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Key indicators

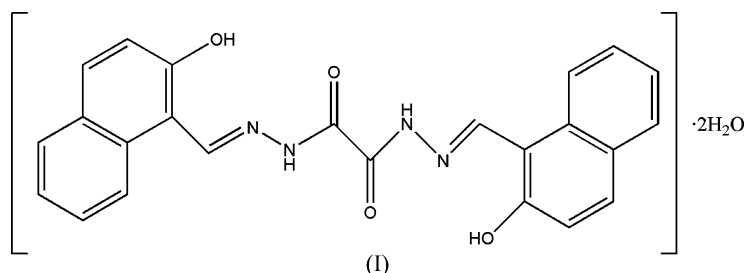
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.053
 wR factor = 0.135
Data-to-parameter ratio = 12.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(2-hydroxy-1-naphthaldehyde) oxaloyl-
dihydrazone dihydrate

The title compound, $\text{C}_{24}\text{H}_{18}\text{N}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$, has a centre of symmetry at the mid-point of the central C—C bond. The uncoordinated water links molecules along the c axis *via* O—H \cdots O hydrogen bonds. N—H \cdots O hydrogen bonds complete a two-dimensional hydrogen-bond network. π — π interactions exist between the imino group and the aromatic ring.

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Comment

Much attention has been paid to hydrogen bonds and π — π stacking interactions due to their important roles in the control of molecular arrangement in the crystalline state and the properties of materials (Jones *et al.*, 2001). Intramolecular hydrogen bonds, and phenol—imine or keto—amine forms, are generally observed in 2-hydroxyaldimine Schiff bases.



The molecule of (I) (Fig. 1) has a centre of symmetry at the mid-point of the central C—C bond. The molecule is planar, the largest deviation from the mean plane being 0.038 Å (C5). The molecule adopts the phenol—imine form and displays strong intramolecular O—H \cdots N hydrogen bonds (Hökelek *et al.*, 2004), with an O \cdots N distance of 2.554 (9) Å (Table 1). The organic molecule and water molecules are linked by O—H \cdots O and N—H \cdots O hydrogen bonds into a two-dimensional network (Table 1 and Fig. 2). π — π interactions exist between the aromatic ring and the non-cyclic π -system of an imine group: C3 \cdots C11ⁱ = 3.562 (4), C4 \cdots C11ⁱ = 3.468 (4) and C5 \cdots C11ⁱ = 3.469 (4) Å [symmetry code: (i) $x, 1 + y, z$] (Fig. 3).

Experimental

The title compound was synthesised by a modification of the procedure described by Lal *et al.* (2002). A hot aqueous solution (10 ml) of oxaloyldihydrazine (0.12 g, 1.00 mmol) was added to a hot ethanol solution (10 ml) of manganese(II) acetate tetrahydrate (0.25 g, 1.00 mmol) with stirring. The mixture was refluxed for 6 h with the addition of 4 drops of 0.2 M NaOH. A hot ethanol solution (10 ml) of 2-hydroxy-1-naphthaldehyde (0.34 g, 2.00 mmol) was then added dropwise. The mixture was refluxed for 6 h. The volume of the solution was reduced to 10 ml. The brown—orange precipitate of the

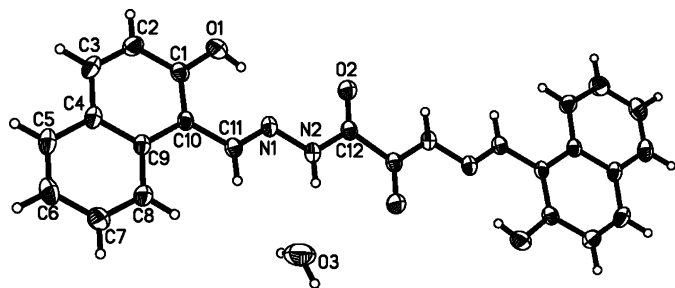


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operator $(-x, -y, -z)$.

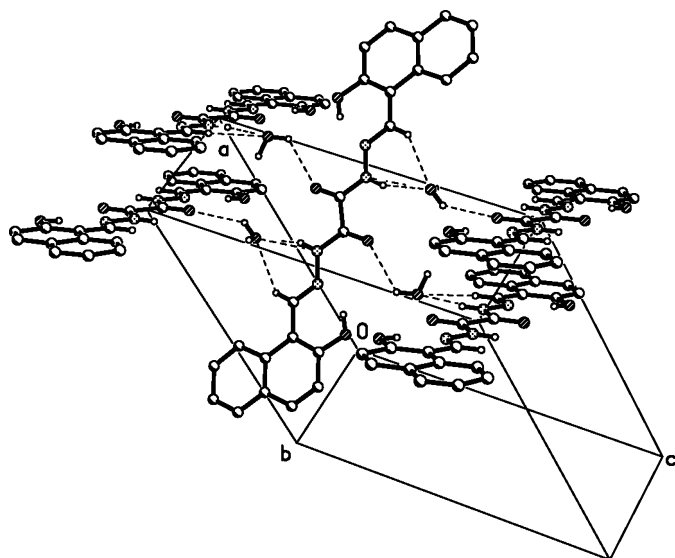


Figure 2
One-dimensional chains generated by hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

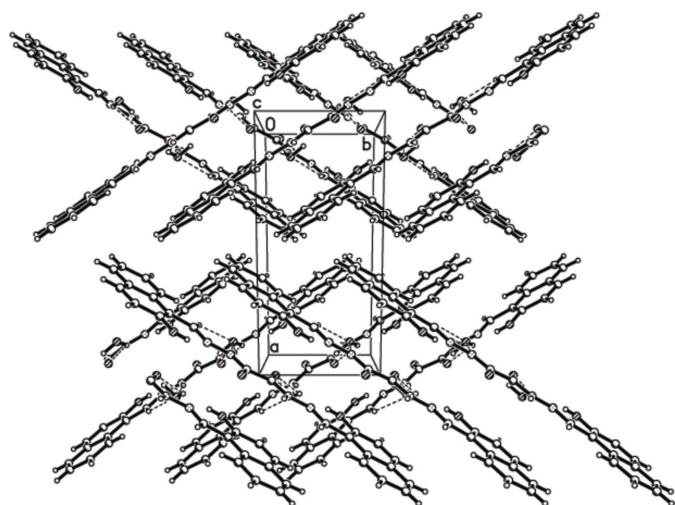


Figure 3
The molecular packing, viewed along the c axis (dashed lines represent hydrogen bonds).

manganese complex of the title compound (Lal *et al.*, 2002) was filtered off while hot, and dried under vacuum. The dried precipitate (0.05 g) and pyridine (20 ml) were added to a 25 ml Teflon-lined stainless steel acid-digestion vessel and heated at 393 K for 1 d under autogenous pressure. After slow cooling to room temperature, yellow needle-shaped crystals of the title compound were obtained (yield 37%). Analysis required for $C_{24}H_{22}N_4O_6$: C 62.33, H 4.80, N 12.1%; found: C 62.36, H 4.78, N 12.1%.

Crystal data

$C_{24}H_{22}N_4O_6 \cdot 2H_2O$
 $M_r = 462.46$
 Monoclinic, $P2_1/c$
 $a = 13.678$ (4) Å
 $b = 5.9250$ (18) Å
 $c = 14.883$ (5) Å
 $\beta = 114.983$ (5)°
 $V = 1093.3$ (6) Å³

$Z = 2$
 $D_x = 1.405$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ (2) K
 Block cut from needle, yellow
 $0.20 \times 0.18 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.822$, $T_{max} = 1.000$

5069 measured reflections
 1918 independent reflections
 1149 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.038$
 $\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.135$
 $S = 1.02$
 1918 reflections
 155 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2643P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.30$ e Å⁻³
 $\Delta\rho_{min} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2 \cdots O3$	0.86	2.10	2.935 (9)	162
$O1-H1 \cdots N1$	0.82	1.83	2.554 (9)	146
$O3-H3WB \cdots O2^{ii}$	0.86	2.40	2.933 (9)	121

Symmetry code: (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

All H atoms were located in a difference Fourier map and refined as riding, with $C-H = 0.93$ Å, $N-H = 0.86$ Å and $O-H = 0.82-0.89$ Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N,O)$.

Data collection: APEX2 (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2003); software used to prepare material for publication: SHELXTL.

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