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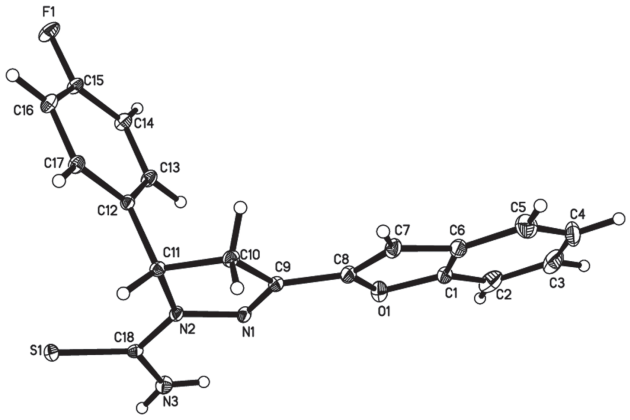
Gamal A. El-Hiti*, Bakr F. Abdel-Wahab, Mohammed Baashen, Hazem A. Ghabbour and Obaid S. Alruqi

Crystal structure of 3-(benzofuran-2-yl)-5-(4-fluorophenyl)-4,5-dihydro-1*H*pyrazole-1-carbothioamide, $C_{18}H_{14}FN_3OS$



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Abstract

$C_{18}H_{14}FN_3OS$, monoclinic, $P2_1/c$ (no. 14), $a = 14.9084(6)$ Å, $b = 11.2339(4)$ Å, $c = 9.4417(4)$ Å, $\beta = 102.331(2)°$, $V = 1544.81(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0365$, $wR_{ref}(F^2) = 0.0953$, $T = 100$ K.

***Corresponding author: Gamal A. El-Hiti,** Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, P. O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa
Bakr F. Abdel-Wahab: Department of Chemistry, College of Science and Humanities, Shaqra University, Duwadimi, Saudi Arabia; and Applied Organic Chemistry Department, National Research Centre, Dokki, Giza, Egypt
Mohammed Baashen: Department of Chemistry, College of Science and Humanities, Shaqra University, Duwadimi, Saudi Arabia
Hazem A. Ghabbour: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P. O. Box 2457 - Riyadh 11451, Kingdom of Saudi Arabia; and Department of Medicinal Chemistry, Faculty of Pharmacy, Mansoura University, Mansoura 35516, Egypt
Obaid S. Alruqi: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P. O. Box 2457 - Riyadh 11451, Kingdom of Saudi Arabia

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Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow, block, size 0.25×0.27×0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.31 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	66.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	34811, 5916
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4687
$N(param)_{refined}$:	227
Programs:	Bruker programs [4], SHELX [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*e*	0.4975	0.3870	0.9891	0.032
H(3)	4*e*	0.5955	0.2254	1.0639	0.037
H(4)	4*e*	0.5762	0.0472	0.9382	0.040
H(5)	4*e*	0.4550	0.0198	0.7381	0.034
H(7)	4*e*	0.3000	0.1539	0.5550	0.025
H(10A)	4*e*	0.1299	0.2713	0.4248	0.018
H(10B)	4*e*	0.2120	0.3033	0.3441	0.018
H(11)	4*e*	0.0513	0.4258	0.3016	0.014
H(13)	4*e*	0.2599	0.5939	0.3420	0.018
H(14)	4*e*	0.3200	0.6739	0.1517	0.019
H(16)	4*e*	0.1259	0.4971	−0.1399	0.019
H(17)	4*e*	0.0663	0.4178	0.0514	0.016
H(3A)	4*e*	0.149(1)	0.655(2)	0.669(2)	0.027(5)
H(3B)	4*e*	0.073(1)	0.739(2)	0.622(2)	0.026(4)
H(1A)	4*e*	0.3467	0.4583	0.7859	0.024

Source of material

The title compound was synthesized from reaction of 1-(benzofuran-2-yl)-3-(4-fluorophenyl)prop-2-en-1-one with thiosemicarbazide in ethanolic sodium hydroxide under reflux

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	−0.00709(2)	0.66889(3)	0.34385(3)	0.0138(2)	0.0148(2)	0.0151(2)	0.0025(1)	0.0024(1)	0.0015(1)
F(1)	4*e*	0.26018(6)	0.64126(8)	−0.11869(8)	0.0263(4)	0.0292(4)	0.0127(3)	−0.0093(3)	0.0060(3)	0.0038(3)
O(1)	4*e*	0.35344(7)	0.37899(9)	0.7549(1)	0.0208(5)	0.0197(5)	0.0193(5)	0.0059(4)	0.0029(4)	0.0041(4)
N(1)	4*e*	0.20148(7)	0.4912(1)	0.5763(1)	0.0126(5)	0.0185(5)	0.0108(4)	0.0005(4)	0.0011(4)	0.0027(4)
N(2)	4*e*	0.13062(7)	0.52637(9)	0.4629(1)	0.0141(5)	0.0139(5)	0.0089(4)	0.0022(4)	0.0013(4)	−0.0003(3)
N(3)	4*e*	0.10302(8)	0.6789(1)	0.6059(1)	0.0212(5)	0.0159(5)	0.0139(5)	0.0020(4)	0.0027(4)	−0.0037(4)
C(1)	4*e*	0.41904(9)	0.2969(1)	0.8189(1)	0.0167(6)	0.0187(6)	0.0182(6)	0.0030(5)	0.0059(5)	0.0063(5)
C(2)	4*e*	0.4892(1)	0.3133(1)	0.9386(2)	0.0298(7)	0.0306(8)	0.0183(6)	−0.0121(6)	0.0036(5)	0.0010(5)
C(3)	4*e*	0.5466(1)	0.2177(2)	0.9814(2)	0.0162(6)	0.049(1)	0.0243(7)	−0.0082(6)	−0.0025(5)	0.0150(7)
C(4)	4*e*	0.5346(1)	0.1107(2)	0.9068(2)	0.0215(7)	0.0385(9)	0.0405(9)	0.0129(6)	0.0089(6)	0.0238(7)
C(5)	4*e*	0.4635(1)	0.0939(1)	0.7876(2)	0.0332(8)	0.0182(7)	0.0356(8)	0.0032(6)	0.0129(7)	0.0032(6)
C(6)	4*e*	0.40436(9)	0.1899(1)	0.7427(1)	0.0137(6)	0.0217(6)	0.0177(6)	−0.0009(5)	0.0034(5)	0.0046(5)
C(7)	4*e*	0.32714(8)	0.2099(1)	0.6267(1)	0.0149(6)	0.0287(7)	0.0187(6)	−0.0015(5)	0.0013(4)	0.0066(5)
C(8)	4*e*	0.30022(9)	0.3224(1)	0.6379(1)	0.0137(6)	0.0286(7)	0.0180(6)	0.0021(5)	0.0051(5)	0.0095(5)
C(9)	4*e*	0.22666(8)	0.3861(1)	0.5450(1)	0.0127(5)	0.0173(6)	0.0130(5)	0.0008(4)	0.0042(4)	0.0037(4)
C(10)	4*e*	0.17170(9)	0.3351(1)	0.4060(1)	0.0191(6)	0.0123(5)	0.0134(5)	0.0022(4)	0.0050(4)	0.0015(4)
C(11)	4*e*	0.11771(8)	0.4455(1)	0.3366(1)	0.0137(5)	0.0116(5)	0.0108(5)	0.0004(4)	0.0031(4)	−0.0011(4)
C(12)	4*e*	0.15669(8)	0.4991(1)	0.2152(1)	0.0121(5)	0.0111(5)	0.0102(5)	0.0016(4)	0.0019(4)	0.0001(4)
C(13)	4*e*	0.23246(8)	0.5746(1)	0.2444(1)	0.0152(5)	0.0178(6)	0.0107(5)	−0.0024(4)	0.0009(4)	−0.0013(4)
C(14)	4*e*	0.26843(9)	0.6222(1)	0.1322(1)	0.0150(6)	0.0187(6)	0.0148(6)	−0.0043(5)	0.0025(4)	0.0002(4)
C(15)	4*e*	0.22693(9)	0.5920(1)	−0.0081(1)	0.0174(6)	0.0164(6)	0.0118(5)	−0.0004(4)	0.0049(4)	0.0030(4)
C(16)	4*e*	0.15248(9)	0.5166(1)	−0.0421(1)	0.0194(6)	0.0164(6)	0.0106(5)	−0.0018(5)	0.0009(4)	−0.0008(4)
C(17)	4*e*	0.11752(8)	0.4700(1)	0.0718(1)	0.0139(5)	0.0133(5)	0.0130(5)	−0.0019(4)	0.0010(4)	−0.0005(4)
C(18)	4*e*	0.07994(8)	0.6236(1)	0.4779(1)	0.0130(5)	0.0121(5)	0.0132(5)	−0.0021(4)	0.0058(4)	0.0009(4)
C(1A)	4*e*	0.35344(7)	0.37899(9)	0.7549(1)	0.0208(5)	0.0197(5)	0.0193(5)	0.0059(4)	0.0029(4)	0.0041(4)
O(7A)	4*e*	0.32714(8)	0.2099(1)	0.6267(1)	0.0149(6)	0.0287(7)	0.0187(6)	−0.0015(5)	0.0013(4)	0.0066(5)

for 12 h. The solid obtained on cooling was recrystallized from dimethylformamide to give colorless crystals of the title compound (Mp. 260–262 °C) [6].

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT [4]. The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX option 13, 23 and 43) [5]. The atoms O1/O7A und C7,H7/C1A,H1A are disordered and were split in the refinement in two parts with occupancy factors of 0.827(7) (O1, C7, H7) and 0.173(7) (O7A, C1A, H1A). The figure shows only one part for clarity.

Discussion

Pyrazolin-1-carbothioamide derivatives show some interesting biological activities such as antibacterial [1], antifungal [2] and anticancer properties [3].

In the title compound, the asymmetric unit contains one independent molecule. The central pyrazol ring (N1/N2/C9—C11) makes the dihedral angles of 15.28(2)° and 83.11(3)° with the benzofuran ring (O1/C1—C8) and the fluorophenyl ring (C12—C17), respectively. Consequently, the fluorophenyl ring is nearly perpendicular to the plane of whole molecule (cf. Figure). The structure shows at least two hydrogen bonds between N3—H2N3···F1^{i} and N3—H1N3···S1^{ii} with symmetry code: (i) *x*, *y*, *z*−1; (ii) *x*, −*y*+1/2, *z*−1/2.

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