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The crystal structure of 2-carboxyl-4-nitroimidazole monohydrate, $C_4H_5N_3O_5$

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Abstract

$C_4H_5N_3O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 6.3017(7)$ Å, $b = 6.9235(7)$ Å, $c = 8.2333(8)$ Å, $\alpha = 100.604(3)^\circ$, $\beta = 90.862(4)^\circ$, $\gamma = 108.102(4)^\circ$, $V = 334.60(6)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0616$, $wR_{ref}(F^2) = 0.1414$, $T = 170.0$ 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.

Source of material

All of reagents were purchased with analysis grade. In representative experiments, 5 ml HNO_3 and 15 ml H_2SO_4

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.08 × 0.05 × 0.04 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ :	0.16 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	3611, 1234, 0.064
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 787
$N(param)_{refined}$:	118
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

were dissolved in 20 ml solvent, then 5.6 g (50 mmol) 2-carboxy-imidazole were added and the target compound were obtained by reaction at 333 K for 6 h. The reaction mixture was then cooled to room temperature and filtered. A single crystal of the title compound was obtained by slow evaporation at 293 K.

Experimental details

Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program using Intrinsic Phasing and refined with the ShelXL [4] refinement package. All H atoms were positioned geometrically and treated as riding, and $U_{iso}(H) = 1.5U_{eq}(O)$, $U_{iso}(H) = 1.2U_{eq}(N)$.

Comment

Nitroimidazole derivatives has been one of the hot topics in energetic materials in recent years [5, 6]. A series of related compounds have been synthesized, for example polyacetylene bearing imidazole moieties, imidazole-functionalized polyfluorene, 2,4-dinitroimidazole, 2-aminoimidazole, 1-methyl-2,4,5-trinitroimidazole, etc. [7–9]. The title compound which features a carboxy group attached to the nitroimidazole ring is expected to become a new function material. Here we report the crystal structure of the title compound (cf. the figure).

The asymmetric unit of the title structure contains one target molecule and one water molecule. The imidazole ring is almost flat, and the geometric parameters are all in

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.7478 (6)	0.7629 (5)	0.4856 (5)	0.0295 (9)
C2	0.5642 (6)	0.6561 (5)	0.3547 (4)	0.0272 (9)
C3	0.3473 (6)	0.4387 (5)	0.1621 (4)	0.0286 (9)
C4	0.2550 (6)	0.5943 (5)	0.2003 (5)	0.0335 (10)
H4A	0.121864	0.603495	0.151350	0.040*
N1	0.3967 (5)	0.7320 (4)	0.3236 (4)	0.0342 (8)
H1	0.382820	0.850293	0.374736	0.041*
N2	0.5397 (5)	0.4748 (4)	0.2564 (4)	0.0288 (8)
N3	0.2606 (5)	0.2532 (5)	0.0371 (4)	0.0357 (8)
O1	0.3673 (5)	0.1296 (4)	0.0159 (4)	0.0465 (8)
O2	0.0857 (5)	0.2272 (4)	−0.0448 (4)	0.0475 (8)
O3	0.7564 (4)	0.9268 (4)	0.5765 (3)	0.0363 (7)
O4	0.8883 (4)	0.6617 (4)	0.4925 (3)	0.0356 (7)
H4	0.987 (7)	0.711 (6)	0.576 (5)	0.053*
O5	0.7819 (5)	0.1910 (4)	0.2834 (4)	0.0450 (8)
H5A	0.718 (8)	0.277 (7)	0.268 (6)	0.067*
H5B	0.766 (8)	0.102 (7)	0.195 (6)	0.067*

expected ranges [10, 11]. Both molecules forming this structure are connected by classical hydrogen bonds.

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