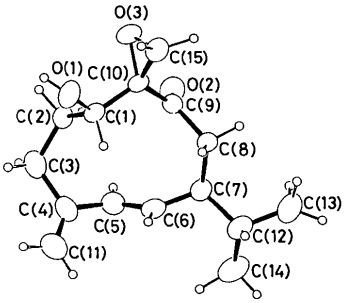

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the molecule with 50% probability ellipsoids. H atoms are represented by circles of radius 0·08 Å.

C(1)–C(2) group in the present compound, the torsion angle C(10)–C(1)–C(2)–C(3) being 148·1 (4)°. The corresponding values in periplanone B and periplanol B, which involve an epoxide moiety at the C(1)–C(2) bond, are −1·7 (5) and 0·7 (6)°, respectively.

References

ADAMS, M. A., NAKANISHI, K., STILL, W. C., ARNOLD, E. V. & PERSOONS, C. J. (1979). *J. Am. Chem. Soc.* **101**, 2493–2498.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HAUPTMANN, H., MÜHLBAUER, G. & WALKER, N. P. C. (1986). *Tetrahedron Lett.* **27**, 1315–1318.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

PERSOONS, C. J., VERWIEL, P. E. J., RITTER, F. J. & NOOYEN, W. J. (1982). *J. Chem. Ecol.* **8**, 439–451.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

SHIZURI, Y., YAMAGUCHI, S., YAMAMURA, S., ISHIHARA, M., OHBA, S., SAITO, Y., NIWA, M., TERADA, Y. & MIYAZAKI, M. (1987). *Tetrahedron Lett.* **28**, 3831–3834.

Acta Cryst. (1987). C**43**, 2447–2448

Structure of 6-Ethyl-3,3-dimethyl-1,2,3,4-tetrahydro-1,5-benzodiazocin-4-one

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Abstract. $C_{14}H_{18}N_2O$, $M_r = 230{\cdot}31$, orthorhombic, $Pca2_1$, $a = 8{\cdot}405$ (1), $b = 8{\cdot}960$ (1), $c = 17{\cdot}020$ (1) Å, $V = 1281{\cdot}8$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}194$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5{\cdot}66$ cm^{-1}, $F(000) = 496$, $T = 291$ K, $R = 0{\cdot}0372$, $wR = 0{\cdot}0358$ for 1049 unique observed reflections. The diazocine ring is boat-shaped. The dihedral angles between the least-squares planes defining the boat conformation are: plane 1 (N1,-C2,C6,C6a,C10a) and plane 2 (C2,C3,N5,C6) 54·9°, plane 2 and plane 3 (C3,C4,N5) 123·7 (2)°. The benzene ring is essentially planar with a maximum deviation from the least-squares plane of 0·008 (4) Å (C8,C10a). Bond lengths and angular properties of the diazocine ring are in fair agreement with previously studied benzodiazocines. Subsequent molecules are linked together into a chain by hydrogen bonds running in the direction of the *a* axis.

Experimental. Crystals from CH_3CN, thin yellow transparent plates, size of specimen 0·50 × 0·40 × 0·05 mm. Stoe four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan, scan width 1·05–1·35°, scan speed 1·21–3·64° min^{-1}. 1161 reflections measured, $2{\cdot}60 \le \theta \le 63{\cdot}94°$, 1073 unique and 1049 with $I \ge 2\sigma(I)$ considered observed, $0 \le h \le 9$, $0 \le k \le 10$, $0 \le l \le 19$, three reflections checked every 60 min, variation in intensity below ±1%. No absorption correction was applied. Lattice parameters from least-squares refinement of 17 reflections, $5{\cdot}58 \le \theta \le 20{\cdot}13°$. Structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement with *SHELX*76 (Sheldrick, 1976) minimizing $\sum w(\Delta F)^2$. Non-H atoms anisotropic, H1 isotropically and individually refined, remaining H atoms in calculated positions with isotropic group temperature factors. $R = 0{\cdot}0372$, $wR = 0{\cdot}0358$, $w = 1/[\sigma^2(F) + 0{\cdot}0001F^2]$, for 1049 reflections with $F \ge 4\sigma(F)$ and 186 parameters. Max. $\Delta/\sigma = 0{\cdot}002$ for any parameter, max. and min. peaks in final $\Delta\rho$ map 0·12 and −0·19 e Å^{-3}. The inverse structure slightly worsened the *R* factors, although this difference was not statistically significant enough to identify the correct enantiomer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
N1	0·2084 (6)	0·0112 (2)	0·4387 (2)	6·1 (1)
C2	0·1533 (5)	−0·1391 (3)	0·4475 (2)	4·5 (1)
C3	0·2790 (5)	−0·2447 (3)	0·4818 (1)	3·7 (1)
C3a	0·3245 (6)	−0·1990 (5)	0·5649 (2)	6·3 (1)
C3b	0·2108 (6)	−0·4044 (3)	0·4819 (2)	5·5 (1)
C4	0·4230 (5)	−0·2397 (2)	0·4275 (1)	3·3 (1)
N5	0·3866 (4)	−0·2628 (2)	0·3491 (1)	3·3 (1)
C6	0·3584 (5)	−0·1608 (2)	0·2991 (1)	3·4 (1)
C6a	0·3683 (5)	0·0022 (2)	0·3158 (1)	3·4 (1)
C7	0·4572 (5)	0·0887 (3)	0·2627 (1)	4·3 (1)
C8	0·4818 (6)	0·2396 (3)	0·2726 (2)	5·0 (1)
C9	0·4139 (6)	0·3101 (3)	0·3367 (2)	4·7 (1)
C10	0·3262 (5)	0·2296 (2)	0·3893 (2)	4·2 (1)
C10a	0·3015 (5)	0·0751 (2)	0·3815 (1)	3·7 (1)
O11	0·5603 (4)	−0·2279 (2)	0·4499 (1)	4·6 (1)
C12	0·3130 (5)	−0·2057 (3)	0·2170 (1)	4·5 (1)
C13	0·3158 (7)	−0·3691 (3)	0·1998 (2)	6·6 (1)

Table 2. *Bond lengths* (Å), *angles* (°) *and hydrogen-bond distances* (Å) *and angle* (°) *with e.s.d.'s in parentheses*

N1–C2	1·432 (4)	C6–C12	1·504 (3)
N1–C10a	1·375 (4)	C6–C6a	1·490 (3)
C2–C3	1·533 (5)	C6a–C7	1·406 (4)
C3–C3a	1·521 (4)	C6a–C10a	1·411 (4)
C3–C3b	1·541 (4)	C7–C8	1·378 (4)
C3–C4	1·524 (5)	C8–C9	1·383 (4)
C4–O11	1·220 (5)	C9–C10	1·366 (4)
C4–N5	1·385 (3)	C10–C10a	1·406 (3)
N5–C6	1·271 (3)	C12–C13	1·493 (4)

C2–N1–C10a	130·5 (2)	N5–C6–C12	118·5 (2)
N1–C2–C3	113·4 (3)	C6a–C6–C12	117·0 (2)
C2–C3–C4	107·3 (2)	C6–C6a–C10a	125·6 (2)
C2–C3–C3b	108·5 (3)	C6–C6a–C7	116·6 (2)
C2–C3–C3a	111·2 (2)	C7–C6a–C10a	117·7 (2)
C3b–C3–C4	108·8 (2)	C6a–C7–C8	122·8 (2)
C3a–C3–C4	110·8 (3)	C7–C8–C9	118·8 (2)
C3a–C3–C3b	110·0 (2)	C8–C9–C10	119·9 (2)
C3–C4–N5	113·9 (3)	C9–C10–C10a	122·5 (2)
C3–C4–O11	124·4 (2)	N1–C10a–C10	115·2 (2)
N5–C4–O11	121·5 (3)	C6a–C10a–C10	118·2 (2)
C4–N5–C6	125·3 (2)	N1–C10a–C6a	126·5 (2)
N5–C6–C6a	124·6 (2)	C6–C12–C13	116·2 (2)

N1···O11($x-\frac{1}{2},-y,-z+1$)	2·985 (4)	N1–H1···O11	165 (3)
H1···O11	2·18 (4)		

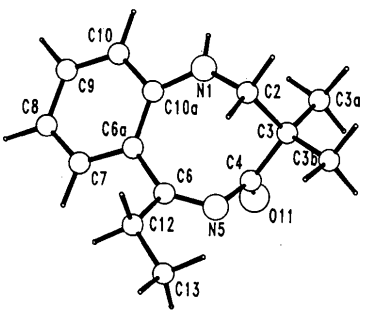

Fig. 1. Perspective view of the molecule with numbering scheme.

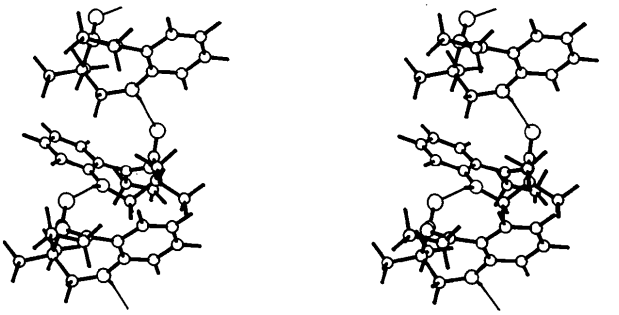

Fig. 2. Fragment of the hydrogen-bonding pattern, involving three molecules, shown in stereo (hydrogen bonds indicated by thin lines).

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1;* Table 2 lists bond lengths, angles and hydrogen-bond data. Fig. 1 shows the molecule with numbering scheme; Fig. 2 illustrates the hydrogen bonding. All drawings by *PLUTO* (Motherwell & Clegg, 1978), geometrical calculations by *PARST* (Nardelli, 1983).

Related literature. This paper reports the first benzodiazocin-4-one; structures reported earlier are 2-ones (Andronati, Dvorkin, Simonov, Danilin, Malinowsky & Bogatsky, 1979, 1982; Dvorkin, Simonov, Andronati & Danilin, 1985).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44271 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ANDRONATI, S. A., DVORKIN, A. A., SIMONOV, YU. A., DANILIN, V. V., MALINOWSKY, T. I. & BOGATSKY, A. V. (1979). *Dokl. Akad. Nauk SSSR*, **248**(5), 1140–1144 (in Russian).

ANDRONATI, S. A., DVORKIN, A. A., SIMONOV, YU. A., DANILIN, V. V., MALINOWSKY, T. I. & BOGATSKY, A. V. (1982). *Acta Cryst.* B**38**, 638–640.

DVORKIN, A. A., SIMONOV, YU. A., ANDRONATI, S. A. & DANILIN, V. V. (1985). *Kristallografiya*, **30**, 91–95.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *PARST. A System of Computer Routines for Calculating Molecular Parameters from the Results of Crystal Structure Analysis*. Univ. of Parma, Italy.

SHELDRICK, G. M. (1976). *SHELX*76. A program for crystal structure determination. Univ. of Cambridge, England.