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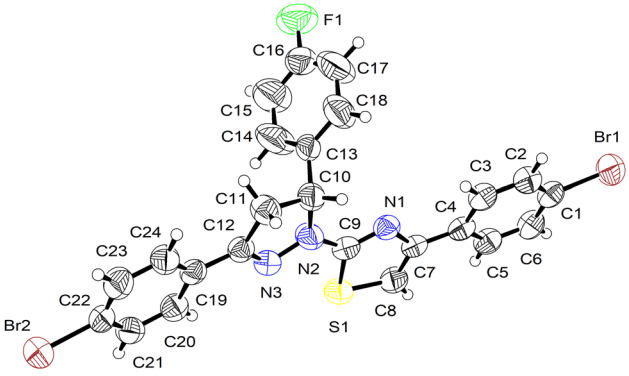
The crystal structure of 4-(4-bromophenyl)-2-(3-(4-bromophenyl)-5-(4-fluorophenyl)-4,5-dihydro-1*H*-pyrazol-1-yl)thiazole, $C_{24}H_{16}Br_2FN_3S$



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Abstract

$C_{24}H_{16}Br_2FN_3S$, monoclinic, $P2_1/n$ (no. 14), a = 14.9517(9) Å, b = 5.4857(3) Å, c = 27.9582(17) Å, β = 102.434(6)°, V = 2239.4(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0444, $wR_{ref}(F^2)$ = 0.1237, T = 296 K.

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The molecular structure is shown in the Figure. 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:	0.48 × 0.16 × 0.03 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	3.74 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	30.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20610, 5655, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3186
$N(param)_{refined}$:	280
Programs:	CrysAlis^{PRO} [1], SHELX [2,3], WinGX/ORTEP [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.7046 (2)	0.1745 (7)	0.31907 (12)	0.0594 (9)
C2	−0.6780 (2)	0.0354 (6)	0.28376 (13)	0.0602 (9)
H2	−0.708105	−0.110059	0.273626	0.072*
C3	−0.6070 (2)	0.1112 (6)	0.26339 (13)	0.0589 (9)
H3	−0.589847	0.016723	0.239233	0.071*
C4	−0.5597 (2)	0.3285 (6)	0.27836 (11)	0.0539 (8)
C5	−0.5887 (3)	0.4639 (6)	0.31446 (12)	0.0612 (9)
H5	−0.558633	0.608682	0.325203	0.073*
C6	−0.6605 (3)	0.3897 (7)	0.33470 (13)	0.0643 (9)
H6	−0.678817	0.483577	0.358569	0.077*
C7	−0.4837 (2)	0.4053 (6)	0.25641 (11)	0.0504 (8)
C8	−0.4252 (3)	0.5922 (7)	0.27169 (13)	0.0652 (9)
H8	−0.428670	0.694161	0.297805	0.078*
C9	−0.3965 (2)	0.3726 (6)	0.20322 (12)	0.0571 (8)
C10	−0.4055 (2)	0.0842 (7)	0.13268 (13)	0.0625 (9)
H10	−0.417361	−0.053416	0.152791	0.075*
C11	−0.3265 (3)	0.0202 (7)	0.10734 (15)	0.0706 (10)
H11A	−0.298022	−0.133208	0.119302	0.085*
H11B	−0.347918	0.010744	0.072081	0.085*
C12	−0.2612 (2)	0.2314 (6)	0.12162 (12)	0.0537 (8)
C13	−0.4930 (2)	0.1594 (6)	0.09792 (12)	0.0585 (9)
C14	−0.4992 (4)	0.3699 (10)	0.0719 (2)	0.126 (2)
H14	−0.448857	0.473421	0.076428	0.151*
C15	−0.5785 (4)	0.4343 (11)	0.0389 (2)	0.127 (2)
H15	−0.580933	0.577966	0.021032	0.152*

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Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C16	−0.6503 (3)	0.2909 (10)	0.03303 (16)	0.0843 (12)
C17	−0.6503 (3)	0.0951 (11)	0.0600 (2)	0.119 (2)
H17	−0.702816	0.000732	0.057266	0.143*
C18	−0.5703 (3)	0.0322 (9)	0.09273 (19)	0.0997 (15)
H18	−0.570569	−0.105721	0.112033	0.120*
C19	−0.1782 (2)	0.2724 (6)	0.10361 (11)	0.0530 (8)
C20	−0.1209 (2)	0.4708 (6)	0.11898 (13)	0.0596 (9)
H20	−0.137362	0.584519	0.140218	0.071*
C21	−0.0408 (2)	0.5014 (7)	0.10335 (13)	0.0627 (9)
H21	−0.003486	0.635080	0.113836	0.075*
C22	−0.0160 (2)	0.3343 (7)	0.07223 (12)	0.0606 (9)
C23	−0.0705 (3)	0.1368 (7)	0.05612 (14)	0.0730 (10)
H23	−0.053277	0.023915	0.034974	0.088*
C24	−0.1516 (3)	0.1082 (7)	0.07185 (13)	0.0652 (10)
H24	−0.189012	−0.024575	0.060787	0.078*
N1	−0.46641 (19)	0.2790 (5)	0.21664 (9)	0.0563 (7)
N2	−0.3645 (2)	0.2870 (6)	0.16439 (11)	0.0701 (8)
N3	−0.28394 (19)	0.3727 (5)	0.15408 (10)	0.0602 (7)
S1	−0.34395 (7)	0.61773 (18)	0.23686 (4)	0.0689 (3)
Br1	−0.80516 (3)	0.06964 (9)	0.34513 (2)	0.08368 (18)
Br2	0.09586 (3)	0.37308 (10)	0.05165 (2)	0.08594 (18)
F1	−0.72759 (19)	0.3550 (6)	0.00048 (11)	0.1255 (11)

Source of material

A mixture of 3-(4-bromophenyl)-5-(4-fluorophenyl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide (0.77 g, 2.0 mmol) and 2-bromo-1-(4-bromophenyl)ethanone (0.56 g, 2.0 mmol) in anhydrous ethanol (10 mL) was stirred under reflux for 3 h. The solid obtained on cooling was filtered, washed with cold ethanol, dried, and recrystallized from dimethylformamide to give colourless crystals (86% yield) of the title compound.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The methine, methylene and hydrogens bonded to sp^2 C atoms were idealized during refinement using options AFIX 13, AFIX 23 and AFIX 43, respectively in the SHELXL-2018 program [3]. The U_{iso} values of the hydrogen atoms were set to $1.2U_{eq}$(C).

Comment

Heterocyclic compounds containing both pyrazole and thiazole moieties have many pharmacological and medicinal applications [5–10]. The syntheses of thiazolyl-pyrazolines are of general interest. They are biologically active and act as antibacterial [11], antifungal [12], and antioxidant [13] agents. The X-ray crystal structures of related compounds have been reported [14–17].

The asymmetric unit of the title structure consists of one molecule. The molecule comprises bromophenyl (A [C1–C6, Br1], D [C19–C24, Br2]), thiazolyl (B [C7–C9, N1, S1]), pyrazolyl (C [C10–C12, N2, N3]) and fluorophenyl (E[C13–C18, F1]) ring systems. In the molecule, *A*, *B*, *C* and *D* are almost coplanar with twist angles: *A*/*B* = 8.9(2)°, *B*/*C* = 11.4(3)° and *C*/*D* = 4.0(3)°. The fluorophenyl ring is almost perpendicular to the *ABCD* plane with a *C*/*E* twist angle of 86.07(13)°.

In the crystal, a weak intermolecular C–Br···F–C interaction is observed with geometry: Br2···F1 = 3.26(2) Å, Br2···F1–C16 = 111.5(9)°. In addition, the planar *ABCD* segments are oriented in an edge-to-face manner to neighbouring molecules related by the 2_1 screw axis parallel to the *b* axis with an angle between the least-squares planes through the *ABCD* segments of 71.8°.

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