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2,3-Epoxy-*trans*-1,3-dimethyl-4a,9a-diaza-1,2,3,4,4a,9,9a,10-octahydroanthracene-9,10-dione*

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Abstract. $C_{14}H_{14}N_2O_3$. Monoclinic, $P2_1/n$, a = 12·5288 (11), b = 8·3247 (5), c = 12·0593 (9) Å, β = 97·46 (1)°, Z = 4, D_x = 1·37 g cm^{-3}. The structure has been solved by direct methods and refined to an R value of 0·047 for 1939 observed reflexions. The intramolecular distances are compared, by means of statistical tests, with those of the *cis* compound.

Introduction. Continuing the project on the tetracycline analogues with potential pharmacological activity mentioned in a previous work (Foces-Foces, Cano & García-Blanco, 1977) we have determined the crystal structure of the title compound. This kind of compound seems to offer a feasible method of obtaining different substituted tetracycline analogues, because of the reactivity of the epoxide group.

A crystal, 0·32 × 0·36 × 0·50 mm, was used in the measurement of the cell dimensions and for the collection of intensities, on a Philips PW 1100 four-circle diffractometer, with graphite-monochromated Cu $K\alpha$ radiation. Cell constants were determined by a least-squares fit to the settings for 85 reflexions. 2121 independent reflexions up to θ = 65° were recorded in

Table 1. *Final positional parameters* (×10^4; *for* H ×10^3) *and their standard deviations, with bond distances* (Å) *for the hydrogen atoms*

	x	y	z		x	y	z	C–H bond
C(1)	1936 (1)	2987 (2)	596 (1)	H(1)	182 (2)	199 (2)	20 (2)	0·96 (2)
C(2)	945 (2)	3327 (2)	1123 (2)	H(2)	28 (2)	304 (3)	62 (2)	0·99 (2)
C(3)	941 (2)	3411 (2)	2329 (2)	H(4*a*)	202 (2)	199 (3)	332 (2)	1·01 (2)
C(4)	1992 (2)	3141 (2)	3054 (1)	H(4*b*)	205 (2)	382 (3)	371 (2)	0·96 (2)
C(5)	5851 (2)	4076 (2)	3187 (2)	H(5)	585 (2)	467 (3)	392 (2)	1·01 (3)
C(6)	6789 (2)	3657 (3)	2775 (2)	H(6)	745 (3)	394 (3)	320 (2)	0·95 (2)
C(7)	6762 (2)	2684 (3)	1835 (2)	H(7)	742 (2)	241 (3)	156 (2)	0·96 (3)
C(8)	5790 (2)	2131 (3)	1286 (2)	H(8)	577 (2)	146 (3)	61 (2)	0·99 (2)
C(9)	3796 (1)	2109 (2)	1057 (1)	H(15*a*)	222 (2)	535 (3)	18 (2)	0·98 (3)
C(10)	3872 (2)	3844 (2)	3125 (1)	H(15*b*)	152 (2)	446 (4)	−79 (2)	1·00 (3)
C(11)	4866 (1)	3531 (2)	2637 (1)	H(15*c*)	280 (2)	414 (2)	−59 (2)	1·00 (3)
C(12)	4837 (1)	2572 (2)	1689 (1)	H(17*a*)	−6 (2)	368 (4)	355 (2)	0·98 (3)
N(13)	2919 (1)	3499 (2)	2474 (1)	H(17*b*)	−3 (2)	190 (4)	317 (2)	1·01 (3)
N(14)	2894 (1)	2694 (2)	1438 (1)	H(17*c*)	−67 (3)	317 (4)	230 (3)	0·99 (3)
C(15)	2150 (2)	4321 (3)	−208 (2)					
O(16)	905 (1)	4878 (2)	1678 (1)					
C(17)	−40 (2)	3040 (3)	2870 (2)					
O(18)	3873 (1)	4346 (2)	4079 (1)					
O(19)	3724 (1)	1350 (2)	178 (1)					

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* Crystal and Molecular Structure of Diazapolycyclic Compounds. II.

Table 2. *Bond distances* (Å), *bond angles and torsion angles* (°) *describing the geometry of the molecule*

C(1)–C(2)	1·494 (3)	C(6)–C(7)	1·390 (3)
C(1)–C(15)	1·521 (3)	C(7)–C(8)	1·388 (3)
C(1)–N(14)	1·488 (2)	C(8)–C(12)	1·395 (3)
C(2)–C(3)	1·456 (3)	C(9)–C(12)	1·475 (2)
C(2)–O(16)	1·458 (2)	C(9)–O(19)	1·227 (2)
C(3)–C(4)	1·499 (2)	C(9)–N(14)	1·364 (2)
C(3)–O(16)	1·449 (2)	C(10)–C(11)	1·468 (3)
C(3)–C(17)	1·497 (3)	C(10)–O(18)	1·224 (2)
C(4)–N(13)	1·462 (2)	C(10)–N(13)	1·371 (2)
C(5)–C(6)	1·378 (3)	C(11)–C(12)	1·391 (2)
C(5)–C(11)	1·399 (3)	N(13)–N(14)	1·415 (2)

C(15)–C(1)–C(2)	111·0 (2)	C(12)–C(9)–O(19)	122·8 (2)
C(15)–C(1)–N(14)	111·2 (2)	N(14)–C(9)–O(19)	120·3 (2)
C(2)–C(1)–N(14)	112·4 (1)	C(11)–C(10)–N(13)	117·0 (2)
C(1)–C(2)–C(3)	123·0 (2)	C(11)–C(10)–O(18)	122·7 (2)
C(1)–C(2)–O(16)	116·3 (2)	N(13)–C(10)–O(18)	120·3 (2)
C(3)–C(2)–O(16)	59·6 (1)	C(10)–C(11)–C(12)	119·9 (2)
C(2)–C(3)–C(4)	117·3 (2)	C(5)–C(11)–C(12)	120·2 (2)
C(2)–C(3)–O(16)	60·2 (1)	C(5)–C(11)–C(10)	119·6 (2)
C(2)–C(3)–C(17)	122·3 (2)	C(9)–C(12)–C(11)	120·1 (2)
O(16)–C(3)–C(17)	116·1 (2)	C(8)–C(12)–C(11)	120·4 (2)
C(4)–C(3)–O(16)	114·0 (2)	C(8)–C(12)–C(9)	119·4 (2)
C(4)–C(3)–C(17)	115·0 (2)	C(4)–N(13)–C(10)	117·1 (1)
C(3)–C(4)–N(13)	112·5 (1)	C(4)–N(13)–N(14)	113·4 (1)
C(6)–C(5)–C(11)	119·3 (2)	C(10)–N(13)–N(14)	121·5 (1)
C(5)–C(6)–C(7)	120·6 (2)	C(1)–N(14)–N(13)	116·7 (1)
C(6)–C(7)–C(8)	120·6 (2)	C(1)–N(14)–C(9)	117·3 (1)
C(7)–C(8)–C(12)	119·0 (2)	C(9)–N(14)–N(13)	122·9 (1)
C(12)–C(9)–N(14)	116·6 (1)	C(2)–O(16)–C(3)	60·1 (1)

C(1)–C(2)–C(3)–C(4)	0·1 (2)
C(2)–C(3)–C(4)–N(13)	24·2 (2)
C(3)–C(4)–N(13)–N(14)	−52·7 (2)
C(4)–N(13)–N(14)–C(1)	57·6 (2)
N(13)–N(14)–C(1)–C(2)	−30·3 (2)
N(14)–C(1)–C(2)–C(3)	1·9 (2)
C(1)–N(14)–C(9)–C(12)	149·7 (1)
C(1)–N(14)–C(9)–O(19)	−24·5 (2)
N(13)–N(14)–C(9)–C(12)	−9·5 (2)
N(13)–N(14)–C(9)–O(19)	176·4 (1)
C(4)–N(13)–C(10) C(11)	154·6 (1)
C(4)–N(13)–C(10)–O(18)	−23·4 (2)
N(14)–N(13)–C(10)–C(11)	7·7 (2)
N(14)–N(13)–C(10)–O(18)	−170·3 (2)
C(15)–C(1)–C(2)–O(16)	−53·9 (2)
H(1)–C(1)–C(2)–H(2)	−39·6 (20)

the $\omega/2\theta$ scan mode. Based on the criterion that a reflexion is observed if $I > 2\sigma(I)$, 182 reflexions were taken as unobserved. The data were corrected for Lorentz and polarization factors, but not for absorption ($\mu = 8·2$ cm^{-1}).

The structure was solved with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) from the 100 highest E values. The electron-density map corresponding to the solution with the highest combined figure of merit revealed the positions of all the non-hydrogen atoms in the molecule, which were then refined by full-matrix least-squares methods. The 14 H atoms were located in a difference map and included in the refinement. At this stage the weighting scheme used was $w = K/[f(F_o)]^2 f(s)$ where $K = 0·60$ and $f(F_o) = 0·34 + 0·03\,F_o$, $f(s) = 2·04 - 2·33(\sin\theta/\lambda)$. The final R and R_w values for the observed reflexions were 0·047 and 0·062 respectively. The scattering factors were taken from *International Tables for X-ray Crystallography* (1974).* A final difference synthesis revealed no features higher than 0·20 e Å^{-3}. Positional parameters, bond distances and angles and torsion angles are given in Tables 1 and 2.

Discussion. A perspective view of the molecule with the numbering of the atoms is shown in Fig. 1. The two N atoms (14 and 13) show mean bond angles of 118·9 and 117·4° respectively. The conformational parameters given by Winkler & Dunitz (1971) for amide groups are (in degrees) χ_N (−20·9; −33·1), χ_C (−5·9; −2·0) and τ (−17·0; −7·9).

The comparison with the *cis* isomer (Foces-Foces *et al.*, 1977) has been carried out by half-normal probability plot methods (Abrahams & Keve, 1971) for the intramolecular distances (slope = 1·51, intercept = 0, correlation = 0·984 and $\chi^2 = 0·026$). The significant differences can be assigned to the different position of the epoxide ring with respect to the C(15) methyl group and the conformation of the piperidazine ring (Table 2). But there are other differences, mainly in the bond distances N(14)–C(1), C(10)–N(13), C(7)–C(8) and C(2)–O(16). In spite of the significant difference in the last bond, the epoxide area remains constant, 0·915 Å^2 (*versus* 0·914 Å^2, Foces-Foces *et al.*, 1977).

With the N(14)–N(13) bond taken as the origin of measurement and with the quantities defined by Cano, Foces-Foces & García-Blanco (1977) for six-membered rings, the actual conformational parameters for the piperidazine ring in both compounds (*cis* and *trans*, respectively) are, in degrees, τ_m (−15·8; 27·8), q

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33165 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

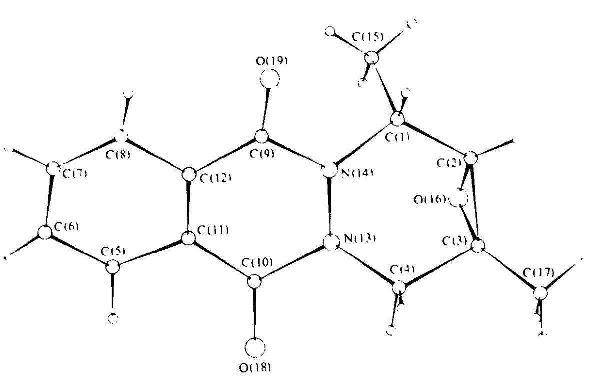

Fig. 1. A perspective view of the molecule showing the numbering system.

(35·2; 31·6), Σ (233·2; 48·2), δ (233·3; 57·0), α_1 (233·3; 52·6) and α_2 (−0·1; −4·4). These parameters show that the conformation for the *cis* compound is diplanar at the C(2)—C(3) and C(4)—N(13) bonds, distorted towards a half chair at the C(2)—C(3) bond. For the *trans* compound the conformation is envelope at N(13) slightly distorted towards a half-chair at the C(2)—C(3) bond.

The packing in the crystal is entirely due to van der Waals forces.

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1,2,4,5-Tetrabenzylhexahydro-*s*-tetrazine

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Abstract. $C_{30}H_{32}N_4$, triclinic, $P\bar{1}$, a = 7·016 (2), b = 9·796 (2), c = 10·444 (2) Å, α = 92·94 (2), β = 107·37 (2), γ = 109·18 (2)°, D_m = 1·17 (1), Z = 1, D_c = 1·167 g cm^{-3}. The structure was solved by direct methods and refined by block-diagonal least squares to R = 0·063. The centrosymmetric molecule has a chair conformation, with one of the two independent benzyl groups axial and the other equatorial.

Introduction. The preparation of the title compound has recently been described, together with PMR and CMR spectra and dipole-moment measurements (Baker, Katritzky, Majoral, Martin & Sullivan, 1976).

Crystals suitable for X-ray analysis were grown from petroleum ether (40–60 °C) solutions. Cell parameters were determined by a least-squares refinement of the setting angles of 15 reflections centred on a Syntex $P2_1$ automatic diffractometer with graphite-monochromatized Mo $K\alpha$ radiation.

Preliminary Weissenberg photographs revealed a triclinic lattice. Intensity data were obtained at room temperature from a crystal (0·30 × 0·55 × 0·15 mm) mounted on the diffractometer (Mo $K\alpha$, monochromatized, θ–2θ scan, 3° < 2θ < 52°). Of the 2487 unique reflections recorded, 1632 with $F_o > 3\sigma(F_o)$ were considered observed and used in the analysis. No absorption correction was applied [μ(Mo $K\alpha$) = 0·65 cm^{-1}].

A statistical analysis of the intensities indicated that the space group was centrosymmetric ($\langle|E|\rangle$ = 0·761, $\langle|E^2-1|\rangle$ = 1·021, $\langle|E^2|\rangle$ = 1·000, $\langle|E|\rangle^2/\langle|E^2|\rangle$ = 0·580, $|E| > 1$ = 30·20%, $|E| > 2·0$ = 4·75%, $|E| >$ 3·0 = 0·68%) and the space group $P\bar{1}$ with half a molecule in the asymmetric unit was assumed. The structure was determined by the application of direct methods with the weighted multisolution tangent refinement technique (Germain, Main & Woolfson, 1971).

The E map computed with the phases from the set with the highest figure of merit revealed the positions of all but one of the non-hydrogen atoms. This remaining atom was located by successive Fourier syntheses. The