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(*E*)-4-Methoxybenzaldehyde *O*-(*N*-phenylcarbamoyl)oxime

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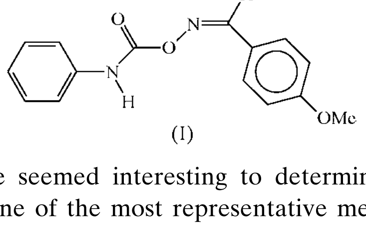
There are two molecules, $C_{15}H_{14}N_2O_3$, in the asymmetric unit of the title compound, the first structurally characterized carbamoyl benzaldehyde oxime derivative; these molecules do not differ significantly. Intermolecular N—H···O hydrogen bonds connect molecules into infinite ...*ABAB*... ribbons along the [100] direction.

Comment

There are some 35 structures of benzaldehyde oxime derivatives in the Cambridge Structural Database (CSD; Version of February 2005; Allen, 2002); however, there are no examples of carbamoyl derivatives. Moreover, there is only one structure, namely [2-methyl-2-(methylthio)propionaldehyde *O*-methylcarbamoyl]oxime (Takusagawa & Jacobson, 1977), containing the NH—C(═O)—O—N═C group, and that structure is relatively poorly determined (*R* = 9.3%, no H atoms). This lack of structures is even more surprising taking into account that chemical literature dealing with *O*-phenylcarbamoylated aldoximes is extensive, for example, acylation of aldoximes (Hauser *et al.*, 1935; Rainsford & Hauser, 1939) and conversion of aldehydes into nitriles (Coşkun & Arikan, 1999). These compounds also have biological activity; for instance, the analgesic anidoxime has the structure of phenylcarbamoylated oxime (Grainger *et al.*, 1977; Watzman & Buckley, 1980) and aldicarb, [2-methyl-2-(methylthio)propionaldehyde *O*-methylcarbamoyl]oxime, is used as a pesticide.



(I)

It therefore seemed interesting to determine the crystal structure of one of the most representative members of this family, the title compound, (I). The asymmetric unit contains two molecules (*A* and *B*). Normal probability plot analyses (Abrahams & Keve, 1971; *International Tables for X-ray Crystallography*, 1974, Vol. IV, p. 293) for both bond lengths and angles show that the differences between the symmetry-independent molecules (Fig. 1) are of a statistical nature; the squares of the correlation factors are 0.96 and 0.99 for bond lengths and angles, respectively.

The overall conformation of the molecule of (I) can be described by the dihedral angles between four approximately planar fragments, *viz.* the phenyl ring (p1), the N(H)COO group (p2), the CC(H)═NO fragment (p3) and the methoxy-substituted phenyl ring (p4). The maximum deviations are no larger than 0.007 (2) Å for the rings and 0.032 (4) Å for fragments p2 and p3. The dihedral angle between the terminal phenyl ring planes, which might serve as a measure of the overall twist of the molecules, is 60.9 (1)° in molecule *A* and 69.6 (1)° in molecule *B*. There are some minor differences in the conformations of the two molecules (Fig. 1 and Table 1). These differences are deserved in the values of the above-mentioned dihedral angles: the p1/p2 angle is 29.7 (1)° in molecule *A* and 29.2 (1)° in *B*, the p2/p3 angle is 21.0 (2)° in *A* and 30.6 (2)° in *B*, and the p3/p4 angles in molecules *A* and *B* are 10.3 (2) and 9.9 (2)°, respectively.

The bond lengths and angles are typical, with a double C2═N2 bond of 1.278 (3) Å, and the typical asymmetry of C—N bonds around atom N1; for 825 Ph—NH—C(═O)—*R* fragments found in the CSD (only organic compounds, no fused rings, no errors, $R < 0.075$), the mean value of the C_{phenyl}—N bond length is 1.414 (13) Å and that of the N—C(═O) bond length is 1.352 (15) Å.

In both symmetry-independent molecules in the title compound, the oxime group is in a *syn* (*E*) conformation (*e.g.* Ciajolo *et al.*, 1981), as can be seen from the values of the O12—N2═C2—C21 torsion angles [−175.5 (2) and −177.8 (2)°]. The conformation along the N—O bond is *anti*; the values of the C1—O12—N2═C2 torsion angles are close to 180°. It might be noted that a different conformation, *syn* (C═N—O—C torsion angle close to 0°), is observed exclusively for compounds in which the oxime fragment is involved in a cyclic environment. For 281 fragments with an acyclic C═N—O—*Z* (where *Z* is not hydrogen) group, there are only four hits with a C═N—O—*Z* torsion angle smaller than 130°, *viz.* *l*-canavanine (Boyar & Marsh, 1982), D-gluconohydroximo-1.5-lactone 1-*N*-2′,3′,4′,6′-pentabenzoate (Beer & Vasella, 1985), 4-(*N*-methoxymethylimino)-2,2-diphenyl-1,3-dioxa-2-borata-1,2,3,4-tetrahydronaphthalene (Kliegel *et al.*, 1989) and (*Z*)-2-(benzoyl-*aci*-nitro)-2-phenyl-

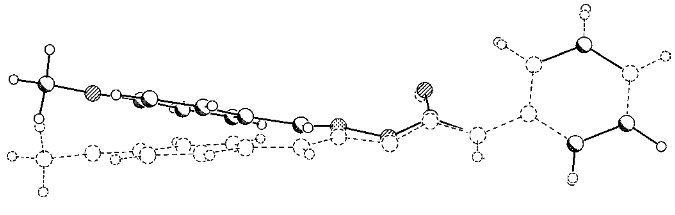


Figure 1
A comparison of the conformation of two symmetry-independent molecules. The terminal phenyl rings were fitted by the least-squares procedure (Siemens, 1989).

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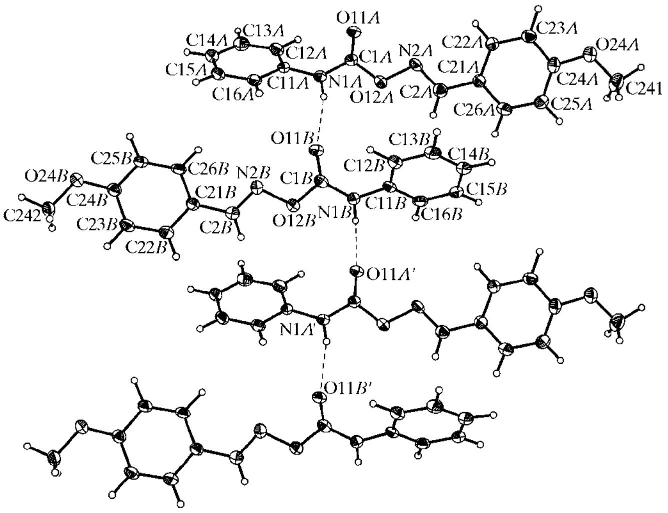

Figure 2
The hydrogen-bonded dimer of molecules *A* and *B*, with the atom-labelling scheme. Anisotropic displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Primes denote symmetry code (1 + *x*, *y*, *z*).

acetonitrile (Powell *et al.*, 1996). The smallest value of this angle is 97°. Almost 90% of these compounds have an absolute value of the C═N—O—*Z* torsion angle larger than 160°.

In the crystal structure, the molecules are arranged in infinite ...*ABAB*... ribbons along the [100] direction by means of strong and directional N—H···O hydrogen bonds (Fig. 2 and Table 1). Using graph-set notation (Etter *et al.*, 1990; Bernstein *et al.*, 1995) at the first order there are two dimers (*DD*), while the most informative is the second-rank graph-set $C_2^2(8)$; additional weak C—H···π contacts and intramolecular C12—H12···O11 hydrogen bonds may play stabilizing roles in the creation of the pattern.

Experimental

The method used for the preparation of (I) has been described elsewhere (Mohamed, 2004). The crystals used for data collection were grown from a methanol solution by slow evaporation.

Crystal data

$C_{15}H_{14}N_2O_3$
$M_r = 270.28$
Triclinic, $P\overline{1}$
$a = 8.8037$ (8) Å
$b = 12.1484$ (15) Å
$c = 12.9823$ (17) Å
$\alpha = 75.275$ (11)°
$\beta = 81.075$ (9)°
$\gamma = 80.746$ (9)°
$V = 1315.9$ (3) Å^3

$Z = 4$
$D_x = 1.364$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3412 reflections
$\theta = 4$–20°
$\mu = 0.10$ mm^{-1}
$T = 100$ (1) K
Plate, colourless
0.3 × 0.2 × 0.05 mm

Data collection

Kuma KM-4 CCD four-circle diffractometer
ω scans
10 887 measured reflections
5124 independent reflections
2749 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.056$
$\theta_{\rm max} = 26.0°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -11 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.128$
$S = 0.94$
5124 reflections
456 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0032 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O11*B*	0.84 (3)	2.10 (3)	2.920 (3)	165 (3)
N1*B*—H1*B*···O11*A*$^{\rm i}$	0.84 (3)	2.16 (3)	2.993 (3)	178 (3)
C12*B*—H12*B*···O11*B*	1.08 (3)	2.27 (2)	2.900 (3)	115 (2)
C12*A*—H12*A*···O11*A*	0.99 (2)	2.41 (2)	2.983 (3)	116 (2)

Symmetry code: (i) $x + 1, y, z$.

C—H distances are in the range 0.90 (2)–1.08 (3) Å.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Sterochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF1002). Services for accessing these data are described at the back of the journal.

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