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6-(2-Hydroxybenzoyl)-2-(4-nitrophenyl)pyrazolo-[1,5-*a*]pyrimidine

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.124
Data-to-parameter ratio = 8.3

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

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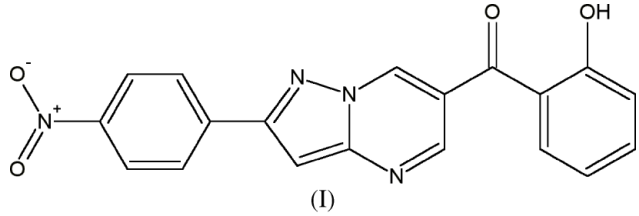
The structure of $C_{19}H_{12}N_4O_4$ contains one strong intramolecular O—H···O bond giving an *S*(6) motif. The molecules are linked together by weak C—H···O and C—H···N hydrogen bonds, forming a complex three-dimensional network of chain and ring motifs.

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*]-pyrimidines starting from 3-formylchromone.



(I)

Geometric and hydrogen-bond parameters for (I) are given in Tables 1 and 2, respectively. The three-dimensional hydrogen-bonding network produced by the weak intermolecular C—H···O and C—H···N hydrogen bonds is very complex, the simpler motifs being three infinite chains based on the following motifs; a *C*(7) motif (Bernstein *et al.*, 1995) involving C5—H5···O62^{i}, a *C*(5) motif involving C66—H66···O67^{iii} and a $C_2^2(12)$ motif involving C66—H66···O67^{iii} and C63—H63···N1^{ii} [symmetry codes: (i) $x-1, y, z$; (iii) $1-x, y+\frac{1}{2}, -z+\frac{1}{2}$]. The chains produced by repeat of these motifs create ring motifs which in turn produce a three-dimensional network. A view of the molecule is shown in Fig. 1.

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-(4-nitrophenyl)pyrazole in ethanol was heated to reflux for 10 min. The title compound precipitated, was separated by filtration and recrys-

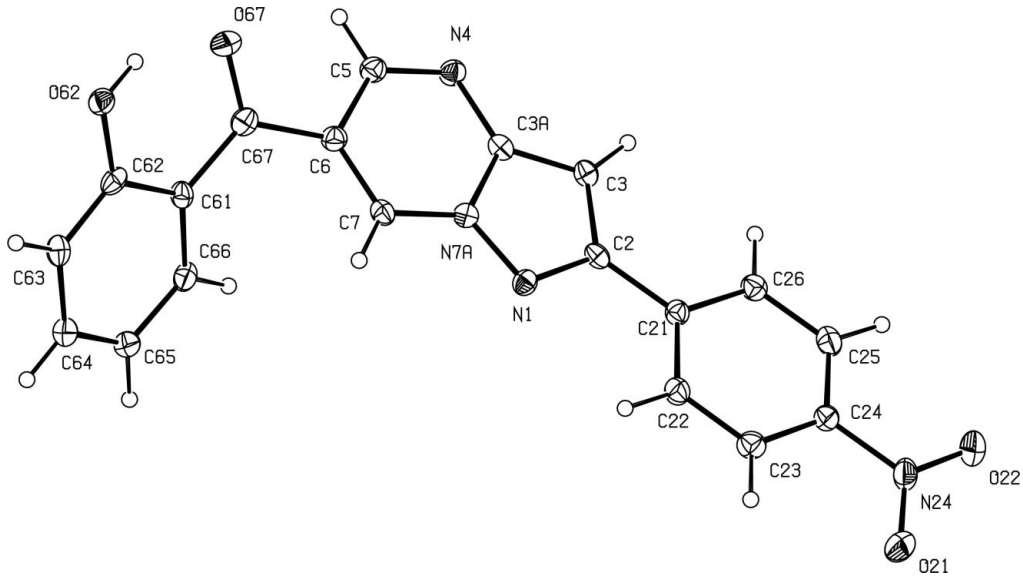


Figure 1
A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

tallized from DMF, affording crystals suitable for X-ray diffraction. M.p. 553–555 K, yield: 90%.

Crystal data

$C_{19}H_{12}N_4O_4$
M_r = 360.33
Orthorhombic, $P2_12_12_1$
a = 5.5421 (2) Å
b = 11.5858 (5) Å
c = 23.8057 (13) Å
V = 1528.56 (12) Å^3
Z = 4
D_x = 1.566 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7067 reflections
θ = 1.0–27.5°
μ = 0.11 mm^{-1}
T = 150 (1) K
Needle, yellow
0.20 × 0.08 × 0.07 mm

Data collection

KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.978, T_{max} = 0.992
7572 measured reflections
2022 independent reflections
1364 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.4°
$h = -7 \rightarrow 7$
$k = -15 \rightarrow 15$
$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.07
2022 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0480P)^2 + 0.3279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.341 (5)	C3A—N4	1.362 (5)
N1—N7*A*	1.353 (4)	C3A—N7*A*	1.391 (5)
C24—N24	1.460 (5)	C7—N7*A*	1.356 (5)
C2—N1—N7*A*	103.6 (3)	C5—N4—C3*A*	115.9 (3)
O21—N24—O22	123.3 (3)	N1—N7*A*—C7	125.1 (3)
O21—N24—C24	118.7 (3)	N1—N7*A*—C3*A*	112.6 (3)
O22—N24—C24	118.0 (3)	C7—N7*A*—C3*A*	122.2 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O62^{i}	0.95	2.56	3.301 (4)	135
C63—H63···N1^{ii}	0.95	2.58	3.519 (5)	168
C66—H66···O67^{iii}	0.95	2.47	3.332 (4)	150
O62—H62···O67	0.90	1.78	2.583 (4)	147

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

H atoms were treated as riding with distances C—H = 0.95 Å and O—H = 0.90 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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