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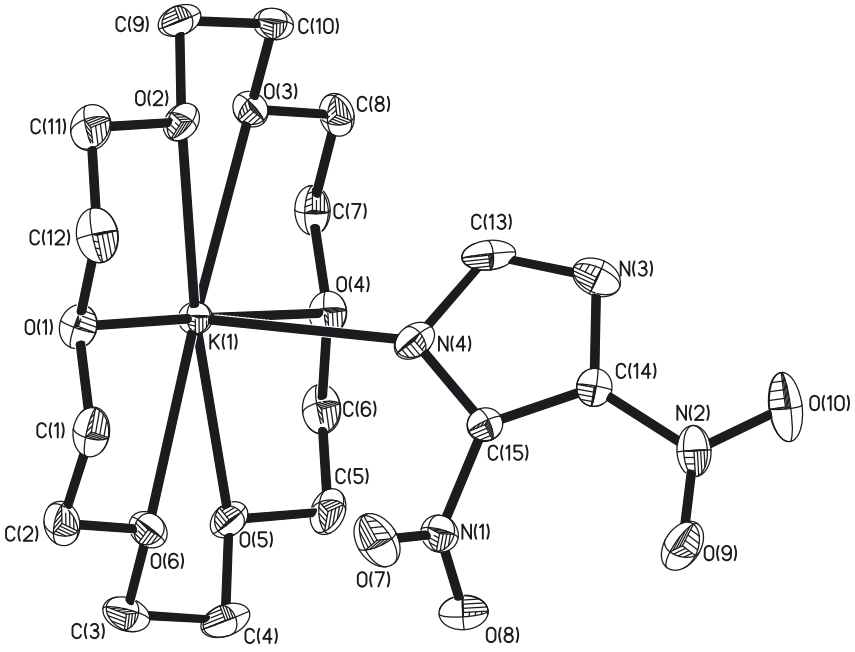
The crystal structure of the coordination compound *catena*-poly[(18-crown-6-ether-κ^6O^6)(4,5-dinitroimidazolato-κ^1O)potassium(I)]



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Abstract

$C_{15}H_{25}KN_4O_{10}$, monoclinic, $P2_1$ (no. 4), a = 8.3669(3) Å, b = 13.6610(4) Å, c = 9.6179(3) Å, β = 104.578(1)°, V = 1063.94(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0391, $wR_{ref}(F^2)$ = 0.0863, 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.09 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.31 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12736, 4284, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3768
$N(param)_{refined}$:	271
Programs:	Olex2,[1] SHELX,[2,3] Bruker[4]

1 Source of material

Add 20 mL of N,N-dimethylformamide (DMF), 3.16 g of 4,5-dinitroimidazole, 1.2 g of KOH and 5.30 g of 18-crown-6 ether to the reactor. The temperature of the reactor was raised to 323 K and the reaction lasted 1 h. After wards the reaction was complete and the temperature of the reactor was lowered to 298 K. The reaction solution is filtered and remove the filtrate and retain the yellow solid precipitate. The yellow solid is dissolved in acetone and the solution is volatilized at room temperature to obtain a yellow block crystal.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1532 (5)	0.4282 (3)	0.7906 (4)	0.0431 (10)
H1A	0.052060	0.439171	0.824192	0.052*
H1B	0.228005	0.484674	0.821344	0.052*
C2	0.2359 (5)	0.3365 (3)	0.8546 (4)	0.0461 (11)
H2A	0.246120	0.335654	0.959356	0.055*
H2B	0.169923	0.278986	0.811227	0.055*
C3	0.4837 (5)	0.2464 (3)	0.8830 (4)	0.0467 (10)
H3A	0.426292	0.188033	0.833227	0.056*
H3B	0.490155	0.240240	0.986836	0.056*
C4	0.6535 (5)	0.2534 (3)	0.8599 (4)	0.0462 (10)
H4A	0.705357	0.316175	0.898407	0.055*
H4B	0.723128	0.199236	0.910359	0.055*
C5	0.7938 (4)	0.2649 (3)	0.6750 (5)	0.0473 (11)
H5A	0.878498	0.219052	0.729110	0.057*
H5B	0.831624	0.332571	0.702138	0.057*
C6	0.7721 (4)	0.2508 (3)	0.5194 (5)	0.0441 (10)
H6A	0.880680	0.253522	0.495983	0.053*
H6B	0.722361	0.185845	0.490271	0.053*
C7	0.6273 (5)	0.3135 (3)	0.2920 (4)	0.0401 (9)
H7A	0.556745	0.254907	0.264718	0.048*
H7B	0.729678	0.303850	0.259970	0.048*
C8	0.5382 (5)	0.4022 (3)	0.2219 (4)	0.0385 (9)
H8A	0.601454	0.461812	0.260195	0.046*
H8B	0.526973	0.399620	0.116989	0.046*
C9	0.1249 (5)	0.4914 (3)	0.2230 (4)	0.0395 (9)
H9A	0.058099	0.546883	0.173404	0.047*
H9B	0.066604	0.429763	0.187310	0.047*
C10	0.2921 (5)	0.4923 (3)	0.1929 (4)	0.0358 (8)
H10A	0.280409	0.495468	0.088025	0.043*
H10B	0.354636	0.550565	0.237713	0.043*
C11	−0.0086 (4)	0.4944 (3)	0.4125 (5)	0.0457 (10)
H11A	−0.063591	0.431316	0.380040	0.055*
H11B	−0.081781	0.547982	0.364737	0.055*
C12	0.0210 (5)	0.5034 (3)	0.5713 (5)	0.0489 (11)
H12A	0.083970	0.563906	0.604762	0.059*
H12B	−0.085902	0.507265	0.597604	0.059*
C13	0.5963 (5)	0.6093 (3)	0.5403 (4)	0.0446 (10)
H13	0.537060	0.605495	0.442222	0.054*
C14	0.7680 (4)	0.6524 (2)	0.7252 (4)	0.0299 (8)
C15	0.6690 (4)	0.5809 (2)	0.7595 (4)	0.0284 (7)
K1	0.38167 (8)	0.36761 (6)	0.53694 (7)	0.02689 (17)
N1	0.6682 (4)	0.5387 (2)	0.8965 (3)	0.0381 (7)
N2	0.9024 (4)	0.7023 (2)	0.8204 (4)	0.0407 (8)
N3	0.7200 (4)	0.6712 (2)	0.5832 (3)	0.0418 (8)
N4	0.5568 (4)	0.5513 (2)	0.6414 (3)	0.0377 (7)
O1	0.1115 (3)	0.42033 (19)	0.6383 (3)	0.0365 (6)
O2	0.1457 (3)	0.49940 (19)	0.3741 (3)	0.0341 (6)
O3	0.3785 (3)	0.40584 (17)	0.2498 (3)	0.0322 (6)
O4	0.6667 (3)	0.32630 (18)	0.4436 (3)	0.0362 (6)
O5	0.6403 (3)	0.24766 (19)	0.7098 (3)	0.0372 (6)
O6	0.3960 (3)	0.33260 (18)	0.8272 (3)	0.0377 (6)
O7	0.5439 (4)	0.5506 (2)	0.9400 (3)	0.0579 (8)
O8	0.7888 (4)	0.4916 (3)	0.9599 (3)	0.0608 (9)
O9	0.9219 (3)	0.6900 (2)	0.9496 (3)	0.0542 (8)
O10	0.9909 (4)	0.7545 (3)	0.7669 (4)	0.0716 (10)

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Imidazole compounds and their energetic salts are research hotspots in the field of energetic materials in various countries.[5–8] 4,5-dinitroimidazole is an important imidazole compound that has been widely and deeply studied.[9,10] The title compound was obtained during the process of preparing potassium salt of 4,5-dinitroimidazole from 4,5-dinitroimidazole, KOH and 18-crown-6 ether. The title compound is an organic intermediate with high research value in the preparation of imidazole energetic salts.

The asymmetric unit of the title compound is a potassium salt of 4,5-dinitroimidazolate anion and the 18-crown-6 ether molecule. The bond lengths and angles are in the expected ranges. The two N atoms and three C atoms of the imidazole ring are in a plane. But two obvious angles are formed between the two nitro and imidazole rings. The dihedral angles of the plane formed by nitro group and the plane formed by the imidazole ring are 63.029° and 9°. Compared to 4,5-dinitroimidazole, the degrees of the above two dihedral angles are changed.[9,10] Due to the influence of steric hindrance, where the rotation angle of C–N bond of one nitro group is greater than C–N bond of another nitro group. So the degrees of the two dihedral angles are quite different.

The title compound is coordination compound of potassium salt of 4,5-dinitroimidazole and 18-crown-6 ether. In previous literature, 18-crown-6 ethers have a recognition effect on potassium ions.[11,12] In the crystal structure of the title compound, one potassium atom connects eight atoms. The eight atoms are six O atoms of a 18-crown-6 ether molecule and two N atoms of two 4,5-dinitroimidazole molecules. The title compounds form long chains according to the above arrangement law.

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