

syn-2,3;4,5-Diepoxy-12-methyl-12-aza[4.4.3]propellane-11,13-dione

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(Received 26 September 1977; accepted 14 October 1977)

Abstract. C₁₃H₁₅NO₄, $M_r = 249.27$, monoclinic, $P2_1/c$, $a = 12.081$ (8), $b = 11.370$ (7), $c = 8.526$ (7) Å, $\beta = 98.02$ (7)°, $Z = 4$, $D_x = 1.428$ g cm⁻³. The unsubstituted six-membered ring is in a chair conformation. The epoxy rings are *syn* with respect to the five-membered ring. The molecule deviates significantly from the mirror-plane symmetry expected from its formula.

Introduction. The configuration of the title compound is of interest in connexion with stereochemical studies of [4.4.*X*]propella-2,4-dienes (*X* ≡ substituted rings) (Landheer & Ginsburg, 1977).

Intensities from a colourless crystal, 0.30 × 0.20 × 0.20 mm, were collected on a semi-automatic Stoe-Weissenberg diffractometer with graphite-monochromatized Mo *K* α radiation ($\lambda = 0.71069$ Å) and the $\omega/2\theta$ scanning technique; 1572 symmetry-independent reflexions were measured, including 188 with zero net intensity. The structure was solved with *MULTAN 74* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and refined by least-squares analysis in two separate blocks with unit weights for all reflexions. H atoms were located on a difference map, but included in

the refinement at positions calculated from the heavy-atom positions. Scattering factors for C, N, and O were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965). The final *R* for 1384 non-zero reflexions was 0.079. Final positional parameters are listed in Table 1.* Interatomic distances, angles and torsion angles involving C, N and O atoms are shown in Fig. 1; the C–H distances (not shown) lie in the range 0.81–1.12 Å.

Discussion. The two epoxy rings are *syn* with respect to the central five-membered ring (with two carbonyl groups attached to it). The same configuration was found in *syn*-8,9-epoxy-12-methyl-12-aza[4.4.3]-propell-3-ene-11,13-dione (Kaftory & Dunitz, 1976). The *anti* configuration was found in *syn,syn,syn,-syn*-1,2;3,4;5,6;7,8-*anti*-9,10-naphthalene pentoxide

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33090 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Atomic coordinates for non-hydrogen atoms ($\times 10^4$) and for hydrogen atoms ($\times 10^3$) (e.s.d.'s in parentheses)

	x	y	z		x	y	z
O(1)	3726 (3)	2912 (3)	4585 (4)	H(2)	459 (4)	390 (4)	300 (6)
O(2)	1452 (3)	3093 (3)	3667 (4)	H(3)	377 (4)	200 (5)	234 (6)
O(3)	3228 (3)	5153 (3)	6133 (4)	H(4)	175 (4)	173 (5)	205 (6)
O(4)	399 (2)	6045 (3)	2208 (5)	H(5)	72 (4)	354 (4)	120 (5)
N	1666 (3)	5687 (3)	4418 (5)	H(71)	134 (5)	514 (5)	-13 (6)
C(1)	3112 (3)	4852 (4)	3251 (5)	H(72)	250 (6)	437 (7)	-25 (9)
C(2)	3830 (3)	3744 (4)	3353 (6)	H(81)	255 (5)	670 (5)	46 (7)
C(3)	3309 (4)	2592 (4)	2969 (6)	H(82)	298 (5)	607 (5)	-126 (7)
C(4)	2080 (4)	2524 (4)	2580 (6)	H(91)	438 (5)	518 (6)	97 (7)
C(5)	1437 (4)	3587 (4)	2119 (6)	H(92)	443 (4)	654 (4)	94 (6)
C(6)	2011 (3)	4772 (4)	2064 (5)	H(101)	344 (4)	674 (4)	315 (6)
C(7)	2116 (4)	5016 (5)	336 (6)	H(102)	455 (4)	591 (4)	368 (5)
C(8)	2864 (4)	6048 (5)	22 (6)	H(131)	40 (5)	645 (6)	516 (8)
C(9)	3974 (4)	5928 (5)	1098 (7)	H(132)	149 (6)	667 (6)	638 (8)
C(10)	3822 (4)	5923 (4)	2823 (6)	H(133)	84 (6)	563 (7)	649 (9)
C(11)	2716 (3)	5195 (4)	4796 (6)				
C(12)	1263 (3)	5605 (4)	2839 (6)				
C(13)	1046 (4)	6206 (5)	5604 (7)				

(Vogel, Breuer, Sommerfeld, Davis & Liu, 1977) and in the *endo*-peroxidation product of a norcardiene (Paquette, Liotta, Liao, Wallis, Eickman, Clardy & Gleiter, 1976). In the latter, however, the two epoxy rings are *syn* with respect to the urazole ring (with two carbonyl groups attached to it).

The dimensions of the epoxy rings agree well with those found in other epoxides (Sands, 1972; Birnbaum, 1973; Kaftory & Dunitz, 1976; Vogel, Breuer, Sommerfeld, Davis & Liu, 1977).

The molecule is appreciably distorted from mirror symmetry (Fig. 2). The distortion can be described as a twist of the molecule around C(1)–C(6) (the torsion angle at this bond is 41° , Fig. 1). As a result of the distortion, the non-bonded distances O(1)···C(11) and O(2)···C(12) are not equal; O(2) is equidistant from C(11) and C(12) (2.929 and 2.943 Å respectively) while O(1)···C(11) and O(1)···C(12) are 2.884 and 4.383 Å respectively. A similar distortion was found by Kaftory & Dunitz (1976). The same explanation given

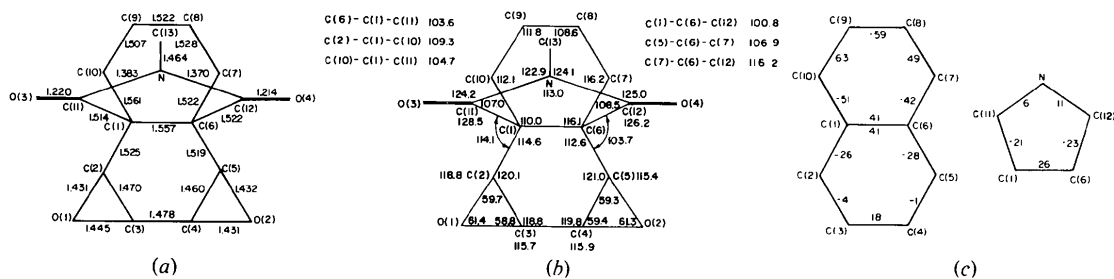


Fig. 1. (a) Bond distances (Å), (b) bond angles ($^\circ$) and (c) torsion angles ($^\circ$). The e.s.d.'s of bond distances are typically 0.005–0.008 Å, of bond angles 0.3–0.4 $^\circ$.

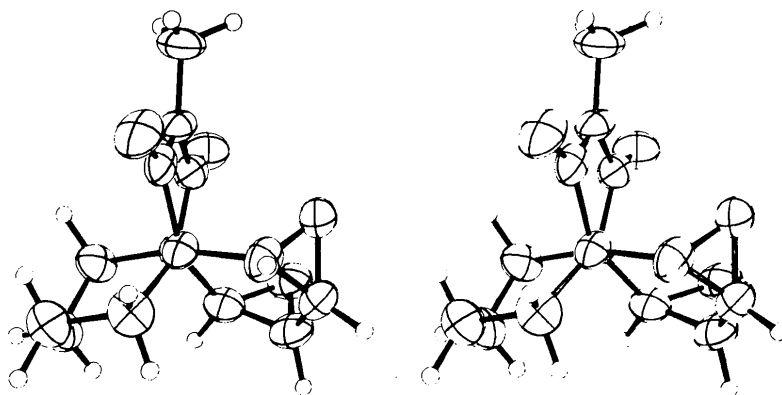


Fig. 2. Stereoscopic view of the molecule down C(6)–C(1), showing vibration ellipsoids at the 50% probability level (Johnson, 1965).

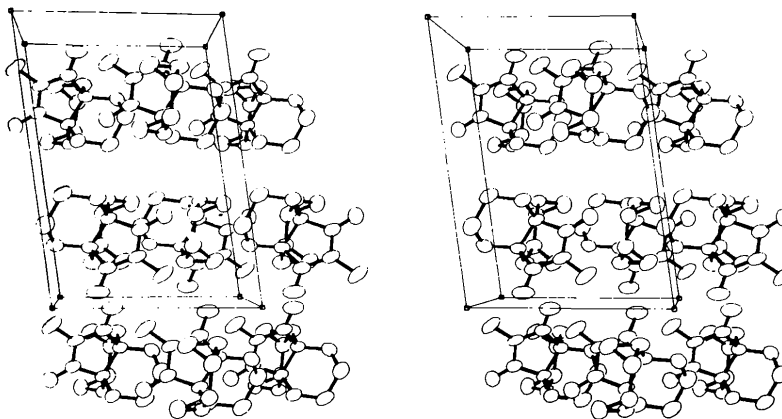


Fig. 3. Packing of molecules in the unit cell. The origin is at the lower left-hand corner of the unit cell. The *a* axis is up the page, *b* is into the page, and *c* is horizontal.

there might also hold here. An attractive intramolecular O(2)···C(11)=O(3) interaction (Bürgi, Dunitz & Shefter, 1974) might be the driving force towards such a distortion. Although the lone-pair orbital of O(2) cannot point exactly towards C(11), the tendency is to reduce the O(2)···C(11)=O(3) approach angle (116.5°) to the optimal value (100–110°) (Bürgi, Dunitz & Shefter, 1974).

The unsubstituted six-membered ring adopts the chair conformation. Torsion angles (Fig. 1) are related by 'mirror symmetry' passing through C(6) and C(9). The six-membered rings in [4.4.4]propellane (Ermer, Gerdil & Dunitz, 1971) also adopt the chair conformation but the 'mirror symmetry' of the torsion angles passes through C(7) and C(10) (the notation is according to the present work), parallel to the C(1) and C(6) bridging atoms. The difference is attributed to the distortion described above.

The five-membered ring has an envelope shape with C(6) as the flap. Mean-plane calculations show that C(1), C(11), C(12), and N are planar (out of plane displacement range: –0.03 to 0.03 Å) while C(6) is found to lie –0.42 Å from that plane; O(4) and C(13) are displaced by 0.11 Å towards the epoxides.

The packing of the molecules is shown in Fig. 3. Intermolecular distances shorter than the sum of the van der Waals radii were not observed.

The author thanks Professor D. Ginsburg and Dr I. J. Landheer for providing the sample.

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Acta Cryst. (1978). **B34**, 954–956

N,N'-Dibenzoyl-*p*-phenylenediamine

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(Received 12 August 1977; accepted 20 October 1977)

Abstract. C₂₀H₁₆N₂O₂, orthorhombic, *Pbca*, *Z* = 4, *a* = 9.540 (7), *b* = 18.273 (9), *c* = 9.387 (3) Å, *V* = 1636 Å³, *D_x* = 1.285, *D_m* = 1.29 g cm⁻³ (by flotation in aqueous CsCl), λ(Mo Kα) = 0.7107 Å, *R* = 0.089 for 1876 reflections. Centrosymmetric molecules are linked together by N–H···O hydrogen bonds. The angle between the phenylenediamine segment and the amide

plane, expressed as the torsion angle C(7)–N–C(8)–C(10), is –56.3°. The amide plane is nearly flat with a torsion angle about the C(7)–N bond [O–C(7)–N–C(8)] of +1.4°.

Introduction. The compound was prepared by Dr Fred Arnold of the Air Force Materials Laboratory and recrystallized by vacuum sublimation at 330°C to yield single crystals in the form of clear plates. Weissenberg and precession photographs were used to identify the crystal system and space group. A platelet with a

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