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2-Amino-5-iodobenzohydroxamic acid: supramolecular aggregation through two-dimensional networks of N—H—O, O—H—N and C—H—O interactions

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Vembu *et al.* $\cdot C_7H_7IN_2O_2$

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Key indicators

Single-crystal X-ray study T = 160 KMean σ (C–C) = 0.004 Å R factor = 0.037 wR factor = 0.091 Data-to-parameter ratio = 17.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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2-Amino-5-iodobenzohydroxamic acid: supramolecular aggregation through two-dimensional networks of N— $H \cdots O$, O— $H \cdots N$ and C— $H \cdots O$ interactions

In the title compound, $C_7H_7IN_2O_2$, the dihedral angle between the mean planes of the benzene ring and the hydroxamic acid group is 39.12 (14)°. The molecules are linked *via* N-H···O, O-H···N and C-H···O interactions, forming two-dimensional layers parallel to (100) in the crystal structure. There is an I···I intermolecular van der Waals contact. Received 22 September 2006 Accepted 13 October 2006

Comment

As a continuation of our study of the molecular and supramolecular architectures of hydroxamic acids, as described in our previous paper (Vembu *et al.*, 2006), the structure of the title compound, (I), is reported here. This study may serve as a forerunner to the study of the correlation between the molecular and supramolecular features of this compound and its biological activity.



The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the mean planes through atoms O10/C7/N8/O11 and atoms C1–C6 is 39.12 (14)°, which indicates a significant deviation from coplanarity of these groups, as observed in *o*-methoxy-*N*-phenylbenzohydroxamic acid [63.75 (7)°; Saad *et al.*, 2003] and 2,4-dichlorobenzohydroxamic acid [49.3 (2)°; Shang *et al.*, 2005]. In contrast, a near-coplanar orientation is observed for similar groups in 2-methoxybenzohydroxamic acid, the dihedral angles being 4.50 (16) and 10.10 (11)° for two independent molecules (Vembu *et al.*, 2006).

The crystal structure of (I) is stabilized by the interplay of $N-H\cdots O$, $C-H\cdots O$, $O-H\cdots N$, $N-H\cdots I$ and van der Waals interactions (Table 1). The intramolecular N9– H9 $A\cdots O10$ interaction generates a loop with a graph-set motif (Bernstein *et al.*, 1995; Etter, 1990) of S(6). The N9– H9 $A\cdots O10^{ii}$ and O11–H11 $\cdots N9^{iv}$ interactions (see Table 1 for symmetry codes) each link pairs of molecules into centrosymmetric dimers and the patterns can be described by graph-set motifs of $R_2^2(12)$ and $R_2^2(14)$, respectively. The N8– H8 $\cdots O10^i$ interaction joins these dimeric groups into one-dimensional ribbons which extend along the [010] direction,

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Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids.



Figure 2

The crystal structure of (I). Dashed lines indicate hydrogen bonds.

while the C6–H6···O11^v interaction cross-links the ribbons into two-dimensional layers parallel to (100). The bifurcated interactions involving N9–H9A and two symmetry-related O10 atoms as acceptors generate another ring motif, $R_2^2(4)$ (Fig. 2).

The N9-H9B···Iⁱⁱⁱ interaction can be considered as a possible hydrogen bond, as the D-H···A angle is almost linear, even though the (N)H···I distance is slightly longer than the average distance of 2.96 Å found (Palusiak *et al.*, 2005) for (N)H···I interactions in the Cambridge Structural

Database (CSD, Version of November 2003; Allen, 2002). There is one other intermolecular short contact, *viz*. I···I(-x, -y, -z) = 3.8508 (2) Å.

Experimental

The title compound, (I), was prepared by a reported method (Devocelle *et al.*, 2003; Lee *et al.*, 2005). Single crystals suitable for X-ray diffraction were obtained from an ethyl acetate–acetone solution $(1:1 \ v/v)$ by slow evaporation.

Crystal data C₇H₇IN₂O₂

 $M_r = 278.05$

Monoclinic, $P2_1/c$

a = 13.5332 (4) Å

b = 4.8500 (2) Å

c = 13.2820 (4) Å

 $\beta = 104.6614 (19)$ $V = 843.39 (5) \text{ Å}^3$ Z = 4 $D_x = 2.190 \text{ Mg m}^{-3}$ Mo K\alpha radiation $\mu = 3.76 \text{ mm}^{-1}$ T = 160 (1) KPrism, colourless $0.18 \times 0.18 \times 0.08 \text{ mm}$

Data collection

Nonius KappaCCD area-detector diffractometer φ and ω scans Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997) $T_{\min} = 0.512, T_{\max} = 0.773$

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.091$ S = 1.182458 reflections 138 parameters All H-atom parameters refined 22968 measured reflections 2458 independent reflections 2084 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.057$ $\theta_{\text{max}} = 30.0^{\circ}$

$$\begin{split} &w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 \\ &+ 0.3155P] \\ &where \ P = (F_o^2 + 2F_c^2)/3 \\ (\Delta/\sigma)_{max} = 0.001 \\ \Delta\rho_{max} = 2.18 \ e \ A^{-3} \\ \Delta\rho_{min} = -1.18 \ e \ A^{-3} \\ Extinction correction: \ SHELXL97 \\ (Sheldrick, 1997) \\ Extinction coefficient: 0.0189 (12) \end{split}$$

 Table 1

 Hydrogen-bond geometry (Å, °).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
N8-H8···O10 ⁱ	0.91 (5)	1.86 (5)	2.718 (4)	157 (4)
N9−H9A···O10	0.82(4)	2.22 (4)	2.855 (4)	135 (3)
N9-H9 A ···O10 ⁱⁱ	0.82(4)	2.39 (4)	3.018 (4)	134 (3)
N9-H9 B ···I ⁱⁱⁱ	0.87(5)	3.09 (5)	3.925 (3)	163 (4)
$O11 - H11 \cdots N9^{iv}$	0.93 (4)	1.85 (5)	2.777 (4)	173 (4)
$C6-H6\cdots O11^{v}$	0.91 (4)	2.46 (4)	3.292 (4)	152 (3)

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

All H atoms were located in a difference map and their positions and isotropic displacement parameters were refined freely. The C–H, N–H and O–H bond lengths are 0.90 (5)–1.00 (3), 0.82 (4)–0.91 (5) and 0.93 (4) Å, respectively.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO*–SMN (Otwinowski & Minor, 1997); data reduction: *DENZO*–SMN and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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