places the centres of positive charge as far apart as possible. Translational stacking is not surprising for pyridoxine since it is nondipolar in the crystal. However, minimizing electrostatic repulsions is not the sole determinant of packing behaviour, as PM.2H_2O contains translational stacks of PM molecules in spite of the fact that PM is a zwitterion.

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N-Isobutyrylbenzamide, $C_{11}H_{13}NO_2$

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Abstract. $M_r = 191\cdot2$, orthorhombic, $Pca2_1$, $a = 8\cdot878\ (2)$, $b = 13\cdot464\ (4)$, $c = 9\cdot009\ (2)$ Å, $U = 1077\ (1)$ Å^3, $Z = 4$, $D_x = 1\cdot179$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0\cdot71073$ Å, $\mu = 0\cdot088$ mm^{-1}, $F(000) = 408$, $T = 293$ K. Final $R = 0\cdot048$ for 385 observed reflections. A product of an unusual oxidation reaction is shown to be the title compound, apparently the first acyclic imide to be studied by X-ray crystallography. Unlike cyclic imides, this imide has O atoms in a 'U' conformation. Electrostatic repulsions between O atoms are relieved by twisting the π system and by opening the O—C—N and C—N—C angles. Bifurcated hydrogen bonds from HN to the O atoms of an adjacent molecule join adjacent molecules in the crystal.

Introduction. During efforts to synthesize the antitumor agent deoxybouvardin, we hydrogenated the azlactone prepared by condensing acetone with hippuric acid and obtained, among other products (Bates & Janda, 1984), a highly crystalline substance in 2% yield whose ^{1}H NMR spectrum indicated the presence of an isopropyl group and a phenyl group. A crystal-structure analysis was performed on this substance when it appeared to be the best method of establishing the arrangement of atoms between the isopropyl group and phenyl group.

Experimental. Crystal dimensions $0\cdot4 \times 0\cdot1 \times 0\cdot1$ mm. Syntex $P2_1$ four-circle diffractometer. Cell constants from 25 reflections with $7 \le 2\theta \le 14°$. No absorption correction. $2\theta_{\max} = 45°$. Range of hkl: 0–9, 0–14, 0–9. Three check reflections collected after every 200 data points showed no appreciable crystal decay. 385 of 764 reflections with $I > 3\sigma(I)$ used in F^2 refinement, $R_{\rm int} = 0\cdot048$. Structure solved by *MULTAN*80 (Main *et al.*, 1980) using 180 highest E values; 11 non-H atoms found in first E map; other three non-H atoms put in calculated positions. Refinement of non-H atoms with isotropic temperature factors gave $R = 0\cdot102$, final refinement (126 parameters) was of non-H atoms with anisotropic temperature factors after adding H atoms in calculated positions with isotropic temperature factors; $R = 0\cdot048$, $wR = 0\cdot054$, $S = 2\cdot0$, weighting scheme of Corfield, Doedens & Ibers (1967), with $p = 0\cdot04$. $(\Delta/\sigma)_{\max} = 0\cdot007$. $\Delta\rho = -0\cdot3$–$0\cdot3$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1962). Least-squares-refinement program *NUCLS* (Doedens & Ibers, 1967), plotter program *ORTEP* (Johnson, 1965).

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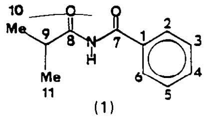
Discussion. Fig. 1 depicts the final bond lengths and angles, Table 1 lists the final positional parameters and isotropic thermal parameters, and the unit-cell contents are shown in Fig. 2.* The compound, totally unexpected, is the unsymmetrical imide (1). Its formation involves oxidative loss of a C atom from an azlactone, a process we have recently brought about in 60% yield (Bates, Fletcher, Janda & Miller, 1984).



(1)

This compound is apparently the first acyclic imide to be studied by X-rays. Unlike the imides in five- and six-membered rings, where the O atoms must be at the ends of a 'W' conformation, the O atoms in acyclic imides have a choice, and in this structure adopt a twisted 'U' conformation. The gradual twist about the π system is indicated by torsion angles of 10·4 and 12·0° about C(9)—C(8)—N—C(7) and C(8)—N—C(7)—C(1), respectively, and a 24·3° angle between the least-squares planes formed by N—C(7)—O(7)—C(1) and the benzene ring (continuing the twist in the same direction). The O—C—N and C—N—C angles are all greater than 120°, presumably to give a greater distance between the O atoms with their partial negative charges. The isopropyl group is rotated to put C(11) almost halfway between O(8) and HN, with C(10) close to O(8) [C(10)—C(9)—C(8)—O(8) = 25·9°] and HC(9) close to HN.

As can be seen in Fig. 2, the molecules in the crystal are held together by an infinite series of bifurcated hydrogen bonds (dotted lines) in the **c** direction from the HN of one molecule to the two carbonyl O atoms of the next molecule. HN thus fulfills a role played by a metal in metal acetylacetonates. The distances and angles observed are typical of a bifurcated system (Czochralska, Shugar, Arora, Bates & Cutler, 1977): N—O(7), 2·92; N—O(8), 3·15; HN—O(7), 2·10; HN—O(8), 2·28 Å; N—H—O(7), 137; N—H—O(8), 144°.

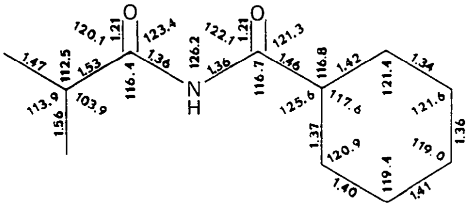


Fig. 1. Bond lengths (Å) and angles (°). E.s.d.'s in bond lengths are 0·02–0·03 Å, and in angles 0·9–1·2°.

Table 1. *Final atomic positional parameters* (× 10³) *and isotropic thermal parameters for non*-H *atoms with e.s.d.'s in parentheses*

Temperature factors are of the form exp[$-B(\sin^2\theta/\lambda^2)$].

	x	y	z	B(Å²)
C(1)	35 (1)	835 (1)	666	3·4 (4)
C(2)	−11 (2)	929 (1)	724 (2)	4·6 (5)
C(3)	−121 (2)	982 (1)	661 (2)	6·1 (6)
C(4)	−192 (1)	950 (1)	535 (2)	6·5 (6)
C(5)	−148 (2)	858 (1)	472 (2)	6·1 (6)
C(6)	−34 (2)	803 (1)	539 (2)	3·8 (4)
C(7)	155 (2)	784 (1)	745 (2)	3·9 (5)
C(8)	337 (2)	650 (1)	721 (2)	3·9 (4)
C(9)	438 (2)	596 (1)	608 (2)	5·1 (5)
C(10)	480 (2)	496 (1)	658 (3)	6·7 (6)
C(11)	574 (2)	668 (1)	587 (3)	9·4 (7)
N	238 (1)	718 (1)	665 (2)	3·3 (3)
O(7)	177 (1)	798 (1)	876 (2)	5·0 (3)
O(8)	343 (1)	628 (1)	852 (2)	5·9 (4)

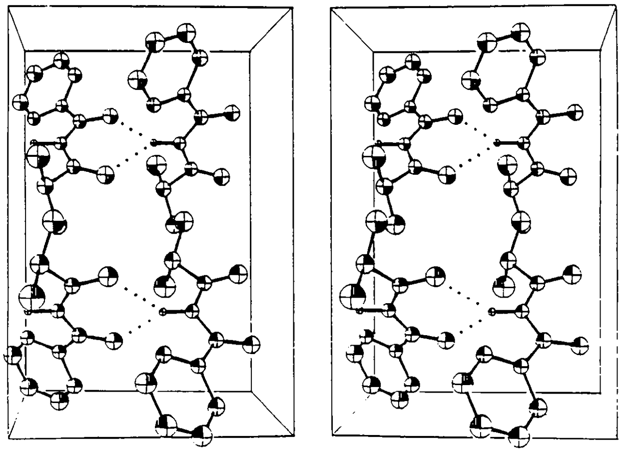


Fig. 2. Stereoscopic illustration of a unit cell, **c** horizontal, **b** vertical, with 30% probability thermal spheres.

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* 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 39807 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.