

MARTIN, S. F. & PACOFSKY, G. J. (1989). In preparation.
 RILEY, P. E. & DAVIS, R. E. (1976). *Acta Cryst.* B32, 381–386.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1985). *SHELXS-86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRUGER & R. GODDARD. Oxford Univ. Press.

SHELDRIK, G. M. (1987). *SHELXTL-PLUS*. Nicolet XRD, Madison, Wisconsin, USA.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.
 SVIRIDOV, A. F., ERMOLENKO, M. S., YASHUNSKY, D. V., BORODKIN, V. S. & KOCHETKOV, N. K. (1987). *Tetrahedron Lett.* 28, 3835–3838, 3839–3842.

Acta Cryst. (1989). C45, 975–976

Structure of 2-Methyl-3-phenyl-5H-1,4-benzodiazepine

BY DEREK J. CHADWICK, KENNETH R. RANGLES AND RICHARD C. STORR

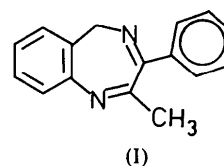
The Robert Robinson Laboratories, Department of Chemistry, Liverpool University, PO Box 147, Liverpool L69 3BX, England

(Received 27 September 1988; accepted 23 December 1988)

Abstract. C₁₆H₁₄N₂, $M_r = 234.30$, monoclinic, $P2_1/c$, $a = 10.046$ (2), $b = 15.842$ (3), $c = 8.066$ (2) Å, $\beta = 91.81$ (2)°, $V = 1283.05$ Å³, $Z = 4$, $D_m = 1.21$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 4.86$ cm⁻¹, $F(000) = 496$, $T = 293$ K, $R = 0.060$ for 2041 unique observed reflections. The heterocyclic ring adopts a bent conformation with the N=C–C=N torsion angle 50.3 (3)°. This means that one of the methylene H atoms is in a pseudo-equatorial position, almost planar to the fused benzene ring, whereas the other is pseudo-axial and almost perpendicular to the ring, thus explaining the difference of ca $\delta 0.9$ between their NMR chemical shifts. No structures have previously been reported for the 5H-benzodiazepine ring system.

Experimental. Compound (I) isolated as major isomer from reaction between *o*-aminobenzylamine and 3-phenyl-2,3-propanedione. Crystals obtained by slow evaporation of a solution in methanol. Density measured by flotation in mixture of hexane and tetrachloromethane. A ca 0.30 mm cuboidal crystal mounted on glass fibre for X-ray analysis. Intensities measured by SERC service with an Enraf–Nonius CAD-4 diffractometer and ω – 2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $35 < \theta < 42$ °. Data collected to $(\sin\theta)/\lambda$ of 0.60 Å⁻¹, $-12 < h < 12$, $0 < k < 18$, $0 < l < 9$; empirical absorption correction applied, transmission factors 0.999–0.859. Three standard reflections ($\bar{1}$, $\bar{1}$, 1, 821, 475) varied $\pm 2\%$; linear drift correction applied. 2707 unique reflections measured, 666 reflections with $F < 5\sigma(F)$ considered unobserved. Solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least squares on F values with *SHELX76* (Sheldrick, 1976). Non-H atoms refined

anisotropically, H atoms calculated geometrically and allowed to 'ride' on associated heavy atoms with four common isotropic temperature factors for methylene, phenyl, fused benzene and methyl hydrogens, respectively, for a total of 171 variables. $R = 0.060$, $wR = 0.081$ where weight = $1.000/[\sigma^2(F) + 0.0129F^2]$. Extinction correction of form $F^* = F(1 - 0.001XF^2/\sin\theta)$ where $X = 0.045$ applied. Final $(\Delta/\sigma)_{\text{max}} < 0.59$ (methyl rotor), < 0.05 elsewhere, $\Delta\rho_{\text{max}} = 0.18$ and $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³ on final difference map. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom parameters are listed in Table 1 and bond lengths and angles in Table 2. The molecule and numbering scheme are shown in Fig. 1.



Related literature. Structures of 50 compounds containing the basic ring connectivity of the benzo-1,4-diazepine system are listed in the Cambridge Crystallographic Database (version of June 1988) but none of these have the double-bond pattern of the system reported here (*i.e.* they are not 5H-benzodiazepines). The structure described here is therefore unique in that respect.

† Lists of structure factors, H-atom coordinates and isotropic temperature factors, and anisotropic temperature factors for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51697 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

KRR is indebted to the SERC for a research studentship.

Table 1. Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s

$$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$\bar{U}(\text{\AA}^2)$
N(1)	0.2367 (2)	0.0106 (1)	0.0161 (2)	0.0671 (10)
N(4)	0.1729 (1)	-0.0739 (1)	0.3457 (2)	0.0632 (9)
C(2)	0.2853 (2)	-0.0563 (1)	0.0838 (2)	0.0645 (11)
C(3)	0.2796 (2)	-0.0768 (1)	0.2659 (2)	0.0571 (10)
C(5)	0.0518 (2)	-0.0469 (1)	0.2537 (3)	0.0739 (12)
C(6)	-0.0158 (2)	0.1067 (2)	0.2828 (3)	0.0770 (13)
C(7)	-0.0001 (2)	0.1905 (2)	0.2438 (3)	0.0824 (14)
C(8)	0.0963 (2)	0.2144 (1)	0.1350 (3)	0.0795 (13)
C(9)	0.1752 (2)	0.1545 (1)	0.0632 (3)	0.0710 (12)
C(10)	0.1612 (2)	0.0694 (1)	0.1028 (2)	0.0578 (9)
C(11)	0.0639 (2)	0.0449 (1)	0.2136 (2)	0.0608 (10)
C(12)	0.3503 (3)	-0.1193 (2)	-0.0261 (3)	0.0985 (18)
C(13)	0.4061 (2)	-0.1000 (1)	0.3549 (2)	0.0560 (10)
C(14)	0.5278 (2)	-0.0698 (1)	0.3026 (3)	0.0751 (13)
C(15)	0.6439 (2)	-0.0889 (2)	0.3899 (4)	0.0885 (16)
C(16)	0.6411 (2)	-0.1387 (2)	0.5279 (3)	0.0844 (15)
C(17)	0.5210 (2)	-0.1705 (2)	0.5814 (3)	0.0799 (13)
C(18)	0.4035 (2)	-0.1508 (1)	0.4964 (2)	0.0664 (11)

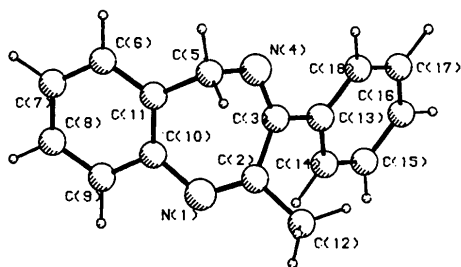


Fig. 1. A view of the molecule drawn with PLUTO (Motherwell & Clegg, 1978).

Table 2. Bond distances (\AA) and angles ($^\circ$) involving non-H atoms, with e.s.d.'s in parentheses

N(1)	C(2)	1.282 (2)	C(7)	C(8)	1.381 (3)		
N(1)	C(10)	1.402 (2)	C(8)	C(9)	1.377 (3)		
N(4)	C(3)	1.269 (2)	C(9)	C(10)	1.394 (3)		
N(4)	C(5)	1.468 (3)	C(10)	C(11)	1.400 (3)		
C(2)	C(3)	1.507 (3)	C(13)	C(14)	1.391 (3)		
C(2)	C(12)	1.498 (3)	C(13)	C(18)	1.397 (3)		
C(3)	C(13)	1.485 (2)	C(14)	C(15)	1.377 (3)		
C(5)	C(11)	1.496 (3)	C(15)	C(16)	1.365 (4)		
C(6)	C(7)	1.374 (3)	C(16)	C(17)	1.389 (3)		
C(6)	C(11)	1.393 (3)	C(17)	C(18)	1.382 (3)		
C(2)	N(1)	C(10)	122.8 (2)	N(1)	C(10)	C(11)	122.3 (2)
C(3)	N(4)	C(5)	117.0 (2)	C(9)	C(10)	C(11)	119.4 (2)
N(1)	C(2)	C(3)	124.6 (2)	C(5)	C(11)	C(6)	123.1 (2)
N(1)	C(2)	C(12)	117.8 (2)	C(5)	C(11)	C(10)	118.0 (2)
C(3)	C(2)	C(12)	117.6 (2)	C(6)	C(11)	C(10)	118.9 (2)
N(4)	C(3)	C(2)	123.0 (2)	C(3)	C(13)	C(14)	121.0 (2)
N(4)	C(3)	C(13)	119.2 (2)	C(3)	C(13)	C(18)	120.0 (2)
C(2)	C(3)	C(13)	117.8 (1)	C(14)	C(13)	C(18)	119.0 (2)
N(4)	C(5)	C(11)	108.7 (2)	C(13)	C(14)	C(15)	120.5 (2)
C(7)	C(6)	C(11)	121.1 (2)	C(14)	C(15)	C(16)	120.3 (2)
C(6)	C(7)	C(8)	119.9 (2)	C(15)	C(16)	C(17)	120.2 (2)
C(7)	C(8)	C(9)	120.2 (2)	C(16)	C(17)	C(18)	120.0 (2)
C(8)	C(9)	C(10)	120.5 (2)	C(13)	C(18)	C(17)	119.9 (2)
N(1)	C(10)	C(9)	117.9 (2)				

References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. 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*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1989). **C45**, 976–978

Structure of Ethyl 6-Methyl-6-phenyl-5,6-dihydro-4H-1,2-oxazine-3-carboxylate

BY DEREK J. CHADWICK, THOMAS L. GILCHRIST AND WAYNE STRETCH

The Robert Robinson Laboratories, Department of Chemistry, Liverpool University, PO Box 147, Liverpool L69 3BX, England

(Received 30 September 1988; accepted 23 December 1988)

Abstract. $\text{C}_{14}\text{H}_{17}\text{NO}_3$, $M_r = 247.29$, monoclinic, $P2_1$, $a = 10.59$ (2), $b = 5.84$ (1), $c = 11.17$ (2) \AA , $\beta = 109.8$ (2) $^\circ$, $V = 650.7$ \AA^3 , $Z = 2$, $D_m = 1.26$, $D_x = 1.26$ g cm^{-3} , $\lambda(\text{Cu K}\alpha) = 1.5418$ \AA , $\mu = 6.40$ cm^{-1} , $F(000) = 264$, $T = ca$ 293 K, $R = 0.051$ for 930 unique observed reflections. The phenyl group occupies an

axial position and the methyl group an equatorial position in an essentially twist-chair conformation of the 1,2-oxazine ring in which the O(1)–N(2)–C(3)–C(4) torsion angle is 2.8 (6) $^\circ$ and the deviations of C(5) and C(6) from the least-squares plane through the dihydro-oxazine ring are -0.33 (1) and 0.34 (1) \AA ,