

## Structure of an Epoxytetradecahydropyrene

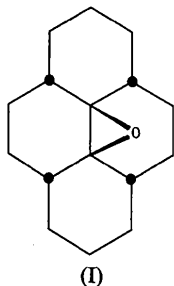
BY MARTIN NIEGER\* AND ERNST EGERT†

Institut für Anorganische Chemie der Universität, Tammannstraße 4, D-3400 Göttingen, Germany

(Received 6 January 1992; accepted 1 March 1992)

**Abstract.** 1,2,3,*r*-3a,4,5,*c*-5a,6,7,8,*c*-8a,9,10,*c*-10a,-10b,10c-hexadecahydro-10b,10c-epoxy-pyrene, C<sub>16</sub>H<sub>24</sub>O, *M<sub>r</sub>* = 232.4, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 15.96 (1), *b* = 5.339 (5), *c* = 16.97 (2) Å, β = 117.43 (6)°, *V* = 1284 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.202 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.7107 Å, μ = 0.07 mm<sup>-1</sup>, *F*(000) = 512, *T* = 298 K, final *R* = 0.089 for 1511 observed reflections. The molecule possesses *C<sub>s</sub>* symmetry. The H atoms at the stereogenic centres [C(3a), C(5a), C(8a) and C(10a)] are *cis* to the epoxy group.

**Experimental.** The title compound (I) was prepared by treatment of tetradecahydropyrene-*t*-10b,*c*-10c-diol (Jussofie, 1984) with glacial acetic acid and formic acid (W. Lüttke & M. I. Jussofie, unpublished).



Colourless crystal 0.2 × 0.5 × 0.7 mm was used for data collection on a Stoe-Siemens four-circle diffractometer, with graphite-monochromated Mo *K*α radiation, and in profile-fitting mode involving variable scan width and speed (Clegg, 1981). Cell constants were determined from ±2θ values of 60 reflections with 20 < 2θ < 25°; increased e.s.d.'s of cell constants (as well as *R<sub>int</sub>* and final *R*) were probably caused by broad reflection profiles. 3864 reflections were measured (2θ<sub>max</sub> = 50°). Three check reflections measured every 100 reflections showed no significant intensity variation. 2245 reflections were unique (*R<sub>int</sub>* = 0.069, index ranges after merging: *h* - 17 → 16, *k* 0 → 6, *l* 0 → 20), of which 1511 with

\* Present address: Institut für Anorganische Chemie der Universität, Gerhard-Domagk-Straße 1, D-5300 Bonn, Germany.

† Present address: Institut für Organische Chemie der Universität, Niederurseler Hang, D-6000 Frankfurt am Main, Germany.

Table 1. Atomic coordinates (× 10<sup>4</sup>) and equivalent isotropic displacement coefficients (Å<sup>2</sup> × 10<sup>3</sup>)Equivalent isotropic *U* is defined as one third of the trace of the orthogonalized *U<sub>ij</sub>* tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
C(1)	4098 (2)	-36 (8)	6643 (2)	52 (2)
C(2)	3628 (2)	93 (9)	7239 (2)	62 (2)
C(3)	2567 (2)	-29 (8)	6665 (2)	51 (2)
C(3a)	2182 (2)	2119 (7)	5998 (2)	38 (1)
C(4)	1134 (2)	1633 (8)	5391 (2)	48 (2)
C(5)	719 (2)	3229 (8)	4555 (2)	51 (2)
C(5a)	1210 (2)	2739 (7)	3984 (2)	41 (1)
C(6)	986 (2)	207 (7)	3510 (2)	53 (2)
C(7)	1488 (2)	-113 (8)	2945 (2)	58 (2)
C(8)	2542 (3)	232 (7)	3498 (2)	53 (2)
C(8a)	2786 (2)	2757 (7)	3966 (2)	42 (2)
C(9)	3846 (2)	3226 (8)	4525 (2)	52 (2)
C(10)	4254 (2)	1695 (8)	5359 (2)	53 (2)
C(10a)	3829 (2)	2125 (7)	5983 (2)	40 (1)
C(10b)	2758 (2)	2636 (6)	5498 (2)	32 (1)
C(10c)	2270 (2)	3045 (6)	4513 (2)	34 (1)
O	2560 (2)	5176 (4)	5109 (1)	42 (1)

Table 2. Bond lengths (Å) and angles (°)

C(1)—C(2)	1.513 (6)	C(1)—C(10a)	1.525 (5)
C(2)—C(3)	1.517 (5)	C(3)—C(3a)	1.528 (5)
C(3a)—C(4)	1.529 (4)	C(3a)—C(10b)	1.537 (6)
C(4)—C(5)	1.521 (5)	C(5)—C(5a)	1.524 (6)
C(5a)—C(6)	1.529 (5)	C(5a)—C(10c)	1.515 (4)
C(6)—C(7)	1.515 (6)	C(7)—C(8)	1.514 (5)
C(8)—C(8a)	1.521 (5)	C(8a)—C(9)	1.532 (5)
C(8a)—C(10c)	1.506 (6)	C(9)—C(10)	1.499 (5)
C(10)—C(10a)	1.517 (6)	C(10a)—C(10b)	1.542 (4)
C(10b)—C(10c)	1.499 (4)	C(10b)—O	1.477 (4)
C(10c)—O	1.449 (4)		
C(2)—C(1)—C(10a)	113.4 (3)	C(1)—C(2)—C(3)	108.7 (3)
C(2)—C(3)—C(3a)	113.1 (3)	C(3)—C(3a)—C(4)	108.2 (3)
C(3)—C(3a)—C(10b)	113.9 (3)	C(4)—C(3a)—C(10b)	113.9 (3)
C(3a)—C(4)—C(5)	113.8 (3)	C(4)—C(5)—C(5a)	111.5 (3)
C(5)—C(5a)—C(6)	115.0 (3)	C(5)—C(5a)—C(10c)	111.5 (3)
C(6)—C(5a)—C(10c)	109.5 (3)	C(5a)—C(6)—C(7)	111.7 (3)
C(6)—C(7)—C(8)	110.8 (3)	C(7)—C(8)—C(8a)	112.0 (3)
C(8)—C(8a)—C(9)	114.3 (3)	C(8)—C(8a)—C(10c)	109.4 (3)
C(9)—C(8a)—C(10c)	111.3 (3)	C(8a)—C(9)—C(10)	111.8 (3)
C(9)—C(10)—C(10a)	115.9 (3)	C(1)—C(10a)—C(10)	109.5 (3)
C(1)—C(10a)—C(10b)	113.2 (3)	C(10)—C(10a)—C(10b)	113.3 (2)
C(3a)—C(10b)—C(10a)	118.1 (3)	C(3a)—C(10b)—C(10c)	120.4 (3)
C(10a)—C(10b)—C(10c)	120.0 (3)	C(3a)—C(10b)—O	110.6 (3)
C(10a)—C(10b)—O	111.0 (3)	C(10c)—C(10b)—O	58.3 (2)
C(5a)—C(10c)—C(8a)	113.7 (3)	C(5a)—C(10c)—C(10b)	120.6 (3)
C(8a)—C(10c)—C(10b)	121.4 (3)	C(5a)—C(10c)—O	114.1 (3)
C(8a)—C(10c)—O	114.9 (3)	C(10b)—C(10c)—O	60.1 (2)
C(10b)—O—C(10c)	61.6 (2)		

|*F*| > 3σ(*F*) were used for all calculations (*SHELXTL*; Sheldrick, 1985). No absorption or extinction corrections were applied. The structure was solved by direct methods and blocked-cascade refinement on *F* converged to *R* = 0.089, *wR* = 0.092 and *S* = 1.58. In the refinement all non-H atoms

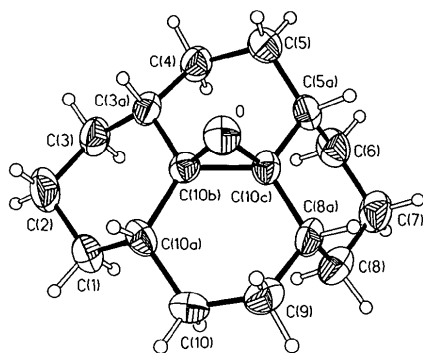


Fig. 1. A 50% thermal-ellipsoid plot with the atom numbering.

were anisotropic; all H atoms, found by  $\Delta\rho$  map, were refined using a riding model and idealized geometry [C—H = 0.96 Å,  $U(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ ]. 154 parameters were refined, with the weighting scheme  $w^{-1} = \sigma^2(F) + 0.0005F^2$ , which led to a featureless analysis of variance in terms of  $\sin\theta$  and  $F_o$ ; maximum  $\Delta/\sigma = 0.01$  in the last cycle. Maximum and minimum heights in the final  $\Delta\rho$  map were 0.31 and  $-0.31 \text{ e \AA}^{-3}$ , respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are

*Acta Cryst.* (1992). **C48**, 2045–2047

## 2,11-Dithia[3.3]metacyclophane-9-carboxylic Acid

BY WILFRIED ASSENMACHER AND MARTIN JANSEN\*

*Institut für Anorganische Chemie, Universität Bonn, Gerhard-Domagk-Strasse 1, W-5300 Bonn 1, Germany*

AND RALF GÜTHER AND FRITZ VÖGTLE

*Institut für Organische Chemie, Universität Bonn, Gerhardt-Domagk-Strasse 1, W-5300 Bonn 1, Germany*

(Received 3 December 1991; accepted 6 March 1992)

**Abstract.** 3,11-Dithiatricyclo[11.3.1.1<sup>5,9</sup>]octadeca-1(17),5,7,9(18),13,15-hexaene-17-carboxylic acid,  $\text{C}_{17}\text{H}_{16}\text{O}_2\text{S}_2$ ,  $M_r = 316.43$ , monoclinic,  $P2_1/n$ ,  $a = 9.285$  (4),  $b = 11.297$  (3),  $c = 14.858$  (5) Å,  $\beta = 103.80$  (2)°,  $V = 1513.5$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.39 \text{ g cm}^{-3}$ ,  $\text{Mo K}\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 3.376 \text{ cm}^{-1}$ ,  $F(000) = 664$ ,  $T = 293 \text{ K}$ , final  $R = 0.051$ ,  $wR = 0.034$  for 3315 independent reflections with  $F > 4\sigma(F)$ . In the solid state the molecules form dimers with two O—H $\cdots$ O hydrogen bonds between a pair of carboxylic acid groups. The plane formed by the two COOH groups is twisted with respect to

given in Table 1,\* bond distances and angles in Table 2. Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

**Related literature.** For partially hydrogenated pyrenes see Jussofie (1984). We have found no structures of hydrogenated pyrenes with boat conformation in the Cambridge Structural Database.

We thank Professor Lüttke and Dr Jussofie (University of Göttingen, Germany) for kindly providing the crystals.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55288 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1003]

### References

- CLEGG, W. (1981). *Acta Cryst.* **A37**, 22–28.  
 JUSSFIE, M. I. (1984). PhD Thesis. Univ. of Göttingen, Germany.  
 SHELDRIK, G. M. (1985). *SHELXTL*. Crystal structure determination program for the DG-Eclipse minicomputer. Univ. of Göttingen, Germany.

the attached arene rings at an angle of 45.9 (1)°. The arene rings are oriented in *syn* conformation forming a dihedral angle of 15.65 (5)°. The S-containing bridges are in a boat–chair arrangement; of three possible *syn* conformers, this has the longest intramolecular S—S distance. The aromatic rings are nearly planar; the substituents, however, display deviations from the aromatic plane.

**Experimental.** The title compound was synthesized from 2,6-bis(bromomethyl)benzoic acid *tert*-butyl ester and 1,3-bis(mercaptomethyl)benzene in benzene/ethanol 1:1 with NaOH as base, using high-dilution conditions (*cf.* Vögtle, Grütze, Nätscher,

\* To whom correspondence should be addressed.