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N^1-(4-Bromophenyl)-N^2-hydroxy-2-oxo-2-phenylacetamidine

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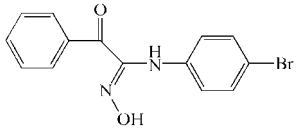
In the title compound, $C_{14}H_{11}BrN_2O_2$, which has the oxime group in an *E* conformation, molecules are linked by strong O—H···O and N—H···O hydrogen bonds into chains of edge-fused rings, unlike closely related compounds.

Comment

The oxime group (C═N—OH) possesses a stronger hydrogen-bonding capability than alcohol, phenol and carboxylic acid groups (Marsman *et al.*, 1999). Intermolecular hydrogen bonding has received considerable attention among the range of directional non-covalent intermolecular interactions, which combine moderate strength and directionality, in the design of compounds with supramolecular structures (Karle *et al.*, 1996). The hydrogen-bonding geometry of molecules containing the oxime group and the nature of the supramolecular interactions have been studied (Glidewell *et al.*, 2004). The crystal structure determination of the title compound, (I), was carried out to determine the strength of



(I)

the hydrogen-bonding capabilities of the oxime group, in order to establish the molecular arrangements and to compare the geometry of the oxime moiety with those found in the related compounds N^1-(2,6-dimethylphenyl)-N^2-hydroxy-α-oxo-α-phenylacetamidine, (II) (Soylu, Taş, Saraçoğlu *et al.*, 2004), *N*-(3-chloro-4-methoxyphenyl)-*N*′-hydroxy-2-oxo-2-phenylacetamidine, (III) (Soylu *et al.*, 2003), 2-[benzoyl-(hydroxyimino)methylamino]benzoic acid, (IV) (Soylu, Taş, Batı *et al.*, 2004), *N*-hydroxy-2-oxo-2,*N*′-diphenylacetamidine, (V) (Büyükgüngör *et al.*, 2003), *N*-hydroxy-*N*′-(1-naphthyl)-2-phenylacetamidin-2-one, (VI) (Hökelek *et al.*, 2004*a*), and *N*-(3-chloro-4-methylphenyl)-*N*′-hydroxy-2-oxo-2-phenylacetamidine, (VII) (Hökelek *et al.*, 2004*b*).

Compound (I) consists of two aromatic rings linked through a monooxime group (Fig. 1 and Table 1). The 4-bromophenyl group shows orientional disorder, with a final C10*A*–C14*A*/Br1*A* to C10*B*–C14*B*/Br1*B* ratio of 49.1 (3):50.9 (3)% and an interplanar angle of 27.6 (2)°. The molecule has an *E* configuration at the C═N bond, with a C2—C1═N1—O1 torsion angle of 172.54 (19)°, which deviates slightly from the values reported in compounds (II)–(VII) (169–174°). Other steric effects of the oxime group may influence the differences; in (I), atom O1 of the oxime group behaves as a donor, resulting in the formation of a nearly linear O1—H1···O2^{i} hydrogen bond (see Table 2). This intermolecular hydrogen bond forms a *C*(6) graph-set chain (Bernstein *et al.*, 1995), *viz*. O1—H1···O2—C2—C1—N1, running along the [001] direction with glide-plane symmetry (x, $1-y$, $\frac{1}{2}+z$) (Fig. 2). Additionally, atom N2 of the amine group acts as a donor in an intermolecular hydrogen bond with atom O1 of an adjacent oxime moiety. This interaction links the molecules into N—

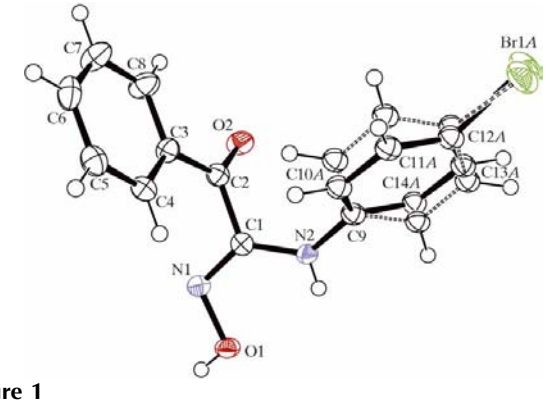


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), with displacement ellipsoids shown at the 50% probability level.

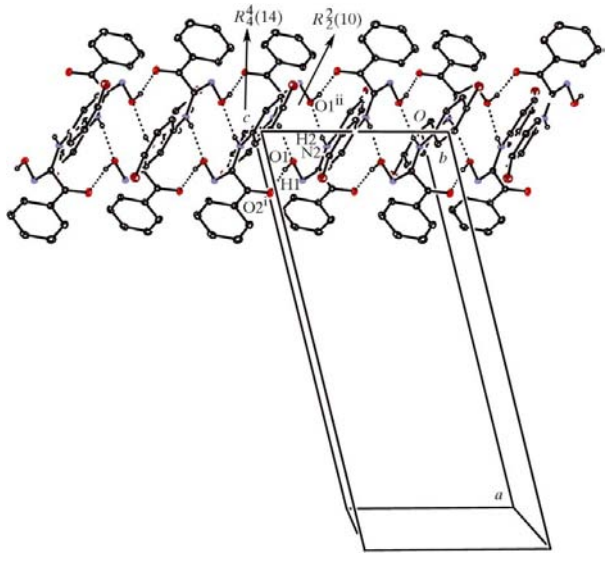


Figure 2
A chain of edge-fused $R_2^2(10)$ and $R_4^4(14)$ rings in (I), viewed down the *a* axis. For clarity, only one chain is shown, and H atoms bonded to C atoms and the minor component of the disordered benzene ring have been omitted. [Symmetry codes: (i) x, $1-y$, $\frac{1}{2}+z$; (ii) $-x$, y, $\frac{3}{2}-z$.]

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H···O hydrogen-bonded dimers that have a graph-set motif of $R_2^2(10)$ (Fig. 2). Propagation by translation of the $R_2^2(10)$ motif linking parallel $C(6)$ chains then generates a polymeric chain of edge-fused rings, with $R_2^2(10)$ and $R_4^4(14)$ motifs, along the c axis (Fig. 2).

The results obtained in this study indicate that there are significant differences between the molecular packings of (I) and of the related oxime compounds (II)–(VII). In these latter structures, the crystal packing is mainly stabilized by intermolecular hydrogen bonds in which the whole hydrogen-bonding pattern of the oxime moiety has a centre of symmetry and can be described according to graph-set notation as $R_2^2(6)$ (Bernstein *et al.*, 1995). In (I), the molecules form polymers with a chain of edge-fused rings as noted above, and there is also one C—H···π interaction involved in the molecular packing (Table 2).

There is also a weak intramolecular N—H···O hydrogen bond (N2—H2···O1; Table 2). This interaction has been reported in the related structures (II)–(VII).

Experimental

The title compound was prepared from a mixture of ω-chloroisonitrosoacetophenone (2.75 g, 0.015 mol) and 4-bromoaniline (2.58 g, 0.015 mol) in ethanol (20 ml). The mixture was stirred for 1 h and then water (20 ml) was added. The precipitated product was filtered off and recrystallized from ethanol.

Crystal data

$C_{14}H_{11}BrN_2O_2$
M_r = 319.16
Monoclinic, $C2/c$
a = 21.593 (5) Å
b = 13.328 (5) Å
c = 9.352 (5) Å
β = 104.008 (5)°
V = 2611.4 (18) Å^3
Z = 8
D_x = 1.624 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8747 reflections
θ = 1.8–28.3°
μ = 3.15 mm^{-1}
T = 293 (2) K
Prism, yellow
0.30 × 0.28 × 0.26 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.452, T_{max} = 0.495
13 425 measured reflections
3210 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 28.3°
$h = -19 \rightarrow 28$
$k = -17 \rightarrow 17$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.089
S = 0.99
3210 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

All H atoms bonded to C atoms were refined using a riding model, with C—H distances of 0.93 Å. The positions of all other H atoms were refined freely. For all H atoms, U_{iso}(H) values were set at 1.2U_{eq}(parent atom). The 4-bromophenyl group shows two-position orientational disorder, the final site-occupancy factors for C10*A*–C14*A*/Br1*A* and C10*B*–C14*B*/Br1*B* being 49.1 (3) and 50.9 (3)%, respectively.

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.426 (3)	N2—C1	1.360 (3)
N1—C1	1.288 (3)	C1—C2	1.515 (3)
N1—O1—H1	107 (3)	C1—N2—C9	125.9 (2)
C1—N1—O1	109.8 (2)	C3—C2—C1	120.0 (2)
O1—N1—C1—N2	4.3 (3)	N1—C1—C2—O2	−112.8 (3)
C9—N2—C1—N1	−158.6 (2)	N2—C1—C2—O2	56.6 (3)

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*3 is the centre of the C9/C10*A*–C14*A* benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.81 (4)	2.05 (4)	2.850 (3)	171 (4)
N2—H2···O1^{ii}	0.78 (3)	2.24 (3)	2.975 (3)	156 (3)
N2—H2···O1	0.78 (3)	2.28 (3)	2.605 (3)	106 (3)
C4—H4···*Cg*3^{ii}	0.93	2.93	3.722 (4)	143

Symmetry codes: (i) $x, -y+1, z+\frac{1}{2}$; (ii) $-x, y, -z+\frac{3}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA1096). Services for accessing these data are described at the back of the journal.

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