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2-Cyano-*N*-(2-hydroxyphenyl)acetamide

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.131
Data-to-parameter ratio = 10.0

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, $C_9H_8O_2N_2$, is described. The crystal structure is stabilized by a hydrogen-bonded network along the [101] and [301] directions.

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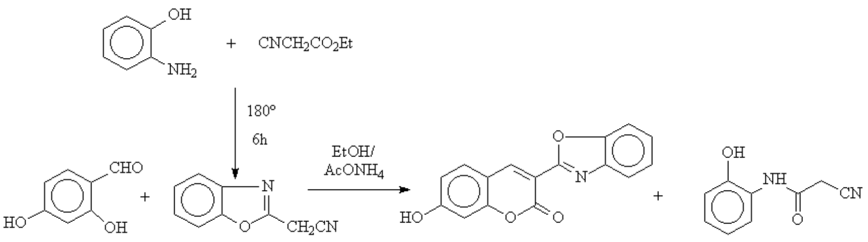
Comment

Coumarins are a family of compounds that has been studied extensively due to its practical applications; optical brightness, laser dyes, sensitizers in phototherapy, *etc*., are some of the uses of this class of compound (Machado & Miranda, 2001).



(1) (2) (3) (4)

The title compound, (4), was obtained during the synthesis of the 3-substituted 7-hydroxycoumarin, (3), from 2,4-dihydroxybenzaldeyde, (1), and benzoxazol-2-ylacetonitrile, (2), as outlined in the Scheme. It is possible that the final mixture contained some of the unreacted precursor (2), this being hydrolysed during the second step of the reaction or during work-up (Luan *et al.*, 2002; Elnagdi *et al.*, 1997). An *ORTEP*-3 (Farrugia, 1997) drawing of (4) is shown in Fig. 1, and selected geometric parameters are presented in Table 1. The 2-hydroxyphenyl and 2-cyanoacetamide moieties are planar and the angle between these systems is 7.48 (18)°.

In the crystal structure, the molecule is linked by two kinds of hydrogen bonds, *viz*. two-center and three-center bonding (Table 2). The two-center hydrogen bond $O1—H1\cdots N2^{ii}$ links the molecules in an infinite zigzag in the [301] direction (Fig. 2*a*; for symmetry codes see Table 2). The three-center hydrogen bond involves the intramolecular interaction $N1—H\cdots O1$ and the intermolecular interaction $N1—H\cdots O2^{i}$, producing an infinite zigzag in the [101] direction (Fig. 2*b*). Atom H is in the plane formed by atoms N1, O1 and O2, indicating the presence of the three-center hydrogen bond (Jeffrey & Maluszynska, 1982). The $N1\cdots O1$ distance of 2.627 (3) Å is clearly indicative of strong intramolecular hydrogen bonding; this distance is significantly shorter than the sum of the van der Waals radii for oxygen and nitrogen (3.07 Å; Bondi, 1964). This intramolecular $N1\cdots O1$ distance is comparable to those observed in 2-[(2-iodophenyl)iminomethyl]phenol [2.624 (5) Å], *N*,*N*′-bis(*p*-chlorosalicylideneamine)-1,2-diaminobenzene [2.615 (6) Å] and 2,2′-azino-

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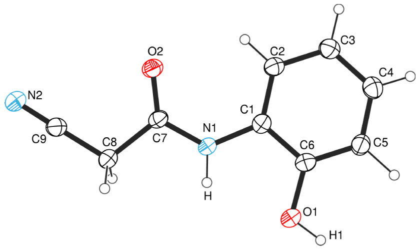
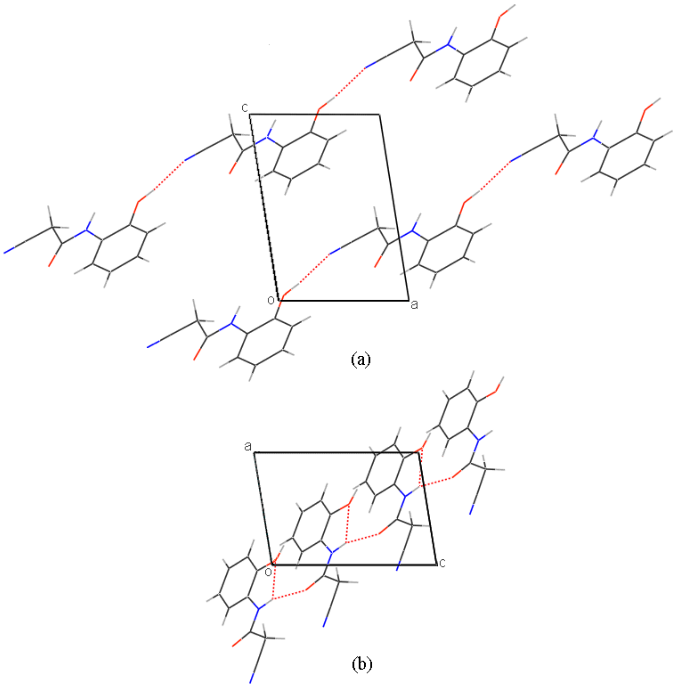


Figure 1
An *ORTEP*-3 view (Farrugia, 1997) of (4), showing 30% probability displacement ellipsoids and the atom-numbering scheme.



(a)

(b)

Figure 2
A *MERCURY* view (CCDC, 2003) of (*a*) the two-center and (*b*) the three-center hydrogen bonds in the crystal structure.

dimethyldiphenol [2.611 (6) Å] (Elmali & Elerman, 1997; Xu *et al.*, 1994; Elerman *et al.*, 1994).

Experimental

The method of preparation included heating *ortho*-aminophenol and ethyl cyanoacetate at 453 K for 6 h, without isolation of the intermediate. After cooling, 2,4-dihydroxybenzaldehyde, ammonium acetate and ethanol were added and the mixture was heated to reflux for 30 min. After cooling, the resulting solid was filtered and washed with water, ethanol and diethyl ether. Possibly the hydrolysis of the benzoxazol-2-ylacetonitrile, (2), produced the title compound (Luan *et al.*, 2002; Elnagdi *et al.*, 1997).

Crystal data

$C_9H_8N_2O_2$
$M_r = 176.17$
Monoclinic, *Cc*
$a = 5.6672$ (3) Å
$b = 18.0609$ (11) Å
$c = 8.1949$ (5) Å
$\beta = 99.135$ (3)°
$V = 828.15$ (8) Å^3
$Z = 4$

$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8167 reflections
$\theta = 1.0$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 120$ (2) K
Prism, yellow
0.12 × 0.06 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans with κ offsets
2565 measured reflections
1436 independent reflections
1301 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.096$
$\theta_{\rm max} = 25°$
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 21$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.131$
$S = 1.06$
1436 reflections
143 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.14 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.367 (3)	N1—C1	1.414 (4)
O2—C7	1.222 (3)	N2—C9	1.148 (4)
N1—C7	1.348 (3)		
C7—N1—C1	127.8 (2)	O1—C6—C1	116.5 (2)
C2—C1—N1	125.1 (2)	O2—C7—N1	125.4 (2)
C6—C1—N1	115.9 (2)	N1—C7—C8	112.7 (2)
O1—C6—C5	122.8 (2)	N2—C9—C8	179.0 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H···O1	0.87 (4)	2.17 (4)	2.629 (4)	111 (3)
N1—H···O2$^{\rm i}$	0.87 (4)	2.08 (4)	2.961 (4)	162 (3)
O1—H1···N2$^{\rm ii}$	0.90 (4)	1.88 (4)	2.762 (4)	164 (4)

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

H atoms were located in a difference Fourier synthesis and refined with a riding model. For methyl H atoms $U_{\rm iso}$ was set equal to $1.5U_{\rm eq}$ of the carrier atom; for other H atoms $U_{\rm iso}$ was set equal to $1.2U_{\rm eq}$. In the absence of significant anomalous scattering effects, the Flack (1983) parameter refinement is essentially meaningless. Friedel pairs were merged before refinement.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *MERCURY* (CCDC, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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