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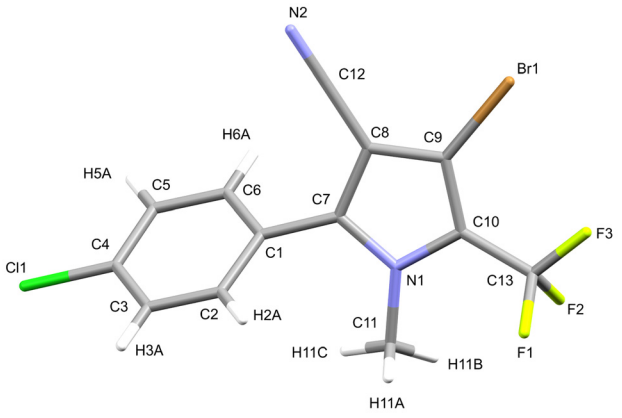
Crystal structure of 4-bromo-2-(4-chlorophenyl)-1-methyl-5-(trifluoromethyl)-1*H*-pyrrole-3-carbonitrile, $C_{13}H_7BrClF_3N_2$



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Abstract

$C_{13}H_7BrClF_3N_2$, monoclinic, $P2_1/c$ (no. 14), a = 9.4183(18) Å, b = 15.185(3) Å, c = 13.1826(18) Å, β = 133.905(8)°, V = 1358.4(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0420, $wR_{ref}(F^2)$ = 0.1015, T = 293 K.

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The molecular 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.

1 Source of materials

In a representative experiment 0.5 g (1.2 mmol) chlorfenapyr was dissolved in anhydrous dichloromethane, boron tribromide 0.4 g (1.5 mmol) was slowly dropped into the solution, and ethyl acetate and water were extracted to obtain 4-bromo-2-(4-chlorophenyl)-1-methyl-5-(trifluoromethyl)-1H-pyrrole-3-carbonitrile. Under the catalysis of iron powder, the title compound was obtained by bromination of the above compounds twice. Crystals of the title compound were obtained by slow evaporation within 3 days in the mixed solution of ethyl acetate and *n*-hexane.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.25 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6787, 2385, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1851
$N(param)_{refined}$:	182
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

2 Experimental details

The collected diffraction data were then processed using the Bruker SAINT program [1], which applies standard corrections for absorption and decay of the X-ray source. The corrected data were then further analyzed using the SHELXT software suite to obtain the crystal structure [2]. The final refinement was performed using the Bruker SHELXL program [3], with the aid of the OLEX2 program for visualizing and refining the structure [4].

3 Comment

Aryl pyrrole insecticides and acaricides are commonly used pesticides with low toxicity, and the representative drug is chlorfenapyr [5]. The title compound is a key intermediate for the synthesis of chlorfenapyr, and it is of good significance to promote the scale production [6–9] of chlorfenapyr.

The 4-bromo-2-(4-chlorophenyl)-1-methyl-5-(trifluoromethyl)-1*H*-pyrrole-3-carbonitrile was synthesized and characterized through a combination of analytical methods [10]. There is one molecule in the asymmetric unit. The benzene ring and pyrrole ring are non-coplanar, with a dihedral angle

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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}
Br1	1.10859 (5)	0.65623 (3)	0.70360 (4)	0.05429 (18)
C1	0.8392 (4)	0.6293 (2)	0.1822 (3)	0.0336 (8)
C2	0.6855 (5)	0.6737 (2)	0.0567 (4)	0.0388 (8)
H2A	0.587817	0.701409	0.046158	0.047*
C3	0.6790 (5)	0.6764 (2)	−0.0509 (4)	0.0401 (8)
H3A	0.576055	0.705277	−0.134574	0.048*
C4	0.8254 (5)	0.6363 (2)	−0.0344 (4)	0.0382 (8)
C5	0.9785 (5)	0.5947 (2)	0.0883 (4)	0.0439 (9)
H5A	1.078000	0.568977	0.099097	0.053*
C6	0.9858 (5)	0.5907 (2)	0.1966 (4)	0.0424 (8)
H6A	1.089953	0.561943	0.279885	0.051*
C7	0.8520 (4)	0.6248 (2)	0.2995 (3)	0.0331 (7)
C8	1.0065 (4)	0.6491 (2)	0.4392 (3)	0.0341 (7)
C9	0.9507 (4)	0.6318 (2)	0.5123 (3)	0.0339 (7)
C10	0.7643 (4)	0.59720 (19)	0.4175 (3)	0.0327 (7)
C11	0.5218 (5)	0.5535 (2)	0.1602 (4)	0.0494 (9)
H11A	0.415589	0.594638	0.115938	0.074*
H11B	0.494048	0.500988	0.184368	0.074*
H11C	0.535511	0.539026	0.096437	0.074*
C12	1.1789 (5)	0.6950 (2)	0.4921 (4)	0.0421 (8)
C13	0.6322 (5)	0.5702 (2)	0.4360 (4)	0.0450 (9)
Cl1	0.81462 (16)	0.63860 (7)	−0.17157 (10)	0.0596 (3)
F1	0.4589 (3)	0.61039 (19)	0.3446 (3)	0.0721 (7)
F2	0.5931 (4)	0.48453 (16)	0.4157 (3)	0.0839 (8)
F3	0.7063 (3)	0.58747 (19)	0.5621 (2)	0.0760 (7)
N1	0.7057 (4)	0.59309 (16)	0.2883 (3)	0.0332 (6)
N2	1.3138 (5)	0.7340 (2)	0.5339 (4)	0.0688 (10)

of 54.32°. Several important bond angle data are involved as follows C1–C7–C8 = 129.1°, C1–C7–N1 = 124.3°.

The title compound has been reported for the first time in this paper. The geometric parameters are in the expected ranges [11].

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