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6-(2-Hydroxybenzoyl)-2-phenylpyrazolo[1,5-*a*]-pyrimidine

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.136
Data-to-parameter ratio = 14.0

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

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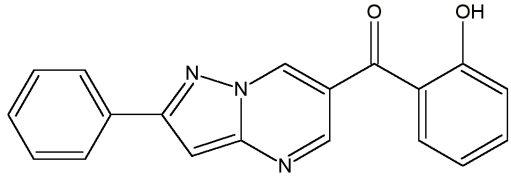
There are both weak and strong hydrogen bonds in the title compound, $C_{19}H_{13}N_3O_2$, The strong bond is an O—H···O intramolecular bond which forms an *S*(6) ring motif, whereas the weak C—H···N bond forms a *C*(9) motif forming chains which run parallel to the *b* axis.

Comment

Since becoming readily available, 3-formylchromone has been used to prepare a variety of heterocyclic systems (Jones & Albrecht, 1976; Haas *et al.*, 1981; Pene & Hubert-Habart., 1980; Sigg *et al.*, 1982). In our investigation of pyrazolo[1,5-*a*]pyrimidines, we have established that the cyclocondensation reaction of 5-amino-1*H*-pyrazoles with α,β-unsaturated aromatic ketones is a versatile and efficient method for the preparation of these compounds (Orlov *et al.*, 1988; Quiroga *et al.*, 1994, 1999), but the reaction with 3-formylchromone was not previously investigated. We have recently applied the above methodology to prepare several pyrazolo[1,5-*a*]-m 3-formylchromone.



(I)

Geometric parameters for the title compound, (I), are given in Table 1 and a view of the molecule is shown in Fig. 1. A strong intramolecular hydrogen bond, O62—H62···O61, forms an *S*(6) (Bernstein *et al.*, 1995) ring motif. The molecules are linked into chains by the weak C63—H63···N1^{i} hydrogen bond, which form a *C*(9) motif [symmetry code: (i) $-x+\frac{1}{2}$, $y+\frac{1}{2}$, $-z+\frac{1}{2}$]. A series of antiparallel chains run parallel to the *b* axis. Details of the hydrogen bonds are given in Table 2.

Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.

Experimental

An equimolar mixture of 3-formylchromone and 5-amino-3-phenylpyrazole in ethanol was heated to reflux for 10 min. The title compound precipitated, was separated by filtration and recrystallized from DMF, affording crystals suitable for X-ray diffraction. M.p. 290–291 K, yield: 86%.

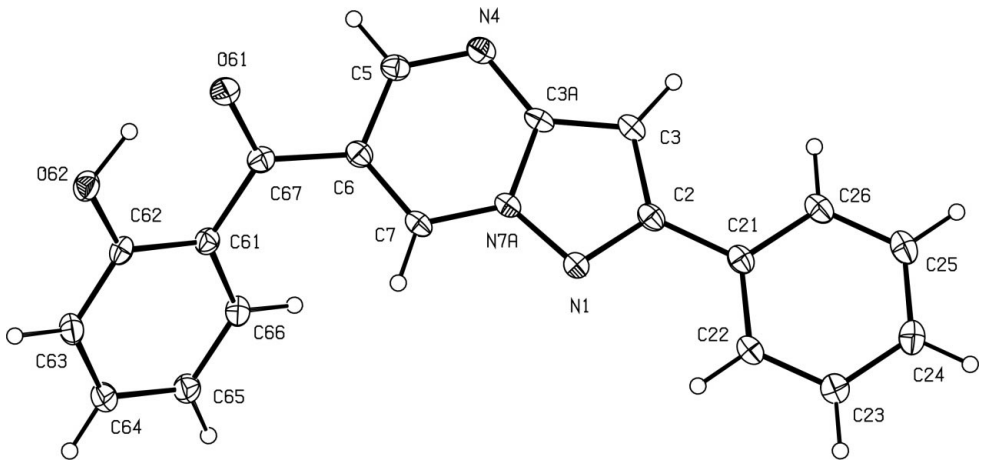


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Crystal data

$C_{19}H_{13}N_3O_2$	D_x = 1.463 Mg m^{-3}
M_r = 315.32	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 9215 reflections
a = 5.6606 (2) Å	θ = 1.0–27.5°
b = 11.7048 (5) Å	μ = 0.10 mm^{-1}
c = 21.6815 (10) Å	T = 150 (1) K
β = 94.7970 (15)°	Needle, yellow
V = 1431.50 (10) Å^3	0.44 × 0.10 × 0.08 mm
Z = 4	

Data collection

KappaCCD diffractometer	3032 independent reflections
φ and ω scans with κ offsets	2168 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)	R_{int} = 0.051
	θ_{max} = 27.5°
T_{min} = 0.958, T_{max} = 0.992	$h = -6 \rightarrow 6$
12 852 measured reflections	$k = -14 \rightarrow 15$
	$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.046	$w = 1/[\sigma^2(F_o^2) + (0.0846P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.136	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.04	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
3032 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
217 parameters	

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.3459 (19)	C3A—N7*A*	1.3922 (19)
N1—N7*A*	1.3580 (19)	N4—C5	1.306 (2)
C3A—N4	1.354 (2)	C7—N7*A*	1.3519 (19)
C2—N1—N7*A*	103.67 (12)	C7—N7*A*—C3*A*	122.59 (14)
C5—N4—C3*A*	116.24 (14)	N1—N7*A*—C3*A*	112.37 (12)
C7—N7*A*—N1	125.03 (13)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C63—H63···N1^{i}	0.95	2.56	3.512 (2)	174
O62—H62···O61	1.03	1.62	2.559 (2)	149

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

H atoms were treated as riding atoms with C—H distances in the range 0.90–0.95 Å and an O—H distance of 1.02 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC, X-ray Crystallographic Service, University of Southampton, using an Enraf—Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. We are grateful to the Ministerio de Educación y Cultura for the award of a grant to one of the authors (AQ).

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