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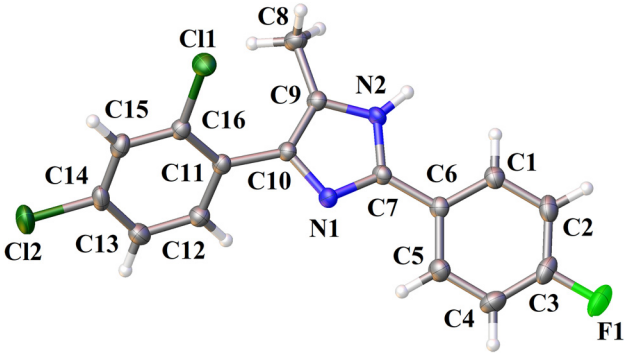
The crystal structure of 4-(2,4-dichlorophenyl)-2-(4-fluorophenyl)-5-methyl-1*H*-imidazole, $C_{16}H_{11}Cl_2FN_2$



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Abstract

$C_{16}H_{11}Cl_2FN_2$, orthorhombic, *Pbca* (no. 61), a = 9.6732(3) Å, b = 15.5602(5) Å, c = 19.0881(6) Å, V = 2873.08(16) Å^3, Z = 8, $R_{gt}(F)$ = 0.0361, w$R_{ref}(F^2)$ = 0.0958, T = 193 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.

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	GaKα radiation (1.34139 Å)
μ:	2.72 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE Metaljet PHOTON II, φ and ω
θ_{max}, completeness:	60.2°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21,290, 3187, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2676
$N(param)_{refined}$:	191
Programs:	Bruker [1], Olex2 [2], Shelx [3, 4]

1 Source of material

Add 4-fluorobenzamidine hydrochloride (2.02 g, 10.0 mmol), 2,4-dichlorophenylacetone (1.75 g, 10.0 mmol), K_2CO_3 (1.52 g, 1.1 equivalent), and dry EtOH (60 mL) in an oven dried 100 mL round bottom flask. Stir the reaction mixture for 12 h at 75 °C. Monitor the reaction progress by TLC. After completion of the reaction evaporate the excess EtOH in a rotary evaporator. Dilute the crude reaction mixture with water (60 mL). Extract the reaction mixture with DCM. Dry the organic layer over anhydrous Na_2SO_4. Remove the organic layer through evaporation under a vacuum. Purify the residue by column chromatography to yield 4-(2,4-dichlorophenyl)-2-(4-fluorophenyl)-5-methyl-4,5-dihydro-1*H*-imidazole as white solid 2.74 g, 85 %. (hexane/ethyl acetate as eluent). The product was dissolved in the mixed solvent (methanol: ethyl acetate = 1:1) and stored at 6 °C to provide white needle crystals.

2 Experimental details

Using Olex2 [2], the structure was solved with the Shelxt [3] structure solution program and refined with the Shelxl [4] refinement package.

3 Comment

Imidazoles are widely used in the various fields such as pharmaceutical industry [5], polymer industry [6], material science [7] and so on. Currently, imidazole and its derivatives are the most commonly used substances in printed circuit boards (PCBs) for protecting copper against oxidation in the presence of water and salt in the air. This is achieved through the formation of an organic solderability preservative (OSP)

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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	0.37982 (18)	0.71881 (11)	0.67183 (9)	0.0254 (4)
H1	0.320198	0.716760	0.711347	0.030*
C2	0.3451 (2)	0.76974 (12)	0.61494 (10)	0.0303 (4)
H2A	0.261626	0.801866	0.614609	0.036*
C3	0.4344 (2)	0.77267 (12)	0.55907 (10)	0.0309 (4)
C4	0.5554 (2)	0.72626 (13)	0.55657 (10)	0.0322 (4)
H4	0.614906	0.729539	0.517068	0.039*
C5	0.58778 (18)	0.67469 (12)	0.61326 (9)	0.0280 (4)
H5	0.670241	0.641547	0.612401	0.034*
C6	0.50133 (16)	0.67052 (11)	0.67173 (9)	0.0210 (3)
C7	0.53886 (16)	0.61660 (11)	0.73151 (8)	0.0202 (3)
C8	0.45144 (18)	0.50648 (13)	0.89341 (9)	0.0291 (4)
H8A	0.449078	0.548851	0.931372	0.044*
H8B	0.502632	0.455541	0.908861	0.044*
H8C	0.356782	0.489992	0.880981	0.044*
C9	0.52105 (16)	0.54453 (11)	0.83113 (8)	0.0213 (3)
C10	0.65103 (16)	0.53515 (11)	0.80352 (8)	0.0200 (3)
C11	0.76826 (16)	0.48138 (11)	0.82622 (9)	0.0210 (3)
C12	0.82737 (18)	0.42600 (11)	0.77729 (9)	0.0256 (4)
H12	0.787086	0.422142	0.732011	0.031*
C13	0.94261 (19)	0.37635 (12)	0.79211 (10)	0.0300 (4)
H13	0.981818	0.339837	0.757521	0.036*
C14	0.99907 (18)	0.38131 (12)	0.85849 (10)	0.0288 (4)
C15	0.94334 (18)	0.43371 (11)	0.90933 (10)	0.0268 (4)
H15	0.982446	0.435620	0.954953	0.032*
C16	0.82888 (17)	0.48374 (11)	0.89273 (9)	0.0223 (3)
Cl1	0.76681 (5)	0.55241 (3)	0.95703 (2)	0.03110 (13)
Cl2	1.14429 (5)	0.31997 (4)	0.87839 (3)	0.04310 (16)
F1	0.40097 (15)	0.82401 (8)	0.50387 (6)	0.0471 (3)
N1	0.66163 (13)	0.58027 (9)	0.74103 (7)	0.0212 (3)
N2	0.45240 (13)	0.59719 (9)	0.78538 (7)	0.0214 (3)
H2	0.366766	0.615394	0.790066	0.026*

film on the surface of the copper [8–10]. The self-assembling ability of imidazole is attributed to its orderly coordinating ability of nitrogen-containing heterocycles on copper surfaces.

The crystal structure mentioned in the title is formed by molecules that exhibit characteristic bonds, including C7–N1, C7–N2, and C9–C10. The lengths of these bonds are 1.328(2), 1.359(2), and 1.371(2) Å, respectively. The angle formed by C16–C11–N1 is 139.3 °C, which was probably influenced by the steric hindrance of the C8–C9 bond [11]. The bond lengths and angles are in the expected ranges.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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