

1-[(4-Formylphenyl)hydrazono]naphthalen-2(1*H*)-one—2-naphthol (1/1)Xin-Gang Liu,^a Ya-Qing Feng,^{a*}
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Key indicators

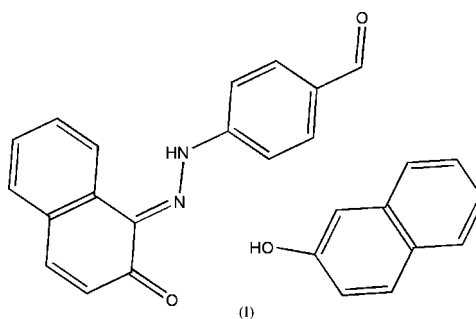
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
Disorder in main residue
R factor = 0.055
wR factor = 0.191
Data-to-parameter ratio = 13.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}_2 \cdot \text{C}_{10}\text{H}_8\text{O}$, shows that the molecules are effectively planar in the solid state and the compound exists in the hydrazone form and not the azo form. The hydrazone H atom and carbonyl O atom are linked by an intramolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bond. The 2-naphthol molecules are disordered over inversion centres, the asymmetric unit containing half each of two such molecules. The packing can be described as a polymeric arrangement of molecules linked through $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.

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Comment

Azo compounds are among the largest group of dyes, with over 1000 compounds on commercial offer. Azo dyes have been developed for colouring fibres, natural and synthetic, and for the coloration of solvents and a wide range of non-textile substrates (Sudesh Kumar & Neckers, 1989). A large fraction of such dyes are derivatives of arylazonaphthols, possessing the azo chromophore. Other substituents known as auxochromes, which augment the chromophore, are methoxy, hydroxyl and amino groups. The position of the azo and hydroxyl groups in these molecules brings into play the azo-hydrazone equilibrium, which has been the subject of intensive investigation in recent years (Antonov *et al.*, 1998, 1999).



1-[(4-Formylphenyl)hydrazono]naphthalen-2(1*H*)-one was synthesized and the structure of the 1:1 adduct, (I), with 2-naphthol determined (Fig. 1). It shows that the naphthalenone molecule is approximately planar in the solid state, with a mean deviation of $0.031(4) \text{ \AA}$, and exists in the hydrazone form and not the azo form. The co-crystallized 2-naphthol molecule shows twofold disorder about an inversion centre. The hydrazone H atom and naphthalenone carbonyl O atom are linked by an intramolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bond. The packing can be described as a polymeric arrangement of molecules linked through $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds involving 2-naphthol, as shown in Fig. 2 and Table 1. Hydroxy atom O3 is hydrogen bonded to atom O1 of the symmetry-related molecule at $(1-x, 2-y, 1-z)$. A comparatively weak $\pi-\pi$

interaction between the naphthalene and benzene rings, with an inter-planar separation of 3.608 (4) Å, is also observed in the crystal structure.

Experimental

Sodium nitrite (5 mmol) in water (10 ml) was added dropwise to 4-aminobenzaldehyde (4 mmol) dispersed in concentrated HCl (2 ml) at 273–278 K and the mixture stirred for 30 min. The resulting solution was added dropwise to sodium carbonate (10 mmol) and 2-naphthol (5 mmol) in water (40 ml) and stirred for 30 min. The red precipitate of (I) was filtered off and washed with water to give a crude product. This was chromatographed through a silica-gel column (100–200 mesh) using petroleum ether–ethyl acetate (5:1 v/v) as eluent to give 1-[(4-formylphenyl)hydrazone]naphthalen-2(1H)-one. ¹H NMR (CDCl₃): δ 6.7–8.47 (d, 10H), 10.00 (s 1H), 16.23 (s, 1H). Recrystallization of the crude product from acetone over a period of 8 d at ambient temperature gave red single crystals of (I) suitable for X-ray analysis.

Crystal data

C ₁₇ H ₁₂ N ₂ O ₂ ·C ₁₀ H ₈ O	Z = 2
M _r = 420.45	D _x = 1.346 Mg m ⁻³
Triclinic, P $\bar{1}$	Mo K α radiation
a = 7.9088 (12) Å	Cell parameters from 1881 reflections
b = 10.7469 (16) Å	θ = 2.6–26.2°
c = 12.3067 (19) Å	μ = 0.09 mm ⁻¹
α = 86.421 (3)°	T = 293 (2) K
β = 89.184 (3)°	Block, red
γ = 83.715 (3)°	0.22 × 0.20 × 0.18 mm
V = 1037.7 (3) Å ³	

Data collection

Bruker SMART CCD area-detector diffractometer	4168 independent reflections
φ and ω scans	2436 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	R _{int} = 0.017
T _{min} = 0.976, T _{max} = 0.984	θ_{max} = 26.4°
5880 measured reflections	h = -9 → 9
	k = -9 → 13
	l = -15 → 14

Refinement

Refinement on F ²	$w = 1/[\sigma^2(F_o^2) + (0.0904P)^2 + 0.3211P]$
R[F ² > 2σ(F ²)] = 0.055	where $P = (F_o^2 + 2F_c^2)/3$
wR(F ²) = 0.191	(Δ/σ) _{max} < 0.001
S = 1.01	$\Delta\rho_{max} = 0.59 \text{ e \AA}^{-3}$
4168 reflections	$\Delta\rho_{min} = -0.26 \text{ e \AA}^{-3}$
303 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2...O1	0.91 (3)	1.86 (3)	2.582 (3)	135 (3)
O3—H3A...O1 ⁱ	0.82	1.94	2.737 (4)	164
O4—H4A...O1	0.85	2.34	3.171 (8)	166

Symmetry code: (i) -x + 1, -y + 2, -z + 1.

The N-bound H atom was located in a difference Fourier map and its coordinates and isotropic displacement parameter were freely refined. O-bound H atoms were located in a difference Fourier map and then refined as riding, with O—H = 0.82–0.85 Å. Other H atoms were positioned geometrically, with C—H = 0.93 Å, and refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The 2-naphthol molecule

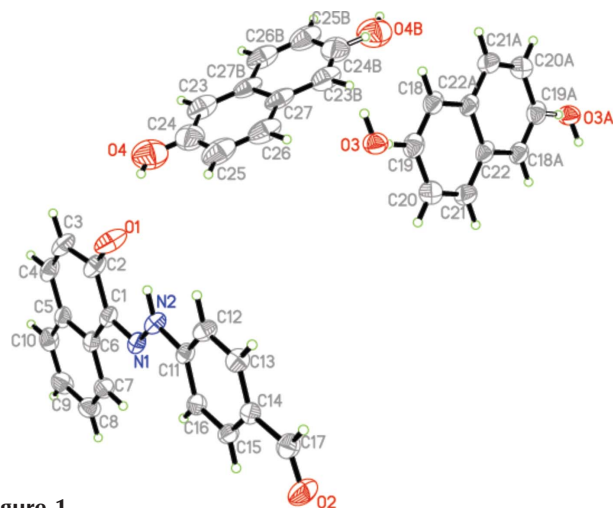


Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (A) 2 - x, 2 - y, 2 - z; (B) 2 - x, 2 - y, 1 - z.]

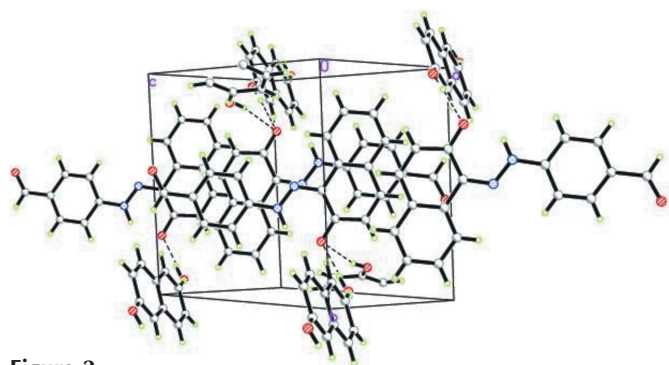


Figure 2

The packing of (I), showing the hydrogen-bonding interactions as dashed lines.

showed a twofold disorder about an inversion centre. Atoms O3 and O4 were also disordered and the ratio of the occupancies was 0.5 (1):0.5 (1).

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); 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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