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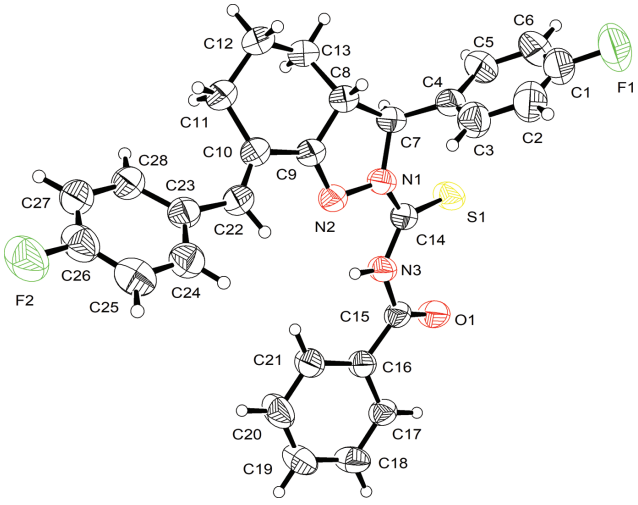
The crystal structure of *N*-(7-(4-fluorobenzylidene)-3-(4-fluorophenyl)-3, 3*a*,4,5,6,7-hexahydro-2*H*-indazole-2-carbonothioyl)benzamide, $C_{28}H_{23}F_2N_3OS$



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Abstract

$C_{28}H_{23}F_2N_3OS$, monoclinic, *I*2/*a* (no. 15), $a = 20.3481(8)$ Å, $b = 10.2647(4)$ Å, $c = 23.6975(11)$ Å, $\beta = 105.317(5)°$, $V = 4773.8(4)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0489$, $wR_{ref}(F^2) = 0.1543$, $T = 296(2)$ K.

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The crystal 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 block
Size:	0.27 × 0.26 × 0.12 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.18 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
θ_{max}, completeness:	29.7°, >93% (up to 25.2, >99%)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	39559, 6270, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3722
$N(param)_{refined}$:	264
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX and ORTEP [4]

Source of materials

The title compound was synthesized from reaction of an equimolar mixture of 7-(4-fluorobenzylidene)-3-(4-fluorophenyl)-3,3*a*,4,5,6,7-hexahydro-2*H*-indazole and benzoyl isothiocyanate in anhydrous ethanol under reflux for 2 h. The crude product was recrystallized from dimethylformamide to give colourless crystals in 82% yield (Mp. 246–248 °C).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. The N—H bond was fixed at 0.86 Å (AFIX 43 instruction in SHELXL [2, 3]), with displacement parameters set to 1.2 times U_{eq}(N). C—H distances for sp^2 carbon atoms were set to 0.93 Å (AFIX 43) and U_{iso}(H) set to 1.2 times U_{eq}(C). The methine C—H distance was set to 0.98 Å (AFIX 13) and U_{iso}(H) set to 1.2 times U_{eq}(C). The methylene C—H distances were set to 0.97 Å (AFIX 23) and U_{iso}(H) set to 1.2 times U_{eq}(C). The phenyl ring is disordered and was refined with restrained geometry to form regular hexagons and restrained displacement parameters

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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	0.29286(13)	−0.5248(3)	0.24724(15)	0.1024(9)
C2	0.30439(13)	−0.3979(3)	0.25847(12)	0.0997(8)
H2	0.303263	−0.362985	0.294425	0.120*
C3	0.31807(12)	−0.3193(3)	0.21515(11)	0.0848(6)
H3	0.326086	−0.230924	0.222449	0.102*
C4	0.32004(10)	−0.3690(2)	0.16194(10)	0.0709(5)
C5	0.30884(14)	−0.5001(2)	0.15249(13)	0.0966(8)
H5	0.310596	−0.536500	0.116986	0.116*
C6	0.29482(16)	−0.5791(3)	0.19591(17)	0.1128(10)
H6	0.286958	−0.667787	0.189478	0.135*
C7	0.33851(10)	−0.28349(19)	0.11630(9)	0.0673(5)
H7	0.334939	−0.333695	0.080434	0.081*
C8	0.41031(9)	−0.2236(2)	0.13749(9)	0.0687(5)
H8	0.426561	−0.237276	0.179833	0.082*
C9	0.39820(9)	−0.08113(19)	0.12669(9)	0.0656(5)
C10	0.45453(9)	0.0119(2)	0.13546(9)	0.0672(5)
C11	0.52028(10)	−0.0441(2)	0.12734(11)	0.0796(6)
H11A	0.558128	0.000816	0.153653	0.095*
H11B	0.522371	−0.025831	0.087694	0.095*
C12	0.52988(10)	−0.1910(2)	0.13815(11)	0.0801(6)
H12A	0.566937	−0.221034	0.122779	0.096*
H12B	0.542544	−0.207232	0.179959	0.096*
C13	0.46581(10)	−0.2688(2)	0.10973(10)	0.0767(6)
H13A	0.452123	−0.253247	0.067875	0.092*
H13B	0.474220	−0.361245	0.116397	0.092*
C14	0.22965(9)	−0.16202(18)	0.07109(9)	0.0630(5)
C15	0.14029(8)	0.00507(18)	0.03167(9)	0.0616(5)
C16[a]	0.1329(3)	0.1480(3)	0.0181(3)	0.061(2)
C17[a]	0.0671(2)	0.1981(6)	0.0045(4)	0.064(2)
H17[a]	0.030541	0.143861	0.004547	0.076*
C18[a]	0.05606(16)	0.3294(6)	−0.0090(3)	0.081(4)
H18[a]	0.012057	0.362997	−0.018094	0.097*
C19[a]	0.1107(2)	0.4106(4)	−0.0090(2)	0.0849(16)
H19[a]	0.103339	0.498396	−0.018125	0.102*
C20[a]	0.17651(17)	0.3604(3)	0.0045(2)	0.0951(17)
H20[a]	0.213106	0.414661	0.004487	0.114*
C21[a]	0.18759(17)	0.2291(3)	0.0181(2)	0.0785(14)
H21[a]	0.231592	0.195525	0.027129	0.094*
C16A[b]	0.1313(3)	0.1489(5)	0.0294(4)	0.0619(19)
C17A[b]	0.0700(3)	0.1954(8)	−0.0060(5)	0.067(2)
H17A[b]	0.038356	0.137217	−0.027968	0.080*
C18A[b]	0.0557(3)	0.3266(8)	−0.0090(4)	0.081(4)
H18A[b]	0.014729	0.356630	−0.033024	0.097*
C19A[b]	0.1018(3)	0.4130(5)	0.0235(3)	0.0840(17)
H19A[b]	0.091479	0.501391	0.022123	0.101*
C20A[b]	0.1631(2)	0.3696(4)	0.0580(3)	0.0930(19)
H20A[b]	0.194948	0.428809	0.078834	0.112*
C21A[b]	0.1774(2)	0.2374(4)	0.0616(3)	0.0762(15)
H21A[b]	0.218433	0.208039	0.085959	0.091*
C22	0.44478(10)	0.1351(2)	0.15006(9)	0.0687(5)
H22	0.400694	0.153165	0.152123	0.082*
C23	0.49145(10)	0.2462(2)	0.16333(8)	0.0669(5)
C24	0.47536(12)	0.3456(2)	0.19732(9)	0.0787(6)
H24	0.435538	0.339254	0.209383	0.094*
C25	0.51670(14)	0.4534(2)	0.21371(11)	0.0895(7)
H25	0.505255	0.518707	0.236592	0.107*
C26	0.57466(13)	0.4613(2)	0.19542(11)	0.0864(7)
C27	0.59160(11)	0.3698(2)	0.16056(11)	0.0842(6)
H27	0.630873	0.379194	0.147888	0.101*
C28	0.55015(10)	0.2624(2)	0.14399(10)	0.0762(6)
H28	0.561429	0.200044	0.119646	0.091*
N1	0.29616(7)	−0.16351(15)	0.10253(7)	0.0657(4)
N2	0.33473(7)	−0.04890(15)	0.10742(7)	0.0660(4)
N3	0.20597(7)	−0.03631(15)	0.05803(7)	0.0664(4)
H3A	0.236086	0.024224	0.067574	0.080*
O1	0.09265(7)	−0.06788(14)	0.01683(8)	0.0878(5)
S1	0.18611(3)	−0.29703(5)	0.05185(3)	0.0797(2)
F1	0.27951(10)	−0.6031(2)	0.28933(9)	0.1500(8)
F2	0.61599(8)	0.56611(15)	0.21233(8)	0.1208(6)

Occupancies: [a] = 0.521(5), [b] = 0479(5).

(DFIX,SIMU) to give final occupancies of 0.479(5) and 0.521(5) for the two components.

Discussion

Many pyrazole derivatives have been synthesized and found to display antimicrobial, antiviral, anti-inflammatory, insecticidal and herbicidal activities [5–8]. The structure of the title compound has been obtained as part of a study of these materials [9].

The asymmetric unit consists of one molecule of the title compound (see the figure). The molecule consists of two fluorophenyl groups [**A** (F1, C1—C6) and **D** (F2, C23—C28)], a hexahydroindazolyl group [**B** (N1, N2, C7—C13)] and a phenyl group [**C** (C16—C21)]. Ring **C** is disordered by a rotation of 35.3(3)° about the C15—C16 bond. The cyclohexane segment (C8—C13) of **B** assumes a chair conformation. The pyrazolyl ring of **B** is planar with a maximum deviation of 0.037(3) Å from the least squares plane of the ring. Related structures include *cis*-1-benzoyl-2-chloroacetyl-4,5-hexamethylenepyrazolidine [10] and 2-benzyl 3-ethyl 6a-methyl 1-(benzoylcarbamothioyl)hexahydrocyclopenta[c] pyrazole-2,3,6a(1H)-tricarboxylate [11] in which the pyrazolyl rings are in envelope conformation. An intramolecular N—H···N contact is observed (N3···N2 distance of 2.575(2) Å; N3—H3A···N2 angle of 112.8°). Two molecules related by inversion symmetry are linked by weak non-classical C—H···O interactions (C17···O1 distance of 3.427(3) Å and C17—H17···O1 angle of 158.4°) to form $R^2_2(10)$ rings. Comparably weak C—H···S interactions also occur in the structure with C27···S1 distances of 3.676(3) Å and C27—H27···S1 angles of 139.7°.

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