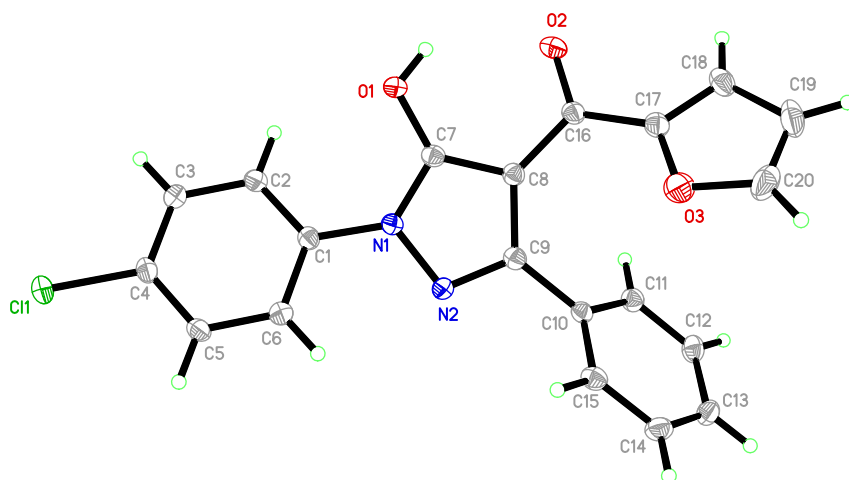


Qiong Wu, Wei-Ya Zhang, Zi-Mo Zhang, Heng-Qiang Zhang and Tong-Yin Jin*

Crystal structure of 1-(4-chlorophenyl)-4-(2-furoyl)-3-phenyl-1*H*-pyrazol-5-ol, C₂₀H₁₃ClN₂O₃



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Abstract

C₂₀H₁₃ClN₂O₃, monoclinic, *P*₂₁/*n* (no. 14), *a* = 6.2256(5) Å, *b* = 18.3976(15) Å, *c* = 14.5582(15) Å, β = 90.555(8)°, *V* = 1667.4(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0443, *wR*_{ref}(*F*²) = 0.1003, *T* = 153(2) 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:	Yellow block
Size:	0.21 × 0.17 × 0.15 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.25 mm ^{−1}
Diffraction, scan mode:	Multiwire proportional, ω
θ _{max} , completeness:	29.3°, >99 %
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	14,927, 4581, 0.039
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2825
<i>N</i> (<i>param</i>) _{refined} :	239
Programs:	CRYSTALIS ^{PRO} [1], SHELX [2, 3], WINGX/ORTEP [4], PLATON [5]

1 Source of material

All reagents were obtained from commercial sources and used without further purification. 3-Methyl-1-*p*-

chlorophenyl-5-pyrazolone was synthesized according to the method proposed by Jensen [6] (yield 85 %; m.p. 441–443 K). 3-Methyl-1-*p*-chlorophenyl-5-pyrazolone (5 g) was dissolved in dioxane (100 mL) by warming and Ca(OH)₂ (3 g) was added. 2-Furoyl chloride (5 mL) was next added, drop by drop, with stirring within 4 min. After refluxing gently for 1.5 h, the orange mixture was cooled and poured with stirring into chilled 2 M HCl (500 mL). A handful of ice-salt mixture was added and vigorous stirring continued for another 30 min, after which, the reaction mixture was kept in a refrigerator until crystallisation occurred. Filtration of the product gave 67 % yield of yellow crystals at room temperature.

*Corresponding author: Tong-Yin Jin, Department of Chemistry, Hebei Normal University for Nationalities, Chengde 067000, People's Republic of China, E-mail: tjyin0521@hbun.edu.cn

Qiong Wu, Wei-Ya Zhang, Zi-Mo Zhang and Heng-Qiang Zhang, Department of Chemistry, Hebei Normal University for Nationalities, Chengde 067000, People's Republic of China. <https://orcid.org/0000-0001-7138-2905> (H.-Q. Zhang)

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.7074 (3)	0.55607 (10)	0.31733 (13)	0.0245 (4)
C2	0.6373 (3)	0.49399 (10)	0.27208 (14)	0.0287 (4)
H2	0.499873	0.476022	0.282109	0.034*
C3	0.7733 (3)	0.45901 (10)	0.21193 (14)	0.0293 (4)
H3	0.728107	0.417203	0.181469	0.035*
C4	0.9762 (3)	0.48641 (10)	0.19734 (14)	0.0278 (4)
C5	1.0458 (3)	0.54871 (10)	0.24083 (14)	0.0281 (4)
H5	1.181974	0.567159	0.229372	0.034*
C6	0.9115 (3)	0.58352 (10)	0.30158 (13)	0.0275 (4)
H6	0.957584	0.625275	0.331890	0.033*
C7	0.3986 (3)	0.57140 (10)	0.42607 (13)	0.0251 (4)
C8	0.3274 (3)	0.62890 (9)	0.47995 (13)	0.0245 (4)
C9	0.4739 (3)	0.68624 (10)	0.46027 (13)	0.0252 (4)
C10	0.4766 (3)	0.76208 (10)	0.49347 (13)	0.0242 (4)
C11	0.2930 (3)	0.80490 (10)	0.49113 (14)	0.0292 (4)
H11	0.165223	0.786174	0.467433	0.035*
C12	0.3000 (3)	0.87551 (11)	0.52407 (15)	0.0337 (5)
H12	0.177208	0.904272	0.521605	0.040*
C13	0.4881 (3)	0.90343 (11)	0.56053 (14)	0.0348 (5)
H13	0.491639	0.950621	0.583388	0.042*
C14	0.6706 (3)	0.86108 (11)	0.56290 (15)	0.0349 (5)
H14	0.797626	0.879666	0.587492	0.042*
C15	0.6651 (3)	0.79121 (10)	0.52888 (14)	0.0300 (4)
H15	0.789518	0.763249	0.529704	0.036*
C16	0.1477 (3)	0.61992 (10)	0.54161 (14)	0.0270 (4)
C17	0.1211 (3)	0.66408 (10)	0.62329 (14)	0.0310 (4)
C18	−0.0551 (4)	0.67513 (11)	0.67463 (15)	0.0373 (5)
H18	−0.191160	0.655778	0.664241	0.045*
C19	0.0068 (5)	0.72165 (13)	0.74699 (17)	0.0561 (7)
H19	−0.081097	0.739646	0.793002	0.067*
C20	0.2162 (5)	0.73494 (13)	0.73680 (17)	0.0589 (8)
H20	0.298410	0.764030	0.775793	0.071*
Cl1	1.14839 (8)	0.44089 (3)	0.12320 (4)	0.03731 (15)
H1	0.214 (4)	0.4968 (14)	0.4427 (19)	0.069 (9)*
N1	0.5723 (2)	0.59412 (8)	0.37947 (11)	0.0255 (3)
N2	0.6192 (2)	0.66617 (8)	0.39968 (11)	0.0268 (4)
O1	0.3338 (2)	0.50323 (7)	0.41839 (10)	0.0290 (3)
O2	0.0142 (2)	0.57168 (8)	0.52497 (10)	0.0397 (4)
O3	0.2934 (3)	0.69965 (8)	0.66070 (11)	0.0459 (4)

2 Experimental details

The hydroxyl H atom was located in a difference Fourier map and refined as riding, with O–H distance restraint of 0.84(3) Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. Other H atoms were placed in calculated positions, with C–H = 0.93 for phenyl and furan, and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for phenyl H.

3 Comment

Acylpyrazolones are an interesting class of β-diketones possessing a prominent structural motif, which is found in numerous active compounds. Due to the easy preparation and its rich biological activity in broad-spectrum of applications they are of general interest [7–9]. Alkaline-earth metal β-diketonate complexes also have recently provoked a growing interest as suitable precursors in the synthesis of high Tc superconducting films [10, 11]. So far research has been restricted to those acylpyrazolones with aryl substituents at four-positions. So far, only a few studies have involved heterocyclic substituents at the four-position. In recent years, we have reported some 4-acylpyrazolones ligands and their complexes [12–14]. Knowledge of the crystal structure of such 4-heterocyclic acylpyrazolones ligands gives us not only sufficient information about nuclearity of the complex molecule, but is important in understanding the behaviour of this compounds in the vapour phase, and the mechanisms of sublimation and decomposition.

The structure of the title compound is shown in Figure. The bond lengths and angles are within normal ranges [15]. The *p*-chlorophenyl ring is slightly twisted by 20.06(5)° with respect to the pyrazolone ring, whereas the furyl rings make dihedral angles of 37.87(5)° with the pyrazolone ring. Atom O1 has a partial anionic character, as shown by the lengthening of the C7=O1 bond [1.322(2) Å] relative to that normally found for carbonyl groups and this atom acts as hydrogen-bond acceptor. The intermolecular O1–H1...O2 hydrogen bond results in the formation of a dimer with an R²₂(12) graph-set motif [16]. It is apparent that the C16=O2 and C8=C16 distances correspond to well defined double bonds. The conjugation effects also cause the pyrazolone and C1–C6 benzene ring to be nearly coplanar, with a mean deviation from the overall plane of 0.003 Å. The clear presence of the hydroxyl H atom in the difference Fourier synthesis and the absence of any residual electronic density in the vicinity of O1 confirm that title compound crystallize as a pure hydroxyl tautomer and that no desmotropism is present [17].

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