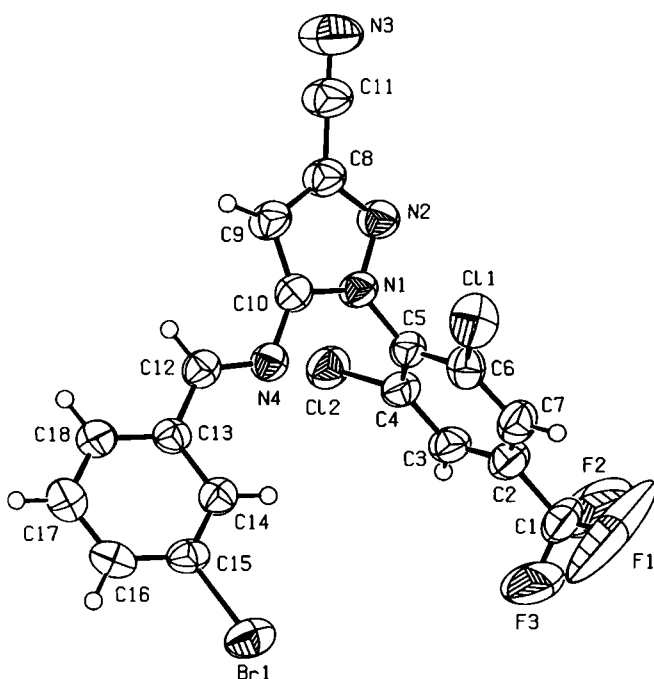


Crystal structure of 1-(2,6-dichloro-4-(trifluoromethyl)phenyl)-5-((3-bromophenyl)methyleneimino)-1*H*-pyrazole-3-carbonitrile, C₁₈H₈BrCl₂F₃N₄

X.-G. Zhang^{*,I,II}, X.-H. Zhang^{II} and P. Zhong^{II}^I Donghua University, School of Chemistry and Chemical Engineering, Shanghai, 201620, P. R. China^{II} Wenzhou University, School of Chemistry and Materials Engineering, Wenzhou 325027, P. R. China

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Abstract

C₁₈H₈BrCl₂F₃N₄, monoclinic, *P*12₁/c1 (no. 14), *a* = 13.064(1) Å, *b* = 14.692(1) Å, *c* = 9.9933(9) Å, β = 101.361(2)°, *V* = 1880.4 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.049, *wR*_{ref}(*F*²) = 0.145, *T* = 273 K.

Source of material

Following the literature method [1], the reaction of 2,6-dichloro-4-(trifluoromethyl)aniline (10 mmol, 2.3 g) with a suspension of sodium nitrite (10 mmol, 0.69 g) and sulfuric acid (3 ml), followed by reaction with a solution of ethyl 2,3-dicyanopropionate (10 mmol, 1.52 g) in acetic acid (5 ml), gave 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-pyrazole. The compound was then reacted with 3-bromobenzaldehyde (10 mmol, 1.85 g) to give the title compound. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

Experimental details

The large displacement parameters of the fluorine atoms represent the rotational disorder. All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of *d*(C–H) = 0.93 Å and *d*(N–H) = 0.86 Å with *U*_{iso} = 1.2*U*_{eq}(parent atom).

Discussion

The title compound is an important intermediate in synthesis of trifluoromethyl-containing pyrazole derivatives, which are good insecticides [2]. Trifluoromethylated compounds have been found diverse applications in the areas of materials science, agrochemistry and biomedical chemistry due to their unique chemical, physical and biological properties [3,4]. In order to continue investigations of such trifluoromethyl-containing pyrazoles, the title compound was synthesized and its crystal structure determined.

The title compound consists of three different rings, the first phenyl ring (C2/C3/C4/C5/C6/C7), the second phenyl ring (C13/C14/C15/C16/C17/C18) and the pyrazolyl ring (N1/N2/C8/C9/C10), which are all almost planar. The dihedral angles between pyrazolyl ring and phenyl (C2–C7) and phenyl (C13–C18) rings are 86.7° and 153.5°, respectively. In the crystal structure, all bond lengths and angles are normal. Furthermore, there are no obvious π–π interactions between pyrazolyl ring and phenyl rings.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.17 × 0.24 × 0.38 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	25.10 cm ^{−1}
Diffractionmeter, scan mode:	Bruker SMART APEX CCD, φ/ω
2θ _{max} :	50.48°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	9892, 3388
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2681
<i>N</i> (<i>param</i>) _{refined} :	253
Programs:	SHELXS-97 [5], SHELXL-97 [6], 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(3)	4e	0.6826	0.8206	0.4275	0.066
H(7)	4e	0.5152	0.6878	0.6637	0.073
H(9)	4e	0.8890	0.3590	0.5194	0.067
H(12)	4e	1.0091	0.4691	0.6580	0.058
H(14)	4e	0.9290	0.6287	0.8882	0.056
H(16)	4e	1.2008	0.6318	1.1585	0.071
H(17)	4e	1.2721	0.5328	1.0218	0.078
H(18)	4e	1.1726	0.4808	0.8196	0.066

* Correspondence author (e-mail: xgzhang99@yahoo.com.cn)

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Br(1)	4e	1.00047(4)	0.71947(3)	1.14718(5)	0.0923(4)	0.0588(3)	0.0653(3)	−0.0019(2)	0.0296(3)	−0.0125(2)
Cl(1)	4e	0.5912(1)	0.5091(1)	0.6712(1)	0.0828(8)	0.0783(8)	0.0789(8)	0.0016(6)	0.0282(7)	0.0220(6)
Cl(2)	4e	0.80475(9)	0.68624(8)	0.3426(1)	0.0681(7)	0.0583(6)	0.0748(7)	−0.0026(5)	0.0307(6)	−0.0055(5)
F(1)	4e	0.4743(8)	0.8488(4)	0.641(1)	0.40(1)	0.106(4)	0.49(1)	0.113(6)	0.37(1)	0.073(6)
F(2)	4e	0.4882(5)	0.8884(4)	0.4587(6)	0.223(6)	0.137(4)	0.173(5)	0.125(4)	−0.060(4)	−0.052(3)
F(3)	4e	0.5977(4)	0.9166(3)	0.6172(7)	0.141(4)	0.107(3)	0.304(7)	0.057(3)	−0.055(5)	−0.119(4)
N(1)	4e	0.7382(2)	0.5224(2)	0.4818(3)	0.052(2)	0.040(2)	0.050(2)	0.007(1)	0.004(2)	−0.002(1)
N(2)	4e	0.6882(3)	0.4689(2)	0.3803(3)	0.059(2)	0.046(2)	0.055(2)	0.005(2)	0.002(2)	−0.000(2)
N(3)	4e	0.6932(5)	0.2658(3)	0.2188(6)	0.137(5)	0.060(3)	0.095(4)	0.017(3)	−0.035(3)	−0.017(3)
N(4)	4e	0.8835(3)	0.5319(2)	0.6645(3)	0.049(2)	0.044(2)	0.051(2)	0.005(1)	0.007(2)	0.001(1)
C(1)	4e	0.5376(4)	0.8547(4)	0.5667(6)	0.066(3)	0.087(4)	0.086(4)	0.033(3)	0.002(3)	−0.030(3)
C(2)	4e	0.5898(4)	0.7664(3)	0.5477(5)	0.061(3)	0.056(3)	0.058(3)	0.015(2)	0.005(2)	−0.010(2)
C(3)	4e	0.6652(3)	0.7669(3)	0.4671(4)	0.061(3)	0.042(2)	0.059(3)	0.006(2)	0.005(2)	−0.005(2)
C(4)	4e	0.7135(3)	0.6865(3)	0.4472(4)	0.052(2)	0.047(2)	0.047(2)	0.003(2)	0.011(2)	−0.007(2)
C(5)	4e	0.6903(3)	0.6062(3)	0.5074(4)	0.044(2)	0.045(2)	0.047(2)	0.007(2)	0.003(2)	−0.004(2)
C(6)	4e	0.6175(3)	0.6076(3)	0.5909(4)	0.050(2)	0.061(3)	0.049(2)	0.005(2)	0.007(2)	0.006(2)
C(7)	4e	0.5657(4)	0.6874(3)	0.6098(5)	0.058(3)	0.073(3)	0.055(2)	0.012(2)	0.017(2)	−0.006(2)
C(8)	4e	0.7482(3)	0.3954(3)	0.3895(4)	0.065(3)	0.042(2)	0.060(3)	0.005(2)	0.006(2)	−0.001(2)
C(9)	4e	0.8362(3)	0.4018(3)	0.4945(4)	0.063(3)	0.043(2)	0.056(2)	0.013(2)	0.002(2)	0.001(2)
C(10)	4e	0.8274(3)	0.4852(3)	0.5523(4)	0.049(2)	0.044(2)	0.047(2)	0.002(2)	0.010(2)	0.002(2)
C(11)	4e	0.7182(4)	0.3228(3)	0.2945(5)	0.086(3)	0.047(3)	0.074(3)	0.011(2)	−0.012(3)	−0.005(2)
C(12)	4e	0.9781(3)	0.5107(3)	0.7079(4)	0.052(2)	0.044(2)	0.050(2)	0.003(2)	0.015(2)	0.002(2)
C(13)	4e	1.0407(3)	0.5485(2)	0.8324(4)	0.049(2)	0.039(2)	0.049(2)	−0.005(2)	0.013(2)	0.001(2)
C(14)	4e	0.9977(3)	0.6091(2)	0.9144(4)	0.051(2)	0.040(2)	0.050(2)	−0.001(2)	0.013(2)	0.004(2)
C(15)	4e	1.0582(3)	0.6388(3)	1.0331(4)	0.063(3)	0.037(2)	0.053(2)	−0.009(2)	0.020(2)	−0.004(2)
C(16)	4e	1.1613(4)	0.6110(3)	1.0765(5)	0.063(3)	0.054(2)	0.058(3)	−0.015(2)	0.005(2)	−0.008(2)
C(17)	4e	1.2032(3)	0.5520(3)	0.9952(5)	0.049(2)	0.067(3)	0.076(3)	−0.003(2)	0.006(2)	−0.005(2)
C(18)	4e	1.1435(3)	0.5208(3)	0.8738(4)	0.053(2)	0.052(2)	0.060(3)	−0.002(2)	0.015(2)	−0.007(2)

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