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Structure of Ethyl 1-Cyano-1,2-dihydro-2-isoquinolinecarboxylate

By E. Tykarska, M. Jaskólski and Z. Kosturkiewicz*

Department of Crystallography, Faculty of Chemistry, A. Mickiewicz University, ul. Grunwaldzka 6, 60–780 Poznań, Poland

Abstract. $C_{13}H_{12}N_2O_2$, $M_r = 228 \cdot 26$, triclinic, $P\bar{1}$, $a = 8 \cdot 536$ (1), $b = 9 \cdot 011$ (1), $c = 8 \cdot 305$ (1) Å, $\alpha = 90 \cdot 88$ (1), $\beta = 79 \cdot 39$ (1), $\gamma = 109 \cdot 62$ (1)°, $V = 590 \cdot 7$ (2) Å^3, $Z = 2$, $D_m = 1 \cdot 29$, $D_x = 1 \cdot 28$ Mg m^{-3}, λ(Cu $K\alpha$) = $1 \cdot 54178$ Å, $\mu = 0 \cdot 732$ mm^{-1}, $F(000) = 240$, room temperature, $R = 0 \cdot 045$ for 1333 observed reflections. The heterocyclic fragment of the molecule is intermediate between 1,3-diplanar and sofa conformations. The C(3)–N(2)–C(13)–O(14) torsion angle [$-171 \cdot 3$ (3)°] characterizes the urethane bond as *anti*. The NCOO group is planar. The C(13)–O(15)–C(16)–C(17) torsion angle [$171 \cdot 4$ (3)°] reveals the twist of the ethyl fragment relative to this plane.

Introduction. The present work is the fourth of a series of investigations of Reissert compounds (Reissert, 1905). It is of interest to determine the effects of substituents on the molecular conformation in such a group of compounds. Our aim is to compare the disposition of the side fragments relative to the central amide or urethane group depending on the character and dimensions of those fragments. So far we have investigated the structure of 2-benzoyl-1,2,3,4-tetra-hydro-1-isoquinolinecarbonitrile (I) (Pływaczyk, Tykarska, Jaskólski & Kosturkiewicz, 1984) and its dihydro analog (II) (Tykarska, Jaskólski & Kosturkiewicz, 1985) as well as ethyl 1-cyano-1,2,3,-4-tetrahydro-2-isoquinolinecarboxylate (III) (Gzella,

Jaskólski, Rychlewska & Kosturkiewicz, 1984). The present compound (IV) is a dihydro analog of (III) and closes the series.

Experimental. Suitable crystals (prisms) obtained from ethanol; D_m by flotation; crystal $0 \cdot 4 \times 0 \cdot 2 \times 0 \cdot 2$ mm, Syntex $P2_1$ diffractometer; cell parameters from least-squares refinement of setting angles of 15 reflections, $20 \le 2\theta \le 30°$; profiles measured for 1552 reflections with $2\theta \le 115°$ and $h = \pm 9$, $k = \pm 9$, $l = 0$–9; profile analysis according to Lehmann & Larsen (1974); no significant intensity variation for two standard reflections; no absorption correction; 1333 observed reflections with $I \ge 1 \cdot 96\sigma(I)$; structure solved by direct methods using *MULTAN*80; full-matrix least-squares refinement on F; $w^{-1} = \sigma^2(F)$; anisotropic thermal parameters for all non-H atoms; 8 extinction-affected reflections excluded from final refinement; H atoms located in $\Delta\rho$ map and included in refinement with isotropic thermal parameters; methyl group refined as rigid body with anisotropic C(17) and rotation angle around C(16)–C(17); $R = 0 \cdot 045$, $wR = 0 \cdot 053$, max. shift/e.s.d. = $0 \cdot 005$, $S = 5 \cdot 13$; largest peak in final $\Delta\rho$ map = $0 \cdot 17$ e Å^{-3}, largest hole = $-0 \cdot 19$ e Å^{-3}; computer programs: *MULTAN*80 (Main *et al.*, 1980); *SHELX*76 (Sheldrick, 1976); and local programs (Jaskókski, 1982); molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates and bond lengths and angles are given in Tables 1 and 2, respectively.* The labelling scheme is shown below.
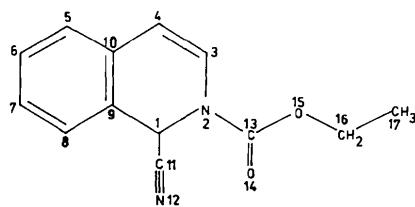
* Lists of structure factors, anisotropic thermal parameters and hydrogen atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42747 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent iso-tropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
C(1)	0·1833 (3)	0·8579 (3)	0·4177 (3)	0·050
N(2)	0·1172 (2)	0·7387 (2)	0·5532 (2)	0·048
C(3)	0·1930 (3)	0·6244 (3)	0·5641 (3)	0·056
C(4)	0·3447 (3)	0·6397 (3)	0·4762 (3)	0·062
C(5)	0·6141 (3)	0·8106 (3)	0·3005 (4)	0·064
C(6)	0·7049 (4)	0·9460 (4)	0·2058 (4)	0·065
C(7)	0·6299 (4)	1·0554 (4)	0·1815 (4)	0·064
C(8)	0·4621 (3)	1·0285 (3)	0·2550 (3)	0·059
C(9)	0·3704 (3)	0·8916 (3)	0·3482 (3)	0·047
C(10)	0·4446 (3)	0·7799 (3)	0·3734 (3)	0·052
C(11)	0·0851 (3)	0·8067 (3)	0·2844 (4)	0·057
N(12)	0·0118 (3)	0·7671 (3)	0·1807 (3)	0·085
C(13)	−0·0303 (3)	0·7363 (3)	0·6561 (3)	0·060
O(14)	−0·1052 (2)	0·8246 (2)	0·6342 (2)	0·073
O(15)	−0·0776 (2)	0·6265 (2)	0·7758 (2)	0·060
C(16)	−0·2336 (4)	0·6141 (4)	0·8904 (4)	0·067
C(17)	−0·2776 (4)	0·4695 (4)	0·9979 (4)	0·074

Table 2. *Bond distances* (Å) *and angles* (°) *and their standard deviations*

C(1)—N(2)	1·461 (3)	C(9)—C(10)	1·394 (4)
N(2)—C(3)	1·399 (3)	C(9)—C(1)	1·522 (3)
C(3)—C(4)	1·328 (4)	C(1)—C(11)	1·486 (4)
C(4)—C(10)	1·447 (4)	C(11)—N(12)	1·140 (4)
C(5)—C(10)	1·396 (4)	N(2)—C(13)	1·379 (3)
C(5)—C(6)	1·370 (4)	C(13)—O(14)	1·210 (3)
C(6)—C(7)	1·379 (5)	C(13)—O(15)	1·321 (3)
C(7)—C(8)	1·390 (4)	O(15)—C(16)	1·458 (4)
C(8)—C(9)	1·375 (4)	C(16)—C(17)	1·490 (5)
C(1)—N(2)—C(3)	120·1 (2)	C(5)—C(10)—C(9)	118·1 (2)
N(2)—C(3)—C(4)	121·4 (2)	C(4)—C(10)—C(5)	123·0 (2)
C(3)—C(4)—C(10)	121·8 (2)	C(9)—C(1)—C(11)	109·2 (2)
C(4)—C(10)—C(9)	118·9 (2)	N(2)—C(1)—C(11)	109·9 (2)
N(2)—C(1)—C(9)	112·9 (2)	C(1)—C(11)—N(12)	179·0 (2)
C(1)—C(9)—C(10)	119·5 (2)	C(1)—N(2)—C(13)	116·5 (2)
C(5)—C(6)—C(7)	120·2 (3)	C(3)—N(2)—C(13)	123·0 (2)
C(6)—C(7)—C(8)	119·6 (3)	N(2)—C(13)—O(15)	111·8 (2)
C(7)—C(8)—C(9)	120·1 (2)	N(2)—C(13)—O(14)	122·5 (2)
C(8)—C(9)—C(10)	120·8 (2)	O(14)—C(13)—O(15)	125·7 (2)
C(1)—C(9)—C(8)	119·7 (2)	C(13)—O(15)—C(16)	115·8 (2)
C(6)—C(5)—C(10)	121·1 (2)	O(15)—C(16)—C(17)	107·0 (2)

The molecule consists of a heterocyclic dihydro-genated ring condensed with an aromatic ring and two side chains, a nitrile group bonded to C(1) and an ethoxycarbonyl group joined to N(2). The N(2)—C(13) bond length [1·379 (3) Å] is similar to that found in the benzoyl derivative (II). The C=O [1·210 (3)] and C—O [1·321 (3) Å] bond distances are typical for such bonds (Soriano-Garcia, Toscano, García, Larraza & Sánchez, 1984). A stereoview of the molecule is shown in Fig. 1, and Table 3 reports the torsion angles characterizing the molecular conformation. The urethane group in the present structure ($\chi^2 = 5\cdot39$) and in the tetrahydro analog (III) is planar as is the amide group in the benzoyl derivatives (I) and (II). The sum of the valence angles around C(13) equals 360·0 (3)° in all Reissert compounds investigated so far. The sum of the valence angles in the bond system around N(2) varies slightly, 360·0 (3)° in (I), 359·1 (3)° in (II) and 358·7 (3)° in (III), and is 359·6 (3)° in the present structure. The τ, χ_C and χ_N parameters (Winkler & Dunitz, 1971) determining the twist and out-of-plane distortions of the N-substituted urethane and amide groups are as follows: $\tau = 8\cdot1$, 7·9, 2·3 and 4·9°, $\chi_C = 0\cdot7$, −0·6, 0·6 and −1·0°, $\chi_N = -1\cdot0$, 13·0, 11·3 and 6·6° for (I), (II), (III) and (IV), respectively. In all these structures the carbonyl group is *anti* with respect to the C(3)—N(2) bond. Maximum conjugation of the urethane(amide)-group electrons and the aromatic system of the isoquinoline moiety requires these fragments to be coplanar. This seems to be more easily realized by the *anti* disposition of the carbonyl group as
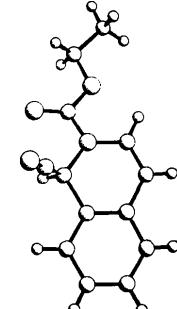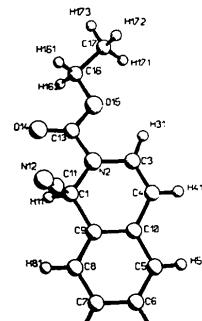

Fig. 1. Stereodrawing of the molecule.

Table 3. *Torsion angles* (°) *characterizing the molecular conformation*

|			
C(9)—C(1)—N(2)—C(3)	−26·5 (3)	C(1)—N(2)—C(13)—O(14)	2·1 (3)
C(1)—N(2)—C(3)—C(4)	14·8 (3)	C(1)—N(2)—C(13)—O(15)	−178·9 (2)
N(2)—C(3)—C(4)—C(10)	4·3 (3)	C(3)—N(2)—C(13)—O(14)	−171·3 (3)
C(3)—C(4)—C(10)—C(9)	−9·1 (3)	C(3)—N(2)—C(13)—O(15)	7·7 (3)
C(4)—C(10)—C(9)—C(1)	−4·8 (3)	N(2)—C(13)—O(15)—C(16)	−179·5 (3)
C(10)—C(9)—C(1)—N(2)	21·5 (3)	O(14)—C(13)—O(15)—C(16)	−0·5 (3)
C(11)—C(1)—N(2)—C(13)	−78·0 (3)	C(13)—O(15)—C(16)—C(17)	171·4 (3)
C(9)—C(1)—N(2)—C(13)	159·9 (2)		
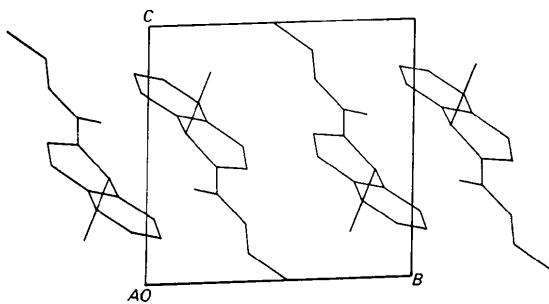
Fig. 2. Projection of the structure down **a**.

a consequence of steric hindrance between O(14) and H(C3) in the other possible rotational isomer. In a molecule with a bulky dimethoxybenzoyl substitutent at C(1) (Pavkovic, Glowinski, Feng & Brown, 1981) the carbonyl group is oriented *syn* relative to the N(2)—C(3) bond, indicating that the disposition of the C(3)—O(14) bond is a compromise between steric interactions with the neighboring substituents at C(1) and C(3).

The heterocyclic ring has a sofa conformation in (I), a 1,3-diplanar form in (II) and a half-chair form in (III). In the present compound (IV) it is intermediate between 1,3-diplanar and sofa conformations. Asymmetry parameters (Duax & Norton, 1975) $\Delta C_2^{1,2} = 7.99$ and $\Delta C_s^1 = 7.02$ indicate better consistency with the sofa conformation.

Fig. 2 presents the molecular packing of (IV) in the unit cell. In the racemic structure two chiral molecules [asymmetric C(1)] across the inversion center $(00\tfrac{1}{2})$ are joined by a weak C(1)—H(11)$\cdots$O(14) hydrogen bond. The H(11)$\cdots$O(14) distance equals 2.33 (2) Å and according to Taylor & Kennard (1982) can be

considered as a hydrogen bond. All other contacts are in the ranges of the van der Waals distances.

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Structure of N^2-(p-Methoxyphenyl)-N^1,N^1-pentamethylenebenzamidine

BY E. TYKARSKA, M. JASKÓLSKI AND Z. KOSTURKIEWICZ*

Department of Crystallography, Faculty of Chemistry, A. Mickiewicz University, 60–780 Poznań, Poland

Abstract. 4-Methoxy-N-(α-piperidinobenzylidene)-aniline, $C_{19}H_{22}N_2O$, $M_r = 294.40$, orthorhombic, $P2_12_12_1$, $a = 9.525$ (1), $b = 19.680$ (2), $c = 8.757$ (1) Å, $V = 1641.4$ (3) Å^3, $Z = 4$, $D_m = 1.18$, $D_x = 1.19$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1.54178$ Å, $\mu = 5.05$ cm^{-1}, $F(000) = 632$, room temperature, $R = 0.047$ for 1189 observed reflexions. The benzamidine phenyl ring and the methoxyphenyl fragment (both approximately planar) are twisted with respect to the central amidine plane by 61.6 (4) and 54.6 (3)°, respectively. The piperidine ring is in a chair conformation. The C—N^1 and C—N^2 bonds are different [1.283 (5) and 1.372 (5) Å, respectively].

* To whom correspondence should be addressed.