Crystal structure of 4-benzoyl-3-methyl-1-phenyl-2-pyrazolin-5-one sulfadiazine dimethylformamide monosolvate, C₃₀H₂₉N₇O₄S

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Abstract

$\text{C}_{30}\text{H}_{29}\text{N}_7\text{O}_4\text{S}$, triclinic, $P\bar{1}$ (no. 2), $a = 7.7793(3)$ Å, $b = 9.1053(4)$ Å, $c = 21.7108(9)$ Å, $\alpha = 97.433(2)^\circ$, $\beta = 93.025(2)^\circ$, $\gamma = 111.055(2)^\circ$, $V = 1414.9$ Å³, $Z = 2$, $R_{\text{w}}(F) = 0.0399$, $wR_{\text{ref}}(F^2) = 0.1084$, $T = 200$ K.

Table 1. Data collection and handling.

Crystal:	yellow platelets, size 0.086×0.360×0.507 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	1.64 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{\max}$:	56.74°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	38010, 7042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6091
$N(\text{param})_{\text{refined}}$:	426
Programs:	SHELXS, SAINT, SADABS, SHELXL, ORTEP-3; PLATON, MERCURY [11–16]

Source of material

The reagents were of analytical grade and were used as obtained. To a solution of synthesized 4-benzoyl-3-methyl-1-phenyl-2-pyrazolin-5-one (2.0 mmol, 0.56 g) in methanol (10 mL) was added slowly to sulfadiazine (2.0 mmol, 0.50 g) in methanol (10 mL) while stirring, and the mixture was refluxed for 3 h. The pale yellow precipitate obtained was filtered, washed with methanol, dried at room temperature and stored over fused CaCl_2 . Yellow cubic single crystals of the titled ketoimine Schiff base good enough for x-ray diffraction were grown at room temperature from slow evaporation of dimethylformamide solution.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density (HFIX 137 in the SHELX program suite [11]), with $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{C})$.

The nitrogen-bound H atoms were located on a difference Fourier map and refined freely. The solvent dimethylformamide is positionally disordered and various anisotropic displacement parameters and bond lengths for the solvent were restrained.

Discussion

The chemistry of acylpyrazolone derived heterocycles has received a lot of research attention because of their interesting reactivity and useful applications [1]. Their strong chelating property, the reason they are excellent analytical reagents, have been improved by way of forming superior group of coordination compounds known as Schiff bases that are bioactive [2, 3]. The therapeutic significant of sulfa drugs derivatives have been well established [4, 5]. The amine $-NH_2$ group they possess is an excellent nucleophile target site for electrophilic carbonyl carbon in aldehydes and ketones to form Schiff bases [6, 7]. We have reported the crystal structure of 4-benzoyl-3-methyl-1-phenyl-2-pyrazolin-5-one Schiff bases with phenylhydrazine derivatives [3, 8]. In a probe into the possible synergistic effect attributed to the combination of sulfadiazine and 4-acylpyrazolone, presented herein is the crystal and molecular structure of a 4-benzoyl-3-methyl-1-phenyl-2-pyrazolin-5-one Schiff base with sulfadiazine. In the asymmetric unit the title compound occurs together with one dimethylformamide solvent molecule. There are two notable intramolecular hydrogen bonds, both with O1, namely $N3-H3\cdots O1$ and $C12-H12\cdots O1$ that hold the phenylpyrazolone backbone essentially planar together with extended resonance. The least square plane through the atoms N1-N3, O1, C1-C3, C5, and C11-C16 has a r.m.s. of 0.0671 Å. Adjacent molecules are held together at the sulfadiazine group with two short $N4-H4\cdots N6$ hydrogen bonds with lengths of 2.062(19) Å which in terms of graph-set analyses [9, 10] can be described by means of a $R_2^2(8)$ descriptor on the unary level. There are also a large number of $\pi\cdots\pi$ ring interactions. The two shortest interactions occur with the phenyl ring C31-C36 which has two $\pi\cdots\pi$ ring interactions; one with an adjacent pyrazole ring N1, N2, C1-C3, and one with the phenyl ring C11-C16. The centroid to centroid distances are 4.2512(8) and 4.2530(9) Å, respectively. The crystal contains the solvent dimethylformamide positionally disordered in a 0.82:0.18 ratio.

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(3)	2i	0.047(2)	0.942(2)	0.2669(8)	0.048(5)
H(4)	2i	0.562(2)	0.610(2)	0.0528(9)	0.041(4)
H(4A)	2i	−0.2662	0.6735	0.4337	0.074
H(4B)	2i	−0.3631	0.7728	0.4756	0.074
H(4C)	2i	−0.1439	0.8186	0.4867	0.074
H(7)	2i	0.2081	0.2030	0.1803	0.058
H(8)	2i	0.0906	0.0282	0.0874	0.066
H(9)	2i	0.1914	0.1181	−0.0049	0.056
H(12)	2i	−0.2714	1.2294	0.2686	0.050
H(13)	2i	−0.4423	1.3869	0.2505	0.057
H(14)	2i	−0.6622	1.4072	0.3156	0.061
H(15)	2i	−0.7155	1.2655	0.3990	0.063
H(16)	2i	−0.5421	1.1116	0.4196	0.051
H(22)	2i	0.1920	0.8998	0.4533	0.046
H(23)	2i	0.2664	0.7236	0.5088	0.058
H(24)	2i	0.1791	0.4578	0.4647	0.063
H(25)	2i	0.0193	0.3653	0.3656	0.058
H(26)	2i	−0.0516	0.5411	0.3084	0.046

Table 2. continued.

Atom	Site Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(32)	2i	0.3556	0.7837	0.3296	0.039
H(33)	2i	0.5666	0.7108	0.2723	0.039
H(35)	2i	0.3543	0.8095	0.1153	0.042
H(36)	2i	0.1343	0.8707	0.1715	0.041
H(71_a)	2i 0.821	0.6264	0.0735	0.0489	0.108
H(72A_a)	2i 0.821	0.9685	0.5046	0.1175	0.137
H(72B_a)	2i 0.821	0.8766	0.4823	0.0475	0.137
H(72C_a)	2i 0.821	1.0400	0.4191	0.0609	0.137
H(73A_a)	2i 0.821	0.8841	0.2614	0.1705	0.115
H(73B_a)	2i 0.821	0.6728	0.1497	0.1479	0.115
H(73C_a)	2i 0.821	0.7275	0.3369	0.1699	0.115
H(81_b)	2i 0.179	0.9766	0.4269	0.0437	0.108
H(82A_b)	2i 0.179	1.0490	0.4666	0.1460	0.138
H(82B_b)	2i 0.179	0.9491	0.3261	0.1843	0.138
H(82C_b)	2i 0.179	0.8586	0.4545	0.1737	0.138
H(83A_b)	2i 0.179	0.7227	0.0885	0.1298	0.101
H(83B_b)	2i 0.179	0.6013	0.1005	0.0701	0.101
H(83C_b)	2i 0.179	0.5974	0.1945	0.1373	0.101

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
S(1)	2i	0.65134(4)	0.71256(4)	0.14880(2)	0.0290(2)	0.0400(2)	0.0296(2)	0.0072(1)	0.0086(1)	−0.0018(1)
O(1)	2i	−0.0871(1)	1.0861(1)	0.27987(5)	0.0474(6)	0.0444(5)	0.0352(5)	0.0257(5)	0.0126(4)	0.0148(4)
O(2)	2i	0.7569(2)	0.8474(1)	0.12137(5)	0.0421(6)	0.0431(6)	0.0415(6)	−0.0034(5)	0.0184(5)	−0.0031(5)
O(3)	2i	0.7472(1)	0.6494(2)	0.18952(5)	0.0348(5)	0.0673(7)	0.0391(6)	0.0242(5)	0.0042(4)	−0.0014(5)
N(1)	2i	−0.2821(2)	1.0651(1)	0.35996(5)	0.0392(6)	0.0357(6)	0.0303(5)	0.0206(5)	0.0070(4)	0.0078(4)
N(2)	2i	−0.3276(2)	0.9769(1)	0.40889(5)	0.0447(6)	0.0415(6)	0.0318(6)	0.0244(5)	0.0104(5)	0.0112(5)
N(3)	2i	0.0921(2)	0.8812(1)	0.28630(5)	0.0391(6)	0.0376(6)	0.0290(5)	0.0218(5)	0.0078(4)	0.0075(4)
N(4)	2i	0.5405(2)	0.5793(1)	0.08783(5)	0.0394(6)	0.0329(6)	0.0258(5)	0.0062(5)	0.0106(5)	0.0017(4)
N(5)	2i	0.3696(2)	0.3865(2)	0.14430(6)	0.0437(6)	0.0413(6)	0.0311(6)	0.0118(5)	0.0072(5)	0.0100(5)
N(6)	2i	0.3584(2)	0.3353(1)	0.03312(5)	0.0362(6)	0.0329(6)	0.0316(6)	0.0080(5)	0.0049(4)	0.0037(4)
C(1)	2i	−0.1501(2)	1.0310(2)	0.32707(6)	0.0364(6)	0.0319(6)	0.0296(6)	0.0167(5)	0.0047(5)	0.0044(5)
C(2)	2i	−0.1049(2)	0.9187(2)	0.35976(6)	0.0385(7)	0.0320(6)	0.0293(6)	0.0174(5)	0.0056(5)	0.0064(5)
C(3)	2i	−0.2242(2)	0.8917(2)	0.40871(6)	0.0423(7)	0.0383(7)	0.0321(6)	0.0224(6)	0.0091(5)	0.0082(5)
C(4)	2i	−0.2517(3)	0.7795(2)	0.45520(8)	0.060(1)	0.061(1)	0.0494(9)	0.0390(8)	0.0272(8)	0.0290(8)
C(5)	2i	0.0191(2)	0.8488(2)	0.33967(6)	0.0347(6)	0.0287(6)	0.0279(6)	0.0134(5)	0.0038(5)	0.0028(5)
C(6)	2i	0.4179(2)	0.4266(2)	0.08923(6)	0.0287(6)	0.0336(6)	0.0309(6)	0.0123(5)	0.0062(5)	0.0054(5)
C(7)	2i	0.2474(2)	0.2375(2)	0.14221(8)	0.0508(9)	0.0496(9)	0.0411(8)	0.0091(7)	0.0062(7)	0.0222(7)
C(8)	2i	0.1770(3)	0.1333(2)	0.08781(8)	0.055(1)	0.0421(8)	0.052(1)	−0.0031(7)	−0.0033(8)	0.0188(7)
C(9)	2i	0.2375(2)	0.1881(2)	0.03370(8)	0.0511(9)	0.0367(7)	0.0409(8)	0.0036(6)	−0.0032(7)	0.0044(6)
C(11)	2i	−0.3873(2)	1.1582(2)	0.34669(6)	0.0352(6)	0.0323(6)	0.0326(6)	0.0164(5)	−0.0001(5)	0.0022(5)
C(12)	2i	−0.3601(2)	1.2380(2)	0.29594(7)	0.0482(8)	0.0511(8)	0.0366(7)	0.0283(7)	0.0068(6)	0.0114(6)
C(13)	2i	−0.4629(2)	1.3307(2)	0.28506(8)	0.0536(9)	0.0557(9)	0.0434(8)	0.0306(8)	0.0025(7)	0.0163(7)
C(14)	2i	−0.5933(2)	1.3426(2)	0.32322(8)	0.0545(9)	0.057(1)	0.057(1)	0.0375(8)	0.0046(8)	0.0140(8)
C(15)	2i	−0.6234(2)	1.2597(2)	0.37279(9)	0.0537(9)	0.063(1)	0.059(1)	0.0398(9)	0.0164(8)	0.0171(8)
C(16)	2i	−0.5209(2)	1.1678(2)	0.38504(7)	0.0458(8)	0.0476(8)	0.0440(8)	0.0264(7)	0.0105(6)	0.0130(7)
C(21)	2i	0.0645(2)	0.7370(2)	0.37544(6)	0.0329(6)	0.0334(6)	0.0334(6)	0.0172(5)	0.0089(5)	0.0084(5)
C(22)	2i	0.1579(2)	0.7913(2)	0.43517(7)	0.0450(8)	0.0427(7)	0.0321(7)	0.0211(6)	0.0073(6)	0.0065(6)
C(23)	2i	0.2012(2)	0.6868(2)	0.46820(8)	0.0521(9)	0.067(1)	0.0384(8)	0.0329(8)	0.0081(7)	0.0181(7)
C(24)	2i	0.1494(2)	0.5295(2)	0.44193(9)	0.056(1)	0.059(1)	0.062(1)	0.0366(8)	0.0179(8)	0.0303(9)
C(25)	2i	0.0552(2)	0.4745(2)	0.38310(9)	0.0491(9)	0.0348(7)	0.070(1)	0.0219(7)	0.0148(8)	0.0144(7)
C(26)	2i	0.0126(2)	0.5785(2)	0.34922(7)	0.0379(7)	0.0338(7)	0.0462(8)	0.0159(6)	0.0064(6)	0.0048(6)
C(31)	2i	0.2240(2)	0.8334(2)	0.25587(6)	0.0322(6)	0.0282(6)	0.0277(6)	0.0120(5)	0.0062(5)	0.0027(5)
C(32)	2i	0.3537(2)	0.7870(2)	0.28603(6)	0.0351(6)	0.0411(7)	0.0232(6)	0.0162(5)	0.0050(5)	0.0042(5)
C(33)	2i	0.4801(2)	0.7456(2)	0.25219(6)	0.0314(6)	0.0387(7)	0.0277(6)	0.0148(5)	0.0034(5)	0.0038(5)
C(34)	2i	0.4803(2)	0.7550(2)	0.18908(6)	0.0293(6)	0.0299(6)	0.0266(6)	0.0077(5)	0.0072(5)	0.0007(5)
C(35)	2i	0.3529(2)	0.8027(2)	0.15859(6)	0.0397(7)	0.0416(7)	0.0253(6)	0.0137(6)	0.0081(5)	0.0090(5)
C(36)	2i	0.2239(2)	0.8401(2)	0.19212(6)	0.0380(7)	0.0382(7)	0.0303(6)	0.0171(6)	0.0061(5)	0.0111(5)
N(7_a)	2i 0.821	0.7975(5)	0.2830(5)	0.0847(1)	0.066(2)	0.066(1)	0.039(2)	0.037(1)	0.011(1)	0.012(1)
O(7_a)	2i 0.821	0.7234(8)	0.1756(6)	−0.0149(1)	0.264(6)	0.158(4)	0.038(1)	0.128(4)	−0.009(2)	0.002(2)
C(71_a)	2i 0.821	0.7098(6)	0.1701(4)	0.0385(2)	0.125(3)	0.094(2)	0.064(2)	0.063(2)	−0.011(2)	0.002(2)
C(72_a)	2i 0.821	0.9312(5)	0.4342(4)	0.0770(2)	0.093(2)	0.077(2)	0.123(3)	0.042(2)	0.060(2)	0.032(2)
C(73_a)	2i 0.821	0.7681(5)	0.2555(4)	0.1484(1)	0.105(2)	0.104(2)	0.050(2)	0.066(2)	0.029(2)	0.024(2)
N(8_b)	2i 0.179	0.827(3)	0.294(2)	0.0979(7)	0.066(2)	0.066(1)	0.039(2)	0.037(1)	0.011(1)	0.012(1)
O(8_b)	2i 0.179	0.812(3)	0.257(3)	−0.0079(9)	0.14(1)	0.15(2)	0.12(1)	0.01(1)	−0.05(1)	0.00(1)
C(81_b)	2i 0.179	0.875(3)	0.329(2)	0.0427(6)	0.125(3)	0.094(2)	0.064(2)	0.063(2)	−0.011(2)	0.002(2)
C(82_b)	2i 0.179	0.929(2)	0.393(2)	0.1551(7)	0.09(1)	0.074(9)	0.10(1)	0.021(8)	0.023(8)	−0.017(8)
C(83_b)	2i 0.179	0.675(2)	0.159(2)	0.1097(6)	0.079(8)	0.070(7)	0.065(8)	0.040(6)	0.015(6)	0.015(6)

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