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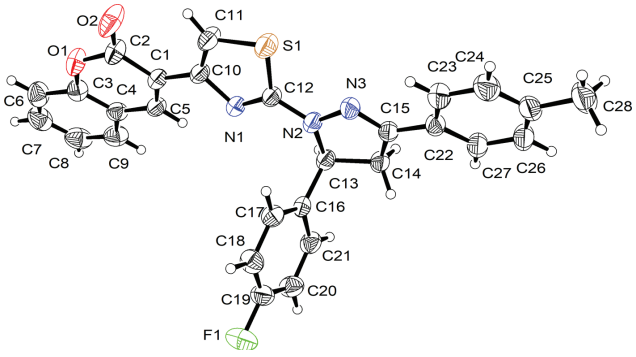
Crystal structure of 3-(2-(5-(4-fluorophenyl)-3-(4-methylphenyl)-4,5-dihydro-1*H*-pyrazol-1-yl) thiazol-4-yl)-2*H*-chromen-2-one, $C_{28}H_{20}FN_3O_2S$



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Abstract

$C_{28}H_{20}FN_3O_2S$, triclinic, $P\bar{1}$ (no. 2), $a = 9.1325(7)$ Å, $b = 11.5184(9)$ Å, $c = 11.6535(9)$ Å, $\alpha = 74.682(7)°$, $\beta = 84.253(6)°$, $\gamma = 76.720(6)°$, $V = 1149.68(15)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0574$, $wR_{ref}(F^2) = 0.1438$, $T = 296(2)$ 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.

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Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.27 × 0.16 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.18 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8916, 5371, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3323
$N(param)_{refined}$:	316
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	0.1169(3)	0.9835(2)	0.2910(2)	0.0435(5)
C2	0.1309(3)	1.1006(2)	0.3110(2)	0.0547(6)
C3	−0.1315(3)	1.1361(2)	0.3741(2)	0.0501(6)
C4	−0.1501(3)	1.0316(2)	0.3454(2)	0.0472(6)
C5	−0.0192(3)	0.9543(2)	0.3071(2)	0.0456(6)
H5	−0.028348	0.880682	0.292655	0.055*
C6	−0.2495(3)	1.2147(3)	0.4163(2)	0.0643(7)
H6	−0.233364	1.282813	0.437781	0.077*
C7	−0.3903(4)	1.1906(3)	0.4259(3)	0.0735(9)
H7	−0.470727	1.242712	0.454459	0.088*
C8	−0.4148(3)	1.0899(3)	0.3938(3)	0.0716(8)
H8	−0.511818	1.076008	0.397977	0.086*
C9	−0.2958(3)	1.0103(3)	0.3556(2)	0.0634(7)
H9	−0.312440	0.941127	0.336331	0.076*
C10	0.2511(3)	0.9040(2)	0.2520(2)	0.0447(5)
C11	0.3852(3)	0.9331(2)	0.2113(2)	0.0584(7)
H11	0.407209	1.009785	0.203622	0.070*
C12	0.3697(3)	0.7263(2)	0.2201(2)	0.0434(5)
C13	0.2927(3)	0.5250(2)	0.2330(2)	0.0438(5)
H13	0.202208	0.567602	0.188277	0.053*
C14	0.3852(3)	0.4229(2)	0.1757(2)	0.0481(6)
H14A	0.387672	0.341846	0.228688	0.058*
H14B	0.344204	0.426431	0.100930	0.058*
C15	0.5387(3)	0.4522(2)	0.15642(19)	0.0438(5)
C16	0.2500(2)	0.47624(19)	0.36272(19)	0.0408(5)
C17	0.3128(3)	0.4993(2)	0.4549(2)	0.0474(6)

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H17	0.383503	0.548820	0.438136	0.057*
C18	0.2720(3)	0.4497(2)	0.5725(2)	0.0548(6)
H18	0.314053	0.465710	0.634665	0.066*
C19	0.1696(3)	0.3774(2)	0.5946(2)	0.0550(6)
C20	0.1048(3)	0.3507(2)	0.5069(2)	0.0589(7)
H20	0.035000	0.300331	0.524899	0.071*
C21	0.1464(3)	0.4010(2)	0.3908(2)	0.0528(6)
H21	0.103771	0.383927	0.329463	0.063*
C22	0.6760(3)	0.3764(2)	0.1166(2)	0.0486(6)
C23	0.8139(3)	0.4104(3)	0.1098(2)	0.0622(7)
H23	0.819715	0.480260	0.133403	0.075*
C24	0.9424(3)	0.3410(3)	0.0681(3)	0.0724(8)
H24	1.033761	0.365002	0.063679	0.087*
C25	0.9373(4)	0.2365(3)	0.0328(2)	0.0667(8)
C26	0.8015(4)	0.2030(3)	0.0418(2)	0.0683(8)
H26	0.796548	0.132282	0.019511	0.082*
C27	0.6710(3)	0.2712(2)	0.0831(2)	0.0579(7)
H27	0.580369	0.245967	0.088105	0.069*
C28	1.0790(4)	0.1634(3)	−0.0156(3)	0.0968(12)
H28A[a]	1.162152	0.201258	−0.014856	0.145*
H28B[a]	1.099749	0.080646	0.033328	0.145*
H28C[a]	1.064672	0.162218	−0.095685	0.145*
H28D[a]	1.055563	0.094824	−0.036619	0.145*
H28E[a]	1.117966	0.215436	−0.084803	0.145*
H28F[a]	1.153043	0.133863	0.044209	0.145*
N1	0.2415(2)	0.78356(16)	0.25686(16)	0.0446(5)
N2	0.3994(2)	0.60672(17)	0.21527(17)	0.0482(5)
N3	0.5421(2)	0.55653(17)	0.17574(16)	0.0468(5)
O1	0.00688(19)	1.16673(15)	0.35960(17)	0.0607(5)
O2	0.2400(2)	1.14491(18)	0.2892(2)	0.0850(7)
S1	0.50975(8)	0.81025(6)	0.17575(7)	0.0622(2)
F1	0.1273(2)	0.33016(17)	0.70997(13)	0.0830(5)

[a]Occupancy: 0.5.

Source of material

The title compound was synthesized from the reaction of equimolar quantities of 5-(4-fluorophenyl)-3-(4-methylphenyl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide and 3-(2-bromoacetyl)-2*H*-chromen-2-one in anhydrous ethanol under reflux for 2 h. The crude product obtained was collected by filtration, washed with ethanol and recrystallized from dimethylformamide to give colourless crystals (88%).

Experimental details

The electron density for the methyl group hydrogens was distributed over several positions and so these hydrogens were modeled in six positions of equal (0.5) occupancy around the methyl C atom. All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl C—H bonds were fixed at 0.96 Å, with displacement parameters 1.5 times U_{eq}(C). C—H distances for sp^2 hybridized groups were set to 0.93 Å and their U_{iso}(H) set to 1.2 times the U_{eq}(C). Methylene C—H bond distances were set to 0.97 Å and U_{iso}(H) set to 1.2 times the U_{eq}(C). The methine C—H bond distance was set to 0.98 Å and U_{iso}(H) set to 1.2 times the U_{eq}(C). The high R1 value for all reflections is attributable to the weakness of high angle data, particularly above 0.86 Å resolution.

Comment

The synthesis of novel heterocycles containing thiazolyl-pyrazoline moieties are of interest since such compounds show a range of biological and medicinal applications [5–9]. In addition, coumarinyl-thiazole containing heterocycles showed interesting applications [10–12]. Related structures have been reported [13, 14].

In the crystal structure, the asymmetric unit consists of one molecule (see the figure). The molecule comprises five ring systems, namely: **A**, chromenonyl (C1—C9,O1,O2); **B**, thiazolyl (C10—C12,N1,S1); **C**, pyrazolyl (C13—C15,N2,N3); **D**, tolyl (C22—C28) and **E**, fluorophenyl (C16—C21,F1) groups. Rings **A** to **D** are almost co-planar with interplanar angles **A**/**B**, **B**/**C**, **C**/**D** of 15.50(8)°, 12.59(11)° and 7.02(13)° respectively. The angle between **C** and **E** is 67.40(9)°.

The molecule displays an almost weak intramolecular C—H···O contact with a C···O distance of 2.824(3) Å and a C11—H11···O2 angle of 117.3°. In the crystal structure, a close intermolecular C—H···N contact with a C···N distance of 3.511(3) Å and C18—H18···N3 angle of 163.8° is also observed. Fluorophenyl groups of neighbouring pairs of molecules are parallel with a ring centroid separation of 3.84 Å. Chromenonyl groups of adjacent molecules are also parallel with a fused-ring centroid-to-centroid distance of 4.164 Å. Lack of strong directional interactions or steric constraints account for the rotational disorder in the methyl group.

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