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Key indicators

Single-crystal X-ray study
 T = 294 K
 Mean $\sigma(C-C)$ = 0.003 Å
 R factor = 0.040
 wR factor = 0.114
 Data-to-parameter ratio = 14.3

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

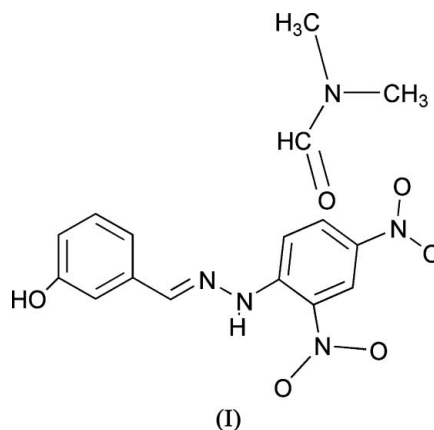
3-[(2,4-Dinitrophenyl)hydrazonomethyl]phenol dimethylformamide solvate

In the title compound, $C_{13}H_{10}N_4O_5 \cdot C_3H_7NO$, the Schiff base is approximately planar. Molecules are connected *via* weak intermolecular O—H...O, N—H...O and C—H...O hydrogen bonds.

Received 23 February 2006
 Accepted 25 February 2006

Comment

Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands (Yu *et al.*, 2005; Deng *et al.*, 2005; Jing, Fan *et al.*, 2005*a,b*; Jing, Wang *et al.*, 2005; Jing *et al.*, 2006), we report here the synthesis and crystal structure of the title compound, (I).



The asymmetric unit of (I) comprises one molecule each of 3-[(2,4-dinitrophenyl)hydrazonomethyl]phenol and dimethyl-

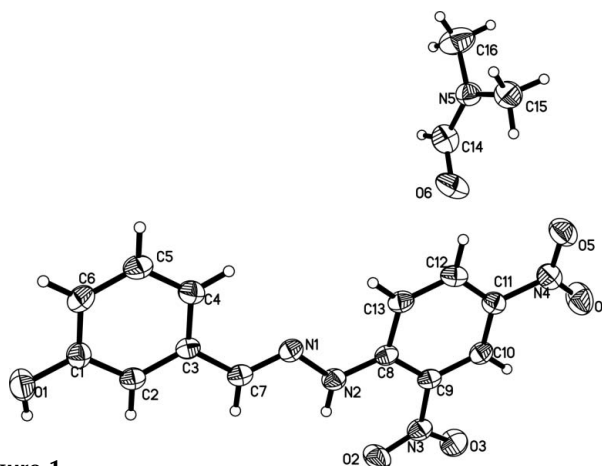


Figure 1
 The structure of (I), showing 30% probability displacement ellipsoids.

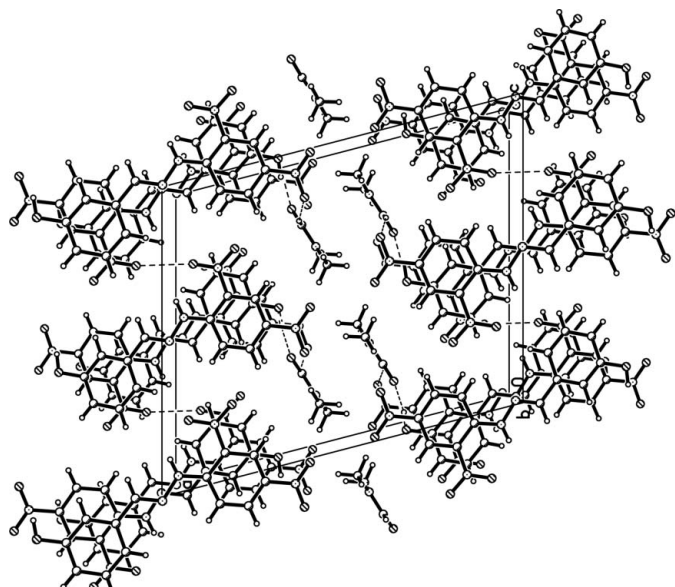


Figure 2

The packing of (I), viewed down the *b* axis, showing intermolecular hydrogen bonds (dashed lines).

formamide (Fig. 1). The Schiff base is approximately planar; the central chromophore (C8–C13/N1–N4) and the 3-hydroxybenzaldehyde group (C1–C7/O1) are each planar, with r.m.s. deviations of 0.020 and 0.008 Å, respectively. The dihedral angle between these planes is 2.44 (3)°.

An intramolecular N–H···O hydrogen bond stabilizes the molecular structure, while intermolecular O–H···O, N–H···O and C–H···O hydrogen bonds stabilize the crystal structure (Table 2 and Fig. 2). Inversion-related Schiff base molecules are linked *via* O–H···O and C–H···O hydrogen bonds involving the dimethylformamide molecules. A short O2···O2(−*x*, *y*, $\frac{1}{2}$ − *z*) contact of 2.750 (2) Å is also observed in the crystal structure.

Experimental

An anhydrous ethanol solution (50 ml) of 3-hydroxybenzaldehyde (1.22 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 2,4-dinitrophenylhydrazine (2.03 g, 10 mmol), and the mixture was stirred at 330 K for 6 h under N₂, whereupon a red precipitate appeared. The product was isolated, recrystallized from *N,N*-dimethylformamide and then dried *in vacuo* to give pure compound (I) in 89% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an *N,N*-dimethylformamide solution.

Crystal data

C₁₃H₁₀N₄O₅·C₃H₇NO
M_r = 375.35
 Monoclinic, *P*2₁/*c*
a = 17.701 (4) Å
b = 7.0661 (18) Å
c = 14.695 (4) Å
 β = 104.607 (4)°
V = 1778.6 (8) Å³
Z = 4

D_x = 1.402 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 2708 reflections
 θ = 2.8–25.7°
 μ = 0.11 mm^{−1}
T = 294 (2) K
 Block, red
 0.40 × 0.32 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 T_{\min} = 0.943, T_{\max} = 0.978
 9686 measured reflections

3634 independent reflections
 2221 reflections with $I > 2\sigma(I)$
 R_{int} = 0.030
 θ_{max} = 26.3°
 h = −17 → 22
 k = −8 → 8
 l = −13 → 18

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.040
 $wR(F^2)$ = 0.114
 S = 1.00
 3634 reflections
 255 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.3148P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}}$ = 0.001
 $\Delta\rho_{\text{max}}$ = 0.16 e Å^{−3}
 $\Delta\rho_{\text{min}}$ = −0.14 e Å^{−3}
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.0309 (18)

Table 1

Selected geometric parameters (Å, °).

N1–C7	1.271 (2)	C3–C4	1.394 (2)
N1–N2	1.3730 (19)	C3–C7	1.457 (2)
N2–C8	1.345 (2)	C8–C13	1.413 (2)
N3–O2	1.2276 (18)	C8–C9	1.419 (2)
N3–C9	1.445 (2)		
C7–N1–N2	116.42 (15)	N1–C7–C3	121.61 (16)
C8–N2–N1	119.53 (15)	N2–C8–C13	120.27 (15)
O2–N3–C9	119.34 (14)	N2–C8–C9	123.72 (15)
C4–C3–C7	122.23 (16)	C8–C9–N3	121.77 (15)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1–H1···O6 ⁱ	0.92 (3)	1.73 (3)	2.634 (2)	168 (2)
N2–H2A···O2 ⁱⁱ	0.855 (18)	2.586 (18)	3.385 (2)	156.1 (15)
N2–H2A···O2	0.855 (18)	2.009 (18)	2.624 (2)	128.1 (15)
C14–H14···O5 ⁱⁱⁱ	0.93	2.39	3.271 (3)	158

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

H atoms attached to N and O atoms were located in a difference Fourier map and refined freely. H atoms bonded to C atoms were included in calculated positions [C–H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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