Zeliha Atioğlu*, Halis Karataş and Zülbiye Kökbudak

The crystal structure of 5-benzoyl-1-[(*E*)-(4-fluorobenzylidene)amino]-4-phenylpyrimidin-2(1*H*)-one, C₂₄H₁₆FN₃O₂

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

<https://doi.org/10.1515/ncrs-2021-0216>

Received May 31, 2021; accepted June 14, 2021;

published online June 24, 2021

Abstract

C₂₄H₁₆FN₃O₂, monoclinic, *P*₂₁ (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) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0409, *wR*_{ref}(*F*²) = 0.0832, *T* = 294(2) K.

CCDC no.: 2065479

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 Kα radiation (0.71073 Å)
μ:	0.10 mm ^{−1}
Diffractometer, scan mode:	Xcalibur, ω
θ _{max} , completeness:	25.0°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	3430, 2195, 0.029
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1680
<i>N</i> (<i>param</i>) _{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

*Corresponding author: Zeliha Atioğlu, Avionics Department, School of Applied Sciences, Cappadocia University, 50420, Mustafapaşa, Ürgüp, Nevşehir, Turkey, E-mail: zeliha.atioglu@kapadokya.edu.tr. <https://orcid.org/0000-0002-1141-5151>

Halis Karataş and Zülbiye Kökbudak, Department of Chemistry, Faculty of Sciences, Erciyes University, 8039 Kayseri, Turkey

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{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**: ν = 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). **¹H 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). **¹³C 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 C₂₄H₁₆FN₃O₂ (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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. 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 fluoro 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.

Acknowledgment: I would like to thank Assoc. Professor Dr. Muhittin Aygün (Dokuz Eylül University, İzmir) for aid with the data collection of the title compound.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The research did not receive funding support.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Oxford Diffraction. CRYSAUS^{PRO}; Oxford Diffraction Ltd: Abingdon, Oxfordshire, England, 2006.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Farrugia L. J. WinGX and ORTEP for Windows: an update. *J. Appl. Crystallogr.* 2012, *45*, 849–854.
5. Spek A. L. Structure validation in chemical crystallography. *Acta Crystallogr.* 2009, *D65*, 148–155.
6. Parsons S., Flack H. D., Wagner T. Use of intensity quotients and differences in absolute structure refinement. *Acta Crystallogr.* 2013, *B69*, 249–259.
7. Machnikova R., Janovska L., Brulikova L. Solid-phase synthetic approach towards new pyrimidines as potential antibacterial agents. *J. Mol. Struct.* 2020, *1200*, 127101.
8. Thiriveedhi A., Nadh R. V., Srinivasu N., Bobde Y., Ghosh B., Sekhar K. V. G. C. Design, synthesis and anti-tumour activity of new pyrimidine-pyrrole appended triazoles. *Toxicol. Vitro* 2019, *60*, 87–96.
9. Quiroga J., Romo P. E., Ortiz A., Isaza J. H., Insuasty B., Abonia R., Nogueras M., Cobo J. Synthesis, structures, electrochemical studies and antioxidant activity of 5-aryl-4-oxo-3,4,5,8-tetrahydropyrido[2,3-*d*]pyrimidine-7-carboxylic acids. *J. Mol. Struct.* 2016, *1120*, 294–301.
10. Aslan H. G., Akkoc S., Kokbudak Z., Aydin L. Synthesis, characterization, and antimicrobial and catalytic activity of a new Schiff base and its metal(II) complexes. *J. Iran. Chem. Soc.* 2017, *14*, 2263–2273.
11. Przybylski P., Huczynski A., Pyta K., Brzezinski B., Bartl F. Biological properties of Schiff bases and azo derivatives of phenols. *Curr. Org. Chem.* 2009, *13*, 124–148.
12. Aslan H. G., Akkoc S., Kokbudak Z. Anticancer activities of various new metal complexes prepared from a Schiff base on A549 cell line. *Inorg. Chem. Commun.* 2020, *111*, 107645.
13. Akkurt M., Öztürk S., Fun H.-K., Önal Z., Altural B. 3-Acetyl-4-benzoyl-2-methyl-5-phenyl-3,3a-dihydropyrazolo[2,3-*c*]pyrimidine-7(6*H*)-thione. *Acta Crystallogr.* 2000, *C56*, e276–e277.
14. Akkurt M., Öztürk S., Önal Z., Altural B., Büyükgüngör O. *N*-(5-Benzoyl-2-oxo-4-phenyl-1,2-dihydropyrimidin-1-yl) benzamide. *Acta Crystallogr.* 2004, *E60*, o1844–o1846.
15. Akkurt M., Hiller W. Structure of 5-benzoyl-1-[4-(dimethylamino)phenylmethyleneamino]-4-phenyl-1*H*-pyrimidin-2-one. *Acta Crystallogr.* 1993, *49*, 747–749.