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Marcin Cymborowski, Maksymilian Chruszcz, Zbigniew Dauter and Wlodek Minor

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3-Iodo-L-tyrosine hemihydrate

Marcin Cymborowski,^a
 Maksymilian Chruszcz,^a
 Zbigniew Dauter^b and Wlodek
 Minor^{a*}

^aUniversity of Virginia, Department of Molecular Physiology and Biological Physics, 1340 Jefferson Park Avenue, Charlottesville, VA 22908, USA, and ^bSynchrotron Radiation Research Section, MCL, National Cancer Institute, Argonne National Laboratory, Argonne, IL 60439, USA

Correspondence e-mail:
 wlodek@iwonka.med.virginia.edu

Key indicators

Single-crystal X-ray study
 $T = 109\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.021
 wR factor = 0.055
 Data-to-parameter ratio = 34.6

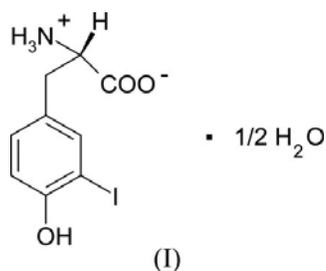
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound [systematic name: (*S*)-2-ammonio-3-(3-iodo-4-hydroxyphenyl)propanoate hemihydrate], $\text{C}_9\text{H}_{10}\text{INO}_3 \cdot 0.5\text{H}_2\text{O}$, is stabilized by ionic interactions and a hydrogen-bond network. The hydrogen bonds, involving a water molecule located on a twofold axis, form bridged layers of 3-iodo-L-tyrosine molecules.

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Comment

3-Iodo-L-tyrosine, of which the hemihydrate structure, (I) (Fig. 1), is reported here, and 3,5-diiodo-L-tyrosine are precursors in the biosynthesis of thyroid hormones. These hormones are very important for the growth and development of vertebrate organisms. Moreover, 3-iodo-L-tyrosine was found to be an inhibitor of tyrosine hydroxylase (Fitzpatrick, 1988, 1991), which catalyzes the first step in the biosynthesis of catecholamine neurotransmitters (Kaufman & Kaufman, 1985). 3-Iodotyrosine is also used in organic synthesis as a starting compound from which numerous tyrosine derivatives are obtained (Palmer *et al.*, 2005; Skaff *et al.* 2005; Knör *et al.*, 2006).



In the Cambridge Structural Database (November 2006 release; Allen, 2002), two structures contain the 3-iodo-L-tyrosine system, namely 3-iodo-L-tyrosine methanol solvate (Okabe & Suga, 1995) and aqua(2,2'-bipyridine)(3-iodo-L-tyrosinato)copper(II) nitrate methanol solvate monohydrate (Zhang *et al.*, 1997). The 3-iodo-L-tyrosine units in these crystal structures have different conformations. The torsion angles $\text{C1}-\text{C7}-\text{C8}-\text{C9}$ and $\text{C8}-\text{C7}-\text{C1}-\text{C2}$ in (I) are -51.5 (3) and 109.7 (2) $^\circ$, respectively, while the equivalent angles in the structure reported by Okabe & Suga (1995) are 66.1 and 86.5 $^\circ$, respectively, and in the structure reported by Zhang *et al.* (1997), these values are 53.4 and -91.4 $^\circ$, respectively.

The water molecule of (I) is located on a twofold axes and not only is involved in hydrogen bonding but also has two symmetry-equivalent contacts of 3.281 (1) \AA with I atoms. The hydrogen bonds formed by the water molecules extend along

[100] (Fig. 2), while the hydrogen bonds between the 3-iodo-L-tyrosine molecules extend in the [010] direction (Fig. 3).

Experimental

3-Iodo-L-tyrosine was purchased from Aldrich. The crystal used for X-ray diffraction was grown by slow evaporation of a solution of (I) in methanol at 293 K.

Crystal data

$C_9H_{10}INO_3 \cdot 0.5H_2O$	$V = 1068.1 (2) \text{ \AA}^3$
$M_r = 316.09$	$Z = 4$
Monoclinic, $C2$	Mo $K\alpha$ radiation
$a = 18.789 (1) \text{ \AA}$	$\mu = 2.98 \text{ mm}^{-1}$
$b = 5.908 (1) \text{ \AA}$	$T = 109 (2) \text{ K}$
$c = 9.624 (1) \text{ \AA}$	$0.48 \times 0.04 \times 0.04 \text{ mm}$
$\beta = 91.04 (1)^\circ$	

Data collection

Rigaku R-AXIS RAPID diffractometer	24658 measured reflections
Absorption correction: multi-scan (Otwinowski <i>et al.</i> , 2003)	4677 independent reflections
$T_{\min} = 0.87, T_{\max} = 0.89$	4517 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$	$\Delta\rho_{\text{max}} = 2.30 \text{ e \AA}^{-3}$
$wR(F^2) = 0.055$	$\Delta\rho_{\text{min}} = -0.67 \text{ e \AA}^{-3}$
$S = 1.08$	Absolute structure: Flack (1983),
4677 reflections	with 2143 Friedel pairs
135 parameters	Flack parameter: $-0.01 (2)$
H atoms treated by a mixture of independent and constrained refinement	

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H2 \cdots O3^i$	0.89	1.86	2.729 (2)	165
$N1-H3 \cdots O1^{ii}$	0.89	2.29	2.920 (2)	128
$N1-H4 \cdots O2^{iii}$	0.89	2.08	2.880 (2)	150
$O1-H1 \cdots O2^{ii}$	0.82	1.80	2.585 (2)	158
$O4-H11 \cdots O3^{iv}$	0.98 (3)	1.87 (3)	2.823 (2)	165 (4)

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

The H atoms of the 3-iodo-L-tyrosine molecule were placed in calculated positions and refined using a riding-model approximation, with C–H bond lengths of 0.93 Å in the case of the benzene ring, 0.97 Å in the case of the CH₂ group and 0.98 Å for the CH. The N–H and O–H distances were constrained to 0.89 and 0.82 Å, respectively. The H atom of the water molecule was located in a difference Fourier map and the O–H bond length and displacement parameter were restrained. $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{N,O})$. The highest residual electron density peak is located 0.67 Å from atom I1.

Data collection: *HKL-2000* (Otwinowski & Minor, 1997); cell refinement: *HKL-2000*; data reduction: *HKL-2000*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990) and *HKL-3000SM* (Minor *et al.*, 2006); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *HKL-3000SM*; molecular graphics: *HKL-3000SM*, *Mercury* (Macrae *et al.*, 2006), *ORTEPIII* (Burnett &

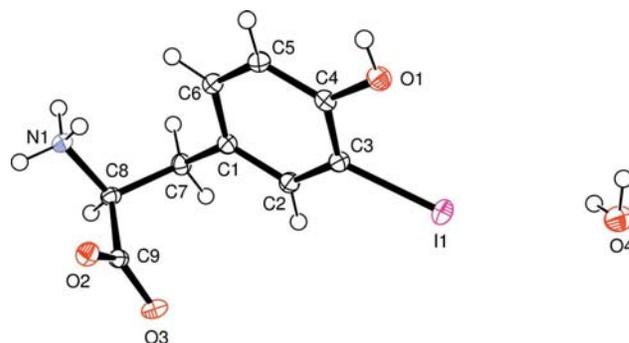


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radius.

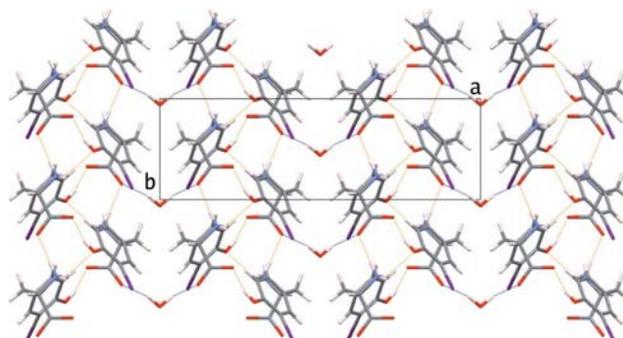


Figure 2
The crystal packing of compound (I), viewed along [001]. Hydrogen bonds formed by water molecules are shown in blue. Hydrogen bonds in which water molecules are not involved are shown in orange.

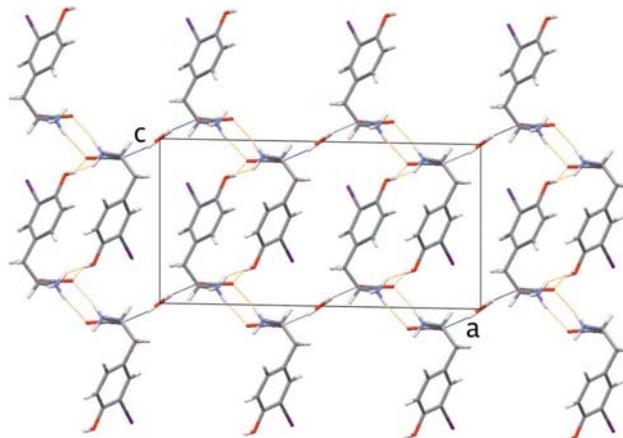


Figure 3
The crystal packing of compound (I), viewed along [010]. Hydrogen bonds formed by water molecules are shown in blue. Hydrogen bonds in which water molecules are not involved are shown in orange.

Johnson, 1996) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *HKL-3000SM*.

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