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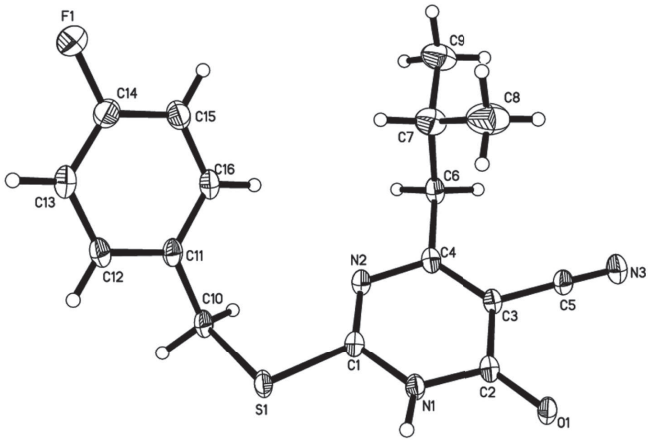
Ali A. El-Emam, Hazem A. Ghabbour*, Omar A. Al-Deeb, Mohammed S. M. Abdelbaky and Santiago García-Granda

# Crystal structure of 2-[(4-fluorobenzyl)sulfanyl]-4-(2-methylpropyl)-6-oxo-1,6-dihydropyrimidine-5-carbonitrile, $C_{16}H_{16}FN_3OS$

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## Abstract

$C_{16}H_{16}FN_3OS$, triclinic, $P\bar{1}$ (no. 2), $a = 5.6885(3)$ Å, $b = 9.4378(4)$ Å, $c = 15.0736(7)$ Å, $\alpha = 84.037(4)°$, $\beta = 81.442(4)°$, $\gamma = 74.271(4)°$, $V = 768.56(7)$ Å$^3$, $Z = 2$, $R_{gt}(F) = 0.0518$, $wR_{ref}(F^2) = 0.1430$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Hazem A. Ghabbour,** Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P.O.Box 2457, Riaydh 11451, Saudi Arabia, e-mail: ghabbourh@yahoo.com
**Ali A. El-Emam and Omar A. Al-Deeb:** Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P.O.Box 2457, Riaydh 11451, Saudi Arabia
**Mohammed S. M. Abdelbaky and Santiago García-Granda:** Departamento de Química Física y Analítica, Facultad de Química, Universidad de Oviedo – CINN, C/ Julian Clavería, 8, 33006 Oviedo, (Asturias), Spain

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless, prism, size 0.0508×0.0622×0.4616 mm |
| Wavelength: | Cu$K_\alpha$ radiation (1.5418 Å) |
| $\mu$: | 20.14 cm$^{-1}$ |
| Diffractometer, scan mode: | Xcalibur, Ruby, Gemini, $\omega$ scans |
| $2\theta_{max}$: | 141.36° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 11233, 2940 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 2544 |
| $N(param)_{refined}$: | 199 |
| Programs: | CrysAlis [18], SHELX [19] |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 2*i* | 0.4381 | 0.6334 | 0.5251 | 0.026 |
| H(13) | 2*i* | −0.4737 | 1.2318 | 0.8092 | 0.035 |
| H(12) | 2*i* | −0.2578 | 1.1887 | 0.6676 | 0.030 |
| H(6A) | 2*i* | 0.9096 | 0.8271 | 0.7583 | 0.030 |
| H(6B) | 2*i* | 1.0731 | 0.6639 | 0.7605 | 0.030 |
| H(16) | 2*i* | 0.3620 | 1.0019 | 0.7788 | 0.032 |
| H(10A) | 2*i* | 0.3864 | 1.0708 | 0.6198 | 0.029 |
| H(10B) | 2*i* | 0.1497 | 1.1572 | 0.5771 | 0.029 |
| H(15) | 2*i* | 0.1479 | 1.0477 | 0.9204 | 0.038 |
| H(7) | 2*i* | 0.5987 | 0.7620 | 0.8599 | 0.046 |
| H(9A) | 2*i* | 0.9170 | 0.8135 | 0.9222 | 0.069 |
| H(9B) | 2*i* | 1.0533 | 0.6451 | 0.9280 | 0.069 |
| H(9C) | 2*i* | 0.8003 | 0.7030 | 0.9872 | 0.069 |
| H(8A) | 2*i* | 0.6347 | 0.5257 | 0.8186 | 0.084 |
| H(8B) | 2*i* | 0.6248 | 0.5259 | 0.9231 | 0.084 |
| H(8C) | 2*i* | 0.8769 | 0.4663 | 0.8637 | 0.084 |

## Source of material

4-Fluorobenzyl chloride (1.45 g, 0.01 mol) and anhydrous potassium carbonate (1.38 g, 0.01 mol) were added to a solution of 6-(2-methylpropyl)-2-thiouracil-5-carbonitrile (2.09 g, 0.01 mol) in *N*,*N*-dimethylformamide (10 mL) and the mixture was stirred at room temperature for 12 hours. Water (15 mL) was gradually added and the mixture was stirred

**Table 3:** Atomic displacement parameters (Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| S(1) | 2*i* | 0.2170(1) | 0.90545(6) | 0.56345(4) | 0.0213(3) | 0.0169(3) | 0.0320(3) | 0.0027(2) | −0.0085(2) | −0.0033(2) |
| O(1) | 2*i* | 0.7895(3) | 0.4171(2) | 0.5397(1) | 0.0205(8) | 0.0153(7) | 0.0312(8) | −0.0002(6) | −0.0056(6) | −0.0036(6) |
| N(1) | 2*i* | 0.5314(3) | 0.6460(2) | 0.5619(1) | 0.0189(9) | 0.0167(9) | 0.028(1) | −0.0004(7) | −0.0077(7) | −0.0031(7) |
| F(1) | 2*i* | −0.3080(3) | 1.1698(2) | 0.9594(1) | 0.0371(9) | 0.054(1) | 0.0331(8) | 0.0011(7) | 0.0015(6) | −0.0048(7) |
| N(2) | 2*i* | 0.5880(3) | 0.8033(2) | 0.6637(1) | 0.0197(9) | 0.0167(9) | 0.029(1) | −0.0010(7) | −0.0038(7) | −0.0022(7) |
| N(3) | 2*i* | 1.2865(4) | 0.3801(2) | 0.6700(2) | 0.024(1) | 0.021(1) | 0.040(1) | −0.0016(8) | −0.0078(8) | −0.0001(8) |
| C(5) | 2*i* | 1.1004(4) | 0.4611(2) | 0.6592(2) | 0.023(1) | 0.017(1) | 0.028(1) | −0.0039(9) | −0.0045(9) | −0.0013(8) |
| C(3) | 2*i* | 0.8740(4) | 0.5667(2) | 0.6426(2) | 0.018(1) | 0.017(1) | 0.027(1) | −0.0012(8) | −0.0046(8) | 0.0001(8) |
| C(1) | 2*i* | 0.4714(4) | 0.7753(2) | 0.6021(2) | 0.018(1) | 0.017(1) | 0.027(1) | −0.0022(8) | −0.0014(8) | −0.0015(8) |
| C(2) | 2*i* | 0.7375(4) | 0.5334(2) | 0.5781(2) | 0.017(1) | 0.016(1) | 0.027(1) | −0.0021(8) | −0.0019(8) | 0.0010(8) |
| C(4) | 2*i* | 0.7908(4) | 0.6957(2) | 0.6862(2) | 0.019(1) | 0.017(1) | 0.027(1) | −0.0030(8) | −0.0017(8) | 0.0011(8) |
| C(13) | 2*i* | −0.3059(4) | 1.1861(3) | 0.8025(2) | 0.020(1) | 0.024(1) | 0.040(1) | −0.0007(9) | −0.003(1) | −0.004(1) |
| C(12) | 2*i* | −0.1764(4) | 1.1600(2) | 0.7183(2) | 0.023(1) | 0.017(1) | 0.035(1) | −0.0023(9) | −0.0091(9) | 0.0000(9) |
| C(6) | 2*i* | 0.9050(4) | 0.7245(2) | 0.7633(2) | 0.022(1) | 0.018(1) | 0.034(1) | −0.0021(8) | −0.0075(9) | −0.0009(9) |
| C(11) | 2*i* | 0.0752(4) | 1.0910(2) | 0.7083(2) | 0.023(1) | 0.013(1) | 0.033(1) | −0.0029(8) | −0.0049(9) | −0.0024(8) |
| C(16) | 2*i* | 0.1945(4) | 1.0486(2) | 0.7849(2) | 0.021(1) | 0.019(1) | 0.038(1) | −0.0005(9) | −0.0070(9) | −0.0032(9) |
| C(14) | 2*i* | −0.1813(5) | 1.1430(3) | 0.8763(2) | 0.031(1) | 0.029(1) | 0.031(1) | −0.004(1) | −0.000(1) | −0.004(1) |
| C(10) | 2*i* | 0.2172(4) | 1.0720(2) | 0.6161(2) | 0.024(1) | 0.015(1) | 0.033(1) | −0.0007(8) | −0.0059(9) | −0.0002(9) |
| C(15) | 2*i* | 0.0678(5) | 1.0750(3) | 0.8694(2) | 0.031(1) | 0.027(1) | 0.035(1) | −0.001(1) | −0.011(1) | −0.003(1) |
| C(7) | 2*i* | 0.7607(5) | 0.6908(3) | 0.8553(2) | 0.042(2) | 0.042(2) | 0.033(1) | −0.013(1) | −0.008(1) | −0.001(1) |
| C(9) | 2*i* | 0.8953(6) | 0.7153(4) | 0.9301(2) | 0.062(2) | 0.050(2) | 0.033(2) | −0.023(2) | −0.014(1) | 0.002(1) |
| C(8) | 2*i* | 0.7207(7) | 0.5384(4) | 0.8661(2) | 0.078(2) | 0.060(2) | 0.039(2) | −0.037(2) | −0.004(2) | 0.002(1) |

for additional 30 minutes. The precipitated crude product was filtered, washed with water, dried, and crystallized from aqueous ethanol to yield 2.29 g (72%) of the title compound. M.p. 481–483 K. Colourless prismatic single crystals were obtained by slow evaporation of an ethanolic solution at room temperature. $^1$H NMR (DMSO-$d_6$, 500.13 MHz): δ 0.92 (d, 6H, $CH_3$, $J = 6.0$ Hz), 2.14 (m, 1H, CH), 2.55 (d, 2H, C***H***$_2$CH, $J = 6.0$ Hz), 4.46 (s, 2H, $CH_2S$), 7.12–7.16 (m, 2H, Ar—H), 7.45–7.47 (m, 2H, Ar—H), 13.70 (s, 1H, NH); $^{13}$C NMR (DMSO-$d_6$, 125.76 MHz): δ 22.6 ($CH_3$), 28.0 (CH), 33.7 ($CH_2S$), 45.4 (***C***$H_2$CH), 96.2 (C-5), 115.5 (CN), 115.8, 131.4, 133.5, 162.9 (Ar—C), 161.0 (C-6), 165.9 (C = O), 174.0 (C-4); ESI-MS, *m/z* (rel. int.): 316.2 (M—H, 100)$^-$.

## Discussion

Various pyrimidine non-nucleoside analogues have been developed as potent chemotherapeutic agents with anticancer, antiviral, antifungal and antibacterial activities. Non-nucleoside pyrimidine-based analogues have emerged as useful therapies against human immunodeficiency viruses (HIV) [1–4], hepatitis B viruses (HBV) [5], herpes simplex viruses (HSV) [6, 7], varicella-zoster virus (VZV) [8] and influenza viruses [9]. A large number of pyrimidine-based antimetabolites are currently used as potent and selective anticancer activity [10–12]. In addition, marked antibacterial and antifungal activities were observed for several pyrimidine-5-carbonitrile derivatives [13–17]. Here, we report the crystal structure of the recently synthesized [16] title compound ($C_{16}H_{16}FN_3OS$).

The crystal structure of the title compound contains one molecule in the asymmetric unit. With respect to the pyrimidinyl ring, (C1/N1/C2/C3/C4/N2), the fluorobenzene ring (C11—C16) form dihedral angle of 51.2 (1)°. The molecular packing is stabilized by one intermolecular hydrogen bond where O1 acts as hydrogen bond acceptor and the NH group (N1) is the hydrogen bond donor. The H···O distance of the N1—H1···O1 hydrogen bond is 1.91(1) Å and the angle is 169.7(1)°.

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