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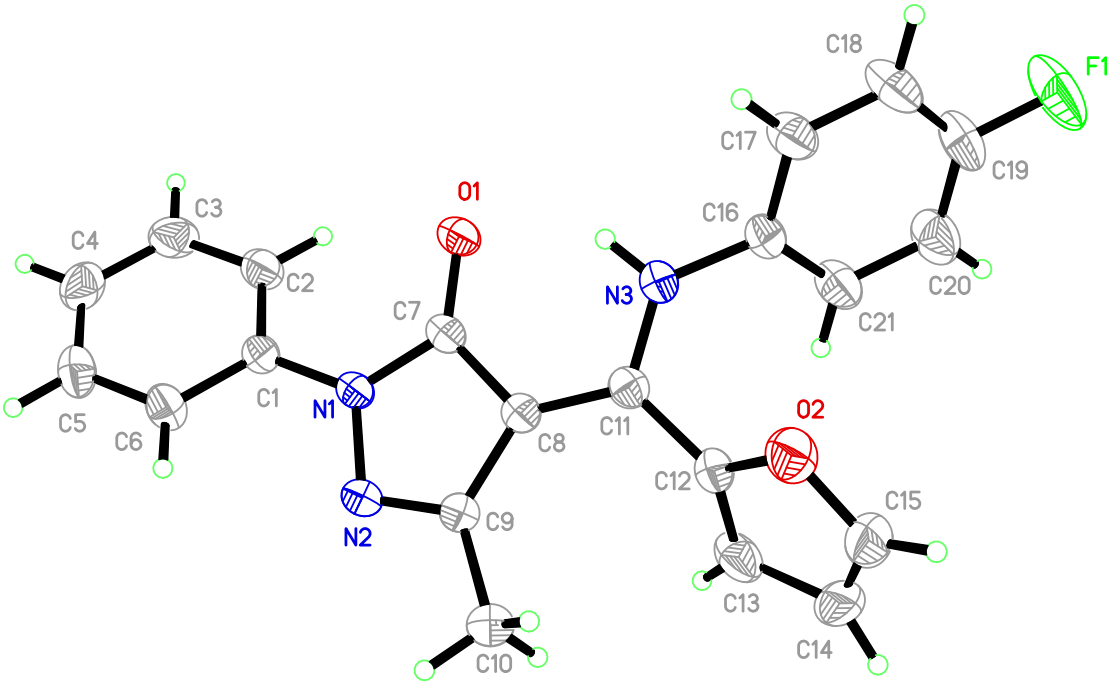
Crystal structure of (Z)-4-(((4-fluorophenyl) amino)(furan-2-yl)methylene)-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one



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Abstract

$C_{22}H_{16}FN_3O_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.7335(18) Å, b = 9.967(2) Å, c = 11.968(3) Å, α = 70.677(4)°, β = 86.259(4)°, γ = 65.759(4) Å, V = 893.4(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0543, wR_{ref} (F^2) = 0.1677, T = 296(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.32 × 0.26 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4758, 3143, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2275
$N(param)_{refined}$:	250
Programs:	Bruker [1], SHELX [2], WinGX/ORTEP [3], PLATON [4]

Source of material

All reagents were obtained from commercial sources and used without further purification. A mixture of a 10 mL HPMFP (2 mmol, 0.5366 g) anhydrous ethanol solution,

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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}
H3A	0.889 (3)	0.317 (3)	0.137 (2)	0.053 (8)*
F1	0.5745 (3)	0.2596 (4)	0.59296 (16)	0.1249 (9)
O1	0.9798 (2)	0.2247 (2)	0.01124 (14)	0.0605 (5)
O2	0.6355 (3)	0.7131 (2)	0.14741 (18)	0.0716 (6)
N1	0.9336 (3)	0.3865 (2)	−0.18788 (16)	0.0467 (5)
N2	0.8553 (3)	0.5467 (2)	−0.25126 (17)	0.0475 (5)
N3	0.8118 (3)	0.3974 (3)	0.15034 (17)	0.0505 (5)
C1	1.0287 (3)	0.2835 (3)	−0.2487 (2)	0.0438 (6)
C2	1.0598 (4)	0.1262 (3)	−0.2036 (2)	0.0576 (7)
H2	1.015731	0.087924	−0.133425	0.069*
C3	1.1558 (4)	0.0280 (3)	−0.2631 (3)	0.0697 (8)
H3	1.178932	−0.077683	−0.231571	0.084*
C4	1.2186 (4)	0.0825 (4)	−0.3685 (3)	0.0696 (8)
H4	1.282999	0.014638	−0.408175	0.083*
C5	1.1851 (4)	0.2384 (4)	−0.4145 (2)	0.0640 (8)
H5	1.225724	0.276401	−0.486369	0.077*
C6	1.0916 (3)	0.3392 (3)	−0.3550 (2)	0.0546 (7)
H6	1.070974	0.444399	−0.386264	0.065*
C7	0.9140 (3)	0.3552 (3)	−0.0680 (2)	0.0466 (6)
C8	0.8038 (3)	0.5050 (3)	−0.0569 (2)	0.0438 (6)
C9	0.7793 (3)	0.6159 (3)	−0.1748 (2)	0.0443 (6)
C10	0.6910 (4)	0.7897 (3)	−0.2175 (3)	0.0649 (8)
H10A	0.723473	0.831082	−0.295128	0.097*
H10B	0.721215	0.829515	−0.163622	0.097*
H10C	0.571362	0.820277	−0.221347	0.097*
C11	0.7452 (3)	0.5199 (3)	0.05040 (19)	0.0431 (6)
C12	0.6120 (3)	0.6629 (3)	0.0608 (2)	0.0467 (6)
C13	0.4582 (3)	0.7500 (4)	0.0036 (2)	0.0700 (9)
H13	0.411302	0.738354	−0.058262	0.084*
C14	0.3817 (4)	0.8674 (4)	0.0616 (4)	0.0921 (13)
H14	0.274449	0.948200	0.042782	0.111*
C15	0.4924 (6)	0.8374 (4)	0.1451 (3)	0.0940 (13)
H15	0.474505	0.894596	0.196074	0.113*
C16	0.7466 (3)	0.3692 (3)	0.2643 (2)	0.0468 (6)
C17	0.8574 (4)	0.2886 (3)	0.3641 (2)	0.0621 (7)
H17	0.972349	0.258713	0.356710	0.075*
C18	0.7990 (4)	0.2517 (4)	0.4755 (2)	0.0769 (9)
H18	0.873732	0.195886	0.543213	0.092*
C19	0.6313 (4)	0.2982 (4)	0.4838 (2)	0.0745 (9)
C20	0.5191 (4)	0.3784 (4)	0.3878 (3)	0.0782 (9)
H20	0.404314	0.409484	0.396599	0.094*
C21	0.5766 (4)	0.4136 (4)	0.2765 (2)	0.0642 (8)
H21	0.500464	0.467533	0.209442	0.077*

and a 10 mL 4-fluoroaniline (2 mmol, 0.2221 g) anhydrous ethanol solution was refluxed for ca. 3 h, adding a few drops of glacial acetic acid as a catalyst. Then ethanol was removed by evaporation and the resulting yellow precipitate formed was filtered off, washed with cold anhydrous ethanol and dried in air. Yellow block crystals suitable for analysis were obtained by slowly evaporation of a solution in anhydrous ethanol at room temperature for a few days.

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program [2]. The H atoms bonded to N3 atoms were located in difference maps and refined freely. Other H atoms were placed in calculated positions, with C–H = 0.93 for phenyl and furyl, 0.96 for methyl H atoms, and refined as riding, with U_{iso}(H) values of 1.2U_{eq}(C) for phenyl and furyl H and 1.5U_{eq}(C) for methyl H.

Comment

4-Acylpyrazolones are an interesting class of β-diketones, containing a pyrazole-bearing moiety. Thus, related metal complexes are used for the separation of elements with similar properties [5]. Some β-diketonate based compounds also have recently provoked a growing interest for various reasons [6–8]. Only a few studies have involved heterocyclic substituents at the 4-position. In recent years, we have reported the Schiff bases derived from 4-heterocyclic acylpyrazolones and their complexes, which possess high antibacterial activation [9, 10]. Knowledge of the crystal structure of such acylpyrazolones derivatives gives us not only sufficient information about nuclearity of the complex, but is important for the understanding of the compounds in the vapour phase, and the mechanisms of sublimation and decomposition.

In the title crystal structure, the compound crystallized with a structural configuration in which the phenyl ring(C(1)–C(6)) is twisted with a dihedral angle of 23.73(15)° with respect to a plane defined by the pyrazole ring. The O1 atom of pyrazole ring and the O(1)/C(7)/C(8)/C(11)/N(3) plane are nearly coplanar with the largest deviation of 0.028 Å. The bond length of C(8)–C(11) (1.383(3) Å) between the usual C–C and C=C bonds indicates the delocalization of the electrons because of the addition of a proton to N(3) is more favourable than to O(1) in the Fourier map (keto-form). The atom O(1) of 1-phenyl-3-methyl-4-(furoyl)-pyrazolone-5 moiety and the N(3) atom of the 4-Fluoroaniline group are on the same side of C(8)–C(11) bond, which are available for coordination with metal cations. A strong intramolecular N(3)–H(3A)···O(1) hydrogen bond is observed, as part of the enamine-keto tautomerism. Two

other weak intramolecular bonds C(6)–H(6)···O(2) and C(2)–H(2)···O(1) are also found, stabilizing the structure. All bond lengths and angles are normal and comparable with those found for related compounds [11–13].

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

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