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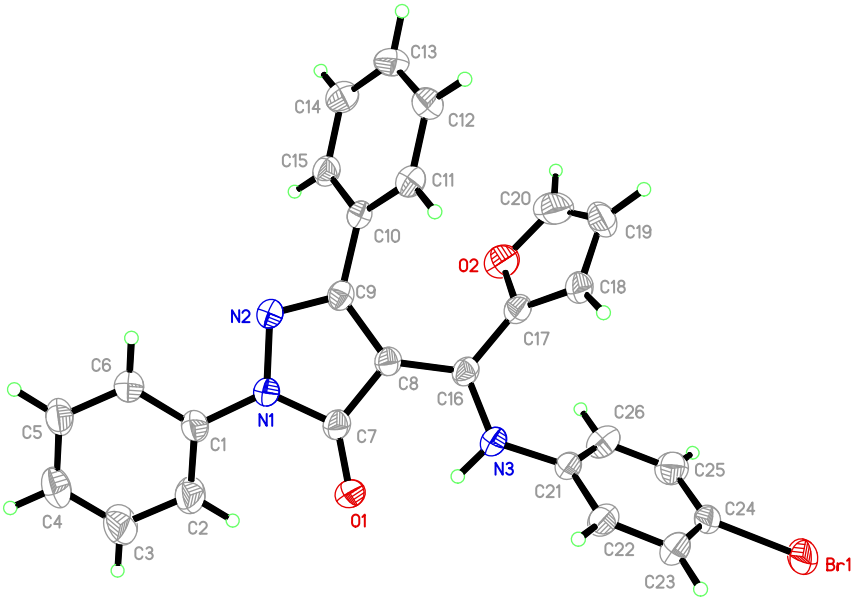
Crystal structure of (*Z*)-4-(((4-bromophenyl) amino)(furan-2-yl)methylene)-2,5-diphenyl-2,4-dihydro-3*H*-pyrazol-3-one, $C_{26}H_{18}BrN_3O_2$



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Abstract

$C_{26}H_{18}BrN_3O_2$, monoclinic, $P2_1/n$ (no. 14), a = 9.5191(14) Å, b = 23.056(3) Å, c = 10.0556(15) Å, β = 95.470(3)°, V = 2196.9(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0640, $wR_{ref}(F^2)$ = 0.1645, T = 296(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.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.18 × 0.15 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.90 mm^{-1}
Diffractometer, scan mode:	Xcalibur, φ and ω
θ_{max}, completeness:	26.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12,679, 4518, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2601
$N(param)_{refined}$:	293
Programs:	CrysAlis^{PRO} [1], SHELX [2], WinGX/ORTEP [3], PLATON [4]

Source of material

All reagents were obtained from commercial sources and used without further purification. The solution of 4-bromoaniline (2.0 mmol) and 1-phenyl-3-phenyl-4-(2-furoyl)-5-pyrazolone (2.0 mmol) in ethanol (15 mL) was refluxed for 7 h and the yellow precipitate was gradually formed. After cooled to the room temperature, the mixture was filtrated and the collected solid was washed with

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.64086 (9)	0.23860 (3)	0.65396 (6)	0.0970 (3)
C1	0.1161 (5)	0.1071 (2)	−0.3497 (5)	0.0536 (12)
C2	0.0849 (7)	0.1655 (2)	−0.3611 (6)	0.0767 (17)
H2	0.104695	0.190266	−0.288805	0.092*
C3	0.0231 (8)	0.1866 (3)	−0.4827 (6)	0.091 (2)
H3	0.004454	0.226094	−0.491807	0.109*
C4	−0.0103 (7)	0.1509 (3)	−0.5878 (6)	0.0767 (17)
H4	−0.052831	0.165631	−0.667822	0.092*
C5	0.0193 (6)	0.0921 (3)	−0.5756 (5)	0.0667 (15)
H5	−0.004674	0.067320	−0.647050	0.080*
C6	0.0840 (6)	0.0705 (2)	−0.4579 (5)	0.0601 (13)
H6	0.106325	0.031310	−0.450811	0.072*
C7	0.2533 (5)	0.1101 (2)	−0.1199 (5)	0.0484 (11)
C8	0.2771 (5)	0.06465 (19)	−0.0206 (4)	0.0427 (10)
C9	0.2148 (5)	0.01324 (18)	−0.0839 (4)	0.0417 (10)
C10	0.2091 (5)	−0.04727 (18)	−0.0357 (4)	0.0423 (10)
C11	0.3288 (5)	−0.0756 (2)	0.0220 (4)	0.0487 (11)
H11	0.414968	−0.056291	0.030889	0.058*
C12	0.3206 (6)	−0.1322 (2)	0.0662 (5)	0.0615 (14)
H12	0.400569	−0.150506	0.106690	0.074*
C13	0.1933 (7)	−0.1615 (2)	0.0501 (5)	0.0699 (16)
H13	0.187731	−0.199759	0.078603	0.084*
C14	0.0749 (6)	−0.1339 (2)	−0.0084 (5)	0.0661 (15)
H14	−0.010730	−0.153562	−0.018964	0.079*
C15	0.0825 (5)	−0.0771 (2)	−0.0515 (4)	0.0507 (12)
H15	0.002063	−0.058874	−0.091365	0.061*
C16	0.3398 (4)	0.07624 (19)	0.1076 (4)	0.0433 (10)
C17	0.3541 (5)	0.03233 (19)	0.2133 (4)	0.0487 (11)
C18	0.4687 (6)	0.0120 (2)	0.2857 (5)	0.0580 (13)
H18	0.561136	0.025033	0.285228	0.070*
C19	0.4193 (10)	−0.0337 (3)	0.3635 (6)	0.092 (2)
H19	0.473596	−0.056803	0.424397	0.110*
C20	0.2801 (11)	−0.0373 (3)	0.3326 (6)	0.096 (2)
H20	0.221316	−0.063763	0.369872	0.115*
C21	0.4501 (5)	0.15269 (19)	0.2593 (5)	0.0494 (12)
C22	0.5754 (6)	0.1823 (2)	0.2636 (5)	0.0617 (14)
H22	0.623378	0.184703	0.187475	0.074*
C23	0.6308 (6)	0.2086 (2)	0.3805 (6)	0.0690 (16)
H23	0.714129	0.229816	0.382619	0.083*
C24	0.5619 (6)	0.2032 (2)	0.4926 (5)	0.0601 (14)
C25	0.4363 (7)	0.1748 (2)	0.4901 (5)	0.0676 (15)
H25	0.389023	0.172588	0.566760	0.081*
C26	0.3794 (6)	0.1493 (2)	0.3719 (6)	0.0658 (15)
H26	0.293518	0.129868	0.369041	0.079*
H3A	0.391 (6)	0.151 (2)	0.060 (6)	0.080 (19)*
N1	0.1752 (4)	0.08339 (16)	−0.2260 (4)	0.0502 (10)
N2	0.1554 (4)	0.02440 (16)	−0.2033 (4)	0.0496 (10)
N3	0.3945 (4)	0.12902 (17)	0.1338 (4)	0.0524 (10)
O1	0.2933 (4)	0.16141 (14)	−0.1159 (3)	0.0621 (9)
O2	0.2360 (4)	0.00314 (16)	0.2386 (4)	0.0695 (10)

additional ethanol and dried in the air. Yellow block crystals were obtained by evaporation of an ethanol/dichloromethane (1:1) mixed solution 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 1.2 U_{eq}(C) for phenyl and furyl H and $1.5_{eq}U$(C)for methyl H.

Comment

4-Heterocyclic acylpyrazolones are an interesting class of β-diketones, containing a pyrazole-bearing chelating arm. Therefore, their metal complexes are widely used for the separation of transition metal elements with similar properties [5]. In our previous work, we have reported the Schiff bases derived from 4-heterocyclic acylpyrazolones and its complexes, which possess high antibacterial activation [6, 7]. In order to further investigate the coordination abilities and the behaviour of pyrazolone based ligands, we synthesized the title compound derived from 1-phenyl-3-phenyl-4-(2-furoyl)-5-pyrazolone (HPPFP), and its structure is reported here.

In the title crystal structure, atoms O1, C7, C8 and C16 of the HPPFP moiety and atom N3 of 4-bromoaniline group are coplanar, the largest deviation being 0.003 Å for atom O1. The dihedral angle between this mean plane and pyrazole ring (N1/N2/C9/C8/C7) of HPPFP is 3.63(2)°. The phenyl ring (C1–C6) is twisted by 59.35(3)° with respect to a plane defined by the pyrazole ring. The bond length of d(C16–C8) = 1.394 (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 N3 is more favorable to the O1 in the Fourier map. The atom O1 of HPPFP and the N3 atom of the 4-bromoaniline group are on the same side of C8–C16 bond, which are available for complexation with metals. A strong intramolecular hydrogen bond N3–H3A···O1 is also indicative of the enamine-keto form. All bond lengths and angles are normal and comparable with those found for related compounds [8, 9]. In the crystal, molecules are linked *via* weak C–H···N and C–H···O hydrogen bonds.

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