Crystal structure of 5-phenylmethylidene-3-(2′-fluorobenzyl)-2-thio-4-imidazolidinone, $C_{17}H_{13}FN_2OS$

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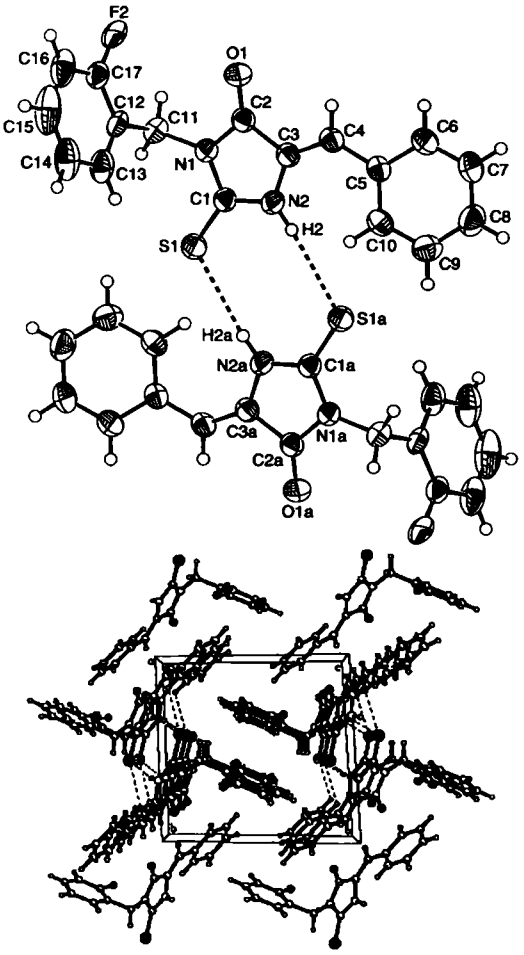

Abstract

$C_{17}H_{13}FN_2OS$, triclinic, $P\bar{1}$ (no. 2), a = 6.465(3) Å, b = 10.658(4) Å, c = 11.080(4) Å, α = 85.153(6)°, β = 78.525(6)°, γ = 79.650(6)°, V = 735.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.095, T = 293 K.

Source of material

In search for biological active imidazolinones [1] we have synthesized the title compound for the first time via tandem aza-Wittig reaction, the light yellow product was purified by double crystallization from the mixtured solvents of CH_3CN, petroleum ether and diethyl ether. Single crystals suitable for the X-ray structure analysis were obtained from CH_2Cl_2.

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Discussion

This highly functionalized material possesses three distinct units: a phenyl ring, an imidazolinone ring, and a fluorobenzyl ring. These units do not lie in the same plane (figure, top). The phenyl ring is linked to imidazolinone ring by a methylidene group, the angle between the planes of these rings is 20.72(7)°. The imidazolinone ring is connected to fluorobenzyl ring by the N1 atom of imidazolinone, the planes of these rings show an angle of 79.15(8)°. The angle between phenyl and fluorobenzyl ring is equal 63.85(8)°. In the unit cell the molecules form layers via sets of nonlinear intermolecular hydrogen bonds, and they are packed in a crossed arrangement when viewed along a-axis (figure, bottom). Among the H bonds, there are two symmetrical hydrogen bonds, N2–H2···S1a and N2a–H2a···S1 (symmetry code: $1-x, 1-y, 1-z$), which are responsible for dimeric subunits. Bond length and bond angle are 3.521 Å and 165.9°, respectively, being in the range known from literature data [2-5].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.30 × 0.36 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.34 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	52.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4257, 2986
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2986
$N(param)_{refined}$:	240
Programs:	SHELXS-97 [6], SHELXL-97 [7], SHELXTL-Plus [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.4290	0.3434	1.0178	0.049
H(4)	2*i*	0.1767	0.0564	1.0545	0.049
H(6)	2*i*	0.2454	–0.0626	1.2391	0.062
H(7)	2*i*	0.4588	–0.1257	1.3837	0.077
H(8)	2*i*	0.7521	–0.0305	1.3832	0.076
H(9)	2*i*	0.8387	0.1256	1.2332	0.067
H(10)	2*i*	0.6276	0.1895	1.0866	0.055
H(11A)	2*i*	–0.0248	0.5261	0.7598	0.052
H(11B)	2*i*	–0.1810	0.4265	0.7973	0.052
H(13)	2*i*	0.3432	0.4194	0.6085	0.067
H(14)	2*i*	0.4508	0.3326	0.4165	0.089
H(15)	2*i*	0.2139	0.2413	0.3335	0.106
H(16)	2*i*	–0.1307	0.2370	0.4426	0.091

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	0.3118(1)	0.5668(1)	0.8619(1)	0.068(1)	0.040(1)	0.054(1)	−0.021(1)	−0.024(1)	0.007(1)
O(1)	2*i*	−0.0507(2)	0.1870(1)	0.8920(1)	0.064(1)	0.056(1)	0.052(1)	−0.030(1)	−0.018(1)	0.000(1)
N(1)	2*i*	0.0984(2)	0.3717(1)	0.8551(1)	0.046(1)	0.041(1)	0.035(1)	−0.013(1)	−0.012(1)	0.000(1)
N(2)	2*i*	0.3299(2)	0.3337(1)	0.9796(1)	0.051(1)	0.039(1)	0.040(1)	−0.014(1)	−0.018(1)	0.001(1)
C(1)	2*i*	0.2478(3)	0.4224(2)	0.8996(1)	0.044(1)	0.038(1)	0.033(1)	−0.009(1)	−0.008(1)	−0.003(1)
C(2)	2*i*	0.0757(3)	0.2511(2)	0.9103(1)	0.047(1)	0.042(1)	0.033(1)	−0.013(1)	−0.006(1)	−0.003(1)
C(3)	2*i*	0.2343(3)	0.2239(2)	0.9928(1)	0.048(1)	0.038(1)	0.033(1)	−0.012(1)	−0.006(1)	−0.004(1)
C(4)	2*i*	0.2631(3)	0.1150(2)	1.0626(2)	0.052(1)	0.035(1)	0.036(1)	−0.013(1)	−0.005(1)	−0.004(1)
C(5)	2*i*	0.4081(3)	0.0743(2)	1.1489(2)	0.050(1)	0.029(1)	0.036(1)	−0.006(1)	−0.005(1)	−0.004(1)
C(6)	2*i*	0.3631(3)	−0.0233(2)	1.2381(2)	0.061(1)	0.041(1)	0.055(1)	−0.017(1)	−0.013(1)	0.007(1)
C(7)	2*i*	0.4914(4)	−0.0614(2)	1.3244(2)	0.078(2)	0.056(1)	0.061(1)	−0.018(1)	−0.023(1)	0.022(1)
C(8)	2*i*	0.6677(4)	−0.0053(2)	1.3237(2)	0.068(1)	0.061(1)	0.063(1)	−0.005(1)	−0.029(1)	0.008(1)
C(9)	2*i*	0.7185(3)	0.0885(2)	1.2346(2)	0.049(1)	0.053(1)	0.067(1)	−0.010(1)	−0.016(1)	−0.002(1)
C(10)	2*i*	0.5909(3)	0.1273(2)	1.1472(2)	0.050(1)	0.036(1)	0.048(1)	−0.008(1)	−0.004(1)	0.001(1)
C(11)	2*i*	−0.0328(3)	0.4357(2)	0.7669(2)	0.044(1)	0.046(1)	0.042(1)	−0.005(1)	−0.015(1)	−0.003(1)
C(12)	2*i*	0.0418(7)	0.3803(5)	0.6407(3)	0.055(3)	0.037(3)	0.038(3)	−0.005(2)	−0.026(2)	0.001(2)
C(13)	2*i*	0.2482(6)	0.3829(5)	0.5753(4)	0.047(3)	0.065(2)	0.046(2)	0.012(2)	−0.011(2)	0.008(2)
C(14)	2*i*	0.3127(7)	0.3308(5)	0.4602(4)	0.073(3)	0.078(3)	0.050(2)	0.031(2)	−0.006(2)	0.007(2)
C(15)	2*i*	0.1707(8)	0.2762(5)	0.4105(3)	0.150(5)	0.059(3)	0.044(3)	0.025(3)	−0.024(3)	−0.016(3)
C(16)	2*i*	−0.0357(8)	0.2736(4)	0.4759(4)	0.145(4)	0.043(2)	0.054(3)	−0.021(3)	−0.048(3)	−0.001(2)
C(17)	2*i*	−0.1002(6)	0.3256(4)	0.5910(3)	0.069(2)	0.038(2)	0.047(2)	−0.015(2)	−0.028(2)	0.008(2)
F(2)	2*i*	−0.2940(3)	0.3172(2)	0.6557(2)	0.070(1)	0.104(2)	0.001(2)	−0.039(1)	−0.031(1)	−0.013(1)

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