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Crystal structure of dihydrazinium 1*H*-pyrazole-3,5-dicarboxylate, C₅H₁₂N₆O₄

Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	0.22 × 0.16 × 0.07 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	33.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	32710, 3560, 0.039
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2985
$N(\text{param})_{\text{refined}}$:	184
Programs:	Bruker programs [1], SHELX [2, 3]

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Abstract

C₅H₁₂N₆O₄, monoclinic, $P2_1/n$ (no. 14), $a = 4.3368(6)$ Å, $b = 15.483(2)$ Å, $c = 13.8852(19)$ Å, $\beta = 97.714(3)^\circ$, $V = 923.9(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0411$, $wR_{\text{ref}}(F^2) = 0.1109$, $T = 200(2)$ K.

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The asymmetric unit of the title crystal 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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Source of material

In an attempt to synthesize the intermediates of 1*H*-pyrazole-3,5-dicarboxylic acid monohydrate with direct synthesis starting from 3,5-pyrazoledicarboxylic acid monohydrate and hydrazine monohydrate in a stoichiometric relationship, a microcrystalline light yellow mixture was obtained with a pair of monocrystals that were mechanically isolated from the mixture and prepared for X-ray analysis.

Experimental details

All hydrogen atoms were identified in difference Fourier map and were refined isotropically.

Discussion

Pyrazole-related molecules have attracted much attention because of their diverse pharmacological properties [4], and also because of their increased use in the synthesis of new functional materials [5]. The derivatives of 3,5-pyrazoledicarboxylic acid (H₃PZDC) are known as building components of either purely organic or organometallic materials. As ligands the H₃PZDC derivatives can display up to six metal coordination sites and various bridging modes [6] that is utilized for the synthesis of polynuclear magnetic solids [7] and MOFs [8]. On the other hand, the uncoordinated H₃PZDC possesses multiple hydrogen bonding sites and can generate extensive hydrogen bonding important for supramolecular organic networks. [9] As a continuation of our research on pyrazole-derived molecules [10, 11], the present

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} */U _{eq}
O1	1.0208(2)	0.37998(5)	0.00034(6)	0.02391(18)
O2	0.95263(19)	0.27289(5)	0.10278(5)	0.01774(16)
O3	0.3217(2)	0.57757(5)	0.34723(6)	0.02178(17)
O4	0.3622(2)	0.43877(6)	0.39166(6)	0.02606(19)
N1	0.7665(2)	0.49729(5)	0.12136(6)	0.01619(17)
N2	0.6304(2)	0.54007(5)	0.18837(6)	0.01656(17)
N3	0.4105(2)	0.19896(6)	0.30831(7)	0.01824(17)
N4	0.0802(2)	0.21603(6)	0.29600(7)	0.01688(17)
N5	−0.1350(2)	0.31100(6)	0.45979(7)	0.01783(17)
N6	0.0646(2)	0.36462(6)	0.52751(6)	0.01631(17)
C1	0.7787(2)	0.41106(6)	0.13768(7)	0.01305(17)
C2	0.6380(2)	0.39651(6)	0.21949(7)	0.01395(17)
C3	0.5512(2)	0.47861(6)	0.24878(7)	0.01327(17)
C4	0.9279(2)	0.35062(6)	0.07503(7)	0.01376(17)
C5	0.3981(2)	0.50030(6)	0.33578(7)	0.01423(17)
H1	0.836(4)	0.5238(11)	0.0767(13)	0.028(4)*
H2	0.601(3)	0.3413(10)	0.2477(11)	0.019(3)*
H31	0.448(4)	0.1762(11)	0.3700(13)	0.028(4)*
H32	0.428(4)	0.1547(11)	0.2676(12)	0.027(4)*
H41	0.035(4)	0.2515(11)	0.3457(13)	0.026(4)*
H42	0.032(4)	0.2416(11)	0.2356(13)	0.027(4)*
H43	−0.042(4)	0.1653(12)	0.3002(12)	0.031(4)*
H51	0.216(4)	0.3295(11)	0.5599(11)	0.023(4)*
H52	−0.046(4)	0.3928(11)	0.5707(13)	0.032(4)*
H53	0.163(4)	0.4018(11)	0.4929(13)	0.031(4)*
H61	−0.290(4)	0.3452(11)	0.4301(12)	0.027(4)*
H62	−0.218(4)	0.2750(11)	0.4960(13)	0.031(4)*

work describes the crystal structure of novel hydrazinium(+1) salt of 3,5-pyrazoledicarboxylic acid, (N₂H₅)₂-HPZDC.

The crystal structures of two hydrazine salts of H₃PZDC have been reported previously. These salts are of the type N₂H₆·(H₂PZDC)₂ [12] and N₂H₅·H₂PZDC·H₂O [9] and both contain the monocarboxylate H₂PZDC[−] anion. The asymmetric unit of title salt (N₂H₅)₂-HPZDC contains the dicarboxylate dianion of H₃PZDC and two hydrazinium counterions. In comparison to previous structures [12, 13] the geometry of species shows expected differences arising from their different protonation states. The crystal structure is stabilized by extensive N—H···O and N—H···N hydrogen bonding between the charged species with H···A distances ranging from 1.84(2) to 2.32(2) Å. The HPZDC anions alone form N1—H1···O1 centrosymmetric dimer [N1—H1···O1ⁱ = 161(2)°, H1···O1 = 1.98(2) Å, (i) = 2 − x, 1 − x, −z].

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