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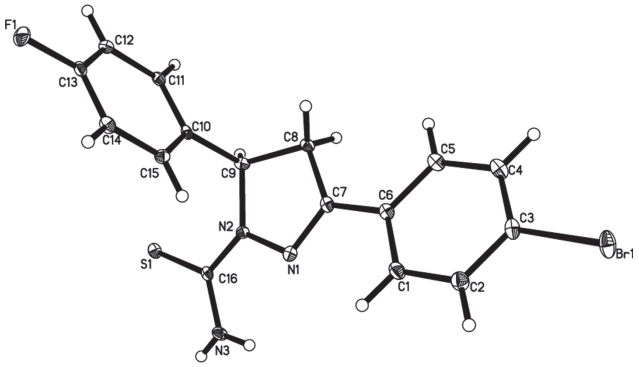
Crystal structure of 3-(4-bromophenyl)-5-(4-fluorophenyl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide, $C_{16}H_{13}BrFN_3S$



DOI 10.1515/ncrs-2016-0057
Received February 15, 2016; accepted June 3, 2016; available online June 11, 2016

Abstract

$C_{16}H_{13}BrFN_3S$, monoclinic, $P2_1/c$, $a = 14.6734(6)$ Å, $b = 11.1226(5)$ Å, $c = 9.4184(4)$ Å, $\beta = 102.524(2)°$, $V = 1500.57(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0473$, $wR_{ref}(F^2) = 0.1182$, $T = 100$ K.

CCDC no.: 1452283

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 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:	Colourless plates
	Size 0.68 × 0.37 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	28.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	58.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21750, 4371, 0.103
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3162
$N(param)_{refined}$:	207
Programs:	SHELX [6], Bruker programs [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.44696(2)	0.15662(3)	0.00851(3)	0.01972(11)
S1	1.01214(5)	0.66563(6)	0.65912(8)	0.01185(15)
F1	0.73592(13)	0.64557(14)	1.11962(18)	0.0181(4)
N1	0.79310(15)	0.5003(2)	0.4233(2)	0.0105(5)
N2	0.86648(15)	0.53044(19)	0.5376(2)	0.0092(4)
N3	0.90095(19)	0.6816(2)	0.3963(3)	0.0128(5)
C1	0.6293(2)	0.4069(3)	0.2376(3)	0.0148(6)
H1A	0.6406	0.4905	0.2306	0.018*
C2	0.5583(2)	0.3529(2)	0.1380(3)	0.0159(6)
H2A	0.5209	0.3989	0.0622	0.019*
C3	0.54196(19)	0.2305(2)	0.1495(3)	0.0131(6)
C4	0.5945(2)	0.1621(2)	0.2604(3)	0.0146(6)
H4A	0.5820	0.0788	0.2679	0.017*
C5	0.66584(18)	0.2170(2)	0.3605(3)	0.0128(6)
H5A	0.7020	0.1710	0.4376	0.015*
C6	0.68495(19)	0.3393(2)	0.3491(3)	0.0109(5)
C7	0.76343(18)	0.3951(2)	0.4513(3)	0.0102(5)
C8	0.81851(19)	0.3397(2)	0.5885(3)	0.0105(5)
H8A	0.8586	0.2735	0.5671	0.013*
H8B	0.7770	0.3087	0.6502	0.013*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C9	0.87727(18)	0.4463(2)	0.6612(3)	0.0099(5)
H9A	0.9441	0.4223	0.6956	0.012*
C10	0.83949(18)	0.5010(2)	0.7848(3)	0.0086(5)
C11	0.87928(18)	0.4708(2)	0.9285(3)	0.0100(5)
H11A	0.9308	0.4171	0.9490	0.012*
C12	0.8441(2)	0.5186(2)	1.0420(3)	0.0128(5)
H12A	0.8712	0.4987	1.1402	0.015*
C13	0.76933(19)	0.5953(2)	1.0087(3)	0.0119(5)
C14	0.72730(19)	0.6266(2)	0.8678(3)	0.0122(6)
H14A	0.6749	0.6790	0.8481	0.015*
C15	0.76382(18)	0.5793(2)	0.7566(3)	0.0109(5)
H15A	0.7368	0.6007	0.6589	0.013*
C16	0.92191(19)	0.6252(2)	0.5241(3)	0.0099(5)
H2N3	0.929(2)	0.734(3)	0.385(4)	0.021(10)*
H1N3	0.852(2)	0.658(2)	0.329(3)	0.004(7)*

Source of material

3-(4-Bromophenyl)-5-(4-fluorophenyl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide was synthesized from reaction of a mixture of (*E*)-1-(4-bromophenyl)-3-(4-fluorophenyl)prop-2-en-1-one, thiosemicarabzide and two mole equivalents of sodium hydroxide in ethanol under reflux for 3 h. The solid was filtered, dried and recrystallized from dimethylformamide to give colorless crystals of the title compound (Mp 270–271°C) [1, 2].

Experimental details

C-bound H atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The nitrogen-bound H atoms was located on a difference Fourier map and refined freely.

Discussion

N^1-thiocarbamoyl-3,5-diaryl-4,5-dihydro-(1*H*)pyrazoles are useful as MAO inhibitors against monoamine oxidases which were isolated and purified from the mitochondrial extracts of rat liver homogenates and human platelets [3]. Corresponding derivatives have antimicrobial and antidepressant activities [4, 5]. In the title compound, $C_{16}H_{13}BrFN_3S$, the asymmetric unit contains only one independent molecule. The central pyrazolyl ring (N1/N2/C7—C9) makes a dihedral angles of 14.71(2)° and 80.41(3)° with the plane of the bromophenyl ring (C1—C6) and the plane of the fluorophenyl ring (C10—C15), respectively. The packing of the structure shows two weak hydrogen bonds between N3—H1N3$\cdots$F1^{i} and C15—H15A$\cdots$Br1^{ii}. (symmetry code: (i) *x*, *y*, *z*-1; (ii) -*x*+1, *y*+1/2, -*z*+1/2.

Acknowledgements: The authors extend their appreciation to the Deanship of Scientific Research and the Research Center, College of Pharmacy, King Saud University for their funding of this research.

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