Crystal Structure of Racemic *trans*-1a,7b-Dihydro-oxireno[*a*]naphthalene-3-spiro-2′-oxiran-2(3*H*)-one

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The title compound crystallizes in the orthorhombic space group *Pbca* with cell dimensions $a = 16{\cdot}35$, $b = 12{\cdot}39$, $c = 8{\cdot}44$ Å, $Z = 8$. The structure was solved from photographic data by the symbolic addition method and refined by a full-matrix least-squares procedure with unit weights to R 0·138 for 312 observed reflections.

CHEMICAL methods have failed to determine the relative disposition of the two epoxide oxygen atoms in an isomer of 1a,7b-dihydro-oxireno[*a*]naphthalene-3-spiro-2′-oxiran-2(3*H*)-one.[1] We now report the results of a three-dimensional single-crystal *X*-ray analysis of this compound.

EXPERIMENTAL

Single crystals were obtained as clear plate-like prisms. A small single-crystal fragment was fashioned from a plate 0·1 mm thick as a cylinder 0·2 × 0·1 mm.

Crystal Data.—$C_{11}H_8O_3$, $M = 188{\cdot}18$, Orthorhombic, $a = 16{\cdot}35 \pm 0{\cdot}05$, $b = 12{\cdot}39 \pm 0{\cdot}04$, $c = 8{\cdot}44 \pm 0{\cdot}02$ Å, $U = 1709{\cdot}75$ Å^3, $D_m = 1{\cdot}45$ (by flotation), $Z = 8$, $D_c = 1{\cdot}46$, $F(000) = 344$. Space group *Pbca* (No. 61, D_{2h}^{15}) uniquely determined from systematic absences, Cu-K_α radiation, $\lambda = 1{\cdot}5418$ Å, μ(Cu-K_α) = 8·95 cm^{-1}.

The crystal data were derived from oscillation and Weissenberg photographs taken with the crystal fragment mounted such that the oscillation axis was parallel to the cylinder axis (later found to be the *a* axis).

Intensity data (766 reflections, of which 312 were classed as observed) were collected from this small crystal by the Weissenberg multiple-film method for the levels 0—12*kl*. Standard Lorentz and polarization corrections were applied and normalized structure factors calculated and sorted on magnitude by use of the initial programmes in the direct-phasing series of Hall[2] the *modus operandi* of which is described by Oh and Maslen.[3] The *E*-statistics confirmed the unique centrosymmetry and the data were then subjected to the symbolic addition procedure.[4] The most probable set of phases produced by this method was used to calculate a three-dimensional Fourier synthesis, from which nine of the most prominent maxima were assigned to

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[1] J. W. Ager, F. W. Eastwood, and R. Robinson, *Tetrahedron*, Suppl. No. 7, 1966, 277.

[2] S. R. Hall, University of Western Australia, ‘Crystallographic Programs for the PDP-6 Computer,’ 1968.

[3] Y. L. Oh and E. N. Maslen, *Acta Cryst.*, 1968, **B24**, 883.

[4] P. Main, M. W. Woolfson, and G. Germain, ‘LSAM: a System of Computer Programmes for the Automatic Solution of Centrosymmetric Crystal Structures,’ University of York, and University of Louvain, Belgium.

nine of the fourteen non-hydrogen atoms. The positions of the remaining heavier atoms were determined from the Fourier and difference Fourier syntheses calculated by use of the phases generated from the partial structure, for which all non-hydrogen atoms were given carbon scattering factors. Residual peaks in the difference synthesis enabled correct assignment of oxygen atoms and it was clear at this stage that the compound was the *trans*-isomer.

Full-matrix least-squares refinement of this model, with isotropic thermal parameters and excluding hydrogen atoms, gave a conventional R 0·138 for the 312 observed reflections; refinement with all data proved difficult owing to the high proportion of unobserved reflections. Final parameters quoted in Table 1 are from observed data only. Observed and calculated structure factors are given in Supplementary Publication No. SUP 20356 (2 pp., 1 microfiche).* Scattering curves were taken from ref. 5.

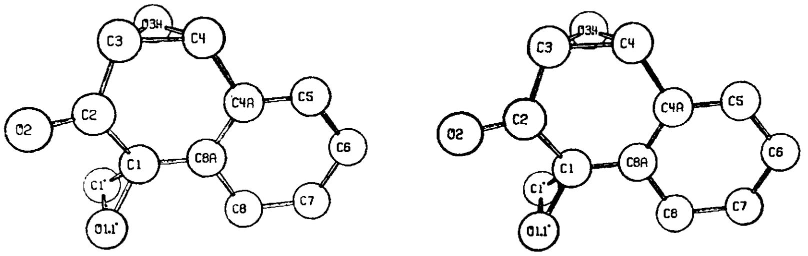

FIGURE 1 A stereoscopic view of one enantiomer of the title compound excluding hydrogen

TABLE 1

Fractional co-ordinates and thermal parameters (Å^2) with estimated standard deviations in parentheses

	x/a	y/b	z/c	B
C(1)	0·142(2)	0·357(2)	0·119(3)	0·7(7)
O(1,1′)	0·192(1)	0·314(2)	−0·009(3)	3·5(6)
C(1′)	0·192(2)	0·252(3)	0·146(4)	3·1(8)
C(2)	0·184(2)	0·463(3)	0·182(4)	1·9(8)
O(2)	0·254(2)	0·487(2)	0·146(2)	3·8(6)
C(3)	0·144(2)	0·529(3)	0·306(4)	3·3(9)
O(3,4)	0·099(1)	0·455(2)	0·425(2)	2·9(5)
C(4)	0·047(2)	0·511(3)	0·306(4)	2·3(8)
C(4A)	0·007(2)	0·433(2)	0·188(3)	1·0(7)
C(5)	−0·077(2)	0·438(3)	0·178(4)	2·7(9)
C(6)	−0·122(2)	0·365(3)	0·078(4)	3·4(8)
C(7)	−0·073(2)	0·288(2)	−0·008(3)	1·4(7)
C(8)	0·020(2)	0·287(3)	0·002(4)	3·2(9)
C(8A)	0·057(2)	0·361(2)	0·108(3)	0·6(7)

To test the *trans*-model, atom C(1′) was given the scattering curve of an oxygen atom and atom O(1,1′) that of a carbon atom. The resultant model was then refined to convergence using the least-squares technique in an identical sequence to that used before. This produced R 0·158 and a high temperature factor, B = 9·6 Å^2, for atom C(1′), assigned as an oxygen atom, and a negative temperature factor, B = −2·6 Å^2, for atom O(1,1′) assigned as a carbon atom. The *trans*-configuration was thus confirmed.

The hydrogen atom positions were sought using Fourier and difference Fourier techniques. However, features which in some cases could be ascribed to hydrogen atoms were not significantly larger than miscellaneous features. The hydrogen atoms were not, therefore, included in the calculations.

DISCUSSION

trans-1a,7a-Dihydro-oxireno[*a*]naphthalene-3-spiro-2′-oxiran-2(3*H*)-one has the molecular structure illustrated in the stereoscopic diagram in Figure 1. In view of the low data-to-parameters ratio together with the high estimated standard deviations of the parameters, it is felt that no detailed discussion of planarity and torsion angles is warranted. Atoms C(4A), C(5)—(8), and C(8A) form a planar hexagon within the accuracy of the determination and atoms C(1) and C(4) are coplanar with this hexagon. Bond lengths and angles are listed in Table 2. The thermal parameters of atoms C(1), C(4A), and C(8A) are relatively low (B 0·7, 1·0, and 0·6 Å^2); these are consistent with the idea that these atoms should be more restrained individually compared with other atoms in the molecule. Slightly different positional or thermal parameters would be expected for

TABLE 2

Bond lengths (Å) and angles (deg.) with estimated standard deviations in parentheses

(*a*) Distances

C(1)–O(1,1′)	1·46(6)	O(3,4)–C(4)	1·49(5)
C(1)–C(1′)	1·56(6)	C(4)–C(4A)	1·53(6)
C(1)–C(2)	1·57(6)	C(4A)–C(5)	1·38(6)
C(1)–C(8A)	1·39(6)	C(4A)–C(8A)	1·39(6)
O(1,1′)–C(1′)	1·52(5)	C(5)–C(6)	1·44(6)
C(2)–O(2)	1·23(5)	C(6)–C(7)	1·44(6)
C(2)–C(3)	1·48(6)	C(7)–C(8)	1·52(6)
C(3)–C(4)	1·60(6)	C(8)–C(8A)	1·41(6)
C(3)–O(3,4)	1·55(5)		

(*b*) Angles

O(1,1′)–C(1)–C(1′)	60·1(65)	C(3)–O(3,4)–C(4)	63·6(65)
O(1,1′)–C(1)–C(2)	108·4(65)	C(3)–C(4)–O(3,4)	60·0(65)
O(1,1′)–C(1)–C(8A)	121·8(65)	C(3)–C(4)–C(4A)	121·1(80)
C(1′)–C(1)–C(2)	114·6(80)	O(3,4)–C(4)–C(4A)	113·3(65)
C(1′)–C(1)–C(8A)	124·3(80)	C(4)–C(4A)–C(5)	116·0(80)
C(2)–C(1)–C(8A)	115·7(80)	C(4)–C(4A)–C(8A)	117·8(80)
C(1)–O(1,1′)–C(1′)	63·4(65)	C(5)–C(4A)–C(8A)	126·1(80)
O(1,1′)–C(1′)–C(1)	56·5(65)	C(4A)–C(5)–C(6)	120·7(80)
C(1)–C(2)–O(2)	122·0(65)	C(5)–C(6)–C(7)	116·0(80)
C(1)–C(2)–C(3)	120·4(80)	C(6)–C(7)–C(8)	121·9(80)
O(2)–C(2)–C(3)	116·9(65)	C(7)–C(8)–C(8A)	117·1(80)
C(2)–C(3)–C(4)	110·0(80)	C(1)–C(8A)–C(4)	125·0(80)
C(2)–C(3)–O(3,4)	109·9(65)	C(1)–C(8A)–C(8)	116·8(80)
O(3,4)–C(3)–C(4)	56·5(65)	C(4A)–C(8A)–C(8)	118·1(80)

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* For details of Supplementary Publications see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. supplied as full size copies).

[5] ‘International Tables for *X*-Ray Crystallography,’ Vol. III, Kynoch Press, Birmingham, 1962.

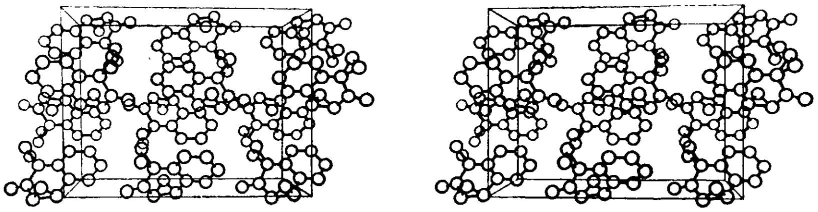

FIGURE 2 A stereoscopic view down the crystallographic *c* axis of the title compound showing the molecular packing arrangement adopted

carbon atoms refined in the absence of associated hydrogen atoms.

The conformation which the molecule adopts in the crystal is the one in which both oxygen atoms O(1,1′) and O(2) are on the same side of the ' plane ' of the molecule. As required by space-group conditions the compound forms a crystal racemate indicating the presence of both enantiomers in equal proportions. Figure 2 is a stereoscopic view down the crystallographic *c* axis and shows the relative disposition of each enantiomer and the manner in which the molecules pack in three dimensions to form the crystal.

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