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# 2-Amino-5-iodobenzohydroxamic acid: supramolecular aggregation through two-dimensional networks of $\mathrm{N}-\mathrm{H} \ldots \mathrm{O}, \mathrm{O}-\mathrm{H} \ldots \mathrm{N}$ and C-H $\ldots$ O interactions <br> Nagarajan Vembu, Anthony Linden, Jean Lee, John G. Kelly, Kevin B. Nolan and Marc Devocelle 

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## Key indicators

Single-crystal X-ray study
$T=160 \mathrm{~K}$
Mean $\sigma(\mathrm{C}-\mathrm{C})=0.004 \AA$
$R$ factor $=0.037$
$w R$ 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 $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$, $\mathrm{O}-\mathrm{H} \cdots \mathrm{N}$ and $\mathrm{C}-\mathrm{H} \cdots \mathrm{O}$ interactions 

In the title compound, $\mathrm{C}_{7} \mathrm{H}_{7} \mathrm{IN}_{2} \mathrm{O}_{2}$, the dihedral angle between the mean planes of the benzene ring and the hydroxamic acid group is $39.12(14)^{\circ}$. The molecules are linked via $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$, $\mathrm{O}-\mathrm{H} \cdots \mathrm{N}$ and $\mathrm{C}-\mathrm{H} \cdots \mathrm{O}$ interactions, forming two-dimensional layers parallel to (100) in the crystal structure. There is an I $\cdots$ I intermolecular van der Waals contact.

## 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.

(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the mean planes through atoms O10/ $\mathrm{C} 7 / \mathrm{N} 8 / \mathrm{O} 11$ and atoms C1-C6 is 39.12 (14) ${ }^{\circ}$, which indicates a significant deviation from coplanarity of these groups, as observed in o-methoxy- $N$-phenylbenzohydroxamic acid [63.75 (7) ${ }^{\circ}$; Saad et al., 2003] and 2,4-dichlorobenzohydroxamic acid [49.3 (2) ${ }^{\circ}$; 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 $10.10(11)^{\circ}$ for two independent molecules (Vembu et al., 2006).

The crystal structure of (I) is stabilized by the interplay of $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}, \mathrm{C}-\mathrm{H} \cdots \mathrm{O}, \mathrm{O}-\mathrm{H} \cdots \mathrm{N}, \mathrm{N}-\mathrm{H} \cdots \mathrm{I}$ and van der Waals interactions (Table 1). The intramolecular N 9 $\mathrm{H} 9 \mathrm{~A} \cdots \mathrm{O} 10$ interaction generates a loop with a graph-set motif (Bernstein et al., 1995; Etter, 1990) of S(6). The N9$\mathrm{H} 9 A \cdots \mathrm{O} 10^{\mathrm{ii}}$ and $\mathrm{O} 11-\mathrm{H} 11 \cdots \mathrm{~N} 9^{\text {iv }}$ 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$\mathrm{H} 8 \cdots \mathrm{O} 10^{\mathrm{i}}$ interaction joins these dimeric groups into onedimensional 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 $\mathrm{C} 6-\mathrm{H} 6 \cdots \mathrm{O} 11^{\mathrm{v}}$ interaction cross-links the ribbons into two-dimensional layers parallel to (100). The bifurcated interactions involving $\mathrm{N} 9-\mathrm{H} 9 A$ and two symmetry-related O10 atoms as acceptors generate another ring motif, $R_{2}^{2}(4)$ (Fig. 2).

The $\mathrm{N} 9-\mathrm{H} 9 B \ldots \mathrm{I}^{\text {iii }}$ interaction can be considered as a possible hydrogen bond, as the $D-\mathrm{H} \cdots A$ angle is almost linear, even though the (N)H•I distance is slightly longer than the average distance of $2.96 \AA$ found (Palusiak et al., 2005) for (N)H $\cdots$ I interactions in the Cambridge Structural

Database (CSD, Version of November 2003; Allen, 2002).
There is one other intermolecular short contact, viz. $\mathrm{I} \cdots \mathrm{I}(-x$, $-y,-z)=3.8508(2) \AA$.

## 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 $\mathrm{v} / \mathrm{v}$ ) by slow evaporation.

## Crystal data

$\mathrm{C}_{7} \mathrm{H}_{7} \mathrm{IN}_{2} \mathrm{O}_{2}$
$M_{r}=278.05$
Monoclinic, $P 2_{1} / c$
$a=13.5332$ (4) $\AA$
$b=4.8500$ (2) A
$c=13.2820(4) \AA$
$\beta=104.6614$ (19) ${ }^{\circ}$
$V=843.39(5) \AA^{3}$

## $Z=4$

$D_{x}=2.190 \mathrm{Mg} \mathrm{m}^{-3}$
Mo $K \alpha$ radiation
$\mu=3.76 \mathrm{~mm}^{-1}$
$T=160$ (1) K
Prism, colourless
$0.18 \times 0.18 \times 0.08 \mathrm{~mm}$

## Data collection

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

## Refinement

Refinement on $F^{2}$
$w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}^{2}\right)+(0.0534 P)^{2}\right.$
$+0.3155 P$ ]
where $P=\left(F_{\mathrm{o}}{ }^{2}+2 F_{\mathrm{c}}{ }^{2}\right) / 3$
$(\Delta / \sigma)_{\text {max }}=0.001$
$\Delta \rho_{\text {max }}=2.18$ e $\AA^{-3}$
$\Delta \rho_{\min }=-1.18$ e $\AA^{-3}$
Extinction correction: SHELXL97
(Sheldrick, 1997)
Extinction coefficient: 0.0189 (12)

Table 1
Hydrogen-bond geometry ( $\mathrm{A},{ }^{\circ}$ ).

| $D-\mathrm{H} \cdots A$ | D-H | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :---: | :---: | :---: | :---: | :---: |
| N8-H8 $\cdots$ O10 ${ }^{\text {i }}$ | 0.91 (5) | 1.86 (5) | 2.718 (4) | 157 (4) |
| N9-H9A $\cdots$ O10 | 0.82 (4) | 2.22 (4) | 2.855 (4) | 135 (3) |
| $\mathrm{N} 9-\mathrm{H} 9 \mathrm{~A} \cdots \mathrm{O} 10^{\text {ii }}$ | 0.82 (4) | 2.39 (4) | 3.018 (4) | 134 (3) |
| N9-H9B $\ldots$ Iii | 0.87 (5) | 3.09 (5) | 3.925 (3) | 163 (4) |
| $\mathrm{O} 11-\mathrm{H} 11 \cdots \mathrm{~N} 9^{\mathrm{iv}}$ | 0.93 (4) | 1.85 (5) | 2.777 (4) | 173 (4) |
| C6-H6 $\cdots{ }^{\text {O }}{ }^{\text {v }}$ | 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) $-x+1,-y+1,-z+1 ;(\mathrm{v})-x+1, y-\frac{1}{2},-z+\frac{1}{2}$.

All H atoms were located in a difference map and their positions and isotropic displacement parameters were refined freely. The $\mathrm{C}-$ $\mathrm{H}, \mathrm{N}-\mathrm{H}$ and $\mathrm{O}-\mathrm{H}$ bond lengths are 0.90 (5)-1.00 (3), 0.82 (4)0.91 (5) and 0.93 (4) A. 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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