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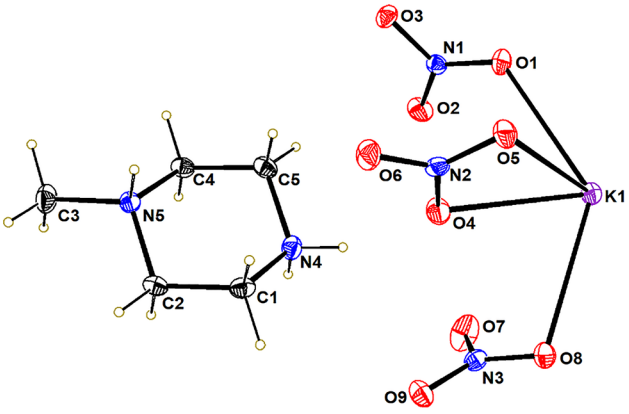
The crystal structure of the double salt potassium 1-methylpiperazine-1,4-di-ium trinitrate, $C_5H_{14}KN_5O_9$



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Abstract

$C_5H_{14}KN_5O_9$, orthorhombic, *Pbca* (no. 61), a = 10.4760(3) Å, b = 14.0476(4) Å, c = 17.3708(5) Å, V = 2,556.33(13) Å^3, Z = 8, T = 100 K, $R_{gt}(F)$ = 0.0226, $wR_{ref}(F^2)$ = 0.0607.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

Deionized water is homemade in the laboratory. 1.002 g 1-Methylpiperazine (10 mmol), 1.011 g KNO_3 (10 mmol), and 1.983 g nitric acid (20 mmol) dissolved into the 30 ml deionized water with stirring. The above mixture was stirred at 50 °C for 30 min. The obtained solution was slowly evaporated at room temperature for two weeks to give colorless and clear crystals.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.23 × 0.19 × 0.15 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.47 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy R, ω
θ_{max}, completeness:	25.0°, >99 %
N $(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12,611, 2,261, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,045
$N(param)_{refined}$:	182
Programs:	SADABS,[1] SHELX,[2,3] Olex2[4]

2 Experimental details

The single crystal diffraction data collection was performed on a XtaLAB Synergy R, HyPix diffractometer. The collected diffraction pattern was indexed and refined in the CrysAlisPro software, and intensities were reduced by integration using the SAINT program, followed by absorption correction in multi-scan using the SADABS[1] program. ShelXT[2] was called in the OLEX2[4] software interface to perform the initial structure solution, and ShelXL[3] was used to perform the refinement. Hydrogen atoms were added by theoretical hydrogenation according to the riding model with a temperature factor U_{eq} of 1.2 or 1.5 times that of the parent atom. The molecular structure (Ortep: 50 %) is shown in the figure.

3 Comment

Molecular perovskite energetic materials refer to new types of energetic materials with a perovskite structure.[5–7] By using molecular self-assembly technology, these materials are constructed in a building block manner, with organic fuels as the A-site, smaller cations as the B-site, and oxidizing anion radicals as the X-site.[8] The oxidizing and reducing

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
K1	0.84277 (3)	0.773620	0.37555 (2)	0.01434 (11)
O1	0.73233 (9)	0.72233 (7)	0.51771 (5)	0.0188 (2)
O2	0.53722 (9)	0.77197 (6)	0.50332 (6)	0.0207 (2)
O3	0.57543 (9)	0.64607 (6)	0.57124 (5)	0.0158 (2)
O4	0.65776 (9)	0.63235 (7)	0.35543 (6)	0.0199 (2)
O5	0.82824 (9)	0.56295 (7)	0.39762 (6)	0.0193 (2)
O6	0.65254 (10)	0.48205 (7)	0.38403 (5)	0.0202 (2)
O7	0.56677 (10)	0.81135 (7)	0.26341 (6)	0.0281 (3)
O8	0.74596 (9)	0.75654 (7)	0.22028 (5)	0.0164 (2)
O9	0.56647 (9)	0.68702 (6)	0.19031 (5)	0.0187 (2)
N1	0.61543 (10)	0.71470 (7)	0.52972 (6)	0.0125 (2)
N2	0.71337 (11)	0.55817 (8)	0.37933 (6)	0.0138 (3)
N3	0.62498 (11)	0.75165 (8)	0.22488 (6)	0.0142 (2)
N4	0.39651 (11)	0.61057 (7)	0.34281 (6)	0.0152 (2)
H4A	0.478805	0.630333	0.337740	0.018*
H4B	0.344662	0.658792	0.327126	0.018*
N5	0.21574 (11)	0.46518 (7)	0.38598 (6)	0.0124 (2)
H5	0.278946	0.415879	0.402467	0.015*
C1	0.37485 (13)	0.52543 (9)	0.29308 (7)	0.0154 (3)
H1A	0.436728	0.474960	0.306849	0.019*
H1B	0.388848	0.542782	0.238495	0.019*
C2	0.24048 (13)	0.48831 (9)	0.30323 (7)	0.0141 (3)
H2A	0.178662	0.536900	0.285508	0.017*
H2B	0.228637	0.430447	0.271484	0.017*
C3	0.08542 (13)	0.42566 (10)	0.39748 (8)	0.0197 (3)
H3A	0.073609	0.409204	0.451843	0.030*
H3B	0.074958	0.368449	0.365817	0.030*
H3C	0.021813	0.473275	0.382366	0.030*
C4	0.23556 (13)	0.55211 (9)	0.43466 (7)	0.0145 (3)
H4C	0.219784	0.536348	0.489384	0.017*
H4D	0.174286	0.602229	0.419176	0.017*
C5	0.37012 (13)	0.58839 (9)	0.42520 (7)	0.0156 (3)
H5A	0.381911	0.646471	0.456689	0.019*
H5B	0.431173	0.539650	0.443618	0.019*

components are assembled within the crystal framework structure, resulting in molecular perovskite energetic materials that are high-density, high-energy, and relatively low-sensitivity.[5,9,10] In this work, we synthesized molecular perovskite energetic materials using nitric acid, potassium nitrate, and 1-methylpiperazine. The prepared molecular perovskite materials have *MeH2PIP* as the A-site, potassium ions as the B-site, and nitrate radical as the X-site. The compound crystallizes in the space group *Pbca* (no. 61) of the orthorhombic crystal system. In the MeH2PIP molecular, the bond lengths of C1–C2 and C4–C5 are 1.5115 and 1.5079 Å, respectively, which are in agreement with the value of a single bond. The bond lengths of N4–C1, N4–C5, N5–C2, and N5–C4 are 1.4927, 1.4906, 1.4964 and 1.4997 Å, respectively, which are in agreement with the value of C=N. Due to its location outside the ring, the bond length of N5–C3 (1.4873 Å) is slightly shorter than that of other carbon-nitrogen bonds.

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