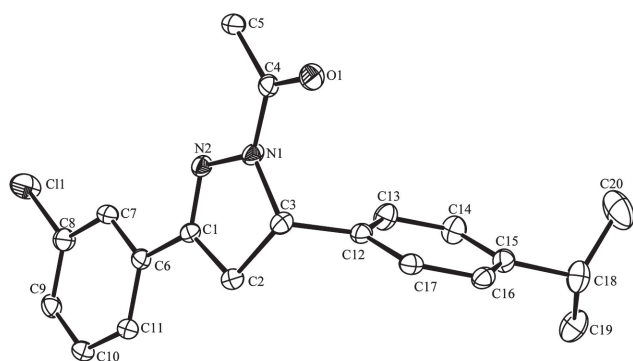


Kai-Xin Zhang, Meng-Ya Dong, Xiang-Dong Mei\*, Yao-Fa Li, Tao Zhang, Dong-Mei She, Wen-Liang Pan, Zhan-Lin Gao and Jun Ning

# Crystal structure of 1-acetyl-3-(3-chlorophenyl)-5-(4-isopropylphenyl)-4,5-dihydro-(1*H*)-pyrazole, $C_{20}H_{21}ClN_2O$



**Table 1:** Data collection and handling.

Crystal:	Colourless block
Size:	0.10 × 0.05 × 0.05 mm
Wavelength:	Mo K $\alpha$ radiation (0.71073 Å)
$\mu$ :	2.3 cm <sup>-1</sup>
Diffractometer, scan mode:	SuperNova, $\omega$ -scans
2 $\theta$ <sub>max</sub> , completeness:	52°, >99%
$N(hkl)$ <sub>measured</sub> , $N(hkl)$ <sub>unique</sub> , $R_{int}$ :	7108, 3465, 0.051
Criterion for $I_{obs}$ , $N(hkl)$ <sub>gt</sub> :	$I_{obs} > 2\sigma(I_{obs})$ , 2210
$N(param)$ <sub>refined</sub> :	220
Programs:	Olex2 [4], Superflip [5], SHELX [6]

DOI 10.1515/ncrs-2016-0207

Received July 2, 2016; accepted November 16, 2016; available online December 17, 2016

## Abstract

$C_{20}H_{21}ClN_2O$ , monoclinic,  $P2_1/n$  (no. 14),  $a = 13.3888(15)$  Å,  $b = 9.6869(10)$  Å,  $c = 13.7238(17)$  Å,  $\beta = 96.445(12)^\circ$ ,  $V = 1768.7(4)$  Å<sup>3</sup>,  $Z = 4$ ,  $R_{gt}(F) = 0.0497$ ,  $wR_{ref}(F^2) = 0.1153$ ,  $T = 180.01(10)$  K.

CCDC no.: 1517484

The asymmetric unit of the title crystal structure is shown in the figure (hydrogen atoms are omitted for clarity). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

**\*Corresponding author: Xiang-Dong Mei**, State Key Laboratory for Biology of Plant Diseases and Insect Pests, Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P. R. China, e-mail: xdmei@ippcaas.cn

**Kai-Xin Zhang, Meng-Ya Dong, Dong-Mei She and Jun Ning:** State Key Laboratory for Biology of Plant Diseases and Insect Pests, Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P. R. China

**Yao-Fa Li, Tao Zhang, Wen-Liang Pan and Zhan-Lin Gao:** Plant Protection Institute, Hebei Academy of Agricultural and Forestry Sciences, IPM Center of Hebei Province; and Key Laboratory of Integrated Pest Management on Crops in Northern Region of North China, Ministry of Agriculture, Baoding Hebei 07100, P. R. China

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{iso}^*/U_{eq}$
C1	0.55300(16)	0.6100(2)	0.90104(18)	0.0266(5)
C2	0.53192(17)	0.7549(2)	0.93277(18)	0.0309(6)
H2A	0.5924	0.8139	0.9336	0.037*
H2B	0.5083	0.7559	0.9986	0.037*
C3	0.44833(17)	0.8023(2)	0.85305(18)	0.0291(6)
H3	0.3868	0.8282	0.8840	0.035*
C4	0.34745(17)	0.6542(2)	0.72821(18)	0.0282(6)
C5	0.33804(17)	0.5177(2)	0.67767(19)	0.0332(6)
H5A	0.3119	0.4494	0.7210	0.050*
H5B	0.4042	0.4881	0.6615	0.050*
H5C	0.2918	0.5259	0.6173	0.050*
C6	0.63006(16)	0.5203(2)	0.95120(18)	0.0278(6)
C7	0.64386(17)	0.3867(2)	0.91664(19)	0.0319(6)
H7	0.6044	0.3541	0.8594	0.038*
C8	0.71544(19)	0.3031(2)	0.9666(2)	0.0366(6)
C9	0.77487(18)	0.3472(2)	1.04955(19)	0.0361(6)
H9	0.8235	0.2873	1.0829	0.043*
C10	0.76237(18)	0.4789(2)	1.08289(19)	0.0358(6)
H10	0.8032	0.5112	1.1394	0.043*
C11	0.69054(17)	0.5650(2)	1.03461(18)	0.0316(6)
H11	0.6823	0.6557	1.0586	0.038*
C12	0.47954(16)	0.9208(2)	0.79161(18)	0.0276(6)
C13	0.54696(18)	0.9025(2)	0.72323(19)	0.0370(7)
H13	0.5715	0.8126	0.7117	0.044*
C14	0.57907(18)	1.0137(2)	0.6716(2)	0.0400(7)
H14	0.6250	0.9990	0.6246	0.048*

Table 2 (continued)

Atom	x	y	z	U <sub>iso</sub> <sup>*</sup> /U <sub>eq</sub>
C15	0.54503(17)	1.1465(2)	0.68734(19)	0.0332(6)
C16	0.47722(16)	1.1634(2)	0.75461(18)	0.0301(6)
H16	0.4523	1.2531	0.7660	0.036*
C17	0.44460(16)	1.0525(2)	0.80593(17)	0.0283(6)
H17	0.3974	1.0671	0.8517	0.034*
C18	0.58222(19)	1.2693(2)	0.6331(2)	0.0460(8)
H18	0.5507	1.3539	0.6581	0.055*
C19	0.6961(2)	1.2853(3)	0.6548(2)	0.0541(9)
H19A	0.7139	1.3005	0.7252	0.081*
H19B	0.7183	1.3644	0.6182	0.081*
H19C	0.7292	1.2013	0.6348	0.081*
C20	0.5506(3)	1.2614(4)	0.5232(3)	0.0764(11)
H20A	0.5807	1.1795	0.4963	0.115*
H20B	0.5736	1.3443	0.4914	0.115*
H20C	0.4772	1.2552	0.5110	0.115*
Cl1	0.73012(6)	0.13562(6)	0.92401(6)	0.0641(3)
N1	0.42942(13)	0.67437(16)	0.79503(15)	0.0294(5)
N2	0.49511(13)	0.56832(16)	0.82576(15)	0.0284(5)
O1	0.28650(11)	0.74757(14)	0.71185(12)	0.0373(4)

### Source of material

The title compound C<sub>20</sub>H<sub>21</sub>ClN<sub>2</sub>O was prepared according to the literature method [1–3]. A mixture of 1-(3-chlorophenyl)-3-(4-isopropylphenyl)prop-2-en-1-one (5 mmol), hydrazine hydrate (10 mmol) in acetic acid (20 mL) was refluxed for 2 h and then poured into ice cold water. The precipitate was separated by filtration, washed free of acid, and then afforded the colourless target blocks after recrystallisation in ethanol.

### Experimental details

All hydrogen atoms were placed at calculated positions.

### Discussion

1-acetyl-3-(3-chlorophenyl)-5-(4-isopropylphenyl)-4,5-dihydro-(1*H*)pyrazole and other chalcone derivatives are important antimicrobial intermediates [1, 7–9]. If using ethanol instead of acetic acid as the solvent of this chemical reaction, it could obtain a similar structure compound without an acetyl group [1]. The molecule in the asymmetric

unit shows chirality at C3: because of the centrosymmetrical space group assignment the crystals are racemates. All bond lengths and angles are in the expected ranges.

**Acknowledgements:** This work was supported by the National Basic Research Program of China (No. 2014CB932200 and 2012CB114104), the NSFC (No. 31321004, 31301914 and 31201553).

### References

- Raghav, N.; Singh, M.: SAR studies of differently functionalized chalcones based hydrazones and their cyclized derivatives as inhibitors of mammalian cathepsin B and cathepsin H. *Bioorgan. Med. Chem.* **22** (2014) 4233–4245.
- Dong, M.; Mei, X.; Li, Y.; Zhang, L.; Pan, W.; Gao, Z.; Ning, J.: Crystal structure of (*E*)-3-(4-tert-butylphenyl)-1-(3-chlorophenyl)prop-2-en-1-one, C<sub>18</sub>H<sub>17</sub>ClO. *Z. Kristallogr.-NCS* **231** (2016) 29–30.
- Nepali, K.; Singh, G.; Turan, A.; Agarwal, A.; Sapra, S.; Kumar, R.; Banerjee, U. C.; Verma, P. K.; Satti, N. K.; Gupta, M. K.; Suri, O. P.; Dhar, K. L.: A rational approach for the design and synthesis of 1-acetyl-3,5-diaryl-4,5-dihydro(1*H*)pyrazoles as a new class of potential non-purine xanthine oxidase inhibitors. *Bioorgan. Med. Chem.* **19** (2011) 1950–1958.
- 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.
- Palatinus, L.; Chapuis, G.: SUPERFLIP – a computer program for the solution of crystal structures by charge flipping in arbitrary dimensions. *J. Appl. Crystallogr.* **40** (2007) 786–790.
- Sheldrick, G.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
- Sudhir, P.; Rajashree, C.; Ashok, B.: Synthesis and biological evaluation of Mannich bases of isoxazoline derivatives as novel anti-microbial agents. *E-Journal of Chem.* **9** (2012) 1760–1772.
- Joshi, V. D.; Kshirsagar, M. D.; Singhal, S.: Synthesis and antimicrobial activities of various pyrazolines from chalcones. *Int. J. Chem. Tech. Res.* **4** (2012) 971–975.
- Lv, P.; Sun, J.; Luo, Y.; Yang, Y.; Zhu, H.: Design, synthesis, and structure-activity relationships of pyrazole derivatives as potential FabH inhibitors. *Bioorg. Med. Chem. Lett.* **20** (2010) 4657–4660.