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Crystal structure of (4*Z*)-4-((4-iodoanilino)(phenyl)methyl]-3-methyl-1-phenyl-1*H*-pyrazol-5-(4*H*)-one, $C_{23}H_{18}IN_3O$

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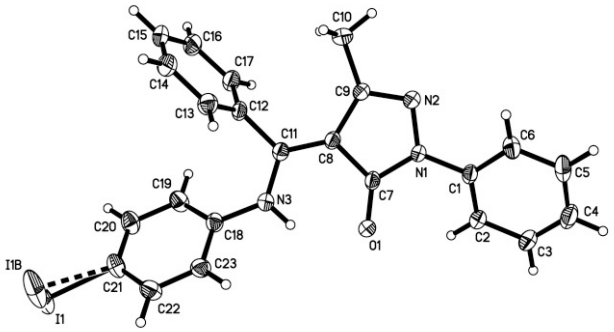

Abstract

$C_{23}H_{18}IN_3O$, triclinic, $P\bar{1}$ (no. 2), a = 7.5861(7) Å, b = 11.0115(9) Å, c = 13.668(1) Å, α = 109.621(2)°, β = 99.416(1)°, γ = 104.785(2)°, V = 1000.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.0659, $wR_{ref}(F^2)$ = 0.1794, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.15×0.22×0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	16.19 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16482, 4932
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3561
$N(param)_{refined}$:	264
Programs:	SHELX [4]

Source of material

A solution of 4-Iodoaniline (1.2 mmol) and 4-benzoyl-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one (1 mmol) in ethanol (10 mL) was refluxed for 5 h and a yellow precipitate was gradually formed. After cooled to room temperature, the mixture was filtrated and the collected solid was washed with additional ethanol and dried in the air. Suitable crystals were obtained by evaporation of an ethanol/dichloromethane (1:1) solution.

Experimental details

All H atoms on C were placed in idealized positions and refined as riding atoms at their parent atoms with C–H distances of 0.93 - 0.96Å, U_{iso}(H) = 1.2 U_{eq}(C) and U_{iso}(H) = 1.5 U_{eq}(C_{methyl}). The H atom bonded to N3 was placed in calculated positions, with N–H = 0.86Å, U_{iso}(H) = 1.2 U_{eq}(N).

Discussion

4-Acylpyrazolones are an interesting class of β-diketones, containing a pyrazole-bearing chelating arm. Thus, their metal complexes are used for the separation of elements with similar properties [1]. In recent years, Schiff bases derived from acylpyrazolones and its complexes have been reported, which possess high antibactrial activation [2-3]. As part of this work, we synthesized the title compound derived from 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone (HPMBP), and its structure is reported here.

In the title crystal structure, atoms O1, C7, C8 and C11 of the HPMBP moiety and atom N3 of the *n*-butylamine group are coplanar, the largest deviation being 0.0707 (11)Å for atom C7. The dihedral angle between this mean plane and pyrazole ring of HPMBP is 4.81(3)°. The bond length of *d*(C8–C11)=1.382 (6) 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 than to O2. The atom O2 of PMBP and the N3 atom of the 4-iodaniline group are on the same side of C8–C11 bond, which are available for coordination with metal cations. A strong intra molecular hydrogen bond N3–H3A···O1 is also indicative of the enamine-keto form.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3A)	2*i*	0.4342	0.8268	0.3278	0.048
H(2B)	2*i*	0.0888	0.9768	0.1308	0.056
H(3B)	2*i*	–0.0651	1.1296	0.1143	0.068
H(4A)	2*i*	–0.3807	1.0763	0.0977	0.072
H(5A)	2*i*	–0.5547	0.8714	0.0989	0.067
H(6A)	2*i*	–0.4020	0.7206	0.1221	0.054
H(10A)	2*i*	–0.2378	0.3609	0.0650	0.076
H(10B)	2*i*	–0.1022	0.3796	0.1726	0.076
H(10C)	2*i*	–0.0332	0.3540	0.0677	0.076
H(13A)	2*i*	0.3177	0.5457	0.4143	0.053
H(14A)	2*i*	0.3433	0.3421	0.4167	0.067
H(15A)	2*i*	0.3702	0.1824	0.2659	0.072
H(16A)	2*i*	0.3662	0.2212	0.1109	0.066
H(17A)	2*i*	0.3305	0.4218	0.1045	0.052
H(19A)	2*i*	0.6826	0.6115	0.2744	0.055
H(20A)	2*i*	0.9729	0.6381	0.3796	0.058
H(22A)	2*i*	0.9378	0.9572	0.6162	0.057
H(23A)	2*i*	0.6461	0.9271	0.5139	0.050

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	2*i*	0.63	1.2466(4)	0.8307(5)	0.5972(3)	0.046(1)	0.098(2)	0.0635(8)	0.028(2)	–0.0039(5)	0.0255(7)
I(1B)	2*i*	0.37	1.235(2)	0.793(3)	0.5994(6)	0.080(3)	0.170(7)	0.056(1)	0.077(3)	0.002(2)	0.020(3)
O(1)	2*i*		0.2513(5)	0.8945(3)	0.2583(3)	0.035(2)	0.030(2)	0.055(2)	0.011(1)	0.002(2)	0.013(1)
N(1)	2*i*		–0.0481(5)	0.7438(4)	0.1445(3)	0.032(2)	0.030(2)	0.040(2)	0.013(1)	0.003(2)	0.014(2)
N(2)	2*i*		–0.1495(6)	0.6049(4)	0.1051(3)	0.038(2)	0.031(2)	0.040(2)	0.013(2)	0.006(2)	0.013(2)
N(3)	2*i*		0.4527(6)	0.7515(4)	0.3233(3)	0.037(2)	0.035(2)	0.044(2)	0.015(2)	0.003(2)	0.014(2)
C(1)	2*i*		–0.1417(7)	0.8354(5)	0.1303(4)	0.039(2)	0.036(2)	0.035(2)	0.020(2)	0.007(2)	0.016(2)
C(2)	2*i*		–0.0398(8)	0.9562(5)	0.1262(5)	0.042(3)	0.038(2)	0.061(3)	0.016(2)	0.007(2)	0.021(2)
C(3)	2*i*		–0.1328(9)	1.0468(6)	0.1151(5)	0.060(3)	0.039(3)	0.071(4)	0.022(2)	0.003(3)	0.023(3)
C(4)	2*i*		–0.321(1)	1.0151(6)	0.1054(5)	0.072(4)	0.057(3)	0.055(3)	0.043(3)	0.004(3)	0.017(3)
C(5)	2*i*		–0.4250(8)	0.8928(7)	0.1068(5)	0.047(3)	0.078(4)	0.056(3)	0.040(3)	0.013(2)	0.028(3)
C(6)	2*i*		–0.3340(7)	0.8025(6)	0.1201(4)	0.037(2)	0.057(3)	0.049(3)	0.021(2)	0.011(2)	0.028(2)
C(7)	2*i*		0.1329(6)	0.7772(4)	0.2069(4)	0.032(2)	0.036(2)	0.039(2)	0.018(2)	0.011(2)	0.017(2)
C(8)	2*i*		0.1494(6)	0.6465(4)	0.2018(3)	0.034(2)	0.031(2)	0.035(2)	0.015(2)	0.009(2)	0.013(2)
C(9)	2*i*		–0.0344(6)	0.5475(4)	0.1376(4)	0.038(2)	0.029(2)	0.035(2)	0.013(2)	0.008(2)	0.013(2)
C(10)	2*i*		–0.1085(8)	0.3971(5)	0.1081(5)	0.046(3)	0.030(2)	0.064(3)	0.009(2)	0.004(2)	0.015(2)
C(11)	2*i*		0.3085(6)	0.6370(4)	0.2602(3)	0.034(2)	0.033(2)	0.029(2)	0.015(2)	0.009(2)	0.010(2)
C(12)	2*i*		0.3230(6)	0.5046(4)	0.2597(4)	0.036(2)	0.037(2)	0.036(2)	0.020(2)	0.007(2)	0.016(2)
C(13)	2*i*		0.3264(7)	0.4803(5)	0.3528(4)	0.048(3)	0.045(3)	0.040(2)	0.016(2)	0.009(2)	0.020(2)
C(14)	2*i*		0.3427(9)	0.3591(6)	0.3545(5)	0.055(3)	0.052(3)	0.061(3)	0.011(3)	–0.002(3)	0.036(3)
C(15)	2*i*		0.3578(9)	0.2636(6)	0.2642(5)	0.054(3)	0.043(3)	0.074(4)	0.018(2)	–0.013(3)	0.026(3)
C(16)	2*i*		0.3549(8)	0.2865(5)	0.1715(5)	0.055(3)	0.040(3)	0.061(3)	0.026(2)	0.001(3)	0.008(2)
C(17)	2*i*		0.3353(7)	0.4068(5)	0.1678(4)	0.049(3)	0.044(3)	0.038(2)	0.024(2)	0.007(2)	0.014(2)
C(18)	2*i*		0.6328(6)	0.7648(5)	0.3840(4)	0.031(2)	0.040(2)	0.040(2)	0.014(2)	0.010(2)	0.018(2)
C(19)	2*i*		0.7326(7)	0.6799(5)	0.3436(4)	0.040(2)	0.054(3)	0.036(2)	0.019(2)	0.008(2)	0.008(2)
C(20)	2*i*		0.9067(7)	0.6965(6)	0.4059(4)	0.039(3)	0.063(3)	0.047(3)	0.026(2)	0.016(2)	0.017(2)
C(21)	2*i*		0.9817(7)	0.8002(6)	0.5074(4)	0.033(2)	0.068(3)	0.042(3)	0.019(2)	0.009(2)	0.025(2)
C(22)	2*i*		0.8855(8)	0.8869(5)	0.5478(4)	0.043(3)	0.044(3)	0.042(3)	0.009(2)	0.008(2)	0.008(2)
C(23)	2*i*		0.7121(7)	0.8689(5)	0.4866(4)	0.037(2)	0.037(2)	0.049(3)	0.011(2)	0.013(2)	0.013(2)

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