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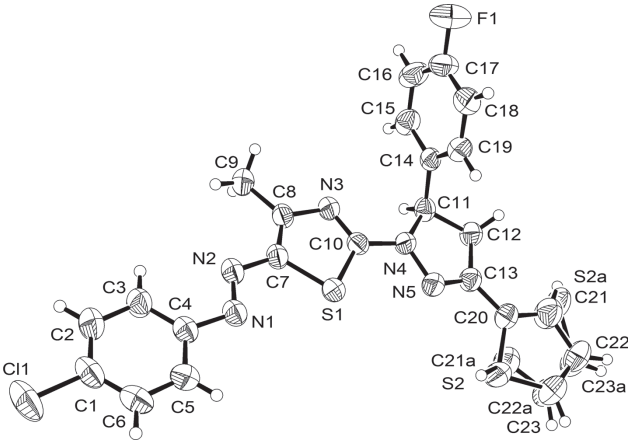
Crystal structure of (*E*)-5-((4-chlorophenyl) diazenyl)-2-(5-(4-fluorophenyl)-3-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazol-1-yl)-4-methylthiazole, $C_{23}H_{17}ClFN_5S_2$



DOI 10.1515/ncrs-2016-0208
Received July 5, 2016; accepted November 16, 2016; available online December 10, 2016

Abstract

$C_{23}H_{17}ClFN_5S_2$, monoclinic, $P2_1/c$ (no. 14), $a = 20.9691(12)$ Å, $b = 11.5316(6)$ Å, $c = 9.2546(4)$ Å, $\beta = 95.484(4)°$, $V = 2227.6(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0468$, $wR_{ref}(F^2) = 0.1126$, $T = 296$ K.

CCDC no.: 1517483

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Table 1: Data collection and handling.

Crystal:	Orange needle
Size:	0.31 × 0.22 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.9 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	59.6°, >83% (>99% for $2\theta_{max} = 50°$)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11261, 5322, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3684
$N(param)_{refined}$:	327
Programs:	CrysAlis^{PRO} [8], SHELX [9], Platon [10]

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 by the reaction of 5-(4-fluorophenyl)-3-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide and 1-chloro-1-((4-chlorophenyl)diazenyl) propan-2-one in ethanol containing two drops of triethylamine under reflux for 1.5 h. The solid obtained was filtered, dried and recrystallized from dimethylformamide to give orange crystals of the title compound (Mp 212–213 °C) [1].

Experimental details

The thiophenyl moiety is disordered and was refined with a ratio of 34.1(3)/65.9(3) with restrained geometry. All H atoms were placed in calculated positions and refined using a riding model. For the methyl groups, C—H bonds were fixed at 0.96 Å and U_{iso} set to $1.5U_{eq}$(C) with free rotation around the C—C bond (HFIX 137 in SHELX [9]). For the rest of the hydrogens, U_{iso} was set to $1.2U_{eq}$(C) with a distance of 0.93 Å for aromatic C—H. For CH and CH_2 groups, the C—H distances were set to 0.98 and 0.97 Å respectively.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.56009(11)	0.2098(2)	−0.0311(2)	0.0600(6)
C2	0.53105(12)	0.1177(2)	−0.1040(3)	0.0651(7)
H2	0.5460	0.0428	−0.0850	0.078*
C3	0.47962(11)	0.1365(2)	−0.2056(2)	0.0590(6)
H3	0.4592	0.0740	−0.2540	0.071*
C4	0.45845(10)	0.24837(19)	−0.2355(2)	0.0465(5)
C5	0.48719(11)	0.3395(2)	−0.1591(2)	0.0579(6)
H5	0.4721	0.4145	−0.1768	0.070*
C6	0.53831(11)	0.3206(2)	−0.0558(3)	0.0663(7)
H6	0.5576	0.3824	−0.0039	0.080*
C7	0.33549(9)	0.21842(18)	−0.5178(2)	0.0430(5)
C8	0.30299(9)	0.13926(18)	−0.6074(2)	0.0431(4)
C9	0.31527(11)	0.01195(19)	−0.6067(3)	0.0584(6)
H9A	0.2752	−0.0288	−0.6211	0.088*
H9B	0.3373	−0.0101	−0.5151	0.088*
H9C	0.3412	−0.0072	−0.6834	0.088*
C10	0.25614(9)	0.29811(17)	−0.6930(2)	0.0403(4)
C11	0.17002(9)	0.31616(17)	−0.8977(2)	0.0427(4)
H11	0.1951	0.2697	−0.9605	0.051*
C12	0.14817(10)	0.42874(18)	−0.9765(2)	0.0472(5)
H12A	0.1592	0.4285	−1.0760	0.057*
H12B	0.1023	0.4397	−0.9765	0.057*
C13	0.18456(10)	0.52095(17)	−0.8887(2)	0.0442(5)
C14	0.11712(9)	0.24294(16)	−0.84667(19)	0.0400(4)
C15	0.10958(11)	0.12884(18)	−0.8897(2)	0.0536(5)
H15	0.1383	0.0959	−0.9483	0.064*
C16	0.05969(13)	0.0630(2)	−0.8464(3)	0.0686(7)
H16	0.0542	−0.0138	−0.8756	0.082*
C17	0.01877(12)	0.1139(2)	−0.7598(3)	0.0680(7)
C18	0.02503(12)	0.2242(2)	−0.7120(3)	0.0653(6)
H18	−0.0033	0.2556	−0.6512	0.078*
C19	0.07501(10)	0.28905(19)	−0.7565(2)	0.0516(5)
H19	0.0804	0.3653	−0.7249	0.062*
S1	0.31014(2)	0.36029(4)	−0.56204(5)	0.04500(14)
S2[a]	0.23680(8)	0.74034(13)	−0.84240(19)	0.0727(5)
C20[a]	0.18176(10)	0.64294(18)	−0.9238(2)	0.0504(5)
C21[a]	0.1410(4)	0.6970(7)	−1.0207(9)	0.0762(18)
H21[a]	0.1096	0.6578	−1.0794	0.091*
C22[a]	0.1492(4)	0.8222(5)	−1.0277(11)	0.0721(18)
H22[a]	0.1239	0.8734	−1.0857	0.087*
C23[a]	0.1998(4)	0.8499(5)	−0.9357(12)	0.0766(18)
H23[a]	0.2138	0.9261	−0.9238	0.092*
S2A[b]	0.1231(2)	0.6918(4)	−1.0543(5)	0.0716(9)
C20A[b]	0.18176(10)	0.64294(18)	−0.9238(2)	0.0504(5)
C21A[b]	0.2191(5)	0.7290(8)	−0.8785(13)	0.072(2)
H21A[b]	0.2516	0.7186	−0.8039	0.086*
C22A[b]	0.2082(9)	0.8435(8)	−0.949(2)	0.075(2)
H22A[b]	0.2311	0.9119	−0.9309	0.090*
C23A[b]	0.1577(9)	0.8248(9)	−1.045(2)	0.074(3)
H23A[b]	0.1416	0.8840	−1.1060	0.089*
N1	0.40778(8)	0.27833(16)	−0.34273(17)	0.0477(4)
N2	0.38302(8)	0.19125(15)	−0.41187(17)	0.0461(4)
N3	0.25676(8)	0.18466(14)	−0.70601(17)	0.0418(4)
N4	0.21285(8)	0.36295(14)	−0.77539(17)	0.0443(4)
N5	0.22023(8)	0.48225(14)	−0.77839(18)	0.0460(4)
F1	−0.03037(9)	0.04929(17)	−0.7155(2)	0.1153(7)
Cl1	0.62511(3)	0.18532(9)	0.09640(8)	0.0954(3)

[a]Occupancy: 0.659(3); [b]Occupancy: 0.341(3).

Discussion

Compounds bearing thiazole and pyrazole nuclei are found to have various antimicrobial activities such as antibacterial, antifungal and antitumor [1–7].

The asymmetric unit (*cf.* the figure) of the crystal structure consists of a molecule of $C_{23}H_{17}ClFN_5S_2$ with a disordered thiophenyl fragment. Apart from the fluorobenzene group, the rest of the molecule is essentially planar; the angles between the planes of neighbouring rings are 13° or less. The plane of the fluorobenzene is perpendicular (90.71(5)°) to the plane of the pyrazolinyl group. In the crystal, the molecules stack along [001] with interplanar distances of about 3.5 Å between glide-plane related molecules.

Acknowledgements: The authors extend their appreciation to the College of Applied Medical Sciences Research Centre and the Deanship of Scientific Research at King Saud University for funding and to Cardiff University for continued support.

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