

La conformation chaise du cycle (Fig. 1) est confirmée par la position des atomes C(7) et C(10) qui se situent de part et d'autre du plan moyen (C) défini par les atomes C(8), C(9), C(11), C(12) avec des distances respectives de $-0,721$ (8) et $0,654$ (8) Å.

A l'intérieur du cristal les molécules sont orientées dans le sens de la longueur parallèlement à l'axe [100]. On note une liaison hydrogène intramoléculaire N—H(N)···O(2) (Fig. 2). L'atome d'hydrogène H(N) est contenu dans le plan (B) de la liaison amide avec un écart au plan de $0,02$ Å. Ce type de liaison est aussi signalé par Sindt & Mackay (1977).

Les trois autres liaisons hydrogène renforcent la cohésion intermoléculaire à l'intérieur du cristal. Elles sont comparables à celles observées dans des carbohydrates (Gatehouse & Poppleton, 1971; Park, Kim & Jeffrey, 1971) et dans le composé déjà cité (Satzke & Mackay, 1975; Sindt & Mackay, 1977).

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Structure of 2-Benzoyl-1,2,3,4-tetrahydro-1-isoquinolinecarbonitrile, C₁₇H₁₄N₂O

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Abstract. $M_r = 262$, monoclinic, $P2_1/a$, $a = 8.8155$ (6), $b = 14.687$ (1), $c = 10.6392$ (8) Å, $\beta = 94.44$ (6)°, $V = 1373.3$ (2) Å³, $Z = 4$, $D_m = 1.267$, $D_x = 1.266$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.54178$ Å, $\mu = 0.558$ mm⁻¹, $F(000) = 552$, room temperature. Final $R = 0.041$ for 1532 observed reflexions. The heterocyclic fragment of the molecule exhibits a flattened sofa conformation. The C(3)—N(2)—C(13)—O(14) torsion angle [-171.9 (3)°] characterizes the amide bond as *anti*. The amide group and the phenyl ring are deconjugated, the corresponding dihedral angle being 55.7 (4)°. This twisting thus minimizes steric interactions between the carbonyl group and neighbouring H atoms.

Introduction. This work initiates a series of investigations of Reissert compounds. The crystal-structure determination of the title compound [synthesized by Brózda (1982)] has been undertaken to study the effect of substituents on the molecular conformation and to facilitate interpretation of ¹³C NMR data (Brózda, 1982).

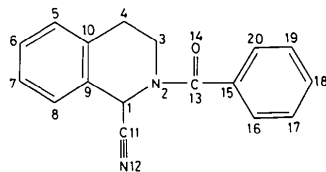
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Experimental. Suitable crystals obtained from ethanol, D_m by flotation, crystal $0.3 \times 0.4 \times 0.4$ mm, Syntex $P2_1$ diffractometer, cell parameters from least-squares treatment of setting angles of 15 reflexions, profiles measured for 2151 reflexions with $2\theta \leq 115^\circ$, range of hkl : $h \pm 9$, $k 0 \rightarrow 16$, $l 0 \rightarrow 11$, profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for two standard reflexions, no absorption correction, 1538 observed reflexions with $I \geq 1.96\sigma(I)$; structure solved by direct methods using *SHELX76* (Sheldrick, 1976), full-matrix least-squares refinement on F , $w^{-1} = \sigma^2(F)$, six extinction-affected reflexions excluded from final refinement, H atoms located in a ΔF map and included as fixed isotropic contributions to F_c , anisotropic thermal parameters for all non-H atoms, $R = 0.041$, $R_w = 0.046$, max. shift/e.s.d. = 0.004 , largest peak in final ΔF map = $0.15 \text{ e } \text{Å}^{-3}$, largest hole = $-0.15 \text{ e } \text{Å}^{-3}$; computer programs: *SHELX76* (Sheldrick, 1976) and local programs (Jaskólski, 1982), molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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Discussion. Atomic coordinates and bond lengths and angles are given in Tables 1 and 2 respectively.* The labelling sequence is shown in the scheme below.



The molecule consists of a heterocyclic hydrogenated ring condensed with an aromatic ring and two side chains: a cyano group bonded to the asymmetric atom C(1) and a benzoyl group joined to N(2). Bond lengths and angles in the aromatic fragments of the molecule do not deviate significantly from standard values. The N(2)–C(1) and N(2)–C(3) bond distances are very similar [1.461 (3) and 1.464 (3) Å] and in good agreement with the values found in piperidine amides (Jaskólski, 1979). The hetero ring contains three sp^2 atoms, C(9), C(10) and N(2). C(1) is bonded to two of these centres and this probably accounts for the opening of the C(9)–C(1)–N(2) angle [114.4 (2)°]. The bond distances and angles in the 1,2,3,4-tetrahydroisoquinoline moiety are in good agreement with the values reported for another 1,2,3,4-tetrahydroisoquinoline derivative (Bellard, Elliott & McDonald, 1982).

A stereoview of the molecule is shown in Fig. 1, while Table 3 reports the torsion angles characterizing the molecular conformation. The N(2)–C(1)–C(9)–C(10)–C(4) fragment of the heterocyclic ring is relatively flat. The torsion angles around the hetero ring are typical for a sofa conformation. The corresponding asymmetry parameter (Duax & Norton, 1975) is $\Delta C_s^3 = 3.3^\circ$. The C=O group is placed *anti* with respect to the C(3)–N(2) bond. Maximum conjugation of the amide-group electrons and the aromatic system of the isoquinoline moiety requires those groups to be coplanar. It seems to be more easily realized by *anti* disposition as a consequence of steric hindrance between O(14) and pseudo-equatorial H(C3) in the other possible amide rotational isomer. The COCN amide group is exactly planar ($\chi^2 = 3.40$). The N(2) amide atom is flat [sum of C–N(2)–C angles = 360.0 (3)°] but the N(2)C₃ plane is slightly non-coplanar with that through the COCN group. The twist angle τ (Winkler & Dunitz, 1971) about the N(2)–C(13) bond is 9.0 (2)°. The aromatic ring of the tetrahydroisoquinoline moiety is exactly planar (χ^2

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
C(1)	0.2290 (3)	0.3920 (1)	0.3760 (2)	0.0462 (8)
N(2)	0.2615 (2)	0.4759 (1)	0.3093 (2)	0.0446 (6)
C(3)	0.3246 (3)	0.5522 (1)	0.3850 (2)	0.0552 (9)
C(4)	0.2272 (3)	0.5738 (1)	0.4905 (2)	0.061 (1)
C(5)	0.1744 (3)	0.4975 (2)	0.6951 (2)	0.064 (1)
C(6)	0.1542 (3)	0.4222 (2)	0.7676 (2)	0.068 (1)
C(7)	0.1594 (3)	0.3374 (2)	0.7139 (2)	0.072 (1)
C(8)	0.1856 (3)	0.3291 (2)	0.5890 (2)	0.063 (1)
C(9)	0.2062 (3)	0.4049 (1)	0.5150 (2)	0.0461 (8)
C(10)	0.2018 (3)	0.4906 (1)	0.5686 (2)	0.0507 (8)
C(11)	0.3527 (3)	0.3254 (2)	0.3601 (2)	0.0554 (9)
N(12)	0.4488 (3)	0.2750 (1)	0.3487 (2)	0.085 (1)
C(13)	0.2325 (3)	0.4757 (1)	0.1819 (2)	0.0487 (8)
O(14)	0.1641 (2)	0.4121 (1)	0.1288 (1)	0.0627 (6)
C(15)	0.2875 (3)	0.5543 (1)	0.1080 (2)	0.0468 (8)
C(16)	0.1857 (3)	0.5992 (1)	0.0242 (2)	0.0521 (8)
C(17)	0.2376 (3)	0.6680 (2)	–0.0508 (2)	0.0594 (9)
C(18)	0.3893 (3)	0.6905 (2)	–0.0437 (2)	0.067 (1)
C(19)	0.4906 (3)	0.6443 (2)	0.0371 (2)	0.075 (1)
C(20)	0.4393 (3)	0.5766 (2)	0.1140 (2)	0.065 (1)

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

C(1)–N(2)	1.461 (3)	C(1)–C(11)	1.485 (3)
N(2)–C(3)	1.464 (3)	C(11)–N(12)	1.138 (4)
C(3)–C(4)	1.499 (3)	N(2)–C(13)	1.360 (3)
C(4)–C(10)	1.504 (3)	C(13)–O(14)	1.225 (3)
C(1)–C(9)	1.520 (3)	C(13)–C(15)	1.497 (3)
C(5)–C(6)	1.368 (4)	C(15)–C(16)	1.383 (3)
C(6)–C(7)	1.373 (4)	C(16)–C(17)	1.386 (3)
C(7)–C(8)	1.372 (3)	C(17)–C(18)	1.373 (4)
C(8)–C(9)	1.383 (3)	C(18)–C(19)	1.371 (4)
C(9)–C(10)	1.383 (3)	C(19)–C(20)	1.386 (4)
C(5)–C(10)	1.390 (3)	C(20)–C(15)	1.375 (4)
C(1)–N(2)–C(3)	117.3 (2)	N(2)–C(1)–C(11)	109.1 (2)
N(2)–C(3)–C(4)	111.0 (2)	C(1)–C(11)–N(12)	179.2 (2)
C(3)–C(4)–C(10)	111.1 (2)	C(1)–N(2)–C(13)	117.0 (2)
C(4)–C(10)–C(9)	120.1 (2)	N(2)–C(13)–O(14)	120.6 (2)
C(10)–C(9)–C(1)	121.6 (2)	N(2)–C(13)–C(15)	118.6 (2)
C(9)–C(1)–N(2)	114.4 (2)	O(14)–C(13)–C(15)	120.9 (2)
C(10)–C(5)–C(6)	121.8 (2)	C(15)–C(16)–C(17)	119.5 (2)
C(5)–C(6)–C(7)	119.3 (2)	C(16)–C(17)–C(18)	120.5 (2)
C(6)–C(7)–C(8)	119.8 (2)	C(17)–C(18)–C(19)	120.0 (2)
C(7)–C(8)–C(9)	121.3 (2)	C(18)–C(19)–C(20)	119.9 (2)
C(8)–C(9)–C(10)	119.3 (2)	C(19)–C(20)–C(15)	120.3 (2)
C(9)–C(10)–C(5)	118.6 (2)	C(20)–C(15)–C(16)	119.8 (2)
C(4)–C(10)–C(5)	121.3 (2)	C(3)–N(2)–C(13)	125.7 (2)
C(1)–C(9)–C(8)	119.1 (2)		

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

C(9)–C(1)–N(2)–C(3)	22.6 (2)	C(1)–N(2)–C(13)–O(14)	9.1 (2)
C(1)–N(2)–C(3)–C(4)	–53.4 (2)	C(3)–N(2)–C(13)–C(15)	8.8 (2)
N(2)–C(3)–C(4)–C(10)	53.9 (2)	O(14)–C(13)–C(15)–C(16)	52.8 (3)
C(3)–C(4)–C(10)–C(9)	–27.3 (2)	N(2)–C(13)–C(15)–C(20)	58.0 (2)
C(1)–C(9)–C(10)–C(4)	–2.9 (2)	C(3)–N(2)–C(13)–O(14)	–171.9 (3)
N(2)–C(1)–C(9)–C(10)	6.2 (2)	C(1)–N(2)–C(13)–C(15)	–170.2 (2)

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39294 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

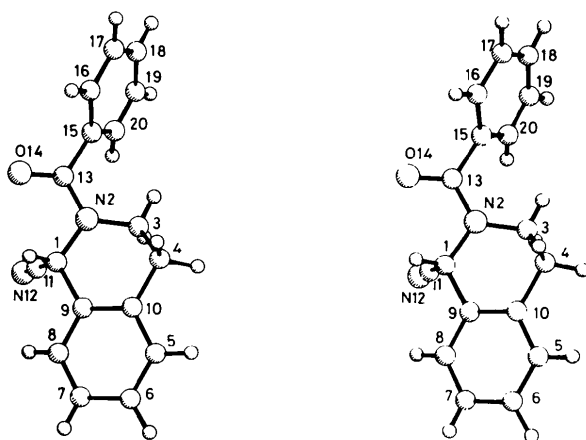


Fig. 1. Stereodrawing of the molecule.

= 7.84) while the phenyl ring of the benzoyl group shows slight but significant deviation from planarity ($\chi^2 = 54.61$). The amide group and the phenyl ring are deconjugated, the dihedral angle between their mean planes being $55.7(4)^\circ$. The lack of conjugation between these two groups is a frequent effect in amides with the aromatic rings at their carbon end (Gdaniec, Jaskólski & Kosturkiewicz, 1979). In the present case it can be attributed to the C=O group being *anti* to the C(3)—N(2) bond. In this situation, if the phenyl ring were to remain coplanar with the C=O group, prohibitive steric interactions between H(C16) or H(C20) and H(C3) would ensue.

The molecular packing is shown in Fig. 2. The chiral molecules compose a racemic crystal. The shortest intermolecular contacts are O(14)···H—C(16) (2.53 Å) and N(12)···H—C(1) (2.67 Å).

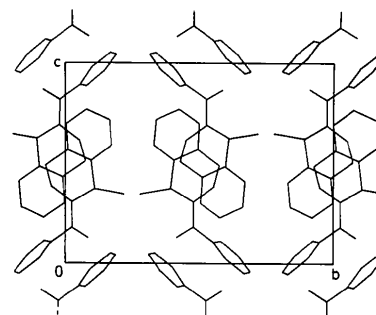


Fig. 2. Projection of the structure down *a*.

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