Zhenpeng Shen, E. Liu, Xinzhe Tian and Fangfang Jian*

Crystal structure of 5-(4-chlorophenyl)-3-(4-fluorophenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, $C_{21}H_{16}ClFN_2$

<https://doi.org/10.1515/ncrs-2018-0553>

Received March 4, 2019; accepted April 11, 2019; available online May 8, 2019

Abstract

$C_{21}H_{16}ClFN_2$, monoclinic, $P2_1/c$ (no. 14), $a = 20.286(4)$ Å, $b = 5.3827(11)$ Å, $c = 16.254(3)$ Å, $\beta = 104.95(3)^\circ$, $Z = 4$, $V = 1714.7(7)$ Å³, $R_{gt}(F) = 0.0548$, $wR_{ref}(F^2) = 0.1199$, $T = 293(2)$ K.

CCDC no.: 1909511

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:	Colourless block
Size:	$0.28 \times 0.25 \times 0.24$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.24 mm^{-1}
Diffractometer, scan mode:	Bruker, ω
$\theta_{\max}$, completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7677, 3018, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2538
$N(\text{param})_{\text{refined}}$:	242
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

A mixture of (2*E*)-3-(4-chlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one (3.05 g, 0.01 mol) and phenyl hydrazine (0.98 mL, 0.01 mol) in 50 mL of glacial acetic acid was refluxed for 6 h. The reaction mixture was cooled and poured into 50 mL ice-cold water. The precipitate was collected by filtration and crystals were obtained from ethanol.

*Corresponding author: Fangfang Jian, School of Chemical Engineering and Pharmaceuticals, Henan University of Science and Technology, Luoyang, Henan 471003, P.R. China, e-mail: Fangfjian@126.com

Zhenpeng Shen, E. Liu and Xinzhe Tian: School of Chemical Engineering and Pharmaceuticals, Henan University of Science and Technology, Luoyang, Henan 471003, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} ^a /U _{eq}
Cl1	0.43470(3)	0.30969(16)	0.31529(5)	0.0811(3)
F1 ^a	1.0298(2)	1.0103(18)	0.3097(6)	0.0895(19)
N1	0.82071(9)	0.5463(4)	0.49712(12)	0.0517(5)
N2	0.76022(9)	0.5170(4)	0.51958(12)	0.0544(5)
C1 ^b	0.8629(5)	0.9861(19)	0.3472(4)	0.0549(18)
H1 ^b	0.8202(5)	1.0575(19)	0.3255(4)	0.066(2)*
C2 ^b	0.9177(5)	1.057(2)	0.3150(6)	0.062(2)
H2 ^b	0.9117(5)	1.174(2)	0.2718(6)	0.074(3)*
C3	0.97817(13)	0.9547(6)	0.34690(19)	0.0776(9)
C4 ^b	0.9823(2)	0.7227(10)	0.3853(3)	0.0719(13)
H4 ^b	1.0194(2)	0.6186(10)	0.3882(3)	0.0863(16)*
C5 ^b	0.9292(2)	0.6521(9)	0.4191(3)	0.0617(11)
H5 ^b	0.9311(2)	0.5003(9)	0.4470(3)	0.0740(14)*
C6	0.87196(11)	0.8094(4)	0.41158(14)	0.0523(6)
C7	0.81574(11)	0.7330(4)	0.44693(13)	0.0472(5)
C8	0.74809(11)	0.8621(4)	0.43008(14)	0.0503(6)
H8a	0.75362(11)	1.0362(4)	0.44573(14)	0.0604(7)*
H8b	0.72336(11)	0.8488(4)	0.37060(14)	0.0604(7)*
C9	0.71143(11)	0.7203(4)	0.48749(14)	0.0495(5)
H9	0.70779(11)	0.8268(4)	0.53503(14)	0.0594(7)*
C10	0.75801(11)	0.3575(4)	0.58632(13)	0.0484(5)
C11	0.80963(12)	0.1855(4)	0.61664(14)	0.0546(6)
H11	0.84770(12)	0.1830(4)	0.59476(14)	0.0655(7)*
C12	0.80461(13)	0.0193(5)	0.67884(16)	0.0649(7)
H12	0.83935(13)	−0.0957(5)	0.69846(16)	0.0778(8)*
C13	0.74903(15)	0.0201(5)	0.71255(16)	0.0713(8)
H13	0.74571(15)	−0.0944(5)	0.75413(16)	0.0855(9)*
C14	0.69855(13)	0.1926(6)	0.68389(16)	0.0676(7)
H14	0.66114(13)	0.1964(6)	0.70703(16)	0.0811(9)*
C15	0.70248(12)	0.3601(5)	0.62135(15)	0.0592(6)
H15	0.66775(12)	0.4756(5)	0.60250(15)	0.0711(8)*
C16	0.64149(11)	0.6230(4)	0.44214(13)	0.0452(5)
C17	0.63451(12)	0.4210(4)	0.38826(15)	0.0554(6)
H17	0.67329(12)	0.3477(4)	0.37842(15)	0.0665(7)*
C18	0.57143(12)	0.3260(5)	0.34892(15)	0.0590(6)
H18	0.56747(12)	0.1881(5)	0.31348(15)	0.0708(8)*
C19	0.51450(12)	0.4369(5)	0.36256(15)	0.0542(6)
C20	0.51948(12)	0.6426(5)	0.41321(17)	0.0622(7)
H20	0.48042(12)	0.7197(5)	0.42075(17)	0.0747(8)*
C21	0.58328(12)	0.7342(5)	0.45296(15)	0.0556(6)
H21	0.58698(12)	0.8736(5)	0.48766(15)	0.0667(7)*
C5A ^c	0.9395(4)	0.7611(16)	0.4582(6)	0.0617(11)
H5A ^c	0.9477(4)	0.6777(16)	0.5100(6)	0.0740(14)*
C4A ^c	0.9936(5)	0.8366(18)	0.4277(6)	0.0719(13)
H4A ^c	1.0384(5)	0.8111(18)	0.4588(6)	0.0863(16)*
C2A ^c	0.9231(9)	1.106(4)	0.3388(11)	0.062(2)
H2A ^c	0.9208(9)	1.258(4)	0.3112(11)	0.074(3)*
C1A ^c	0.8713(10)	1.027(4)	0.3721(9)	0.0549(18)
H1A ^c	0.8338(10)	1.131(4)	0.3670(9)	0.066(2)*
F1A ^d	1.0355(7)	1.088(4)	0.3387(13)	0.0895(19)

^aOccupancy: 0.75(2), ^bOccupancy: 0.632(4), ^cOccupancy: 0.368(4),^dOccupancy: 0.25(2).

Experimental details

The hydrogen atoms were placed in calculated positions and refined using a riding on attached atoms with isotropic thermal parameters 1.2 times those of their carrier atoms.

Comment

Pyrazoline derivatives play an important role in pharmaceuticals as well as medicinal chemistry because they possess a broad spectrum of biological activities including [4–7]. As a result, a great deal of attention has been paid to the investigations on the structure and property of new pyrazolines [8–10]. Continuing our previous work [10], we report the crystal structure of the title compound. The molecular structure of the title compound is shown in the figure, bond lengths and angles are within normal ranges [11]. The fluoro-substituted benzene ring is statistically disordered over two conformations.

Acknowledgements: The authors would like to thank the financial supports from the National Natural Science Foundation of China (Grant No. 21002023).

References

1. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, WI, USA (2002).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
4. Polevoi, L. G.; Ordin, T. N. K. A.: 1-yi(Peruyi) Mosk. Med. Inst., Moscow **159** (1964) 9147d.
5. Jamode, V. S.; Chandak, H. S.; Bhagat, P. R.: Synthesis and antimicrobial activity of 3-(3-phenylsulphonamidophenyl)-5-aryl-2-pyrazolines. *Asian J. Chem.* **16** (2004) 233–236.
6. Ren, F. P.; Tian, X. Z.; Ren, Y. L.; Zhao, S.; Wang, J. J.; Zhao, B.: Nitrogen dioxide-catalyzed aerobic oxidation of benzyl alcohols under cocatalyst and acid-free conditions. *Catal. Commun.* **101** (2017) 98–101.
7. Ren, Y. L.; Wang, W. H.; Zhao, B.; Tian, X. Z.; Zhao, S.; Wang, J. J.; Li, F. W.: Nitrogen dioxide catalyzed oxidative thiocyanation of arenes with ambient air as the terminal oxidant source. *ChemCatChem* **8** (2016) 3361–3366.
8. Li, J. T.; Zhang, X. H.; Lin, Z. P.: An improved synthesis of 1,3,5-triaryl-2-pyrazolines in acetic acid aqueous solution under ultrasound irradiation. *Beilstein J. Org. Chem.* **13** (2007) 13–16.
9. Movassagh, B.; Rooh, H.; Bijanzadeh, H. R.: A mild and hight efficient one-pot synthesis 1,3,5-triaryl-2-pyrazolines. *Chem. Heterocycl. Compd.* **48** (2013) 1719–1721.

10. Zhao, P. S.; Guo, H. M.; Wang, X.; Jian, F. F.: Synthesis, crystal structure and density functional studies on 1*N*-phenyl-3-(2,4-dichlorophenyl)-5-(4-chlorophenyl)-2-pyrazoline. J. Chin. Chem. Soc. **55** (2008) 183–188.
11. Kimura, T.; Kai, Y.; Yasuoka, N.; Kasai, N.: The crystal and molecular structure of two forms of 1,3-diphenyl-5-(*p*-chlorophenyl)-2-pyrazoline. Acta Crystallogr. **B33** (1977) 1786–1792.