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The crystal structure of 1-methyl-1*H*-pyrazol-2-ium nitrate, C₄H₇O₃N₃

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.08 × 0.05 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3531, 737, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 585
$N(\text{param})_{\text{refined}}$:	65
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

C₄H₇O₃N₃, orthorhombic, *Pnma* (no. 62), $a = 14.2289(10)$ Å, $b = 6.1881(4)$ Å, $c = 7.5592(5)$ Å, $V = 665.59(8)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0427$, $wR_{\text{ref}}(F^2) = 0.1098$, $T = 150$ K.

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The molecular 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

An amount of 15.34 g of 15% nitric acid was added to the reactor. Then 3 g of 1-methylpyrazole was added to the

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dropping funnel. Keeping the temperature below 283 K, 1-methylpyrazole was slowly added to 15% nitric acid and reacted for 30 min. After the reaction the reaction solution was poured into the evaporating dish. Colorless rhombic crystals were obtained by evaporation at room temperature.

Experimental details

Hydrogen atom was placed in their geometrically idealized positions and constrained to ride on their parent atoms. All the non-hydrogen atoms were refined anisotropically.

Comment

The compound 1-methyl-3,4,5-trinitropyrazole is a high energy insensitive simple explosive [5–8]. However, most of the processes of the synthesis of 1-methyl-3,4,5-trinitropyrazole have complex steps and low yield [9, 10]. The title 1-methylpyrazole nitrate can be obtained from 1-methylpyrazole by nitration to 1-methyl-3,4,5-trinitropyrazole. It is an organic intermediate with research value and application prospect.

The asymmetric unit of the title compound includes a 1-methylpyrazole cation and a nitrate ion. The bond lengths and angles within these moieties are in the expected ranges [11]. Four carbon atoms and two nitrogen atoms in the 1-methylpyrazole group are in the same plane. More interestingly, the plane formed by 1-methylpyrazole is almost the same as that formed by nitrate ions. Some hydrogen bonds connect anions and cations.

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.55042 (10)	0.250000	0.5349 (2)	0.0363 (5)
O2	0.65501 (11)	0.250000	0.3276 (2)	0.0419 (5)
O3	0.50675 (14)	0.250000	0.2602 (2)	0.0463 (6)
N2	0.36760 (13)	0.250000	0.5856 (2)	0.0273 (5)
N1	0.33810 (13)	0.250000	0.7545 (2)	0.0276 (5)
N3	0.57104 (13)	0.250000	0.3713 (3)	0.0303 (5)
C2	0.24443 (17)	0.250000	0.7564 (4)	0.0359 (7)
H2A	0.205788	0.250000	0.858919	0.043*
C3	0.21338 (16)	0.250000	0.5833 (4)	0.0385 (7)
H3	0.150044	0.250000	0.543535	0.046*
C4	0.29281 (16)	0.250000	0.4807 (3)	0.0339 (6)
H4	0.294106	0.250000	0.355042	0.041*
C1	0.40502 (19)	0.250000	0.8999 (3)	0.0410 (7)
H1C ^a	0.372162	0.282803	1.010671	0.062*
H1B ^a	0.453335	0.359746	0.878360	0.062*
H1A ^a	0.434655	0.107450	0.908807	0.062*
H2	0.4313 (14)	0.250000	0.551 (4)	0.084 (12)*

^aOccupancy: 0.5.

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