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The crystal structure of 5-benzoyl-1-[(*E*)-(4-fluorobenzylidene)amino]-4-phenylpyrimidin-2(1*H*)-one, $C_{24}H_{16}FN_3O_2$

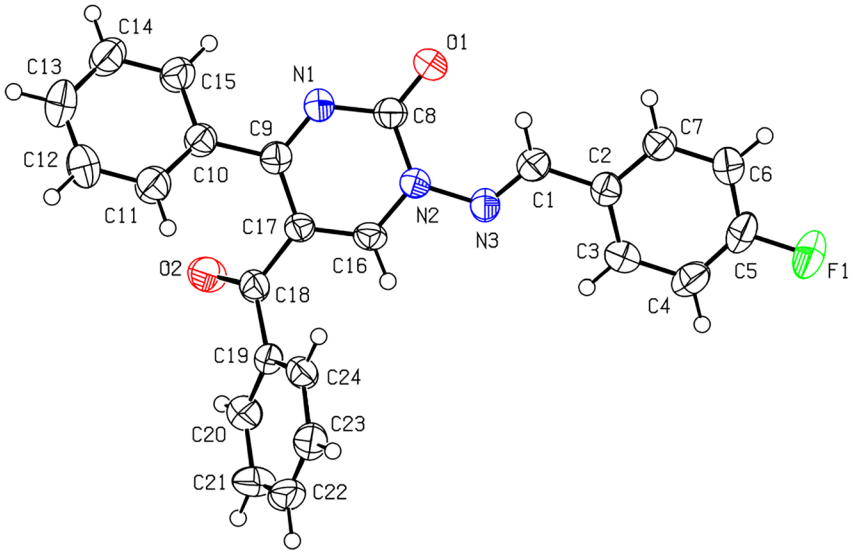

Figure 1: The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

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Abstract

$C_{24}H_{16}FN_3O_2$, monoclinic, $P2_1$ (no. 4), a = 6.0024(5) Å, b = 19.474(2) Å, c = 8.3531(8) Å, β = 91.750(9)°, V = 975.95(16) Å^3, Z = 2, $R_{gt}(F)$ = 0.0409, $wR_{ref}(F^2)$ = 0.0832, T = 294(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters (see Figure 1).

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.30 × 0.28 × 0.22 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3430, 2195, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1680
$N(param)_{refined}$:	271
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4], PLATON [5], structure refinement [6]

Source of material

The title Schiff base was synthesized according to the following procedure. About 1.0 mmol of aminopyrimidine derivative {1-amino-5-benzoyl-4-phenylpyrimidin-2(1*H*)-one} was dissolved in 30 mL of ethyl alcohol, and put to a solution of 4-fluorobenzaldehyde (1.2 mmol). A catalytic amount of *p*-toluene sulfonic acid was used as catalyst. The reaction mixture was refluxed for 6 h and then stirred for 24 h at 25 °C. Ethyl alcohol was aspirated, and diethyl ether

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.4207 (4)	0.28738 (16)	0.1087 (3)	0.0791 (9)
O1	0.6332 (4)	0.34582 (17)	0.7251 (3)	0.0543 (9)
O2	1.2532 (4)	0.55868 (17)	0.4500 (4)	0.0591 (9)
N1	0.8934 (5)	0.42981 (19)	0.7518 (4)	0.0410 (9)
N2	0.6280 (5)	0.43114 (17)	0.5354 (4)	0.0374 (9)
N3	0.4484 (5)	0.40886 (18)	0.4361 (4)	0.0419 (9)
C1	0.3085 (6)	0.3668 (2)	0.4894 (5)	0.0432 (11)
H1	0.324181	0.349847	0.593261	0.052*
C2	0.1208 (6)	0.3451 (2)	0.3843 (5)	0.0383 (10)
C3	0.0859 (7)	0.3726 (2)	0.2318 (5)	0.0473 (12)
H3	0.186226	0.404403	0.192823	0.057*
C4	−0.0956 (7)	0.3531 (3)	0.1380 (5)	0.0544 (13)
H4	−0.120613	0.371454	0.036286	0.065*
C5	−0.2381 (7)	0.3057 (3)	0.1993 (5)	0.0518 (13)
C6	−0.2093 (6)	0.2766 (3)	0.3458 (5)	0.0508 (13)
H6	−0.309460	0.244095	0.382256	0.061*
C7	−0.0266 (6)	0.2965 (2)	0.4396 (5)	0.0451 (11)
H7	−0.002663	0.277079	0.540383	0.054*
C8	0.7159 (6)	0.3977 (3)	0.6754 (5)	0.0410 (11)
C9	0.9907 (6)	0.4836 (2)	0.6921 (5)	0.0374 (10)
C10	1.1779 (6)	0.5125 (2)	0.7918 (5)	0.0412 (11)
C11	1.2220 (7)	0.5821 (3)	0.7999 (5)	0.0552 (13)
H11	1.132344	0.612730	0.741724	0.066*
C12	1.3981 (8)	0.6065 (3)	0.8937 (6)	0.0686 (16)
H12	1.426222	0.653441	0.898224	0.082*
C13	1.5313 (7)	0.5623 (3)	0.9797 (6)	0.0654 (15)
H13	1.652549	0.578989	1.039870	0.078*
C14	1.4860 (7)	0.4932 (3)	0.9774 (6)	0.0594 (14)
H14	1.573843	0.463213	1.038699	0.071*
C15	1.3099 (6)	0.4681 (3)	0.8842 (5)	0.0493 (12)
H15	1.279576	0.421298	0.883255	0.059*
C16	0.7334 (6)	0.4853 (2)	0.4729 (5)	0.0374 (10)
H16	0.676756	0.504300	0.377982	0.045*
C17	0.9192 (6)	0.5135 (2)	0.5426 (4)	0.0351 (10)
C18	1.0509 (6)	0.5639 (2)	0.4506 (5)	0.0398 (10)
C19	0.9352 (6)	0.6188 (2)	0.3553 (5)	0.0372 (10)
C20	1.0452 (7)	0.6474 (3)	0.2296 (5)	0.0544 (13)
H20	1.185055	0.630978	0.203635	0.065*
C21	0.9491 (9)	0.7003 (3)	0.1416 (6)	0.0667 (15)
H21	1.023022	0.719103	0.055728	0.080*
C22	0.7424 (8)	0.7251 (3)	0.1819 (6)	0.0597 (14)
H22	0.678019	0.761289	0.124294	0.072*
C23	0.6341 (7)	0.6969 (2)	0.3049 (6)	0.0529 (13)
H23	0.494585	0.713686	0.330536	0.064*
C24	0.7271 (6)	0.6434 (2)	0.3939 (5)	0.0423 (11)
H24	0.650722	0.624309	0.478349	0.051*

was put over product. The solid obtained, was filtered after stirred in the cold for 24 h. The final product was purified two times by crystallization in ethyl alcohol. Yield: 70%; m.p.: 175–177 °C; color: white. H NMR **IR**: υ = 3030.0 (aromatic C–H), 1674.1, 1652.9 (C=O), 1622.0 and 1595.0 (C=N and C=C), 780.2–680.1 cm^{-1} (pyrimidine ring). ^{1}**H NMR** (400 MHz, DMSO) δ 9.25 (s, 1H), 8.69 (s, 1H), 8.00 (dd, J = 8.1, 5.4 Hz, 2H), 7.82 (d, J = 7.3 Hz, 2H), 7.60–7.29 (m, 10H). ^{13}**C NMR** (100 MHz, DMSO) δ 192.01, 171.20, 166.05, 163.96, 151.53, 149.23, 137.26, 133.88, 132.07, 131.97, 131.14, 130.18, 129.14, 129.06, 128.69, 116.94, 116.72, 116.06. **Anal. Calcd.** For $C_{24}H_{16}FN_3O_2$ (397.401 g/mol). C, 72.54; H, 4.06; N, 10.57. Found: C, 72.35; H, 3.85; N, 10.35.

Experimental details

All H atoms were positioned at geometrically idealized positions and were refined using a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C). The Flack parameter (0.1(10) from 328 selected quotients) was calculated using Parsons' method [6].

Comment

Pyrimidine derivatives are important classes of heterocyclic molecules that indicate a broad spectrum of biological activities such as antibacterial, anticancer, antioxidant and analgesic activities [7–9]. Therefore, the synthesis of pyrimidine-based molecules has achieved great significance in recent years. Schiff bases, also called imines, are obtained by a condensation reaction between a primary amine and aldehyde or ketone under mild condition. They find use as catalysts, intermediates in organic synthesis, pigments and dyes, polymer stabilizers [10]. Schiff bases also exhibit a wide range of biological activities [11, 12].

All atoms in the pyrimidine ring (N1/N2/C8/C9/C16/C17) are almost coplanar, the largest deviation from the mean plane was calculated to be 0.049(5) Å for C8. The orientations of the substituents with respect to the pyrimidine ring are defined by the dihedral angles of 10.9(2), 32.5(2) and 62.2(2)° for a fluro phenyl ring *A*(C2–C7), and two phenyl rings *B*(C10–C15), *C*(C19–C24), respectively. The other angles between the ring planes are 43.5(2)° for *A*/*B*, 60.0(2)° for *A*/*C* and 73.4 (2)° for *B*/*C*. All bond distances and angles are in good agreement with those in related structures [13–15].

The molecular conformation is affected by a weak intramolecular C1–H1···O1 contact. In the crystal structure, molecules are linked by weak intermolecular C22–H22 ··· F1 (−*x*, *y*+1/2, −*z*) and two C–H ··· π interactions [C7–H7···Cg4 (−*x*+1, *y*−1/2, −*z*+1) and C13–H13···Cg4

(x+1, y, z+1); where Cg4 is the centroid of the C19–C24 phenyl ring], forming a three dimensional network.

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