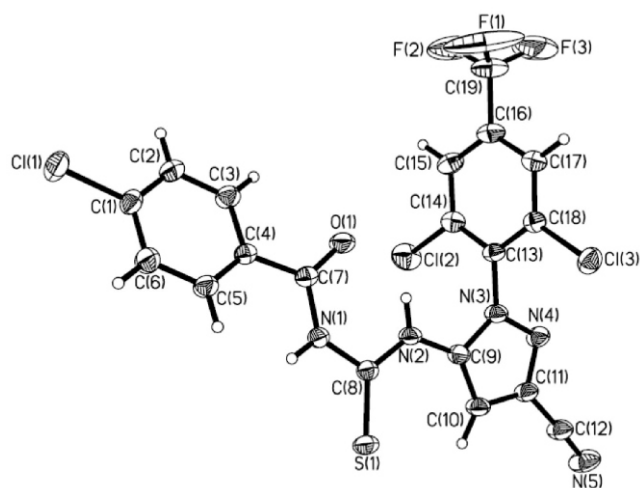


Crystal structure of *N*-[1-(2,6-dichloro-4-trifluoromethyl)-phenyl]-3-cyano-1*H*-pyrazol-5-yl]-*N'*-4-chlorobenzoyl-thiourea, C₁₉H₉Cl₃F₃N₅OS

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Abstract

C₁₉H₉Cl₃F₃N₅OS, monoclinic, *P*2₁/*n* (no. 14), *a* = 16.973(2) Å, *b* = 7.3779(7) Å, *c* = 17.346(2) Å, β = 94.280(2)°, *V* = 2166.0 Å³, *Z* = 4, *R*_g(*F*) = 0.074, *wR*_{ref}(*F*²) = 0.177, *T* = 298 K.

Source of material

Following the method in [1], reaction of 2,6-dichloro-4-trifluoromethylamine (0.01 mol) with a suspension of nitrosyl sulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate (0.01 mol) in acetic acid, gave 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-pyrazole (about 0.005 mol, product 1). Furthermore, according to the method in [2], 4-chlorobenzoyl chloride (0.007 mol) with dry potassium thiocyanate (0.1 mol) was refluxed in anhydrous CH₃CN for two hours at 80 °C and was then filtrated to obtain acylisothiocyanate solution (product 2). The products 1 and 2 were then reacted in anhydrous CH₃CN for about four hours to get the title compound.

Experimental Details

Single crystals suitable for X-ray analysis were obtained by slow evaporation of the solution of acetone (m.p. 482 – 483 K).

IR and NMR data are available in the CIF file.

Discussion

Acyl-thiourea derivatives have attracted much attention due to their chemical properties and biological activity. For example, some thiourea derivatives have been found to be useful as herbicides, inhibitors, anti-HIV and plant-growth regulators [3–6].

Furthermore, the pyrazoles with the functional groups of chloride and trifluoromethyl, like 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulphenylpyrazole or 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonyl pyrazole, show good bioactivity [1]. So we envisioned the acylthiourea derivatives bearing pyrazole moiety might have high biological activity.

The title compound is an acyl-thiourea with an overall U-shape. In the crystal structure, the dihedral angles between the pyrazole and attached phenyl ring and the chlorophenyl are 73.04(1)° and 29.51(1)°, respectively. An intramolecular N2–H2...O1 hydrogen bond with an *d*(N2...O1) = 2.604(4) Å additionally stabilizes the molecule.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.24 × 0.25 × 0.34 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	5.68 cm ^{−1}
Diffractometer, scan mode:	Bruker APEX, φ and ω
2θ _{max} :	50.04°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	10964, 3810
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3387
<i>N</i> (<i>param</i>) _{refined} :	289
Programs:	SHELXS-97, SHELXL-97, SHELXTL [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.8603	0.6914	0.0592	0.050
H(2)	4e	0.9411	0.7291	0.2298	0.053
H(2A)	4e	1.1568	0.7770	−0.0690	0.061
H(3)	4e	1.1085	0.7436	0.0509	0.058
H(5)	4e	0.8873	0.7737	−0.0447	0.058
H(6)	4e	0.9350	0.8150	−0.1644	0.067
H(10)	4e	0.7701	0.5039	0.3135	0.057
H(15)	4e	1.1825	0.7134	0.3189	0.064
H(17)	4e	1.0899	1.1616	0.4066	0.054

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
S(1)	4e	0.76040(6)	0.5722(2)	0.15844(7)	0.0323(6)	0.089(1)	0.0489(7)	−0.0124(6)	0.0022(5)	0.0012(6)
O(1)	4e	1.0141(2)	0.7530(5)	0.1520(2)	0.039(2)	0.087(2)	0.043(2)	−0.015(2)	−0.001(1)	0.006(2)
F(1)	4e	1.2254(3)	1.1789(9)	0.3240(6)	0.066(3)	0.180(6)	0.49(1)	−0.040(3)	0.015(5)	0.204(8)
F(2)	4e	1.2817(2)	0.9446(7)	0.3424(4)	0.045(2)	0.143(4)	0.301(8)	−0.016(2)	0.043(3)	−0.004(5)
F(3)	4e	1.2521(4)	1.087(2)	0.4293(5)	0.138(5)	0.46(2)	0.198(7)	−0.204(8)	0.032(5)	−0.117(8)
N(1)	4e	0.8896(2)	0.6918(5)	0.1018(2)	0.035(2)	0.057(2)	0.032(2)	−0.007(2)	0.000(1)	0.003(2)
N(2)	4e	0.8958(2)	0.6797(5)	0.2341(2)	0.037(2)	0.056(2)	0.039(2)	−0.018(2)	0.004(2)	0.004(2)
N(3)	4e	0.9288(2)	0.6895(5)	0.3684(2)	0.039(2)	0.058(2)	0.037(2)	−0.015(2)	0.001(2)	0.007(2)
N(4)	4e	0.9045(2)	0.6374(5)	0.4380(2)	0.044(2)	0.067(2)	0.037(2)	−0.010(2)	0.006(2)	0.008(2)
N(5)	4e	0.7537(3)	0.4135(7)	0.5217(3)	0.055(3)	0.112(4)	0.064(3)	−0.018(3)	0.009(2)	0.031(3)
C(1)	4e	1.0499(3)	0.7993(6)	−0.1276(2)	0.056(3)	0.038(2)	0.044(2)	−0.004(2)	0.017(2)	−0.001(2)
C(2)	4e	1.1027(3)	0.7774(7)	−0.0639(3)	0.036(2)	0.064(3)	0.054(3)	−0.003(2)	0.011(2)	−0.001(2)
C(3)	4e	1.0735(2)	0.7563(6)	0.0073(3)	0.037(2)	0.058(3)	0.049(3)	0.002(2)	0.004(2)	0.003(2)
C(4)	4e	0.9930(2)	0.7534(5)	0.0155(2)	0.034(2)	0.033(2)	0.045(2)	0.000(2)	0.006(2)	0.000(2)
C(5)	4e	0.9416(2)	0.7752(6)	−0.0494(2)	0.033(2)	0.065(3)	0.046(2)	0.000(2)	0.002(2)	0.003(2)
C(6)	4e	0.9698(3)	0.7991(7)	−0.1209(3)	0.046(3)	0.079(3)	0.042(2)	−0.001(2)	0.001(2)	0.003(2)
C(7)	4e	0.9676(2)	0.7335(5)	0.0954(2)	0.036(2)	0.037(2)	0.047(2)	−0.002(2)	0.006(2)	0.002(2)
C(8)	4e	0.8513(2)	0.6498(5)	0.1681(2)	0.034(2)	0.038(2)	0.043(2)	−0.000(2)	0.003(2)	−0.001(2)
C(9)	4e	0.8767(2)	0.6398(6)	0.3084(2)	0.037(2)	0.046(2)	0.040(2)	−0.007(2)	−0.000(2)	0.003(2)
C(10)	4e	0.8152(3)	0.5529(6)	0.3393(3)	0.039(2)	0.056(3)	0.047(2)	−0.016(2)	0.003(2)	0.006(2)
C(11)	4e	0.8361(2)	0.5550(6)	0.4188(2)	0.037(2)	0.056(3)	0.045(2)	−0.009(2)	0.005(2)	0.010(2)
C(12)	4e	0.7918(3)	0.4768(7)	0.4778(3)	0.044(2)	0.074(3)	0.048(3)	−0.013(2)	0.002(2)	0.012(2)
C(13)	4e	1.0026(2)	0.7800(6)	0.3654(2)	0.031(2)	0.052(2)	0.033(2)	−0.006(2)	−0.002(2)	0.010(2)
C(14)	4e	1.0675(2)	0.6890(6)	0.3401(3)	0.045(2)	0.044(2)	0.049(2)	−0.002(2)	−0.001(2)	0.008(2)
C(15)	4e	1.1398(3)	0.7738(7)	0.3375(3)	0.037(2)	0.060(3)	0.063(3)	0.005(2)	0.008(2)	0.012(2)
C(16)	4e	1.1469(2)	0.9499(7)	0.3632(3)	0.034(2)	0.062(3)	0.059(3)	−0.008(2)	−0.002(2)	0.016(2)
C(17)	4e	1.0838(2)	1.0429(6)	0.3891(2)	0.042(2)	0.045(2)	0.048(2)	−0.006(2)	−0.003(2)	0.006(2)
C(18)	4e	1.0116(2)	0.9583(6)	0.3887(2)	0.037(2)	0.049(2)	0.034(2)	−0.001(2)	−0.003(2)	0.008(2)
C(19)	4e	1.2252(3)	1.043(1)	0.3624(5)	0.039(3)	0.086(4)	0.126(6)	−0.018(3)	0.001(3)	0.014(4)
Cl(1)	4e	1.08635(8)	0.8338(2)	−0.21759(7)	0.0733(8)	0.0760(9)	0.0493(7)	−0.0058(7)	0.0248(6)	0.0004(6)
Cl(2)	4e	1.05809(9)	0.4668(2)	0.3107(1)	0.0751(9)	0.0455(7)	0.103(1)	0.0027(6)	0.0030(8)	−0.0033(7)
Cl(3)	4e	0.93154(7)	1.0775(2)	0.41736(7)	0.0466(6)	0.0748(8)	0.0493(6)	0.0103(5)	0.0046(5)	−0.0030(6)

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