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6-(2-Hydroxybenzoyl)-2-(4-methylphenyl)-5-methyl-7-phenylpyrazolo[1,5-*a*]pyrimidine: complex sheets built from C—H···N and C—H···O hydrogen bonds

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The molecules of the title compound, $C_{27}H_{21}N_3O_2$, contain an intramolecular O—H···O hydrogen bond, and they are linked into complex sheets by a combination of two C—H···N hydrogen bonds and two C—H···O hydrogen bonds.

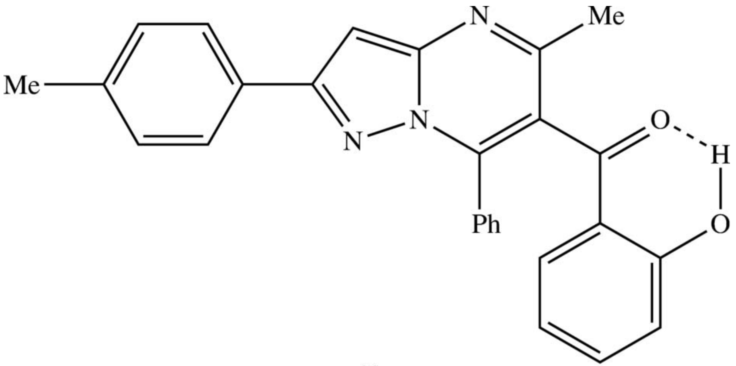
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.121
Data-to-parameter ratio = 16.3

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

Comment

We report here the structure of the title new pyrazolo[1,5-*a*]pyrimidine, (I) (Fig. 1), prepared under solvent-free conditions from the reaction between a 5-aminopyrazole and a 2-benzoylchromone.



(I)

The bond distances in the pyrazolo[1,5-*a*]pyrimidine fragment (Table 1) are typical of this ring system (Portilla *et al.*, 2005, 2006) and they are consistent with 10-π electron delocalization reminiscent of the naphthalene type. The aryl ring at C2 is almost coplanar with the heterocyclic system, but the substituents at C6 and C7 are considerably twisted out of this plane, probably to avoid mutually repulsive interactions between their aryl rings (Table 1).

There is a fairly short intramolecular O—H···O hydrogen bond, but the supramolecular aggregation of (I) depends solely on rather long, and thus fairly weak, C—H···N and C—H···O hydrogen bonds (Table 2), which link the molecules into complex sheets. The formation of the sheet can be readily analysed in terms of its simple sub-structures.

Aryl atom C74 in the molecule at (x, y, z) acts as hydrogen-bond donor to hydroxyl atom O62 in the molecule at ($1 - x$, $-y$, $1 - z$), so generating by inversion a centrosymmetric $R_2^2(22)$ (Bernstein *et al.*, 1995) dimer centred at ($\frac{1}{2}$, 0, $\frac{1}{2}$) (Fig. 2), and these dimers are linked by the C—H···N hydrogen bonds. Atoms C3 and C22 in the molecule at (x, y, z) and ($1 - x$, $-y$, $1 - z$) both act as hydrogen-bond donors to the atoms N4 in the molecules at ($\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$) and ($-\frac{1}{2} + x, -\frac{1}{2} - y, -\frac{1}{2} + z$), which themselves lie in the $R_2^2(22)$ dimers centred at (1, $\frac{1}{2}$, 1)

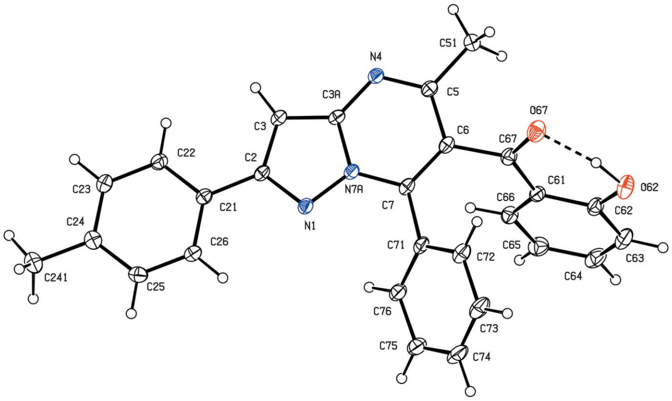


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and the intramolecular O—H···O hydrogen bond (dashed line). Displacement ellipsoids are drawn at the 30% probability level.

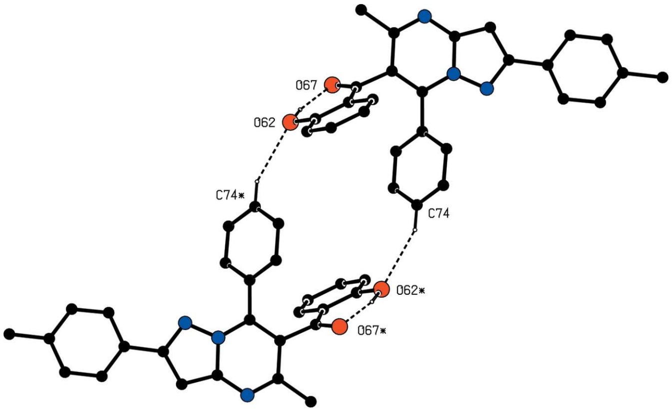


Figure 2
Part of the crystal structure of compound (I), showing the formation of an $R_2^2(22)$ dimer. For the sake of clarity, H atoms bonded to C atoms but not involved in the motifs shown have been omitted. Similarly, the unit-cell outline has been omitted. Atoms marked with an asterisk (*) are at the symmetry position (1 − *x*, −*y*, 1 − *z*).

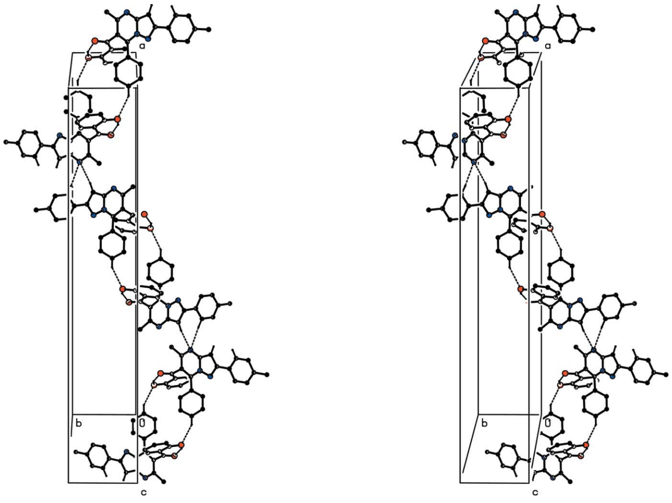


Figure 3
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a chain of rings along [111]. For the sake of clarity, H atoms bonded to C atoms but not involved in the motifs shown have been omitted.

and (0, $-\frac{1}{2}$, 0), respectively. Propagation of these hydrogen bonds then generates a chain of rings along [111], in which $R_2^2(22)$ rings alternate with $R_2^1(7)$ rings (Fig. 3).

Finally, aryl atom C76 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to carbonyl atom O67 in the molecule at (*x*, 1 + *y*, *z*), so generating by translation a *C*(7) chain running parallel to the [010] direction. The combination of [010] and [111] chains generates a sheet parallel to ($10\overline{1}$), but there are no direction-specific interactions between adjacent sheets.

Experimental

Equimolar quantities (1.0 mmol of each component) of 3-(4-methylphenyl)-5-amino-1*H*-pyrazole and 3-benzoyl-2-methylbenzo-4-pyrone were placed in an open Pyrex glass vessel and heated in an oil-bath at 373 K for 2 min. The reaction mixture was then cooled and extracted with ethanol. After removal of the solvent, the product, (I), was recrystallized from dimethylformamide to give yellow crystals suitable for single-crystal X-ray diffraction (m.p. 471–473 K, yield 60%).

Crystal data

$C_{27}H_{21}N_3O_2$
$M_r = 419.47$
Monoclinic, $C2/c$
$a = 38.7649\ (11)$ Å
$b = 6.7864\ (2)$ Å
$c = 16.7002\ (5)$ Å
$\beta = 108.599\ (2)^\circ$
$V = 4163.9\ (2)$ Å^3
$Z = 8$
$D_x = 1.338$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 120\ (2)$ K
Lath, yellow
0.80 × 0.50 × 0.20 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.964$, $T_{\max} = 0.983$
30599 measured reflections
4751 independent reflections
3797 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.121$
$S = 1.02$
4751 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 3.7732P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.3505 (18)	C5—C6	1.4342 (19)
C2—C3	1.4013 (19)	C6—C7	1.376 (2)
C3—C3*A*	1.377 (2)	C7—N7*A*	1.3675 (18)
C3*A*—N4	1.3525 (18)	N7*A*—N1	1.3603 (16)
N4—C5	1.3141 (18)	C3*A*—N7*A*	1.4011 (17)
N1—C2—C21—C22	179.38 (13)	C6—C67—C61—C62	−176.77 (13)
C5—C6—C67—C61	−110.80 (15)	C6—C7—C71—C72	43.8 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O62—H62$\cdots$O67	0.96	1.68	2.554 (2)	150
C3—H3$\cdots$N4^{i}	0.95	2.61	3.543 (2)	168
C22—H22$\cdots$N4^{i}	0.95	2.57	3.513 (2)	174
C74—H74$\cdots$O62^{ii}	0.95	2.55	3.422 (2)	154
C76—H76$\cdots$O67^{iii}	0.95	2.56	3.444 (2)	155

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

All H atoms were located in a difference map and then treated as riding, with C—H distances of 0.95 (aromatic) or 0.98 Å (methyl) and an O—H distance of 0.96 Å, and with $U_{\rm iso}$(H) = $xU_{\rm eq}$(C,O), where x = 1.5 for the methyl groups and 1.2 for all other H.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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