

2,3-Dihydroxybenzaldehyde thiosemicarbazone hemihydrate

Abdurazag T. Swesi,^a Yang Farina,^a Mohammad Kassim^a and Seik Weng Ng^{b*}^aSchool of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, Bangi 43600, Malaysia, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

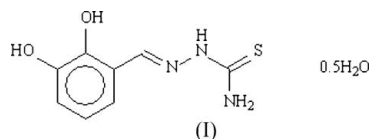
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.057
 wR factor = 0.129
Data-to-parameter ratio = 11.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the Schiff base in the crystal structure of the title compound, $\text{C}_8\text{H}_9\text{N}_3\text{O}_2\text{S}\cdot 0.5\text{H}_2\text{O}$, interacts with symmetry-equivalent molecules and with the water molecule, which lies on a twofold rotation axis, to give a three-dimensional hydrogen-bonded network structure.

Comment

Salicylaldehyde condenses with thiosemicarbazide to yield salicylaldehyde thiosemicarbazone (Chattopadhyay *et al.*, 1988), a ligand that can be used in its deprotonated form to chelate metal ions. A similar reaction with 2,3-dihydroxybenzaldehyde furnishes the corresponding Schiff base, 2,3-dihydroxybenzaldehyde thiosemicarbazone, which possesses two hydroxy substituents on the aromatic ring. 2,3-Dihydroxybenzaldehyde thiosemicarbazone was reported many years ago (Bernstein *et al.*, 1951; Donovick *et al.*, 1950), and it exhibits useful activity as a chemotherapeutic agent. The compound crystallizes as the title hemihydrate, (I) (Fig. 1), with the water molecule lying on a twofold rotation axis.



Bond lengths and angles in the molecule are normal and are similar to those found in salicylaldehyde thiosemicarbazone itself. However, owing to an additional hydroxy group and the presence of the water molecule, the crystal structure exhibits extensive hydrogen bonding (Table 1), which gives rise to a three-dimensional network structure.

Experimental

An ethanol (25 ml) solution of 2,3-dihydroxybenzaldehyde (1.38 g, 10 mmol) was added to an ethanol (10 ml) solution of thiosemicarbazide (0.94 g, 10 mmol). The mixture was heated for 3 h. The product was recrystallized from methanol to afford needle-shaped crystals in 70% yield (m.p. 479–581 K).

Crystal data

$\text{C}_8\text{H}_9\text{N}_3\text{O}_2\text{S}\cdot 0.5\text{H}_2\text{O}$
 $M_r = 220.25$
Monoclinic, $C2/c$
 $a = 21.546$ (2) Å
 $b = 11.658$ (1) Å
 $c = 8.190$ (1) Å
 $\beta = 105.923$ (1)°
 $V = 1978.3$ (3) Å³

$Z = 8$
 $D_x = 1.479$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.31$ mm⁻¹
 $T = 293$ (2) K
Needle, yellow
 $0.50 \times 0.20 \times 0.10$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
 ω and φ scans
 Absorption correction: none
 5025 measured reflections

1740 independent reflections
 1510 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.129$
 $S = 1.19$
 1740 reflections
 150 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 2.2284P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.24 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{Å}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1O...S1 ⁱ	0.84 (1)	2.50 (2)	3.162 (2)	137 (3)
O2—H2O...O1W	0.85 (1)	1.95 (1)	2.788 (3)	173 (4)
N1—H1N1...S1 ⁱⁱ	0.85 (1)	2.56 (1)	3.390 (3)	167 (3)
N1—H1N2...N3	0.85 (1)	2.28 (4)	2.629 (3)	105 (3)
N2—H2N...O1 ⁱ	0.85 (1)	2.22 (2)	3.014 (3)	155 (3)
O1W—H1W...S1 ⁱⁱⁱ	0.85 (1)	2.52 (1)	3.345 (3)	163 (2)

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

The water and amino H atoms were located in a difference Fourier map and were refined with distance restraints of O—H = N—H = 0.85 (1) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O,N})$. The distance between the H atoms of the water molecule, which lies on a twofold rotation axis, was restrained to be 1.39 (1) Å. All other H atoms were positioned geometrically and were included in the refinement in the riding-model approximation, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

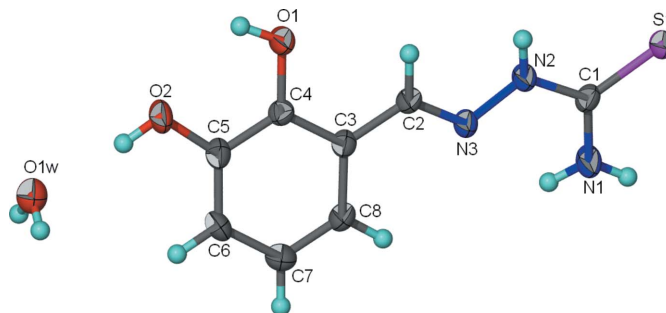


Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

We are grateful to IRPA 09–02–02–0133, Universiti Kebangsaan Malaysia and the University of Malaya for supporting this work.

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Bernstein, J., Yale, H. L., Losee, K., Holsing, M., Martins, J. & Lott, W. A. (1951). *J. Am. Chem. Soc.* **73**, 906–12.
 Bruker (2003). *SAINTE* (Version 6.45a) and *SMART* (Version 6.45a). Bruker AXS Inc., Madison, Wisconsin, USA.
 Chattopadhyay, D., Mazumdar, S. K., Banerjee, T., Ghosh, S. & Mak, T. C. W. (1988). *Acta Cryst.* **C44**, 1025–1028.
 Donovan, R., Pansy, F., Stryker, G. & Bernstein, J. (1950). *J. Bacteriol.* **59**, 667–674.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.