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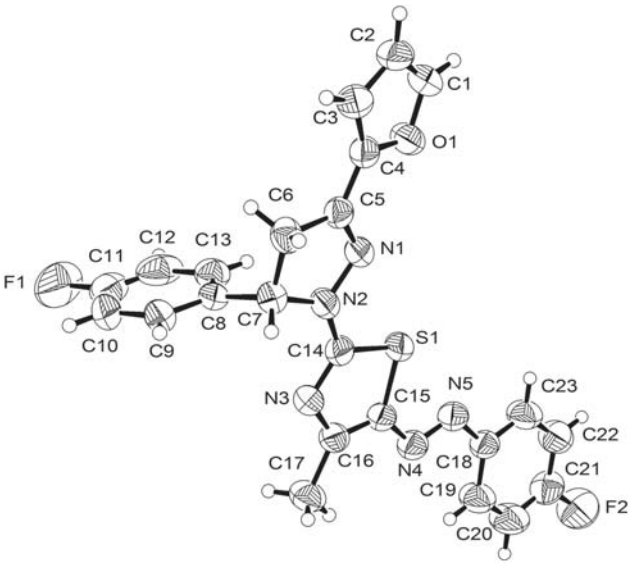
Crystal structure of (*E*)-2-(5-(4-fluorophenyl)-3-(furan-2-yl)-4,5-dihydro-1*H*-pyrazol-1-yl)-5-((4-fluorophenyl)diazenyl)-4-methylthiazole, $C_{23}H_{17}F_2N_5OS$



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Abstract

$C_{23}H_{17}F_2N_5OS$, monoclinic, $P2_1/c$ (no. 14), $a = 5.2272(4)$ Å, $b = 26.7398(15)$ Å, $c = 15.2645(10)$ Å, $\beta = 97.726(7)°$, $V = 2114.2(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0547$, $wR_{ref}(F^2) = 0.1371$, $T = 296(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Orange needle
Size:	0.82 × 0.27 × 0.06 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	2.0 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	59.4°, >84% (99% to 50.4 2*θ)*
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10634, 5088, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3027
$N(param)_{refined}$:	290
Programs:	CrysAlis^{PRO} [14], SHELX [15], PLATON [16]

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized from reaction of a mixture of 1:1 molar ratios of 5-(4-fluorophenyl)-3-(furan-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide and *N′*-(4-fluorophenyl)-2-oxopropanehydrazonoyl chloride in ethanol under reflux condition for 2 h. The solid obtained on cooling was recrystallized from dimethylformamide to give the title compound as orange crystals in 64% yield, Mp. 225–226 °C [1].

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl, methylene and methine C—H bonds were fixed at 0.96 Å, 0.97 Å and 0.98 Å respectively. Displacement parameters were 1.5 times U_{eq}(C) for methyl groups and 1.2 times U_{eq}(C) for methylene and methine hydrogens. Methyl groups were allowed to spin about the C—C bond. Aromatic C—H distances were set to 0.93 Å and their U_{iso} set to 1.2 times U_{eq}(C).

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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}
C1	0.6495(5)	0.04738(10)	0.59780(18)	0.0655(7)
H1	0.7744	0.0224	0.6059	0.079*
C2	0.6361(5)	0.08585(10)	0.65183(18)	0.0649(7)
H2	0.7473	0.0926	0.7033	0.078*
C3	0.4207(5)	0.11438(10)	0.61618(17)	0.0615(7)
H3	0.3617	0.1436	0.6396	0.074*
C4	0.3163(5)	0.09110(8)	0.54141(15)	0.0500(6)
C5	0.0979(5)	0.10124(8)	0.47659(15)	0.0484(5)
C6	−0.0673(5)	0.14694(9)	0.47670(16)	0.0561(6)
H6A	0.0359	0.1772	0.4797	0.067*
H6B	−0.1676	0.1465	0.5257	0.067*
C7	−0.2426(5)	0.14272(8)	0.38738(15)	0.0511(6)
H7	−0.4242	0.1455	0.3963	0.061*
C8	−0.1805(4)	0.18103(8)	0.32111(15)	0.0474(5)
C9	−0.3144(5)	0.22552(9)	0.31469(19)	0.0663(7)
H9	−0.4475	0.2306	0.3485	0.080*
C10	−0.2526(8)	0.26247(12)	0.2587(3)	0.0966(13)
H10	−0.3431	0.2925	0.2542	0.116*
C11	−0.0596(9)	0.25460(16)	0.2104(2)	0.1000(14)
C12	0.0778(7)	0.21132(16)	0.21420(19)	0.0889(11)
H12	0.2106	0.2070	0.1800	0.107*
C13	0.0149(5)	0.17366(10)	0.27052(16)	0.0610(7)
H13	0.1049	0.1436	0.2739	0.073*
C14	−0.3075(5)	0.06521(8)	0.29387(15)	0.0510(6)
C15	−0.4537(5)	0.00457(9)	0.18424(16)	0.0521(6)
C16	−0.5903(5)	0.04815(9)	0.17850(16)	0.0534(6)
C17	−0.8163(5)	0.05989(11)	0.11044(18)	0.0706(8)
H17A	−0.9659	0.0658	0.1391	0.106*
H17B	−0.8485	0.0322	0.0705	0.106*
H17C	−0.7791	0.0892	0.0780	0.106*
C18	−0.3962(5)	−0.11526(9)	0.09743(16)	0.0549(6)
C19	−0.6020(6)	−0.12125(11)	0.0320(2)	0.0775(9)
H19	−0.7214	−0.0956	0.0198	0.093*
C20	−0.6318(7)	−0.16490(13)	−0.0153(2)	0.0914(10)
H20	−0.7712	−0.1691	−0.0594	0.110*
C21	−0.4549(6)	−0.20178(11)	0.00311(19)	0.0728(8)
C22	−0.2510(6)	−0.19752(11)	0.0663(2)	0.0793(9)
H22	−0.1328	−0.2235	0.0777	0.095*
C23	−0.2219(6)	−0.15362(11)	0.1137(2)	0.0751(8)
H23	−0.0816	−0.1499	0.1576	0.090*
N1	0.0261(4)	0.07057(7)	0.41315(13)	0.0538(5)
N2	−0.1851(4)	0.09153(7)	0.36206(13)	0.0564(5)
N3	−0.5072(4)	0.08338(7)	0.24102(13)	0.0548(5)
N4	−0.5024(4)	−0.03614(8)	0.13060(13)	0.0567(5)
N5	−0.3458(4)	−0.07204(8)	0.15071(13)	0.0579(5)
O1	0.4563(3)	0.04935(6)	0.52908(11)	0.0641(5)
F1	0.0032(6)	0.29140(10)	0.15608(16)	0.1654(13)
F2	−0.4863(4)	−0.24579(7)	−0.04261(13)	0.1111(7)
S1	−0.20373(13)	0.00572(2)	0.27253(4)	0.0553(2)

Discussion

Many pyrazolylthiazoles have been synthesized using different procedures and showed antinociceptive, anti-inflammatory and antimicrobial activities [2–10]. The X-ray crystal structures for related compounds have been published recently [11, 12].

The asymmetric unit consists of one molecule. In the molecule, the furan(*A*)-pyrazole(*B*)-thiazole(*C*)-fluorophenyl(D) ring system is almost planar. Thus the largest deviation from the least-squares plane through the four rings is 0.22(1) Å (by O1). The greatest difference between the planes through adjacent rings (A and B) is 7.1(2)°. The second fluorophenyl group (E) is almost perpendicular (85.0(5)°) to the A—B—C—D system. In the crystal, pairs of molecules related by an inversion centre interact through two edge-to-face interactions involving rings D and E with centroid-to-centroid distances of 5.3 Å. A short intermolecular O···O contact (2.84 Å) occurs between furan moieties of pairs of molecules related by inversion symmetry. Such contact is not unique, as shown by a search of the CSD [13] which gave 78 hits for contacts within the sum of van-der-Waals radii for furan oxygens.

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