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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.040
 wR factor = 0.107
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.(18-Crown-6- $\kappa^6\text{O}$)potassium 2,4-dinitrophenolate 2,4-dinitrophenol

In the title compound, $[\text{K}(\text{C}_{12}\text{H}_{24}\text{O}_6)](\text{C}_6\text{H}_3\text{N}_2\text{O}_5)\cdot\text{C}_6\text{H}_4\text{N}_2\text{O}_5$, the 18-crown-6-potassium ion is centrosymmetric, with the O atoms from *o*-nitro groups of the anions above and below the plane. The phenolate O atoms of adjacent anions are connected by short symmetrical hydrogen bonds [2.453 (4) Å], forming chains in the *c* direction.

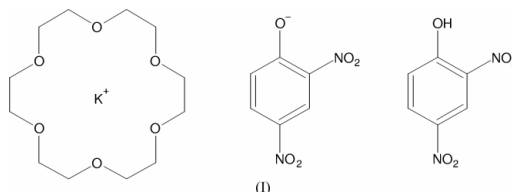
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Comment

In the title compound, (I), the asymmetric unit contains one anion with half a potassium and half a hydrogen ion, each lying on inversion centres. The potassium ion lies in the plane of the 18-crown-6 molecule, with K—O distances 2.777 (2), 2.780 (2) and 2.845 (2) Å. Eightfold coordination of the potassium is completed by O9 and O9' ($-x, 1 - y, 1 - z$) from *o*-nitro groups on either side of the crown [2.756 (2) Å] (Fig. 1). Phenolate atom O7 is 2.453 (4) Å from O7' ($-x, 1 - y, 2 - z$), with the hydrogen ion, H30, at the mid-point (0, 0.5, 1), giving a typical short symmetrical hydrogen bond. These hydrogen bonds connect the molecules into zigzag chains in the *c* direction.



The Cambridge Structural Database (Allen, 2002) lists over 300 structures containing the 18-crown-6-potassium cation. When two additional ligands are available, as in (I), the cation can be centrosymmetric (*e.g.* Bryn & Strouse, 1981). More often, the potassium ion is out of the ring plane and is coordinated by one or two ligand atoms on the side away from the crown. The potassium ion can lie as much as 1.6 Å out of the plane of the crown O atoms (Ziolo *et al.*, 1981), but is usually closer to the plane. For example, in 18-crown-6-potassium propiophenone (Veya *et al.*, 1994), the potassium ion is 0.678 Å out of the plane. In 18-crown-6 [2,6-bis(trifluoromethyl)phenylphosphanyl]potassium (Rudzevich *et al.*, 2002), the potassium ion is 0.465 Å out of the plane. In these examples, the exposed face of the potassium is coordinated by one atom from the anion. In 18-crown-6 potassium picrate (Barnes & Collard, 1988), the potassium ion is 0.678 Å out of the plane and is coordinated by the phenol O atom and an O atom from an *o*-nitro group of the same anion at distances of 2.741 (3) and 2.846 (4) Å, respectively.

Coordination of nitrophenolate anions to potassium ions by O atoms from *o*-nitro groups occurs in potassium picrate (Palenik, 1972) and in potassium 2,4-dinitrophenolate (Chal-

oner *et al.*, 1998), as well as in 18-crown-6 potassium picrate (Barnes & Collard, 1988). The phenolate O atom is usually involved as well. In (I), the phenolate O atom is not involved, because of the opportunity to form a strong hydrogen bond and restricted access to the potassium ion lying in the ring plane. The plane of the substituted phenyl ring makes an angle of 22.9 (1)° with the plane of the crown. The *p*-nitro group is twisted by 12.9 (4)° from the plane of the phenyl group compared with 30.2 (3)° for the *o*-nitro group. These values are typical of *p*- and *o*-nitro groups of nitrobenzenes (Barnes & Chudek, 2003). Coordination by three O atoms from nitro groups of two bridging anions in a dimeric complex has also been observed (Barnes *et al.*, 1994).

Experimental

Aqueous solutions of potassium 2,4-dinitrophenolate (0.1 mmol) and 18-crown-6 (0.1 mmol) were mixed and the product allowed to crystallize.

Crystal data

$C_{12}H_{24}KO_6^+ \cdot C_6H_3N_2O_5^- \cdot$ $C_6H_4N_2O_5$	$Z = 1$
$M_r = 670.63$	$D_x = 1.453 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.4869 (7) \text{ \AA}$	Cell parameters from 25 reflections
$b = 10.3156 (18) \text{ \AA}$	$\theta = 10.0\text{--}12.0^\circ$
$c = 10.6773 (18) \text{ \AA}$	$\mu = 0.25 \text{ mm}^{-1}$
$\alpha = 83.6340 (14)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 69.4840 (11)^\circ$	Lath, yellow
$\gamma = 85.0830 (11)^\circ$	$0.36 \times 0.18 \times 0.09 \text{ mm}$
$V = 766.6 (2) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 25.5^\circ$
ω – 2θ scans	$h = 0 \rightarrow 9$
Absorption correction: none	$k = -12 \rightarrow 12$
3065 measured reflections	$l = -12 \rightarrow 12$
2827 independent reflections	3 standard reflections every 300 reflections
1752 reflections with $I > 2\sigma(I)$	intensity decay: none
$R_{\text{int}} = 0.023$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.0572P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} = 0.003$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
2827 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
206 parameters	
H atoms: see below	

Table 1

Selected geometric parameters (Å, °).

K1–O9 ⁱ	2.756 (2)	K1–O28	2.7801 (16)
K1–O9	2.756 (2)	K1–O22	2.8452 (17)
K1–O25 ⁱ	2.7768 (16)	K1–O22 ⁱ	2.8452 (17)
K1–O25	2.7768 (16)	C1–O7	1.297 (3)
K1–O28 ⁱ	2.7801 (16)	O7–H30	1.2266
O9–K1–O25	106.43 (6)	O9–K1–O22	92.87 (6)
O9–K1–O28	91.94 (6)	O25–K1–O22	60.12 (6)
O25–K1–O28	60.15 (5)	O28–K1–O22	118.87 (5)

Symmetry code: (i) $-x, 1-y, 1-z$.

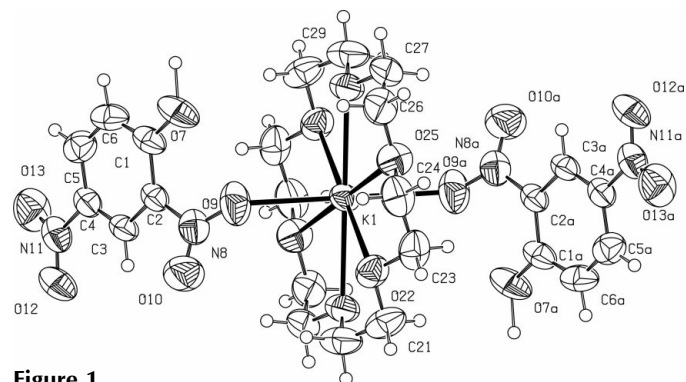


Figure 1

The structure of the centrosymmetric unit in (I), showing ellipsoids at the 50% probability level.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O7-H30 \cdots O7^{ii}$	1.226 (4)	1.226 (4)	2.453 (4)	180

Symmetry code: (ii) $-x, 1-y, 2-z$.

H atoms attached to C atoms were placed in calculated positions and allowed to ride during the refinement. Isotropic displacement parameters were constrained to be $1.3U_{\text{eq}}$ of the parent C atom. A difference synthesis showed atom H30 at the inversion centre (0, 0.5, 1). The coordinates were fixed at this position while the isotropic displacement parameter was allowed to refine.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL97*.

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