

4305 reflections  
289 parameters  
H atoms riding (see text)  
 $w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 1.5671P]$   
where  $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from  
*International Tables for  
Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C9A—S10A	1.664 (4)	C9B—S10B	1.666 (4)
C9A—S11A	1.735 (4)	C9B—S11B	1.738 (4)
C13A—S14A	1.726 (4)	C13B—S12B	1.734 (4)
C13A—S12A	1.732 (4)	C13B—S14B	1.736 (4)
C15A—S14A	1.805 (5)	C15B—S14B	1.801 (5)
S11A—S12A	2.0486 (14)	S11B—S12B	2.0479 (14)
C9A—S11A—S12A	97.25 (14)	C9B—S11B—S12B	96.71 (13)
C13A—S12A—S11A	93.61 (13)	C13B—S12B—S11B	93.99 (13)
C13A—S14A—C15A	104.7 (2)	C13B—S14B—C15B	105.5 (2)

Owing to technical reasons beyond our control, we were forced to measure a slightly restricted data set (up to  $65^\circ$  in  $\theta$  instead of the usual  $67^\circ$ ). In spite of this drawback, a very adequate ratio of reflections to parameters ( $\sim 15$ ) was attained, and refinement on  $F^2$  using the whole data set led to reasonable  $R$  factors, as well as satisfactory s.u. values for parameters involving non-H atoms. H atoms were placed at idealized positions and allowed to ride with isotropic displacement factors 1.25 times larger than those of their hosts.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4-PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *XS* in *SHELXTL/PC* (Sheldrick, 1991). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* (Sheldrick, 1993), *PARST* (Nardelli, 1983) and *CSD* (Allen, Kennard & Taylor, 1983).

Data collection was performed at the Laboratorio Nacional de Difracción (LANADI), La Plata, Argentina.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1441). Services for accessing these data are described at the back of the journal.

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## Four 9-Alkylthiophenanthrenes at 193 K (Alkyl = Methyl, Ethyl, Propyl and Butyl)

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## Abstract

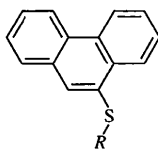
Four 9-alkylthiophenanthrenes, namely, 9-methylthiophenanthrene,  $C_{15}H_{12}S$ , (1), 9-ethylthiophenanthrene,  $C_{16}H_{14}S$ , (2), 9-propylthiophenanthrene,  $C_{17}H_{16}S$ , (3) and 9-butylthiophenanthrene,  $C_{18}H_{18}S$ , (4), have been characterized by X-ray analysis. Compound (1) crystallizes in space group  $P2_1$  with two molecules, and the other three compounds, in a novel display of isostructurality, in space group  $P2_1/c$ , with one molecule per asymmetric unit.

## Comment

Some 9-alkylthiophenanthrenes were recently required as starting materials in connection with our studies of the preparation of new hemithiodithioacetals and their reactions (Kansikas, Leskelä, Sipilä & Hase, 1995; Kansikas, Sipilä & Hase, 1996).

After crystallization, the title compounds (2), (3) and (4) were found to appear as colorless plates, and compound (1) as light brown rods or yellow needles. Melting points were determined in open capillary tubes with an electrothermal apparatus and are uncorrected. Thin yellow needles of (1) have a melting point of 367 K, but the thicker light brown crystals used in the structure determination have a melting point of

364 K. The melting point for compound (2) is in good agreement with the range 352–355 K for 9-ethylthiophenanthrene crystallized from petrol ether–dichloromethane (Node, Nishide, Ohta, Fuji, Fujita, Hori & Inayama, 1983).



- (1)  $R = CH_3$
- (2)  $R = C_2H_5$
- (3)  $R = C_3H_7$
- (4)  $R = C_4H_9$

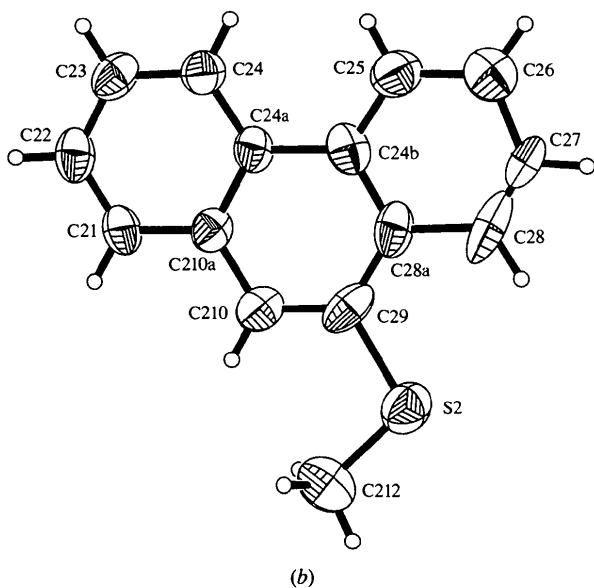
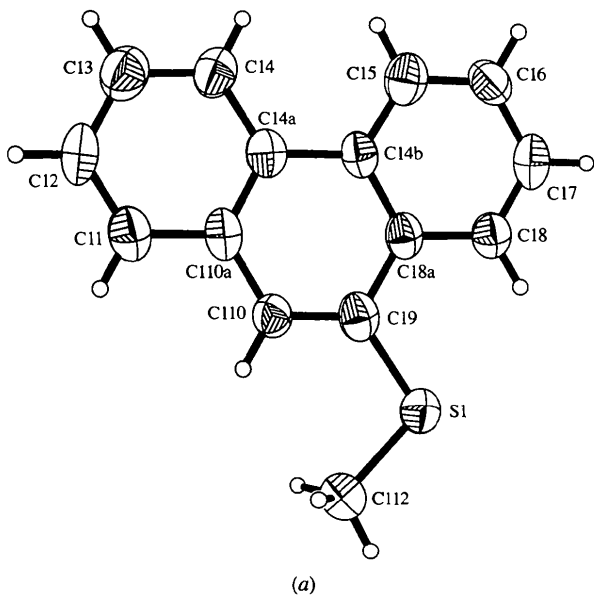


Fig. 1. View of (a) molecule *a* and (b) molecule *b* of compound (1) with atom labels. Displacement ellipsoids are drawn at the 50% probability level.

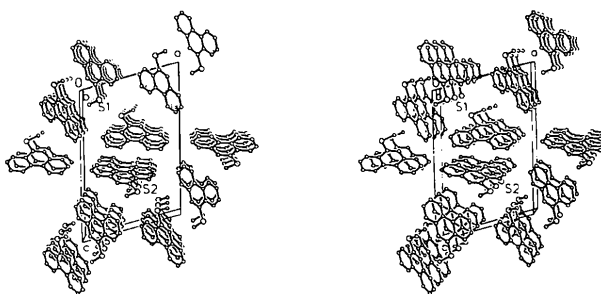


Fig. 2. Stereoscopic view of compound (1) showing molecular packing as seen from the *b*-axis direction.

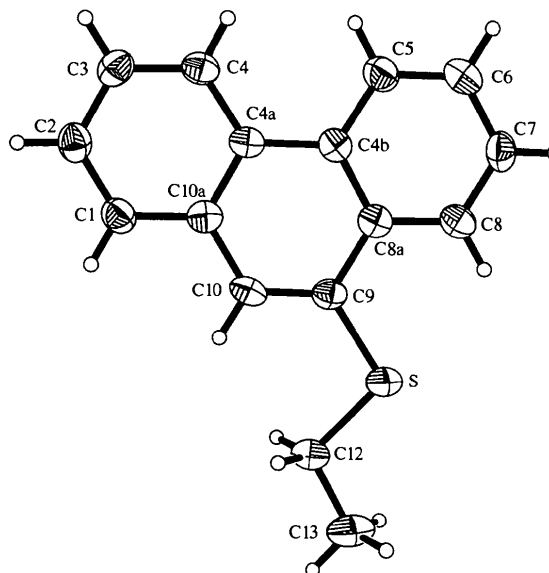


Fig. 3. View of compound (2) with atom labels. Displacement ellipsoids are drawn at the 50% probability level.

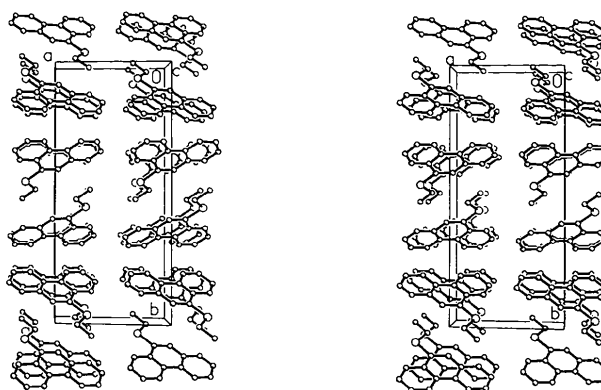


Fig. 4. Stereoscopic view of compound (2) showing molecular packing as seen from the *c*-axis direction.

Bond lengths C4a—C4b and C9—C10 in phenanthrene have been found to differ by 0.122 and 0.100 Å in an X-ray (5*a*) and a neutron (5*b*) study, respectively (Kay, Okaya & Cox, 1971). For 9-trimethylsilyl-

phenanthrene, (6) (Lu, Hseu & Lee, 1977), and the present structures, (1)–(4), the differences are 0.099, 0.100, 0.102, 0.098, 0.092 and 0.073 Å for (1*a*), (1*b*), (2), (3), (4) and (6), respectively. The packing diagram for (4) is similar to Fig. 4.

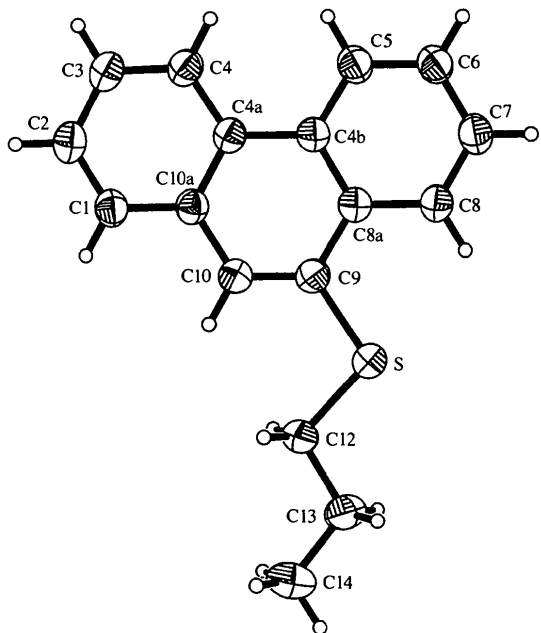


Fig. 5. View of compound (3) with atom labels. Displacement ellipsoids are drawn at the 50% probability level.

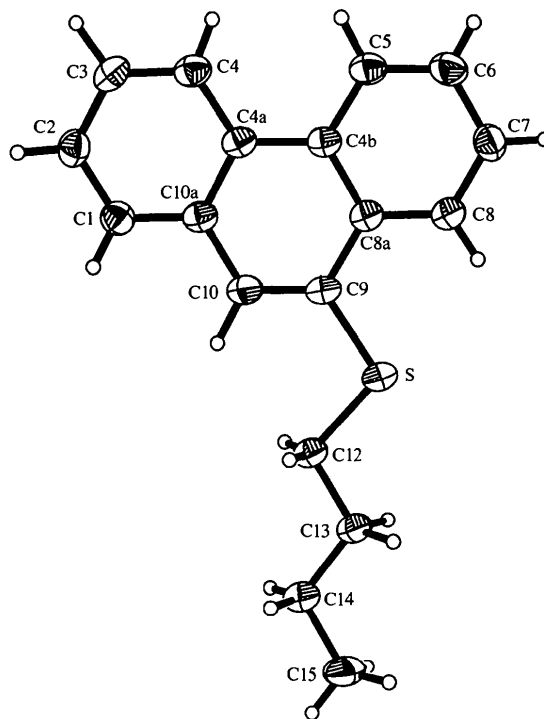


Fig. 7. View of compound (4) with atom labels. Displacement ellipsoids are drawn at the 50% probability level.

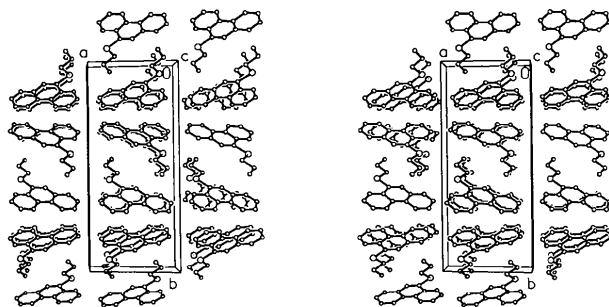


Fig. 6. Stereoscopic view of compound (3) showing molecular packing as seen from the *c*-axis direction.

## Experimental

The 9-alkylthiophenanthrenes were prepared from 9-bromophenanthrene, KOH and alkylthiol in dimethylformamide (Sipilä & Hase, 1997). Crystals suitable for X-ray analysis were obtained by recrystallization from ethanol.

### Compound (1)

#### Crystal data

$C_{15}H_{12}S$   
 $M_r = 224.31$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$

#### Monoclinic

$P2_1$

$a = 11.786 (2) \text{ \AA}$

$b = 5.543 (1) \text{ \AA}$

$c = 17.799 (4) \text{ \AA}$

$\beta = 106.16 (3)^\circ$

$V = 1116.9 (4) \text{ \AA}^3$

$Z = 4$

$D_x = 1.334 \text{ Mg m}^{-3}$

$D_m$  not measured

#### Cell parameters from 20 reflections

$\theta = 5\text{--}10^\circ$

$\mu = 0.255 \text{ mm}^{-1}$

$T = 193 (2) \text{ K}$

Rod

$0.3 \times 0.2 \times 0.2 \text{ mm}$

Light brown

#### Data collection

Rigaku AFC-7S diffractometer

$\omega/2\theta$  scans

Absorption correction: none

2323 measured reflections

2323 independent reflections

1815 reflections with

$I > 2\sigma(I)$

$\theta_{\max} = 26.51^\circ$

$h = 0 \rightarrow 14$

$k = 0 \rightarrow 6$

$l = -22 \rightarrow 21$

3 standard reflections

every 100 reflections  
intensity decay: <1%

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.073$

$wR(F^2) = 0.209$

$S = 1.040$

2306 reflections

291 parameters

$\Delta\rho_{\max} = 0.577 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.571 \text{ e \AA}^{-3}$

Extinction correction:

*SHELXL93*

Extinction coefficient:

0.005 (5)

## H atoms riding

$$w = 1/[\sigma^2(F_o^2) + (0.103P)^2 + 1.09P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

## Scattering factors from

*International Tables for Crystallography* (Vol. C)

Absolute configuration:  
Flack (1983)

$$\text{Flack parameter} = 0.16 \quad (22)$$

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

S1—C19	1.765 (7)	S2—C29	1.797 (7)
S1—C112	1.797 (9)	S2—C212	1.741 (9)
C14—C14a	1.422 (9)	C24—C24a	1.410 (10)
C14a—C14b	1.465 (9)	C24a—C24b	1.470 (9)
C14a—C110a	1.399 (10)	C24a—C210a	1.409 (10)
C14b—C15	1.402 (10)	C24b—C25	1.366 (11)
C14b—C18a	1.415 (9)	C24b—C28a	1.416 (11)
C18a—C19	1.448 (10)	C28a—C29	1.417 (11)
C19—C110	1.363 (9)	C29—C210	1.372 (10)
C110—C110a	1.430 (9)	C210—C210a	1.447 (10)
C19—S1—C112	103.1 (4)	C29—S2—C212	104.7 (4)
C14—C14a—C14b	122.2 (7)	C24—C24a—C24b	122.9 (7)
C14b—C14a—C110a	119.1 (6)	C24b—C24a—C210a	118.2 (6)
C14a—C14b—C15	122.9 (6)	C24a—C24b—C25	123.1 (7)
C14a—C14b—C18a	119.0 (6)	C24a—C24b—C28a	119.0 (7)
C14b—C18a—C19	120.1 (6)	C24b—C28a—C29	120.8 (6)
C110—C19—C18a	120.0 (6)	C210—C29—C28a	121.1 (7)
C110—C19—S1	124.0 (6)	C210—C29—S2	118.5 (7)
C18a—C19—S1	116.0 (5)	C28a—C29—S2	120.3 (6)
C19—C110—C110a	121.3 (6)	C29—C210—C210a	119.8 (7)
C14a—C110a—C110	120.5 (6)	C24a—C210a—C210	121.1 (6)

## Compound (2)

## Crystal data

C<sub>16</sub>H<sub>14</sub>SM<sub>r</sub> = 238.35

Monoclinic

P2<sub>1</sub>/c

a = 9.054 (4) Å

b = 20.644 (9) Å

c = 6.659 (3) Å

β = 98.10 (3)°

V = 1232.2 (10) Å<sup>3</sup>

Z = 4

D<sub>x</sub> = 1.285 Mg m<sup>-3</sup>D<sub>m</sub> not measured

## Data collection

Rigaku AFC-7S diffractometer

ω/2θ scans

Absorption correction: none

1897 measured reflections

1851 independent reflections

1389 reflections with

I &gt; 2σ(I)

## Refinement

Refinement on F<sup>2</sup>R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.068wR(F<sup>2</sup>) = 0.155

S = 1.030

1843 reflections

155 parameters

H atoms riding

## Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 20 reflections

θ = 5–10°

μ = 0.235 mm<sup>-1</sup>

T = 193 (2) K

Plate

0.3 × 0.2 × 0.1 mm

Colourless

R<sub>int</sub> = 0.037θ<sub>max</sub> = 25.00°

h = -10 → 10

k = 0 → 24

l = 0 → 7

3 standard reflections

every 100 reflections

intensity decay: none

(Δ/σ)<sub>max</sub> < 0.001Δρ<sub>max</sub> = 0.316 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.343 e Å<sup>-3</sup>

Extinction correction:

SHELXL93

Extinction coefficient:

0.0032 (19)

$$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 1.426P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

## Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 2. Selected geometric parameters (Å, °) for (2)

S—C9	1.769 (4)	C4b—C8a	1.422 (6)
S—C12	1.794 (4)	C8a—C9	1.449 (5)
C4—C4a	1.412 (6)	C9—C10	1.360 (6)
C4a—C4b	1.462 (5)	C10—C10a	1.426 (6)
C4a—C10a	1.405 (6)	C12—C13	1.526 (6)
C4b—C5	1.406 (6)		
C9—S—C12	104.1 (2)	C10—C9—C8a	119.9 (4)
C4—C4a—C4b	123.1 (4)	C10—C9—S	123.9 (3)
C4b—C4a—C10a	118.2 (4)	C8a—C9—S	116.2 (3)
C4a—C4b—C5	121.6 (4)	C9—C10—C10a	121.8 (4)
C4a—C4b—C8a	119.9 (4)	C4a—C10a—C10	120.7 (4)
C4b—C8a—C9	119.4 (4)	C13—C12—S	108.2 (3)

## Compound (3)

## Crystal data

C<sub>17</sub>H<sub>16</sub>SM<sub>r</sub> = 252.37

Monoclinic

P2<sub>1</sub>/c

a = 9.067 (3) Å

b = 21.123 (7) Å

c = 7.007 (3) Å

β = 99.12 (3)°

V = 1325.05 (9) Å<sup>3</sup>

Z = 4

D<sub>x</sub> = 1.265 Mg m<sup>-3</sup>D<sub>m</sub> not measured

## Data collection

Rigaku AFC-7S diffractometer

ω/2θ scans

Absorption correction: none

2654 measured reflections

2599 independent reflections

2419 reflections with

I &gt; 2σ(I)

## Refinement

Refinement on F<sup>2</sup>R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.050wR(F<sup>2</sup>) = 0.186

S = 1.040

2585 reflections

227 parameters

H atoms: refined coordinates

and isotropic U

## Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 20 reflections

θ = 4–10°

μ = 0.223 mm<sup>-1</sup>

T = 193 (2) K

Plate

0.3 × 0.3 × 0.2 mm

Colourless

R<sub>int</sub> = 0.036θ<sub>max</sub> = 26.49°

h = -11 → 11

k = 0 → 26

l = 0 → 8

3 standard reflections

every 100 reflections

intensity decay: none

$$w = 1/[\sigma^2(F_o^2) + (0.142P)^2 + 0.179P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.004$$

$$\Delta\rho_{\max} = 0.474 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.295 \text{ e } \text{Å}^{-3}$$

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 3. Selected geometric parameters (Å, °) for (3)

S—C9	1.764 (2)	C4b—C8a	1.418 (2)
S—C12	1.806 (2)	C8a—C9	1.452 (3)
C4—C4a	1.411 (2)	C9—C10	1.363 (3)
C4a—C4b	1.462 (2)	C10—C10a	1.430 (2)
C4a—C10a	1.411 (3)	C12—C13	1.524 (3)
C4b—C5	1.407 (3)	C13—C14	1.511 (3)
C9—S—C12	104.06 (9)	C10—C9—S	123.68 (14)
C4—C4a—C4b	123.1 (2)	C8a—C9—S	116.26 (12)
C4b—C4a—C10a	118.6 (2)	C9—C10—C10a	121.7 (2)

C4a—C4b—C5	121.9 (2)	C4a—C10a—C10	120.3 (2)
C4a—C4b—C8a	119.8 (2)	C13—C12—S	107.45 (13)
C4b—C8a—C9	119.47 (15)	C14—C13—C12	111.3 (2)
C10—C9—C8a	120.1 (2)		

**Compound (4)***Crystal data*C<sub>18</sub>H<sub>18</sub>S $M_r = 266.38$ 

Monoclinic

 $P2_1/c$  $a = 9.010 (4) \text{ \AA}$  $b = 23.124 (8) \text{ \AA}$  $c = 6.888 (3) \text{ \AA}$  $\beta = 97.81 (3)^\circ$  $V = 1421.8 (10) \text{ \AA}^3$  $Z = 4$  $D_x = 1.244 \text{ Mg m}^{-3}$  $D_m$  not measured*Data collection*

Rigaku AFC-7S diffractometer

 $\omega/2\theta$  scans

Absorption correction: none

2851 measured reflections

2851 independent reflections

2273 reflections with

 $I > 2\sigma(I)$ *Refinement*Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.060$  $wR(F^2) = 0.166$  $S = 1.964$ 

2839 reflections

227 parameters

H atoms riding

 $w = 1/[\sigma^2(F_o^2) + (0.0016P)^2 + 0.1P]$ where  $P = (F_o^2 + 2F_c^2)/3$ Mo  $K\alpha$  radiation $\lambda = 0.71069 \text{ \AA}$ 

Cell parameters from 20 reflections

 $\theta = 4-8^\circ$  $\mu = 0.211 \text{ mm}^{-1}$  $T = 193 (2) \text{ K}$ 

Plate

 $0.3 \times 0.2 \times 0.2 \text{ mm}$ 

Colourless

 $\theta_{\max} = 26.51^\circ$  $h = -11 \rightarrow 11$  $k = -28 \rightarrow 28$  $l = 0 \rightarrow 8$ 

20 standard reflections

every 100 reflections

intensity decay: none

 $(\Delta/\sigma)_{\max} = -0.077$  $\Delta\rho_{\max} = 0.362 \text{ e \AA}^{-3}$  $\Delta\rho_{\min} = -0.631 \text{ e \AA}^{-3}$ 

Extinction correction:

*SHELXL93*

Extinction coefficient:

0.0016 (14)

Scattering factors from

*International Tables for Crystallography* (Vol. C)

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1230). Services for accessing these data are described at the back of the journal.

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 Sipilä, K. & Hase, T. (1997). *Synth. Commun.* **27**, 1391–1393.

*Acta Cryst.* (1997). **C53**, 1131–1133

### 5,8-Bis(4-methoxy-2,3,6-trimethylbenzene-sulfonyl)-1,4-dioxo-5,8-diazocane

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**Abstract**

The molecular shape of the title compound, C<sub>24</sub>H<sub>34</sub>N<sub>2</sub>O<sub>8</sub>S<sub>2</sub>, in the crystalline state is characterized by internal twofold symmetry, with the eight-membered ring adopting a crown conformation.

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (4)

S—C9	1.766 (3)	C8a—C9	1.459 (4)
S—C12	1.803 (3)	C9—C10	1.357 (4)
C4—C4a	1.410 (3)	C10—C10a	1.430 (4)
C4a—C4b	1.449 (4)	C12—C13	1.527 (4)
C4a—C10a	1.416 (4)	C13—C14	1.525 (4)
C4b—C5	1.418 (4)	C14—C15	1.521 (4)
C4b—C8a	1.430 (3)		
C9—S—C12	104.51 (13)	C10—C9—S	123.6 (2)
C4—C4a—C4b	123.1 (2)	C8a—C9—S	115.7 (2)
C4b—C4a—C10a	118.8 (2)	C9—C10—C10a	121.5 (2)
C4a—C4b—C5	122.5 (2)	C4a—C10a—C10	120.3 (2)
C4a—C4b—C8a	120.2 (2)	C13—C12—S	107.5 (2)
C4b—C8a—C9	118.4 (2)	C14—C13—C12	111.7 (2)
C10—C9—C8a	120.7 (2)	C15—C14—C13	112.1 (3)

Observed intensities were corrected for Lorentz and polarization effects and extinction. All non-H atoms were refined anisotropically. H atoms with isotropic  $U$  values were placed in calculated positions and refined using a riding model for (1), (2) and (4). In compound (3), additional H-atom coordinates and isotropic  $U$  values were refined.