Crystal structure of 4-(4-pyridil)-3,5-dimethylpyrozole hydrate, $(C_5H_4N)CH_3(C_3N_2H)CH_3 \cdot H_2O$

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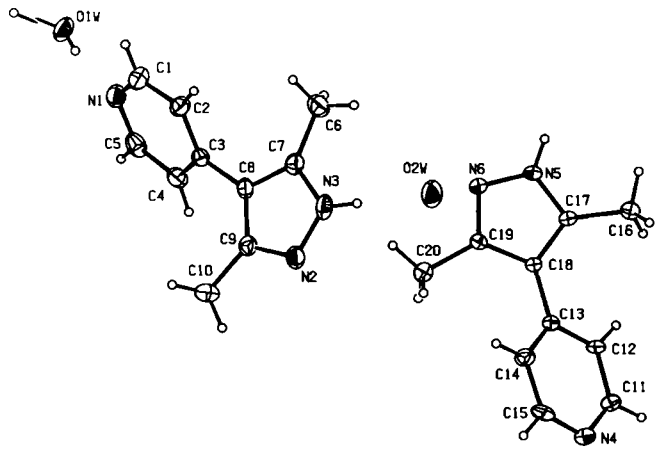

Abbstract

$C_{100}H_{13}N_3O$, triclinic, $P\bar{1}$ (no. 2), $a = 7.475(2)$ Å, $b = 9.171(2)$ Å, $c = 15.536(3)$ Å, $\alpha = 80.88(3)°$, $\beta = 89.91(3)°$, $\gamma = 81.24(3)°$, $V = 1039.0$ Å^3, $Z = 4$, $R_{gt(F)} = 0.063$, $wR_{ref(F^2)} = 0.085$, $T = 293$ K.

Soource of material

A solution of 3.54 g (0.02 mol) 3-(4-pyridyl)pentane-2,4-dione anod 2.14 g (0.02 mol) hydrazine hydrate solution (30 % a.q.) in 30) ml methanol was refluxed for half of an hour. The solvent was remmoved and the residual was washed with water. The product waas recrystallized in acetone. The yellow crystals were obtained byy evaporating the solution at the room temperature.

Exxperimental details

Thhe H atoms of one water molecule (O1W) were located by difference Fourier maps and were refined isotropically. The H atoms of f the other water molecule (O2W) were not found. The positions of f other hydrogen atoms were calculated.

Diiscussion

Ann increasing interest has been payed attention to self-assembling poblymetallic coordination compounds and supermolecules which usually possess interesting structures and potential application suuch as inclusion and shape-selective catalysis [1-4]. Here we describe the crystal structure of the new ligand 4-(4-pyridyl)-3,5-diimethylpyrozole (I) and compare its structure and coordination prroperties with other similar compound.

The structure consists of two independent molecules, which have the same composition but slightly different structures. In the two molecules, the lengths of N—C of pyridyl (py) and C—N and N—N of 3,5-dimethylpyrozole (dipz) are slightly different. The bond angles ∠N–N–C are somewhat different, in one molecule (figure, left) they are 105.6(2)° and 111.3(2)° while in the other molecule (figure, right) they are 103.8(2) and 113.8(2)°.

In **I**, the bond lengths C—N (1.319(4) Å – 1.355(3) Å) and N—N (1.336(2) Å – 1.370(3) Å) of dipz ring agree well with those in similar compounds such as 3,5-dimethylpyrazole in $[C_{10}H_{10}NO_2]_2 \cdot 2H_2O$ (**II**) [2]. The C—N bond lengths are in the range 1.319(4) Å to 1.342(4) Å. The bond angles ∠N–C–C (106.0(2)° – 112.4(2)°) of pz in **I** are similar to those angles in **II**, while the bond angles ∠C–N–N (103.8(2)° – 113.8(2)°) of pz in **I** are shorter than those in **II**. The bond angles ∠N–C–C are from 124.0(3)° to 126.4(3)°. Since the ligand **I** has two coordinating moieties, py and dipz, it has more diverse coordination modes than similar compounds (**II**), namely two monodentate modes, two bidentate modes and one tridentate mode.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.12 × 0.47 × 0.54 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.83 cm^{-1}
Diffractometer, scan mode:	Rigaku R-axis RAPID IP, ω/φ
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6798, 4535
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1570
$N(param)_{refined}$:	254
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	2*i*	−0.3698	0.1650	0.5022	0.079
H(5A)	2*i*	0.2601	0.1823	0.9873	0.056
H(1B)	2*i*	0.0997	0.6676	0.2513	0.085
H(2B)	2*i*	−0.1040	0.5246	0.3086	0.068
H(4B)	2*i*	0.3074	0.1916	0.3800	0.071
H(5B)	2*i*	0.4973	0.3516	0.3221	0.080
H(6B)	2*i*	−0.3549	0.4159	0.5178	0.117
H(6C)	2*i*	−0.2804	0.5093	0.4360	0.117
H(6D)	2*i*	−0.1532	0.4440	0.5176	0.117
H(10A)	2*i*	0.0002	−0.1015	0.3410	0.112
H(10B)	2*i*	0.1680	−0.0207	0.3500	0.112
H(10C)	2*i*	0.0319	0.0344	0.2708	0.112

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(11A)	2*i*	0.3108	−0.5862	1.1151	0.063
H(12A)	2*i*	0.3393	−0.3381	1.0952	0.051
H(14A)	2*i*	0.1734	−0.3059	0.8474	0.062
H(15A)	2*i*	0.1638	−0.5568	0.8753	0.068
H(16A)	2*i*	0.1649	0.0528	1.1185	0.076
H(16B)	2*i*	0.2604	−0.1138	1.1384	0.076
H(16C)	2*i*	0.0602	−0.0764	1.1022	0.076
H(20A)	2*i*	0.4383	−0.0165	0.7543	0.094
H(20B)	2*i*	0.3237	−0.1476	0.7698	0.094
H(20C)	2*i*	0.5213	−0.1710	0.8086	0.094
H(1W)	2*i*	0.631(4)	0.767(3)	0.215(1)	0.091
H(2W)	2*i*	0.485(3)	0.653(3)	0.260(2)	0.091

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.3222(4)	0.5263(3)	0.2814(2)	0.077(2)	0.071(2)	0.055(2)	−0.028(2)	0.014(2)	−0.012(22)
N(2)	2*i*	−0.2030(3)	0.0499(3)	0.4288(2)	0.071(2)	0.072(2)	0.048(2)	−0.025(2)	0.013(2)	−0.016(22)
N(3)	2*i*	−0.2797(3)	0.1675(3)	0.4678(2)	0.067(2)	0.087(2)	0.048(2)	−0.032(2)	0.019(2)	−0.008(22)
N(4)	2*i*	0.2347(3)	−0.5988(3)	0.9970(2)	0.062(2)	0.036(2)	0.061(2)	−0.008(1)	0.003(2)	−0.009(22)
N(5)	2*i*	0.2764(3)	0.1010(2)	0.9654(2)	0.064(2)	0.029(2)	0.050(2)	−0.012(1)	0.008(1)	−0.014(11)
N(6)	2*i*	0.3367(3)	0.0939(2)	0.8849(2)	0.065(2)	0.032(2)	0.046(2)	−0.010(1)	0.010(1)	−0.006(11)
C(1)	2*i*	0.1425(5)	0.5708(4)	0.2785(2)	0.089(3)	0.064(3)	0.059(2)	−0.022(3)	0.012(2)	−0.001(22)
C(2)	2*i*	0.0185(4)	0.4852(3)	0.3123(2)	0.057(2)	0.048(2)	0.060(2)	−0.006(2)	0.006(2)	0.002(22)
C(3)	2*i*	0.0741(4)	0.3399(3)	0.3521(2)	0.050(2)	0.045(2)	0.036(2)	−0.008(2)	−0.001(2)	−0.011(22)
C(4)	2*i*	0.2609(4)	0.2888(3)	0.3549(2)	0.059(2)	0.058(2)	0.063(2)	−0.006(2)	0.010(2)	−0.021(22)
C(5)	2*i*	0.3737(4)	0.3869(5)	0.3192(2)	0.049(2)	0.093(3)	0.069(3)	−0.024(3)	0.015(2)	−0.039(22)
C(6)	2*i*	−0.2508(4)	0.4264(3)	0.4824(2)	0.088(3)	0.074(3)	0.084(3)	−0.030(2)	0.030(2)	−0.036(22)
C(7)	2*i*	−0.1954(4)	0.2881(3)	0.4453(2)	0.057(2)	0.062(2)	0.046(2)	−0.025(2)	0.003(2)	−0.011(22)
C(8)	2*i*	−0.0550(4)	0.2444(3)	0.3916(2)	0.062(2)	0.062(2)	0.040(2)	−0.027(2)	0.014(2)	−0.015(22)
C(9)	2*i*	−0.0668(4)	0.0978(3)	0.3832(2)	0.057(2)	0.054(2)	0.038(2)	−0.010(2)	0.000(2)	−0.004(22)
C(10)	2*i*	0.0432(4)	−0.0069(3)	0.3316(2)	0.094(3)	0.047(2)	0.084(3)	−0.006(2)	0.005(2)	−0.020(22)
C(11)	2*i*	0.2854(4)	−0.5301(3)	1.0600(2)	0.063(2)	0.036(2)	0.053(2)	−0.001(2)	0.003(2)	0.003(22)
C(12)	2*i*	0.3027(4)	−0.3796(3)	1.0485(2)	0.054(2)	0.023(2)	0.052(2)	−0.005(2)	0.005(2)	−0.010(22)
C(13)	2*i*	0.2654(4)	−0.2906(3)	0.9671(2)	0.049(2)	0.033(2)	0.041(2)	−0.010(2)	0.006(2)	−0.009(22)
C(14)	2*i*	0.2067(4)	−0.3598(3)	0.9024(2)	0.066(2)	0.042(2)	0.050(2)	−0.016(2)	−0.002(2)	−0.011(22)
C(15)	2*i*	0.1983(4)	−0.5118(3)	0.9209(2)	0.066(2)	0.042(2)	0.070(3)	−0.010(2)	−0.001(2)	−0.032(22)
C(16)	2*i*	0.1762(3)	−0.0433(3)	1.1002(2)	0.054(2)	0.042(2)	0.053(2)	−0.000(2)	0.006(2)	−0.007(22)
C(17)	2*i*	0.2432(3)	−0.0318(3)	1.0091(2)	0.040(2)	0.027(2)	0.042(2)	−0.004(1)	−0.001(2)	−0.004(22)
C(18)	2*i*	0.2839(3)	−0.1334(3)	0.9529(2)	0.043(2)	0.026(2)	0.037(2)	−0.005(2)	0.004(2)	−0.005(22)
C(19)	2*i*	0.3404(4)	−0.0483(3)	0.8770(2)	0.049(2)	0.034(2)	0.039(2)	−0.001(2)	0.006(2)	−0.008(22)
C(20)	2*i*	0.4124(4)	−0.1006(3)	0.7950(2)	0.088(3)	0.049(2)	0.048(2)	−0.008(2)	0.009(2)	−0.002(22)
O(1W)	2*i*	0.5957(3)	0.7063(2)	0.2708(1)	0.101(2)	0.075(2)	0.061(2)	−0.049(2)	0.016(2)	−0.003(11)
O(2W)	2*i*	−0.5753(3)	0.1609(2)	0.5812(1)	0.144(2)	0.107(2)	0.085(2)	−0.077(2)	0.051(2)	−0.037(22)

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