

C2—C3	1.50 (2)	C7—C8	1.36 (2)
C3—C9	1.50 (2)	C8—C9	1.37 (2)
C3—C11	1.53 (2)	C12—C13	1.53 (2)
C3—C10	1.56 (2)	C12—C15	1.55 (3)
C4—C5	1.41 (2)	C12—C14	1.56 (3)
C4—C9	1.43 (2)		
C2—N1—O	129 (1)	C6—C5—C4	123 (1)
C2—N1—C8	110 (1)	C5—C6—C7	119 (1)
O—N1—C8	122 (1)	C5—C6—C12	121 (1)
N1—C2—C3	114 (1)	C7—C6—C12	120 (1)
C9—C3—C2	98 (1)	C8—C7—C6	117 (1)
C9—C3—C11	111 (1)	C7—C8—C9	126 (1)
C9—C3—C10	113 (1)	C7—C8—N1	127 (1)
C2—C3—C11	111 (1)	C9—C8—N1	107 (1)
C2—C3—C10	113 (1)	C8—C9—C4	118 (1)
C11—C3—C10	110 (1)	C8—C9—C3	111 (1)
C5—C4—C9	117 (1)	C4—C9—C3	131 (1)

The structure was solved by direct methods (*SIMPEL*; Schenk & Hall, 1990) and refined by full-matrix least-squares calculations; anisotropic for non-H atoms and isotropic for H atoms. The H atoms were positioned geometrically and included as riding atoms in the structure-factor calculations. After isotropic refinement an empirical absorption correction (*DIFABS*; Walker & Stuart, 1983) was applied. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* program comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal ADDREF*, *SORTRF*. Program(s) used to solve structure: *Xtal SIMPEL*. Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *Xtal BONDLA*, *CIFIO*.

We thank Ing. J. Fraanje for collecting the X-ray data and Drs. K. Goubitz for his help with the calculations and the preparation of the drawings.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71334 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1054]

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*Acta Cryst.* (1993). **C49**, 1973–1975

## Structure of *rac*-(3*aS*,9*bR*)-3*a*,4,5,9*b*-Tetrahydronaphtho[1,2-*b*]furan-2(3*H*)-one

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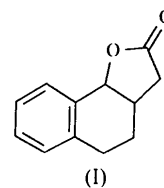
(Received 19 January 1993; accepted 31 March 1993)

## Abstract

The molecule of the title compound presents a *cis* B/C ring junction. The B ring adopts an almost perfect sofa conformation, the C ring an almost perfect envelope.

## Comment

The preparation of the title compound (I) will be reported elsewhere (Ghosh, 1993). Crystals suitable for X-ray diffraction were found in the original sample. The crystal-structure determination has been undertaken to establish the relative stereochemical configuration of the title compound.



There are no unusual geometric features present in the molecule; the C—O bond distances in the five-membered ring fall within the range given by Allen *et al.* (1987). The  $\gamma$ -lactone ring fusion is *cis* and the H3*a*—C3*a*—C9*b*—H9*b* torsion angle is 36 (2)°. The phenyl ring is essentially planar; the maximum deviation of an atom from the six-atom plane is 0.011 (3) Å. The cyclohexene ring adopts a sofa conformation with atoms C5, C5*a*, C9*a*, C9*b* and C3*a* virtually coplanar and atom C4 displaced 0.678 (4) Å out of the plane; the ring displacement asymmetry parameter (Duax, Weeks & Rohrer, 1976)  $\Delta C_s(C4)$  is 1.2°. The  $\gamma$ -lactone ring is in an envelope conformation, in which C3*a*

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sits 0.506 (4) Å out of plane from the other four atoms; the ring displacement asymmetry parameter  $\Delta C_s(C3a)$  is 0.8°. The title compound forms the ring skeleton in the crystal structures of a number of bromo substituted desmotroposantonin compounds: in 2-bromo(-)- $\alpha$ -desmotroposantonin (White & Sim, 1977a), 2,7-dibromo(-)- $\beta$ -desmotroposantonin (White & Sim, 1977b) and 2-bromo(-)- $\beta$ -desmotroposantonin (McPhail, Rimmer, Robertson & Sim, 1967) the B/C ring junction is always found to be *cis*.

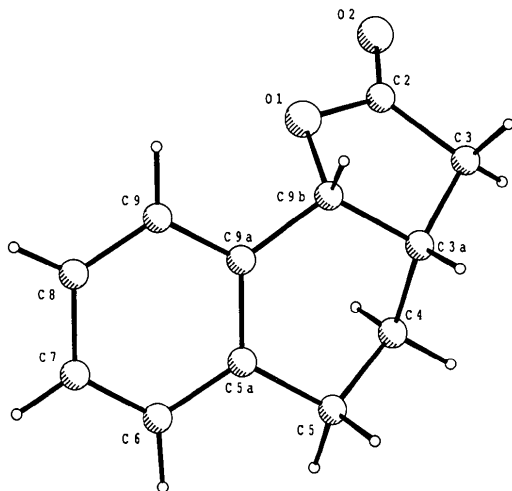


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing showing the numbering system of the title compound. The H atoms are shown but not labelled.

## Experimental

### Crystal data

$C_{12}H_{12}O_2$

$M_r = 188.23$

Monoclinic

$P2_1/c$

$a = 9.433 (3) \text{ \AA}$

$b = 12.674 (3) \text{ \AA}$

$c = 8.276 (3) \text{ \AA}$

$\beta = 101.99 (3)^\circ$

$V = 967.8 (5) \text{ \AA}^3$

$Z = 4$

$D_x = 1.292 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 23 reflections

$\theta = 20.0\text{--}21.5^\circ$

$\mu = 0.081 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block

$0.43 \times 0.43 \times 0.25 \text{ mm}$

Colourless

### Data collection

Enraf-Nonius CAD-4 diffractometer

$\theta/2\theta$  scans

Absorption correction:

None

2963 measured reflections

2927 independent reflections

1514 observed reflections

$[I > 2.5\sigma(I)]$

$\theta_{\max} = 29.89^\circ$

$h = -13 \rightarrow 12$

$k = 0 \rightarrow 17$

$l = 0 \rightarrow 11$

2 standard reflections

frequency: 5000 s

intensity variation: none

### Refinement

Refinement on  $F$

Final  $R = 0.049$

$wR = 0.072$

$S = 0.169$

1514 reflections

176 parameters

$w = 1/(2.01 + F_o$

$+ 0.0091F_o^2)$

$(\Delta/\sigma)_{\max} = 0.092$

$\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.183 \text{ e \AA}^{-3}$

Extinction correction:

Zachariasen (1967)

Extinction coefficient:

1442 (608)

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
O1	0.7167 (2)	0.7584 (1)	0.9241 (2)	0.0578 (9)
C2	0.8589 (3)	0.7507 (3)	0.9235 (3)	0.062 (1)
C3	0.9113 (3)	0.6417 (3)	0.9729 (5)	0.075 (2)
C3a	0.7739 (3)	0.5759 (2)	0.9440 (3)	0.058 (1)
C4	0.7326 (3)	0.5316 (2)	0.7696 (4)	0.064 (2)
C5	0.5835 (3)	0.4818 (2)	0.7396 (4)	0.064 (1)
C5a	0.4711 (3)	0.5600 (2)	0.7699 (3)	0.049 (1)
C6	0.3262 (3)	0.5519 (2)	0.6870 (3)	0.064 (2)
C7	0.2251 (3)	0.6234 (3)	0.7122 (4)	0.070 (2)
C8	0.2629 (3)	0.7056 (2)	0.8223 (4)	0.066 (2)
C9	0.4045 (3)	0.7140 (2)	0.9082 (3)	0.052 (1)
C9a	0.5095 (2)	0.6429 (2)	0.8813 (3)	0.043 (1)
C9b	0.6627 (3)	0.6572 (2)	0.9750 (3)	0.049 (1)
O2	0.9250 (2)	0.8254 (2)	0.8859 (3)	0.087 (1)

Table 2. Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C2	1.345 (3)	C5—C5a	1.510 (4)
O1—C9b	1.474 (3)	C5a—C6	1.400 (4)
C2—C3	1.495 (5)	C5a—C9a	1.395 (3)
C2—O2	1.209 (4)	C6—C7	1.364 (5)
C3—C3a	1.518 (4)	C7—C8	1.381 (4)
C3a—C4	1.523 (4)	C8—C9	1.380 (4)
C3a—C9b	1.530 (4)	C9—C9a	1.390 (3)
C4—C5	1.514 (4)	C9a—C9b	1.502 (3)
C2—O1—C9b	110.0 (2)	C6—C5a—C9a	118.2 (2)
O1—C2—C3	109.8 (2)	C5a—C6—C7	121.3 (3)
O1—C2—O2	120.8 (3)	C6—C7—C8	120.6 (3)
C3—C2—O2	129.4 (3)	C7—C8—C9	119.2 (3)
C2—C3—C3a	104.0 (2)	C8—C9—C9a	120.9 (2)
C3—C3a—C4	113.2 (3)	C5a—C9a—C9	119.9 (2)
C3—C3a—C9b	101.3 (2)	C5a—C9a—C9b	121.4 (2)
C4—C3a—C9b	111.0 (2)	C9—C9a—C9b	118.7 (2)
C3a—C4—C5	110.8 (3)	O1—C9b—C3a	104.4 (2)
C4—C5—C5a	111.0 (2)	O1—C9b—C9a	108.1 (2)
C5—C5a—C6	121.2 (2)	C3a—C9b—C9a	116.9 (2)
C5—C5a—C9a	120.6 (2)		

The structure was solved by direct methods (*SIMPEL*; Schenk & Hall, 1990) and refined by full-matrix least-squares calculations; anisotropic for non-H atoms and isotropic for H atoms. The H atoms were positioned geometrically and refined in the structure-factor calculations. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal ADDREF*, *SORTRF*. Program(s) used to solve structure: *Xtal SIMPEL*. Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *Xtal BONDLA*, *CIFIO*.

The authors thank D. Heijdenrijk for collecting the X-ray data.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71304 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1048]

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*Acta Cryst.* (1993). **C49**, 1975–1976

## Structure of *N,N'*-Dimethylpiperazine-2,3-dithione: Space Group Correction

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(Received 18 March 1993; accepted 19 May 1993)

### Abstract

The crystal structure of *N,N'*-dimethylpiperazine-2,3-dithione,  $C_6H_{10}N_2S_2$ , has been described and refined in space group *Aa* [Servaas, Stufkens, Oskam, Vernooijs, Baerends, De Ridder & Stam (1989). *Inorg. Chem.* **28**,

4104–4113]. It is described properly in space group *A2/a*. Revised coordinates and bond lengths and angles are given.

### Comment

The structure of the title compound was reported in space group *Aa* (Servaas *et al.*, 1989). The six-membered ring of the molecule was found to adopt a half-chair conformation with the twofold axis running through the midpoints of the C–C bonds, parallel to the *b* axis. Furthermore, the two N–C(methyl) bond lengths were very dissimilar [1.439 (5) and 1.489 (5) Å]; there is no reason for them to be unequal. This unusual spread of distances undoubtedly resulted from the refinement of a centrosymmetric structure in a non-centrosymmetric space group (Ermer & Dunitz, 1970; Schomaker & Marsh, 1979). Therefore it seemed likely that the structure should properly be described in space group *A2/a*.

Starting coordinates were from Table 3 of Servaas *et al.* (1989) and averaged in accordance with *A2/a*. Full-matrix minimization of the quantity  $\sum w(F_o - F_c)^2$  with  $w = 1/[\sigma^2(F) + 0.0035F^2]$  was performed. Refinement was anisotropic for the non-H atoms and isotropic for the H atoms. An isotropic extinction coefficient was included in the parameters [Zachariasen, final value  $3.4(5) \times 10^{-5}$ ]. Anomalous dispersion was taken into account but no correction for absorption was applied. The H-atom positions were calculated initially on the basis of standard geometry and refined. A final difference Fourier map revealed residual electron density within the range  $-0.22$ – $0.30 e \text{ \AA}^{-3}$ .

The values obtained for parameters, bond lengths and angles are close to the averages of pairs of equivalent values obtained in the earlier *Aa* refinement. The two N–C(methyl) bond lengths are now equal by symmetry at 1.473 (3) Å. The general description of the structure remains unchanged.

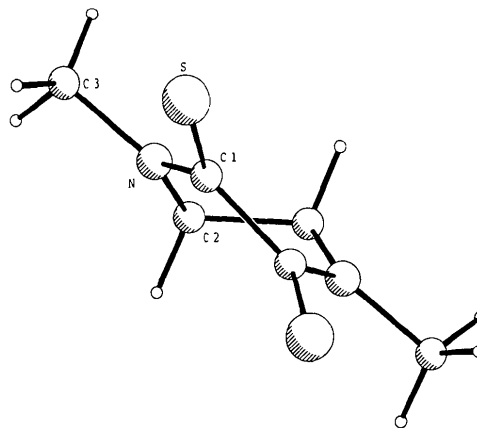


Fig. 1. A *PLUTO* (Motherwell & Clegg, 1978) drawing of  $C_6H_{10}N_2S_2$  showing the labelling of the independent non-H atoms. The view is down the twofold axis, with the crystallographic  $2_1$  axis vertical.

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