Crystal structure of 4-(5-(benzoyloxy)-3-methyl-1*H*-pyrazol-1-yl)benzoic acid monohydrate, $C_{18}H_{16}N_2O_5$

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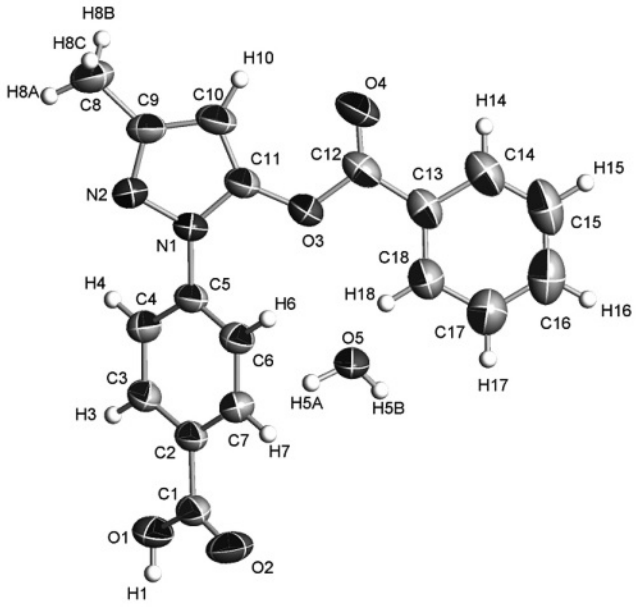

Abstract

$C_{18}H_{16}N_2O_5$, triclinic, $P\bar{1}$ (no. 2), a = 8.716(1) Å, b = 9.969(1) Å, c = 11.435(2) Å, α = 104.535(3)°, β = 104.039(4)°, γ = 111.181(2)°, V = 833.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.0446, $wR_{ref}(F^2)$ = 0.1276, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.22×0.34×0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	55.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7869, 3853
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2200
$N(param)_{refined}$:	237
Programs:	SHELX, DIAMOND, ORTEP-3, MERCURY, PLATON [11–15]

Source of material

The title compound was prepared following the reported procedures [1–4]. 4-Hydrazinobenzoic acid (3.05 g, 0.020 mol) was added to 20 ml anhydrous ethanol solution of ethyl acetoacetate (2.61 g, 0.020 mol) in a 100 ml flask. The mixture was heated at reflux and stirred for 2 h. The solvent was removed, obtaining a the light yellow powder, which was washed with anhydrous ether (20 ml), dried *in vacuo* to constant weight and shown to be 4-(5-hydroxy-3-methyl-1*H*-pyrazol-1-yl)benzoic acid. 4-(5-hydroxy-3-methyl-1*H*- pyrazol-1-yl) benzoic acid (2.20 g, 0.010 mol) and benzoyl chloride (1.42 g, 0.010 mol) were added to 100 ml 1,4-dioxane and heated at reflux and stirred for 1 h. After filtration, the solvent was removed, the sandy-brown powder was dried *in vacuo* to constant weight and shown to be 4-(5-(benzoyloxy)-3-methyl-1*H*-pyrazol-1-yl)benzoic acid (yield 64%). The yellow single crystals were obtained by slow evaporation form an ethylacetate solution at room temperature after several days.

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Experimental details

All H-atoms were positioned with idealized geometry and refined isotropic (U_{iso}(H) = 1.5U_{eq}(C) for hydroxy H atoms and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms) using a riding model with C–H = 0.93 Å, O–H = 0.82 Å.

Discussion

The pyrazole compounds are important targets coordination chemistry [5–7]. The asymmetrical coordination moiety of the pyrazole compounds could play a key role in the catalytic properties of new organometal pyrazole, a vast area of coordination chemistry that is waiting to be further explored and developed, particularly in the case of group VIIIB metals, which are known to be potential catalysts in a variety of applications [8]. The introduction of carboxyl, in the structure of pyrazole compounds, can improve the catalytic ability of metal complexes [9, 10]. In recent years, we have been engaged in the preparation of the derivatives of pyrazole for designing new pyrazole with potential catalytic application. Herein we report the crystal structure of a compound which may be used as a new precursor for obtaining metal complexes catalyst. The title crystal structure is built by the $C_{18}H_{16}N_2O_5$ molecules, in which all bond lengths and angles are in normal ranges. The benzene rings (C2–C7 and C13–C18) of the title compound make dihedral angles of 36.95(10) and 8.25(11)° with the pyrazolone moiety, respectively. In addition, there are several hydrogen bond interactions between the adjacent units involving to the pyridyl, carboxyl and water molecules. Some are listed as follows: O5–H5B···O2, [O···O= 2.775(3) Å, O–H···O= 165(3)°]; O5–H5A···N2$^{\#1}$, [O···N= 2.833(3) Å, O–H···N = 173(2)°]; O1–H1···O5$^{\#2}$, [O···O= 2.563(2) Å, O–H···O = 172°]. Symmetry codes: #1: x, –1+y, z, #2: 2–x,1–y,1–z. These connection construct a one-dimensional (1D) chain structure along the b axis.

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	–0.0323	0.2893	0.5409	0.100
H(6)	2*i*	0.6552	0.2100	0.6106	0.059
H(3)	2*i*	0.1123	0.0750	0.6561	0.061
H(4)	2*i*	0.2888	–0.0359	0.7284	0.062

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(7)	2*i*	0.4790	0.3213	0.5407	0.061
H(8A)	2*i*	0.4918	–0.3895	0.7382	0.108
H(8B)	2*i*	0.6770	–0.3207	0.8493	0.108
H(8C)	2*i*	0.6587	–0.3757	0.7034	0.108
H(10)	2*i*	0.9273	–0.0296	0.8528	0.074
H(14)	2*i*	1.3667	0.5185	1.0049	0.091
H(18)	2*i*	0.9023	0.4697	0.7839	0.085
H(16)	2*i*	1.3293	0.8807	0.9371	0.109
H(15)	2*i*	1.4958	0.7743	1.0309	0.108
H(17)	2*i*	1.0339	0.7268	0.8112	0.106
H(5A)	2*i*	0.711(3)	0.281(3)	0.411(2)	0.105(9)
H(5B)	2*i*	0.830(3)	0.446(3)	0.497(2)	0.092(8)

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}
O(1)	2*i*	0.0284(2)	0.2537(2)	0.5763(1)	0.0535(8)	0.0733(9)	0.089(1)	0.0401(7)	0.0246(8)	0.0397(8)
C(2)	2*i*	0.2787(2)	0.2108(2)	0.5922(2)	0.043(1)	0.041(1)	0.0489(9)	0.0214(8)	0.0078(8)	0.0109(8)
N(1)	2*i*	0.5980(2)	0.0105(2)	0.7246(1)	0.0434(8)	0.0508(9)	0.0555(8)	0.0257(7)	0.0118(7)	0.0236(7)
N(2)	2*i*	0.5201(2)	–0.1410(2)	0.7131(1)	0.0526(9)	0.0499(9)	0.0587(9)	0.0266(8)	0.0148(7)	0.0252(7)
C(6)	2*i*	0.5469(2)	0.1840(2)	0.6210(2)	0.045(1)	0.054(1)	0.055(1)	0.0263(9)	0.0174(8)	0.0222(9)
O(3)	2*i*	0.8691(2)	0.2261(2)	0.8078(1)	0.0424(7)	0.0585(8)	0.0740(8)	0.0229(6)	0.0083(6)	0.0247(7)
C(5)	2*i*	0.4905(2)	0.0790(2)	0.6789(2)	0.044(1)	0.045(1)	0.0464(9)	0.0239(8)	0.0099(8)	0.0153(8)
O(2)	2*i*	0.2097(2)	0.3695(2)	0.4865(2)	0.079(1)	0.086(1)	0.103(1)	0.0579(9)	0.0407(9)	0.0579(9)
C(3)	2*i*	0.2220(2)	0.1030(2)	0.6478(2)	0.042(1)	0.050(1)	0.061(1)	0.0233(8)	0.0157(8)	0.0193(9)
C(4)	2*i*	0.3273(2)	0.0367(2)	0.6912(2)	0.049(1)	0.052(1)	0.060(1)	0.0255(9)	0.0192(9)	0.0261(9)
C(7)	2*i*	0.4409(2)	0.2497(2)	0.5788(2)	0.052(1)	0.049(1)	0.055(1)	0.0254(9)	0.0171(9)	0.0230(9)
C(1)	2*i*	0.1697(2)	0.2857(2)	0.5464(2)	0.049(1)	0.044(1)	0.056(1)	0.0255(9)	0.0116(9)	0.0135(9)
O(4)	2*i*	1.1301(2)	0.2389(2)	0.9151(2)	0.0573(9)	0.087(1)	0.095(1)	0.0388(8)	0.0010(8)	0.0273(9)
C(12)	2*i*	1.0484(2)	0.3028(2)	0.8756(2)	0.044(1)	0.074(1)	0.052(1)	0.028(1)	0.0091(9)	0.015(1)
C(13)	2*i*	1.1212(2)	0.4666(2)	0.8908(2)	0.045(1)	0.064(1)	0.048(1)	0.019(1)	0.0139(8)	0.0097(9)
C(11)	2*i*	0.7758(2)	0.0726(2)	0.7852(2)	0.045(1)	0.054(1)	0.055(1)	0.0268(9)	0.0112(8)	0.0191(9)
C(9)	2*i*	0.6526(3)	–0.1693(2)	0.7653(2)	0.059(1)	0.059(1)	0.054(1)	0.036(1)	0.0156(9)	0.0250(9)
C(8)	2*i*	0.6168(3)	–0.3279(2)	0.7639(2)	0.081(1)	0.067(1)	0.078(1)	0.045(1)	0.020(1)	0.035(1)
C(10)	2*i*	0.8158(3)	–0.0386(2)	0.8121(2)	0.053(1)	0.067(1)	0.066(1)	0.037(1)	0.010(1)	0.024(1)
C(14)	2*i*	1.2989(3)	0.5600(3)	0.9654(2)	0.052(1)	0.083(2)	0.067(1)	0.020(1)	0.011(1)	0.014(1)
C(18)	2*i*	1.0222(3)	0.5308(2)	0.8337(2)	0.055(1)	0.062(1)	0.080(1)	0.019(1)	0.016(1)	0.022(1)
C(16)	2*i*	1.2770(4)	0.7763(3)	0.9250(2)	0.088(2)	0.069(2)	0.081(2)	0.008(2)	0.033(1)	0.016(1)
C(15)	2*i*	1.3758(3)	0.7128(3)	0.9815(2)	0.062(2)	0.086(2)	0.072(2)	0.002(1)	0.016(1)	0.009(1)
C(17)	2*i*	1.1008(3)	0.6845(3)	0.8506(2)	0.080(2)	0.072(2)	0.097(2)	0.024(1)	0.025(1)	0.032(1)
O(5)	2*i*	0.8186(2)	0.3559(2)	0.4778(2)	0.0479(9)	0.054(1)	0.117(1)	0.0251(8)	0.0094(9)	0.0367(9)

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