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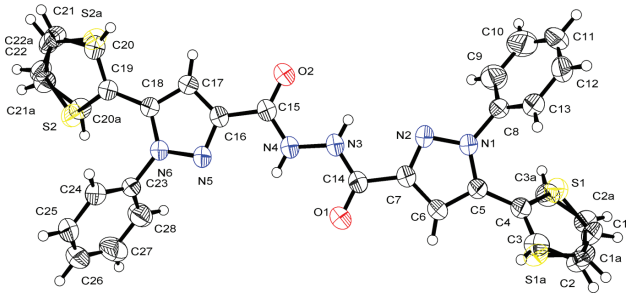
Crystal structure of 1-phenyl-*N'*-(1-phenyl-5-(thiophen-2-yl)-1*H*-pyrazole-3-carbonyl)-5-(thiophen-2-yl)-1*H*-pyrazole-3-carbohydrazide, $C_{28}H_{20}N_6O_2S_2$



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Abstract

$C_{28}H_{20}N_6O_2S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.6738(6)$ Å, $b = 11.7869(7)$ Å, $c = 12.5381(7)$ Å, $\alpha = 112.842(6)°$, $\beta = 91.963(4)°$, $\gamma = 116.129(6)°$, $V = 1264.38(15)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0523$, $wR_{ref}(F^2) = 0.1390$, $T = 296(2)$ K.

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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 an equimolar mixture of 1-phenyl-5-(thiophen-2-yl)-1*H*-pyrazole-3-carbohydrazide and 2-(methoxymethylene)malononitrile in ethanol under reflux for 1.5 h. The solid obtained on cooling was collected by filtration, dried and recrystallized

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Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.30 × 0.19 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.25 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10719, 6002, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4380
$N(param)_{refined}$:	417
Programs:	CrysAlis^{PRO} [9], SHELX [10], WinGX [11]

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

Atom	x	y	z	U_{iso}*/U_{eq}
S1[a]	0.53465(11)	0.34985(13)	0.71518(12)	0.0664(4)
C1[a]	0.4846(5)	0.4560(5)	0.8228(4)	0.0627(12)
H1[a]	0.549379	0.537074	0.891002	0.075*
C2[a]	0.3408(5)	0.4085(6)	0.7972(5)	0.0651(11)
H2[a]	0.292964	0.450287	0.843816	0.078*
C3[a]	0.2758(8)	0.2832(11)	0.6862(9)	0.0656(16)
H3[a]	0.177161	0.234682	0.652859	0.079*
C4[a]	0.3610(2)	0.2385(2)	0.63239(17)	0.0466(5)
S1A[b]	0.2424(7)	0.2796(10)	0.6921(9)	0.0688(14)
C1A[b]	0.3838(13)	0.4266(18)	0.8129(15)	0.057(3)
H1A[b]	0.369843	0.491069	0.875773	0.069*
C2A[b]	0.5156(14)	0.4382(16)	0.8067(13)	0.059(3)
H2A[b]	0.602666	0.507921	0.862585	0.070*
C3A[b]	0.4940(12)	0.3221(17)	0.6974(14)	0.067(3)
H3A[b]	0.569287	0.307032	0.674116	0.081*
C4A[b]	0.3610(2)	0.2385(2)	0.63239(17)	0.0466(5)
C5	0.3135(2)	0.10572(19)	0.52627(17)	0.0421(4)
C6	0.2007(2)	−0.0253(2)	0.49954(18)	0.0473(5)
H6	0.134030	−0.048065	0.544076	0.057*
C7	0.2068(2)	−0.11664(19)	0.39219(18)	0.0447(4)
C8	0.5003(2)	0.19227(19)	0.41823(16)	0.0425(4)
C9	0.6187(3)	0.1766(3)	0.4017(2)	0.0677(7)
H9	0.621872	0.099208	0.402195	0.081*
C10	0.7305(3)	0.2750(4)	0.3846(3)	0.0898(9)
H10	0.811686	0.265428	0.373942	0.108*
C11	0.7275(3)	0.3892(3)	0.3826(2)	0.0809(9)
H11	0.806201	0.456831	0.371479	0.097*
C12	0.6079(3)	0.4026(2)	0.3970(2)	0.0694(7)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H12	0.604777	0.479025	0.394240	0.083*
C13	0.4937(2)	0.3062(2)	0.41518(18)	0.0508(5)
H13	0.412489	0.315899	0.425448	0.061*
C14	0.1114(2)	−0.2710(2)	0.32795(19)	0.0482(5)
C15	0.0942(2)	−0.5481(2)	0.06679(19)	0.0493(5)
C16	0.0018(2)	−0.70304(19)	0.00660(18)	0.0452(5)
C17	0.0174(2)	−0.7993(2)	−0.09108(18)	0.0478(5)
H17	0.085527	−0.779631	−0.135102	0.057*
C18	−0.0892(2)	−0.93014(19)	−0.10910(16)	0.0421(4)
S2[c]	−0.1851(3)	−1.21667(18)	−0.1788(2)	0.0564(5)
C19[c]	−0.1175(2)	−1.0688(2)	−0.19663(17)	0.0440(4)
C20[c]	−0.0800(17)	−1.0914(14)	−0.3047(9)	0.057(2)
H20[c]	−0.042354	−1.022008	−0.330743	0.068*
C2[c]	−0.1048(12)	−1.2311(7)	−0.3716(8)	0.0511(13)
H21[c]	−0.087427	−1.264754	−0.446696	0.061*
C22[c]	−0.1571(10)	−1.3091(8)	−0.3113(5)	0.0500(13)
H22[c]	−0.175656	−1.401789	−0.338654	0.060*
S2A[d]	−0.0696(7)	−1.0881(6)	−0.3229(4)	0.0560(8)
C19A[d]	−0.1175(2)	−1.0688(2)	−0.19663(17)	0.0440(4)
C20A[d]	−0.1586(18)	−1.1886(11)	−0.1788(14)	0.059(3)
H20A[d]	−0.179414	−1.193369	−0.108754	0.071*
C21A[d]	−0.1649(17)	−1.3030(13)	−0.2809(9)	0.056(2)
H21A[d]	−0.200408	−1.394590	−0.289816	0.067*
C22A[d]	−0.1120(19)	−1.2596(10)	−0.3630(12)	0.054(2)
H22A[d]	−0.100014	−1.316450	−0.433204	0.064*
C23	−0.2811(2)	−1.00608(19)	−0.00114(17)	0.0431(4)
C24	−0.4113(2)	−1.0925(2)	−0.0840(2)	0.0565(6)
H24	−0.423716	−1.086218	−0.154914	0.068*
C25	−0.5238(3)	−1.1892(2)	−0.0603(2)	0.0696(7)
H25	−0.613023	−1.247945	−0.115284	0.083*
C26	−0.5048(3)	−1.1988(3)	0.0434(3)	0.0768(8)
H26	−0.580538	−1.264818	0.058451	0.092*
C27	−0.3737(3)	−1.1112(3)	0.1256(3)	0.0794(8)
H27	−0.361020	−1.118217	0.196119	0.095*
C28	−0.2615(3)	−1.0134(3)	0.1042(2)	0.0625(6)
H28	−0.173220	−0.952913	0.160305	0.075*
N1	0.38018(17)	0.08720(15)	0.43565(14)	0.0425(4)
N2	0.31608(18)	−0.05003(16)	0.35200(15)	0.0463(4)
N3	0.14238(19)	−0.33627(16)	0.22880(16)	0.0553(5)
H3B	0.211634	−0.288734	0.204268	0.066*
N4	0.0618(2)	−0.48181(17)	0.16540(17)	0.0599(5)
H4	−0.008465	−0.529077	0.189308	0.072*
N5	−0.10724(18)	−0.76601(16)	0.04906(15)	0.0467(4)
N6	−0.16261(17)	−0.90569(15)	−0.02352(14)	0.0435(4)
O1	0.01181(19)	−0.33486(16)	0.36309(17)	0.0776(6)
O2	0.1941(2)	−0.48422(16)	0.03243(17)	0.0810(6)

Occupancies: [a] = 0.768(3); [b] = 0.232(3); [c] = 0.609(3); [d] = 0.391(3).

from dimethylformamide to colourless crystals (Mp. > 300 °C; 67%).

Experimental details

The two thiophenyl groups are disordered and the occupancies refined to 0.768(3)/0.232(3) and 0.609(3)/0.391(3). Both components of either disordered group were refined with similar geometry and displacement parameters (SAME, SIMU and ISOR instructions in SHELXL [10]). The hydrogen atoms were placed in calculated positions (AFIX 43 instruction in SHELXL [10]) with the U_{iso} values set to 1.2 U_{eq}(C, N).

Comment

N,N′-Diacylhydrazines have been synthesized efficiently using various synthetic approaches as biologically active compounds [1–4]. Also, they show fungicidal and herbicidal activities [5–7]. The X-ray crystal structure for a similar compound has been published [8].

The asymmetric unit comprises one molecule in which both thiophenyl groups are disordered. The two thiophenyl groups are twisted from the least-squares plane of the pyrazole-carbohydrazide group by 45.3(3)° and 31.1(2)° whereas the two phenyl groups are twisted by 53.1(8)° and 59.8(1)°. The pyrazole-carbohydrazide groups of the molecules form planes parallel to $(1\bar{1}1)$ in the crystal. The molecules are arranged in pairs which are linked by edge-to-face contacts between phenyl groups with centroid-to-centroid distances of 4.88 Å. The separation between the planes of the pyrazole-carbohydrazide groups of adjacent molecules is 3.46 Å.

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