The molecules are almost parallel to the (100) plane in the crystal (Fig. 2). Two strong intermolecular hydrogen bonds, O1—HO1···O2 and O3—HO3···N1, are observed between the carboxyl and oxime groups. The O1···O2 and O3···N1 lengths are 2.731 (2) and 2.727 (2) Å, and the O1—HO1···O2 and O3—HO3···N1 angles are 169 (3) and 176 (2)°, respectively. For a comparison of hydrogen-bond lengths with those observed in carboxylic acid dimers and oxime dimers see Maurin, Paul & Curtin (1992). No other close contacts were observed.

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Structure of 3-Benzoylbenzoic Acid Oxime

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Abstract. 3-(α-Hydroxyiminobenzyl)benzoic acid, $C_{14}H_{11}NO_3$, $M_r = 241.25$, monoclinic, $P2_1/n$, $a = 9.169\,(1)$, $b = 9.370\,(1)$, $c = 13.883\,(2)$ Å, $\beta = 92.028\,(4)°$, $V = 1192.0\,(2)$ Å^3, $Z = 4$, $D_x = 1.345$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.89$ cm^{-1}, $F(000) = 504$, $T = 295$ K, $R = 0.0426$, $wR = 0.0454$ for 1538 reflections [$|F| \geq 3.92\sigma(F)$]. Infinite chains of molecules held together by pairs of strong hydrogen bonds between carboxyl and oxime groups were observed.

Introduction. 3-Benzoylbenzoic acid oxime is the third oxime structure solved in our laboratories (Padmanabhan, Paul & Curtin, 1989; Maurin, Paul & Curtin, 1992), which contains the carboxyl as the second functional group. Structures of different complexity are being studied to determine whether the preference for carboxyl–oxime hydrogen-bond formation is a general rule for this type of compound.

Experimental. The title compound was synthesized by the rection of 3-benzoylbenzoic acid with hydroxylamine hydrochloride in alkaline solution. The recrystallization of the solid product from ethanol/water solution gave prismatic crystals with a melting point of 475.5–476.4 K. A crystal with dimensions *ca* 0.22 × 0.10 × 0.55 mm was used for the data collection. The crystal lattice parameters were obtained by a least-squares treatment of 27 reflections within a 2θ range of 9.84–20.15°. The systematic absences ($0k0$, k odd; $h0l$, $h + l$ odd) indicated $P2_1/n$ as the space group. 2091 unique reflections were measured at room temperature using the ω–2θ scan mode on a Syntex $P2_1$ single-crystal diffractometer. Graphite-monochromated Mo $K\alpha$ radiation was used for data collection. Intensities were collected to $2\theta \leq 50°$ ($0 \leq h \leq 16$, $0 \leq k \leq 11$, $-10 \leq l \leq 10$). Four low-order reflections of very high intensity could not be measured reliably and were not included in any further calculations. No significant variation in the intensities of three standard reflections was observed during data collection. The data were corrected for Lorentz and polarization effects but not for absorption or extinction. The structure was solved using the *SHELXS86* program (Sheldrick, 1990), and refined by use of *SHELX76* (Sheldrick, 1976). All of the non-H atoms were located from an E map. Least-squares refinement on F with anisotropic displacement parameters gave $R = 0.081$. All H atoms were located from a $\Delta\rho$ map. The inclusion of the H atoms in the model, refinement of their positional and isotropic displacement parameters, and the use of weights $w_i = 1.0/[\sigma^2(F_i) + 0.0001F_i^2]$ gave final values of $R = 0.0426$ and $wR = 0.0453$; $S = 1.720$. The peaks on the final $\Delta\rho$ map were in the range -0.24–0.17 e Å^{-3}.

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The ratios of shifts to e.s.d.'s in the last cycle of refinement were $(\Delta/\sigma)_{\max} = 0.034$ and $(\Delta/\sigma)_{\text{av}} = 0.008$. The ratio of reflections to refined parameters was 7.43. 207 parameters were refined. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).*

Discussion. The conformation of the molecule and the numbering of the atoms are shown in Fig. 1. The atomic coordinates are given in Table 1. The bond lengths and angles involving the non-H atoms are given in Table 2. A stereoview of the unit-cell contents is shown in Fig. 2. The dihedral angle between the plane of the carboxyl group (O1—C7—O2) and the benzene-ring plane is 11.7 (2)°. The two phenyl rings are twisted from the plane of the oxime group. The dihedral angles between the plane defined by atoms C8, N1, O3 and the planes of rings C1–C6 and C9–C14 are 75.1 (1) and 31.4 (1)°, respectively. The atoms C3 and C9 are almost coplanar with the oxime group. The dihedral angle between the two phenyl rings is 94.6 (1)°. An analysis of bond lengths and angles leads to the conclusion that the observed geometry of the ring C1–C6 is only slightly distorted by thermal motion, while that of the second ring C9–C14 may be affected by libration. However, there is evidence of conjugation between the oxime group and the C9–C14 ring. Such an interaction is suggested by the shorter C8—C9 distance [1.477 (3) Å] than that of C3—C8 [1.498 (3) Å]. The much lower dihedral angle between the plane of the oxime group and the plane of the second ring [31.4 (1)°] in comparison with the angle for the first ring plane [75.1 (1)°] suggests the possible occurrence of such an interaction. Because the sensitivity of bond angles is

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non*-H *atoms*

$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)/3.$$

	x	y	z	U_{eq}
O1	−0.2247 (2)	0.0563 (2)	0.4975 (1)	0.0509 (9)
O2	−0.0780 (2)	0.1550 (2)	0.6108 (1)	0.0471 (9)
O3	−0.5523 (2)	0.3137 (2)	0.8684 (1)	0.0521 (9)
N1	−0.4592 (2)	0.2639 (2)	0.9428 (1)	0.039 (1)
C1	−0.3017 (2)	0.0581 (2)	0.6592 (1)	0.034 (1)
C2	−0.2837 (2)	0.1196 (2)	0.7500 (1)	0.033 (1)
C3	−0.3868 (2)	0.0955 (2)	0.8190 (1)	0.034 (1)
C4	−0.5056 (3)	0.0074 (3)	0.7970 (2)	0.043 (1)
C5	−0.5225 (3)	−0.0551 (3)	0.7070 (2)	0.049 (1)
C6	−0.4208 (3)	−0.0291 (3)	0.6377 (2)	0.043 (1)
C7	−0.1981 (2)	0.0883 (2)	0.5815 (2)	0.037 (1)
C8	−0.3748 (2)	0.1625 (2)	0.9169 (1)	0.033 (1)
C9	−0.2696 (2)	0.1088 (2)	0.9914 (1)	0.034 (1)
C10	−0.2167 (3)	0.1984 (3)	1.0654 (2)	0.043 (1)
C11	−0.1284 (3)	0.1445 (3)	1.1393 (2)	0.053 (1)
C12	−0.0910 (3)	0.0018 (4)	1.1406 (2)	0.060 (2)
C13	−0.1389 (3)	−0.0865 (3)	1.0667 (2)	0.056 (2)
C14	−0.2267 (3)	−0.0333 (3)	0.9918 (2)	0.044 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non*-H *atoms*

C—H and O—H bond lengths are in the range 0.94–0.98 Å.

O3—N1	1.397 (2)	O3—N1—C8	113.5 (2)
C1—C2	1.390 (3)	C2—C1—C6	120.4 (2)
C2—C3	1.388 (3)	C2—C1—C7	121.3 (2)
C3—C4	1.392 (3)	C6—C1—C7	118.3 (2)
C4—C5	1.383 (3)	C1—C2—C3	119.8 (2)
C1—C6	1.388 (3)	C2—C3—C4	119.4 (2)
C5—C6	1.385 (3)	C2—C3—C8	121.9 (2)
O1—C7	1.221 (3)	C4—C3—C8	118.7 (2)
O2—C7	1.318 (3)	C3—C4—C5	120.7 (2)
C1—C7	1.489 (3)	C4—C5—C6	119.8 (2)
N1—C8	1.284 (3)	C1—C6—C5	119.9 (2)
C3—C8	1.498 (3)	O1—C7—O2	123.2 (2)
C8—C9	1.477 (3)	O1—C7—C1	122.3 (2)
C9—C10	1.400 (3)	O2—C7—C1	114.5 (2)
C10—C11	1.380 (3)	N1—C8—C3	122.5 (2)
C11—C12	1.380 (5)	N1—C8—C9	116.2 (2)
C12—C13	1.378 (4)	C3—C8—C9	121.2 (2)
C9—C14	1.388 (3)	C8—C9—C10	120.7 (2)
C13—C14	1.386 (4)	C8—C9—C14	120.5 (2)
		C10—C9—C14	118.8 (2)
		C9—C10—C11	120.3 (2)
		C10—C11—C12	120.2 (3)
		C11—C12—C13	120.0 (3)
		C12—C13—C14	120.3 (3)
		C9—C14—C13	120.3 (2)

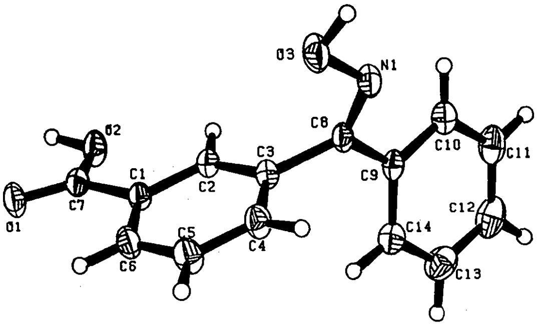

Fig. 1. Conformation of the molecule and the numbering of the atoms.

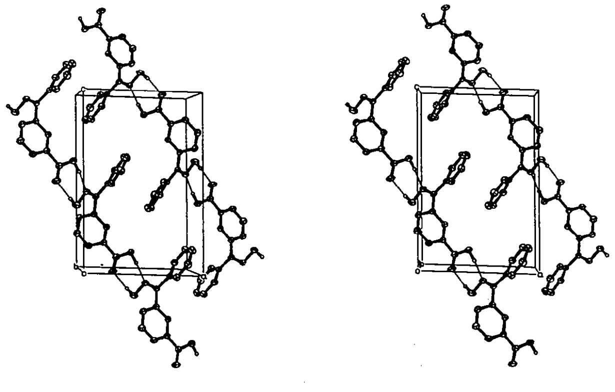

Fig. 2. Stereoview of the unit cell along the *b* axis with **a** across and **c** down the diagram. Only H atoms involved in hydrogen-bond formation are included.

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* Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55292 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0272]

less than that of bond lengths as a result of the influence of thermal motion (Domenicano, Vaciago & Coulson, 1975), the effect on the C8—C9 distance is less significant than that on the C10—C9—C14 bond angle; this value [118.8 (2)°] is lower than 120° suggesting some π-electron delocalization with the —C═N—OH substituent.

The crystal-packing diagram shows distinctly the strong hydrogen bonds between oxime and carboxyl groups related by the n-glide plane. The O2—HO2···N1 hydrogen bond has a length of 2.718 (2) Å with an angle of 174 (3)° while the O1···HO3—O3 hydrogen bond has a length 2.720 (2) Å and an angle of 157 (3)°. The mentioned hydrogen bonds have intermediate values compared with those observed in several oxime–oxime dimers (Bachechi & Zambonelli, 1972, 1973; Brehm & Watson, 1972; Gieren, Huebner & Ruiz-Perez, 1986) and those determined in carboxylic acid dimers [see for example Patil, Curtin & Paul (1985)]. No other hydrogen bonds or close intermolecular contacts were observed.

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Structures of Three Derivatives of 6-Phthalimidopenicillanic Acid

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Abstract. *cis*-6-Phthalimidopenicillanic acid, methyl ester, $C_{17}H_{16}N_2O_5S$ (*A*), $M_r = 360.39$, monoclinic, $P2_1$, $a = 11.137$ (2), $b = 6.976$ (4), $c = 11.755$ (3) Å, $\beta = 111.57$ (1)°, $V = 849$ (1) Å^3, $Z = 2$, $D_x = 1.412$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.2107$ mm^{-1}, $F(000) = 376$, $T = 298$ K, final $R = 0.030$ for 1457 unique diffractometer data and 225 refined parameters. *trans*-6-Phthalimidopenicillanic acid, methyl ester, $C_{17}H_{16}N_2O_5S$ (*B*), $M_r = 360.39$, orthorhombic, $P2_12_12_1$, $a = 10.276$ (2), $b = 10.850$ (2), $c = 15.371$ (3) Å, $V = 1713.8$ (6) Å^3, $Z = 4$, $D_x = 1.397$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.2107$ mm^{-1}, $F(000) = 752$, $T = 298$ K, final $R = 0.045$ for 1232 unique diffractometer data and 225 refined parameters. *cis*-6-Phthalimidopenicillanic acid, α-sulfoxide monohydrate, $C_{16}H_{14}N_2O_6S.H_2O$ (*C*), $M_r = 380.35$, orthorhombic, $P2_12_12_1$, $a = 10.028$ (2), $b = 13.004$ (3), $c = 12.865$ (2) Å, $V = 1677.5$ (6) Å^3, $Z = 4$, $D_x = 1.506$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.2200$ mm^{-1}, $F(000) = 792$, $T = 298$ K, final $R = 0.043$ for 1432 unique diffractometer data and 235 refined parameters. Twist (*T*), envelope E_3, and envelope E_S conformations have been observed for the thiazolidine ring of *A*, *B* and *C*, respectively. Epimerization at C atom C6 does not change the pyramidality of the β-lactam N atom; however, the β-lactam ring is more planar and the overall topology of the molecule is altered. Oxidation of the S atom to sulfoxide diminished pyramidality of the N atom and provided a very good acceptor for hydrogen bonding [O—H···O distance 2.633 (5) Å]. For the *cis* β-lactams *A* and *C*, short intramolecular non-bonding distances between the S atom and atoms from thiazolidine substitutents were observed. The relative orientation of the carboxyl group at C3

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