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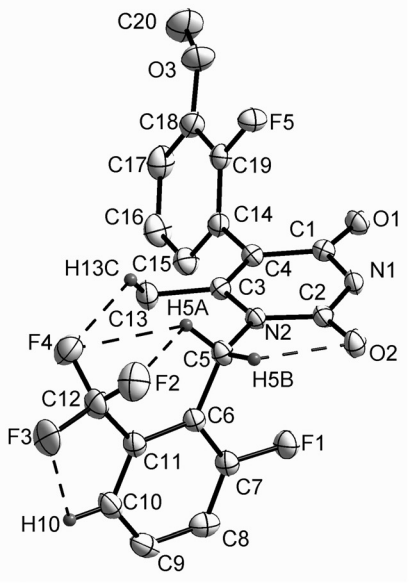
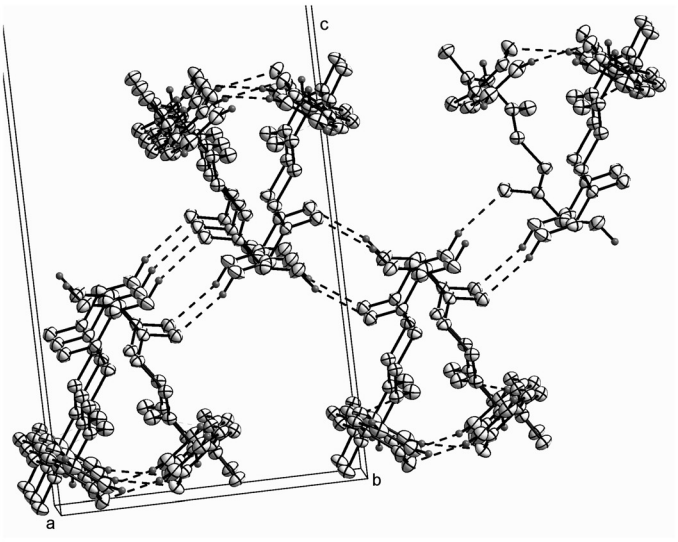
The crystal structure of 5-(2-fluoro-3-methoxyphenyl)-1-(2-fluoro-6-(trifluoromethyl) benzyl)-6-methylpyrimidine-2,4(1*H*,3*H*)-dione, $C_{20}H_{15}F_5N_2O_3$





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Abstract

$C_{20}H_{15}F_5N_2O_3$, orthorhombic, *Pbca* (no. 61), a = 10.9913 (4) Å, b = 11.0451(5) Å, c = 30.3925(14) Å, V = 3689.6 (3) Å^3, Z = 8, $R_{gt}(F)$ = 0.0360, $wR_{ref}(F^2)$ = 0.0927, T = 170 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.45 × 0.15 × 0.09 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.14 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
θ_{max}, completeness:	27.1°, >99%
N $(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	55,383, 4080, 0.061
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3247
$N(param)_{refined}$:	273
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4], PLATON [5–8], Publcif [9]

Source of material

In a representative experiment, approximately 0.4 g (1 mmol) of the title compound was dissolved in a mixed solvent of xylene (5 mL) and dichloromethane (5 mL) placed in a glass vial sealed with perforated tape. Solvent was evaporated slowly at room temperature and the product was obtained as a needle crystals in about two weeks.

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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	0.37628 (8)	0.80102 (8)	0.47806 (3)	0.0324 (2)
F2	0.74812 (9)	1.00525 (10)	0.40788 (4)	0.0463 (3)
F3	0.67307 (11)	1.17325 (9)	0.38586 (4)	0.0540 (3)
F4	0.63691 (10)	1.01055 (9)	0.34989 (3)	0.0443 (3)
F5	0.43702 (8)	0.39478 (8)	0.30865 (3)	0.0299 (2)
O1	0.33282 (10)	0.38100 (9)	0.40013 (3)	0.0289 (2)
O2	0.57698 (10)	0.62480 (9)	0.48081 (3)	0.0290 (2)
O3	0.30526 (11)	0.33727 (11)	0.23971 (4)	0.0408 (3)
N1	0.45547 (11)	0.50533 (10)	0.43923 (4)	0.0240 (3)
H1	0.459553	0.449340	0.459778	0.029*
N2	0.51251 (11)	0.69585 (10)	0.41403 (4)	0.0215 (2)
C1	0.38539 (13)	0.47809 (12)	0.40245 (5)	0.0227 (3)
C2	0.51841 (13)	0.60935 (12)	0.44684 (5)	0.0230 (3)
C3	0.44309 (13)	0.67822 (12)	0.37608 (4)	0.0221 (3)
C4	0.38118 (13)	0.57386 (12)	0.36975 (5)	0.0223 (3)
C5	0.58291 (13)	0.80739 (12)	0.42148 (5)	0.0234 (3)
H5A	0.630002	0.825759	0.394531	0.028*
H5B	0.641898	0.792528	0.445502	0.028*
C6	0.50685 (12)	0.91762 (12)	0.43330 (4)	0.0219 (3)
C7	0.40719 (13)	0.91045 (13)	0.46109 (5)	0.0246 (3)
C8	0.33650 (14)	1.00785 (14)	0.47336 (5)	0.0305 (3)
H8	0.269244	0.997414	0.492625	0.037*
C9	0.36547 (15)	1.12070 (15)	0.45708 (5)	0.0344 (4)
H9	0.316582	1.188805	0.464291	0.041*
C10	0.46622 (15)	1.13428 (14)	0.43017 (5)	0.0329 (4)
H10	0.487401	1.212313	0.419480	0.040*
C11	0.53637 (14)	1.03493 (13)	0.41871 (5)	0.0261 (3)
C12	0.64715 (16)	1.05584 (14)	0.39083 (5)	0.0347 (4)
C13	0.44151 (15)	0.77978 (13)	0.34335 (5)	0.0302 (3)
H13A	0.400238	0.850032	0.356199	0.045*
H13B	0.398055	0.753894	0.316812	0.045*
H13C	0.525217	0.801802	0.335643	0.045*
C14	0.30323 (13)	0.55042 (12)	0.33062 (5)	0.0235 (3)
C15	0.19568 (14)	0.61467 (14)	0.32290 (5)	0.0316 (3)
H15	0.172129	0.678158	0.342188	0.038*
C16	0.12383 (15)	0.58558 (16)	0.28717 (6)	0.0381 (4)
H16	0.050912	0.629636	0.282136	0.046*
C17	0.15589 (15)	0.49354 (15)	0.25854 (5)	0.0361 (4)
H17	0.104958	0.474570	0.234244	0.043*
C18	0.26243 (15)	0.42904 (14)	0.26536 (5)	0.0297 (3)
C19	0.33338 (13)	0.45918 (13)	0.30163 (5)	0.0240 (3)
C20	0.2214 (2)	0.28483 (18)	0.20942 (6)	0.0528 (5)
H20A	0.201834	0.343773	0.186374	0.079*
H20B	0.146795	0.261971	0.225009	0.079*
H20C	0.257859	0.212714	0.196046	0.079*

Experimental details

Using Olex2 [4], the structure was solved with the ShelXT [2] using intrinsic phasing and refined with the ShelXL [3] refinement package.

Coordinates of hydrogen atoms were included using constraints or restraints. Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms for all C(H) groups, all C(H, H) groups, all N(H) groups, and 1.5 U_{eq} for all C(H, H, H) groups.

The secondary CH2, C5(H5A, H5B), were refined with riding coordinates.

The aromatic/amide H, N1(H1), C8(H8), C9(H9), C10(H10), C15(H15), C16(H16), C17(H17), were refined with riding coordinates.

Idealised Me, C13(H13A, H13B, H13C), C20(H20A, H20B, H20C), were refined as rotating group.

Comment

The title compound is an intermediate of products reported by literature [10], which are the most recent synthetic class of antimicrobials active against a number of pathogenic microorganisms and has been approved for a treatment of select gram-positive infections. This research is also part of our continuing interest in synthesis, characterisation and understanding of hydrogen bonding schemes of related compounds [11–13].

The asymmetric unit of the title structure contains one title molecule (*cf*. left part of the figure). There are four intramolecular non-classical hydrogen bonds.

C5 participates in three intramolecular hydrogen bonds (*cf*. left part of the figure). These bonds are C5–H5A···F2, D···A = 2.8713 (17) Å; C5–H5A···F4, D···A = 3.1814 (17) Å; and C5–H5B···O2, D···A = 2.7061 (17) Å. C10 participates in an intramolecular hydrogen bond (C10–H10···F3, D···A = 2.677 (2) Å; *cf*. left part of the figure).

The intermolecular hydrogen bonds (N1–H1···O2′; ′ = 1 − *x*, 1 − *y*, 1 − *z*; D···A = 2.8459 (15) Å; *cf*. right part of the figure) connect N1 and O2 of two adjacent molecules to form an eight-membered ring.

Non-classical intermolecular hydrogen bonds (C9–H9···F1″; ″ = ½ − *x*, ½ + *y*, *z*; D···A = 3.3814 (19) Å) and (C10–H10···O1‴; ‴ = *x*, 1 + *y*, *z*; D···A = 3.2263 (19) Å) run along the b axis (*cf*. right part of the figure. Some atoms are omitted for clarity purpose).

Non-classical intermolecular hydrogen bonds (C17–H17···F5⁗; ⁗ = −½ + *x*, *y*, ½ − *z*, D···A = 3.3387 (18) Å) run along a axis (*cf*. right part of the figure).

In total, the three intermolecular hydrogen bonding interactions construct a network parallel to the *ab* plane (*cf*. right part of the figure).

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