.61 (18)	C15—C14—H14B	109.0
C16—C16A—C20A	119.91 (18)	S13—C14—H14B	109.0
C17—C16A—C20A	118.44 (19)	H14A—C14—H14B	107.8
C18—C17—C16A	121.3 (2)	C15—C16—H16	119.5
C17—C18—C19	120.2 (2)	C16A—C16—H16	119.5
C20—C19—C18	120.3 (2)	C18—C17—H17	119.3
C19—C20—C20A	120.9 (2)	C16A—C17—H17	119.3
C20—C20A—C16A	118.82 (18)	C17—C18—H18	119.9
C20—C20A—C21	122.72 (18)	C19—C18—H18	119.9
C16A—C20A—C21	118.45 (18)	C20—C19—H19	119.9
C22—C21—C20A	118.81 (17)	C18—C19—H19	119.9
C22—C21—C1	118.07 (17)	C19—C20—H20	119.6
C20A—C21—C1	122.77 (18)	C20A—C20—H20	119.6
C21—C22—C15	123.24 (18)	C21—C22—H22	118.4
C21—C1—H1A	109.3	C15—C22—H22	118.4
C21—C1—S2—C3	41.41 (16)	C11—C12—S13—C14	−63.60 (16)
C1—S2—C3—C4	64.53 (16)	C12—S13—C14—C15	−40.79 (16)
S2—C3—C4—C5	34.4 (2)	S13—C14—C15—C16	−75.2 (2)
S2—C3—C4—C11	−146.85 (15)	S13—C14—C15—C22	98.75 (18)
C11—C4—C5—C5A	−1.5 (3)	C22—C15—C16—C16A	−1.5 (3)
C3—C4—C5—C5A	177.25 (17)	C14—C15—C16—C16A	172.45 (17)
C4—C5—C5A—C6	−178.02 (17)	C15—C16—C16A—C17	−178.77 (18)
C4—C5—C5A—C9A	1.1 (3)	C15—C16—C16A—C20A	−1.3 (3)
C5—C5A—C6—C7	178.87 (18)	C16—C16A—C17—C18	176.7 (2)
C9A—C5A—C6—C7	−0.2 (3)	C20A—C16A—C17—C18	−0.8 (3)
C5A—C6—C7—C8	0.6 (3)	C16A—C17—C18—C19	1.0 (3)
C6—C7—C8—C9	−0.4 (3)	C17—C18—C19—C20	−0.4 (3)
C7—C8—C9—C9A	−0.1 (3)	C18—C19—C20—C20A	−0.3 (3)

C8—C9—C9A—C10	−179.07 (18)	C19—C20—C20A—C16A	0.5 (3)
C8—C9—C9A—C5A	0.4 (3)	C19—C20—C20A—C21	−179.02 (19)
C5—C5A—C9A—C10	0.1 (3)	C16—C16A—C20A—C20	−177.49 (18)
C6—C5A—C9A—C10	179.25 (16)	C17—C16A—C20A—C20	0.1 (3)
C5—C5A—C9A—C9	−179.38 (17)	C16—C16A—C20A—C21	2.0 (3)
C6—C5A—C9A—C9	−0.2 (3)	C17—C16A—C20A—C21	179.59 (17)
C9—C9A—C10—C11	178.57 (17)	C20—C20A—C21—C22	179.58 (18)
C5A—C9A—C10—C11	−0.9 (3)	C16A—C20A—C21—C22	0.1 (3)
C9A—C10—C11—C4	0.5 (3)	C20—C20A—C21—C1	6.5 (3)
C9A—C10—C11—C12	−178.11 (16)	C16A—C20A—C21—C1	−172.94 (17)
C5—C4—C11—C10	0.7 (3)	S2—C1—C21—C22	−99.84 (18)
C3—C4—C11—C10	−178.04 (16)	S2—C1—C21—C20A	73.2 (2)
C5—C4—C11—C12	179.27 (16)	C20A—C21—C22—C15	−3.1 (3)
C3—C4—C11—C12	0.6 (3)	C1—C21—C22—C15	170.29 (17)
C10—C11—C12—S13	−39.2 (2)	C16—C15—C22—C21	3.8 (3)
C4—C11—C12—S13	142.21 (15)	C14—C15—C22—C21	−170.44 (17)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···S2[i]	0.95	2.93	3.707 (2)	140
C9—H9···S13[ii]	0.95	3.00	3.6951 (19)	131

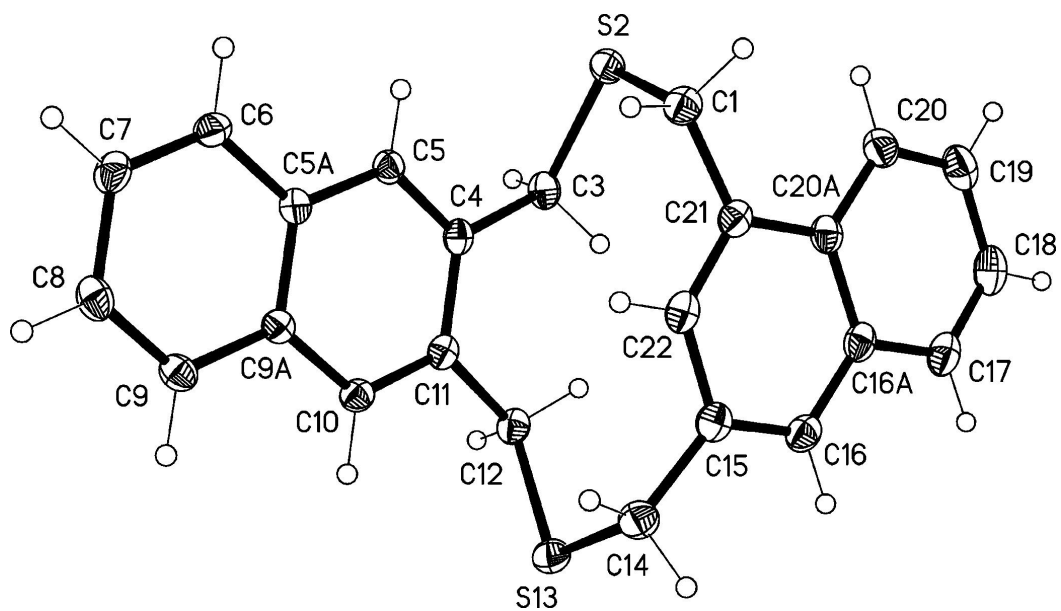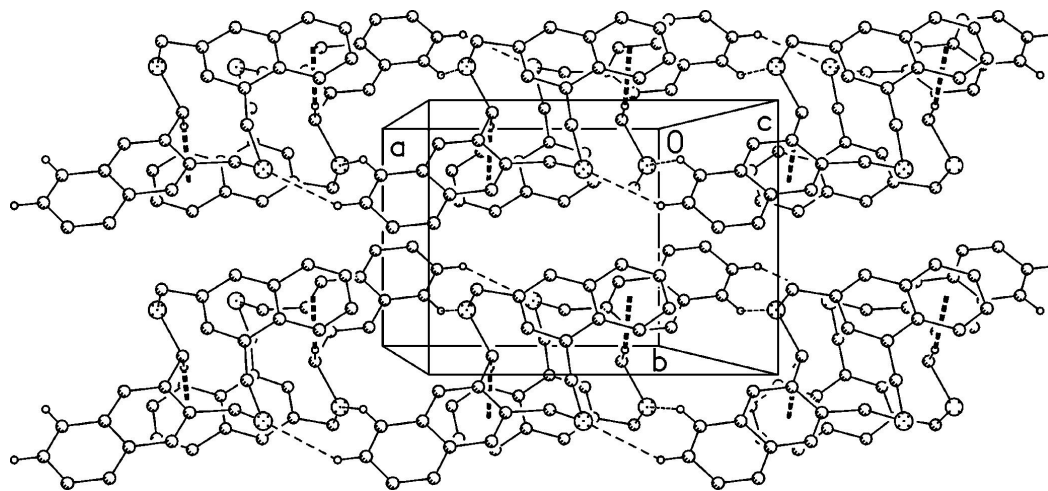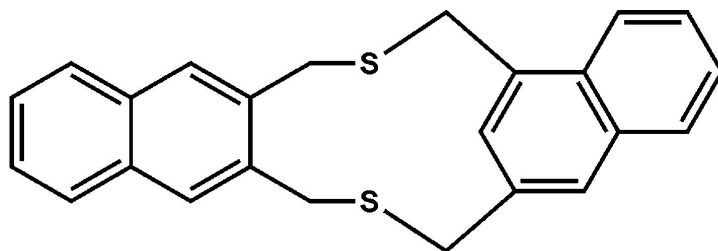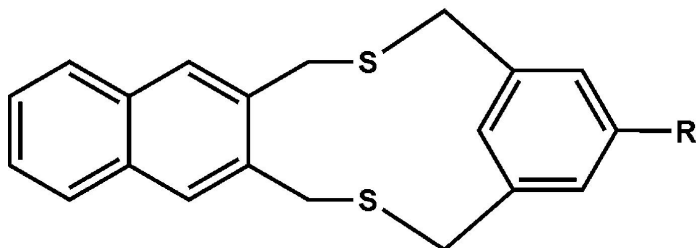Symmetry codes: (i) $x+1, y, z$; (ii) $−x+2, −y+1, −z$.

Fig. 1

Fig. 2

Fig. 3

I

IIa: R = H
IIb: R = CH₃