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Structure of (*E*)-4-Benzoylbutyramide Oxime

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Abstract. 4-(α-Hydroxyiminobenzyl)butyramide, $C_{11}H_{14}N_2O_2$, $M_r = 206.24$, tetragonal, $I4_1cd$, $a = b = 15.582\,(5)$, $c = 17.528\,(5)$ Å, $V = 4255.8$ Å^3, $Z = 16$, $D_x = 1.288$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.54$ cm^{-1}, $F(000) = 1760$, $T = 223$ K, $R = 0.0485$, $wR = 0.0415$ for 845 independent reflections [$|F| > 3.92\sigma(F)$]. Intermolecular hydrogen bonds between amide and oxime groups and between amide N and carbonyl O atoms were observed.

Introduction. The crystal structures of several compounds containing both carboxylic acid and oxime groups have recently been investigated. (*e.g.*, Maurin, Paul & Curtin, 1992). In all crystals so far determined, carboxyl–oxime hydrogen bonding was found to be the dominant packing feature rather than carboxyl–carboxyl or oxime–oxime hydrogen bonds. In order to determine whether similar bonds were observed in crystal structures of molecules containing both amide and oxime groups the structure of the title compound was determined and is reported here.

Experimental. (*E*)-4-Benzoylbutyramide oxime was obtained from 4-benzoylbutyric acid by treatment with thionyl chloride followed by aqueous ammonia to give the amide and, finally, treatment with hydroxylamine hydrochloride. Recrystallization from ethanol/water solution gave the oxime, m.p. 413.8–414.7 K. A fragment (0.30 × 0.30 × 0.36 mm) of an elongated colorless prismatic crystal was used for data collection. The crystal lattice parameters were obtained by a least-squares treatment of 25 reflections with $16.4 \leq 2\theta \leq 24.0°$. 2579 intensities were measured using ω–θ-scan mode on a CAD-4 single-crystal diffractometer; 1326 of them were not related by symmetry. Intensities were collected to $2\theta \leq 56°$ ($0 \leq h \leq 20$, $0 \leq k \leq 20$, $0 \leq l \leq 23$). The systematic absences ($00l$, $l = 4n$; hkl, $h + k + l = 2n$; hhl, $2h + l = 4n$; $0kl$, k, $l = 2n$) indicated $I4_1cd$ as the space group. No significant variation in the intensities of three standard reflections was observed during the data collection. Symmetrically related reflections were averaged (internal consistency $R_i = 0.0249$). The data were corrected for Lorentz–polarization effects but not for absorption or extinction. The structure was solved using the *SHELXS*86 direct-methods program (Sheldrick, 1986) and refined by minimizing $\sum w||F_o| - |F_c||^2$ with the use of the *SHELX*76 program (Sheldrick, 1976). All of the non-H atoms were located from an E map resulting in an agreement factor of 0.19, which decreased to 0.09 after refinement of positional and isotropic thermal parameters. Further refinement with anisotropic thermal parameters gave an R factor of 0.079. The H atoms attached to the C atoms were located according to geometrical conditions. The amide and the oxime-group H atoms were located from a difference Fourier map, calculated mainly from low-angle reflections. The inclusion of the H atoms at fixed positions (except for the oxime and amide H atoms which were allowed to vary) into a model, refinement of positional and anisotropic thermal parameters for the other atoms and, finally, the use of weights $w_i = 1.43/[\sigma^2(F_i) + 0.00025F_i^2]$, resulted in the R factors given in the *Abstract* (GOF = 1.345). The peaks on the final difference map were in the range -0.30–0.21 e Å^{-3}. The ratios of shifts to e.s.d.'s in the last cycle of refinement were less than 0.038 with an average value of 0.005. The ratio of data to refined parameters was 5.35. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).*

Discussion. The molecular structure and the atom numbering are shown in Fig. 1. The atomic coordinates are given in Table 1. The bond lengths and

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* Lists of H-atom coordinates, anisotropic thermal parameters, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55090 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1000]

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angles involving the non-H atoms are listed in Table 2. The oxime has the *E* configuration. A stereoview of crystal packing down the *c* axis is shown in Fig. 2 with the hydrogen bonds marked as thin lines. Previously studied crystal structures of molecules containing both carbonyl and oxime groups (Maurin, Paul & Curtin, 1992) have been dominated by intermolecular carbonyl–oxime hydrogen bonding. Similar features are found in the structure of 4-benzoylbutyramide oxime. The amide and oxime groups are linked by N—H···N and O—H···O hydrogen bonds to form chains of molecules that run in the **b** direction. In addition, the chains are linked in the **c** direction by amide–amide (N—H···O) hydrogen bonds; the amide O atom acts as an acceptor for both oxime O—H···O and amide N—H···O hydrogen atoms. The hydrogen-bond lengths are: O—H···O 2.730 (5) Å with an angle of 166 (4)°, N—H···N 2.969 (6) Å with an angle of 153 (3)° and N—H···O 2.814 Å with an angle of 168 (3)°. No other close contacts were observed.

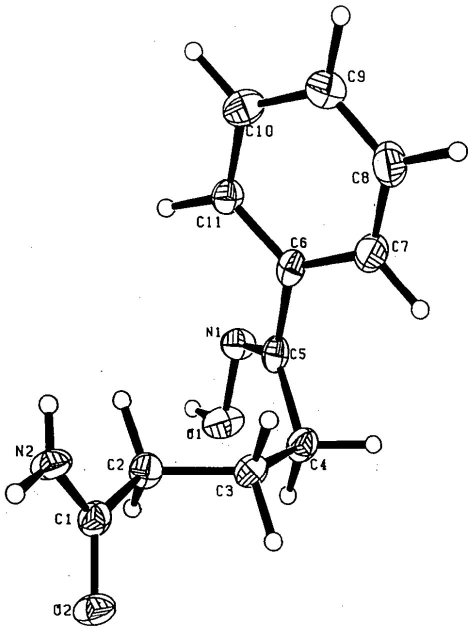

Fig. 1. Conformation of the molecule and numbering of atoms.

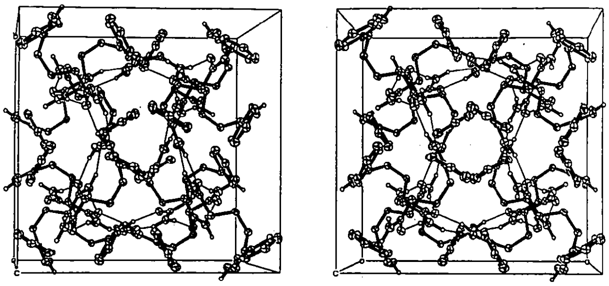

Fig. 2. Stereoview of crystal packing. Hydrogen bonds are marked as thin lines. Only those H atoms involved in hydrogen bonding are represented.

Table 1. *Fractional atomic coordinates and isotropic/equivalent isotropic thermal parameters* (Å^2)

U_{iso} for H atoms. $U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3$.

	x	*y*	*z*	$U_{\text{iso}}/U_{\text{eq}}$
C1	0.2764 (3)	0.3003 (2)	0.2519*	0.034 (2)
C2	0.2293 (3)	0.2160 (2)	0.2537 (4)	0.035 (2)
C3	0.1329 (3)	0.2326 (2)	0.2621 (4)	0.034 (2)
C4	0.0793 (3)	0.1511 (2)	0.2470 (4)	0.038 (2)
C5	0.1029 (2)	0.0782 (3)	0.2995 (4)	0.032 (2)
C6	0.0914 (3)	0.0870 (3)	0.3822 (3)	0.031 (2)
C7	0.0197 (3)	0.1290 (3)	0.4128 (4)	0.043 (3)
C8	0.0073 (3)	0.1334 (3)	0.4913 (4)	0.050 (3)
C9	0.0660 (3)	0.0964 (3)	0.5399 (4)	0.043 (2)
C10	0.1385 (3)	0.0565 (3)	0.5111 (4)	0.043 (2)
C11	0.1511 (3)	0.0527 (3)	0.4328 (3)	0.035 (2)
N1	0.1346 (2)	0.0072 (3)	0.2779 (3)	0.038 (2)
N2	0.3134 (3)	0.3286 (3)	0.3143 (3)	0.038 (2)
O1	0.1470 (2)	0.0020 (2)	0.1984 (3)	0.051 (2)
O2	0.2763 (2)	0.3447 (2)	0.1921 (3)	0.040 (2)
H*a*N	0.316 (2)	0.294 (2)	0.351 (2)	0.022 (5)
H*b*N	0.343 (2)	0.382 (2)	0.316 (2)	0.039 (5)
HO	0.177 (3)	−0.041 (3)	0.192 (3)	0.061 (6)

* *z* coordinate of C1 fixed in order to define origin for non-centrosymmetric space group.

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

N—H bond lengths are 0.84 (3) and 0.96 (3) Å, respectively, O—H is 0.82 (4) Å. All C—H bond lengths were fixed and equal to 1.08 Å.

C1—C2	1.504 (5)	C2—C1—N2	119.3 (3)
C2—C3	1.532 (6)	C2—C1—O2	119.9 (3)
C3—C4	1.543 (6)	N2—C1—O2	120.7 (4)
C4—C5	1.507 (7)	C1—C2—C3	109.5 (3)
C5—C6	1.468 (9)	C2—C3—C4	112.1 (3)
C6—C7	1.401 (7)	C3—C4—C5	112.5 (4)
C7—C8	1.390 (9)	C4—C5—C6	120.2 (4)
C8—C9	1.377 (8)	C4—C5—N1	124.7 (6)
C9—C10	1.384 (7)	C6—C5—N1	115.0 (5)
C6—C11	1.391 (7)	C5—C6—C7	121.2 (5)
C10—C11	1.388 (9)	C5—C6—C11	120.8 (4)
C5—N1	1.269 (6)	C7—C6—C11	117.9 (5)
C1—N2	1.314 (5)	C6—C7—C8	120.8 (5)
N1—O1	1.410 (8)	C7—C8—C9	119.9 (5)
C1—O2	1.257 (5)	C8—C9—C10	120.3 (6)
		C9—C10—C11	119.7 (5)
		C6—C11—C10	121.3 (4)
		C5—N1—O1	113.5 (5)

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Structure of 3-Benzoylbenzamide Oxime

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Abstract. 3-(α-Hydroxyiminobenzyl)benzamide, $C_{14}H_{12}N_2O_2$, $M_r = 240.26$, orthorhombic, *Pbca*, $a = 7.9333\,(7)$, $b = 25.947\,(2)$, $c = 11.855\,(1)$ Å, $V = 2440.30$ Å^3, $Z = 8$, $D_x = 1.308$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.57$ cm^{-1}, $F(000) = 1008$, $T = 295$ K, $R = 0.0423$, $wR = 0.0467$ for 1487 reflections $[|F| \geq 3.92\sigma(F)]$. Intermolecular hydrogen bonds between amide and oxime groups, and between amide groups were observed.

Introduction. As part of our studies on the hydrogen-bond interactions in the packing of molecules containing both amide and oxime groups, compared with molecules containing both carboxylic acid and oxime groups, we determined the crystal structure of the title compound. The results and a comparison of the dominant packing features with those of related compounds are reported herein.

Experimental. 3-Benzoylbenzoic acid was converted to the amide oxime by treatment with $SOCl_2$ followed by aqueous ammonia and, finally, hydroxylamine hydrochloride solution. The product after recrystallization from ethanol/water solution had m.p. 466.8–467.4 K. A pale-yellow prismatic crystal of dimensions *ca* 0.42 × 0.19 × 0.17 mm was used for data collection. The lattice constants were obtained by least-squares treatment of 42 reflections with $9.48 \leq 2\theta \leq 20.53°$. 2143 unique reflections were measured at room temperature using the ω–2θ scan mode on a Syntex $P2_1$ single-crystal diffractometer to $2\theta \leq 50°$ ($0 \leq h \leq 9$, $0 \leq k \leq 30$, $0 \leq l \leq 14$). Graphite-monochromated Mo $K\alpha$ radiation was used for data collection. The systematic absences ($0kl$, k odd; $h0l$, l odd; $hk0$, h odd) indicated *Pbca* as the space group. No significant variation in the intensities of three standard reflections was observed during the data collection. The data were corrected for Lorentz–polarization effects but not for absorption or extinction. The structure was solved using the *SHELXS*86 direct-methods program (Sheldrick, 1986) and refined by use of the *SHELX*76 program (Sheldrick, 1976). All of the non-H atoms were located from an *E* map resulting in an agreement factor of 0.17, which decreased to 0.12 after refinement of positional and isotropic thermal parameters. Further refinement with anisotropic thermal parameters gave an *R* factor of 0.081. The H atoms attached to the benzene rings were located according to geometrical conditions. The amide and the oxime H atoms were located from the difference Fourier map. Refinement of a model with positional and anisotropic thermal parameters for the non-H atoms and with positional and isotropic thermal parameters for the other atoms, and the use of weights $w_i = 2.85/\sigma^2(F_i)$, resulted in $R = 0.0423$, $wR = 0.0467$ and GOF = 1.787. The peaks on the final difference map were in the range −0.19–0.16 e Å^{-3}. The ratios of shifts to e.s.d.'s in the last cycle of refinement were less than 0.005 with an average value of 0.001. The ratio of reflections to refined parameters was 1487/211 = 7.05. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).*

Discussion. The molecular structure and the atom numbering are shown in Fig. 1. The atomic coordinates are given in Table 1. The bond lengths and angles involving the non-H atoms are listed in Table 2. The configuration of the oxime group is such that the hydroxyl group is *syn* to the benzamide ring. The oxime group does not lie in the plane of either phenyl ring. The dihedral angles between the plane defined by atoms C8, N1 and O1, and the planes of the rings C1–C6 and C9–C14 are 60.7 and 23.3°, respectively. The C3 and C9 atoms are almost copla-

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* Lists of H-atom coordinates, bond distances and angles involving H atoms, anisotropic thermal parameters, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55095 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1001]

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