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Potassium Phthalimide

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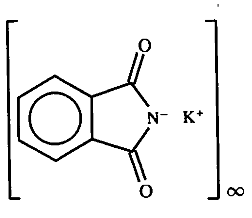
Abstract

Potassium phthalimide, $K^+.C_8H_4NO_2^-$, a widely used reagent for the preparation of anthranilic acid *via* Hofmann degradation or primary alkylamines *via* the Gabriel synthesis, crystallizes in polar layers of potassium cations coordinated by five O and three N centres alternating with apolar layers of stacked benzene subunits.

Comment

Potassium phthalimide, (I), is an industrial product prepared on a technical scale, *e.g.* as a precursor of anthranilic acid (*Ullmann's Encyclopedia of Industrial Chemistry*, 1992) or for the selective preparation of primary amines *via* the Gabriel synthesis (Gibson & Bradshaw, 1968; Gabriel, 1887). Neither its crystal structure nor crystal structures of other alkaline salts of phthalimide have been reported previously.



(I)

The potassium salt crystallizes in space group $P2_1/n$ with one molecular anion and one potassium cation in the asymmetric unit (Fig. 1). The cations are arranged in layers perpendicular to the *b* axis (Fig. 2) and within a layer are surrounded by four potassium neighbours, with K···K distances between 3.841 (1) and 4.120 (1) Å. The coordination sphere of the cation consists of five carbonyl O centres, with contact distances between 2.683 (2) and 3.086 (2) Å, and three N centres, with contact distances ranging from 2.935 (2) to 3.199 (2) Å. The molecular anions are stacked along the *a* axis almost perpendicular to their plane, with an interplanar distance of about 3.4 Å. The offset between the centroids of their benzene rings is about 1.76 Å and al-

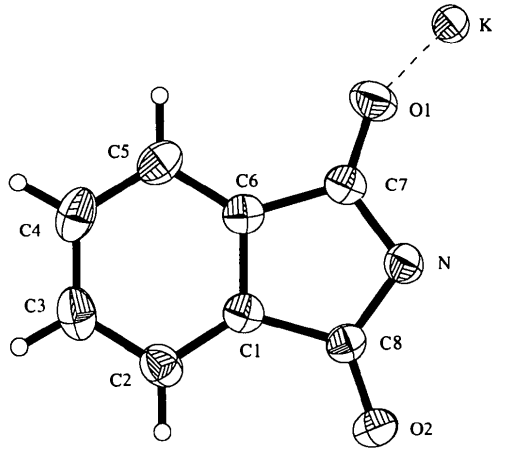


Fig. 1. View of both the anisotropic displacement ellipsoids for the non-H atoms of (I), plotted at the 50% probability level, and the numbering scheme for the asymmetric unit.

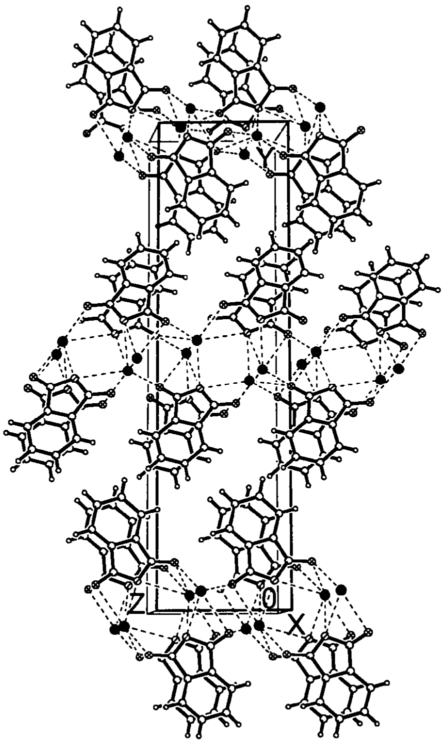


Fig. 2. The crystal packing viewed almost along the *a* axis.

lows favourable interactions between H(C) units and the π system (Hobza, Selzle & Schlag, 1994; Hunter, 1994; Hunter & Sanders, 1990). The imido side of the anions is oriented towards a cation layer, while the benzene fragments of the anions point towards each other and form apolar layers. This bilayer pattern is a frequently encountered packing motif of alkali metal salts with anions consisting of *ortho*-functionalized benzene units. It is found, for example, in the known crystal structures of the salts of benzene-1,2-disulfonic acid (Nagel, Eller & Bock, 1996), as well as in the crystal structure of sodium thiosaccharinate monohydrate (Penavić, Jovanovski & Grupče, 1990).

The packing and cell dimensions of the thiosaccharinate structure are quite similar to those of the structure reported here, though it crystallizes in a different space group ($P2_12_12_1$). The molecular anion is almost planar, with torsion angles less than 3° out of planarity. Only minor structural changes are observed compared with the protonated neutral molecule (Ng, 1992; Matzat, 1972*a*,*b*); the C—N—C bond angle is reduced from 112.8 (2) to 107.1 (2)° and the C=O and C—N bond lengths are increased by about 0.02 Å in the anion.

Experimental

Potassium phthalimide is commercially available. Crystals were obtained by slow cooling of a saturated solution in DMF.

Crystal data

$K^+.C_8H_4NO_2^-$
$M_r = 185.22$
Monoclinic
$P2_1/n$
$a = 3.8410(10)$ Å
$b = 27.234(3)$ Å
$c = 7.3616(8)$ Å
$\beta = 102.090(14)°$
$V = 753.0(2)$ Å^3
$Z = 4$
$D_x = 1.634$ Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 25–50°$
$\mu = 5.791$ mm^{-1}
$T = 293(2)$ K
Thin needle
0.85 × 0.05 × 0.05 mm
Pale pink

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.70$, $T_{max} = 0.75$
3308 measured reflections
1426 independent reflections
1237 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.018$
$\theta_{max} = 69.83°$
$h = 0 \rightarrow 4$
$k = -32 \rightarrow 32$
$l = -8 \rightarrow 8$
3 standard reflections frequency: 90 min intensity decay: 10%

Refinement

Refinement on F^2
$R(F) = 0.0323$
$wR(F^2) = 0.1007$
$S = 1.092$
1424 reflections
110 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.0481P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.332$ e Å^{-3}
$\Delta\rho_{min} = -0.351$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.002 (1)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
K	0.50705 (11)	0.46998 (2)	0.25759 (5)	0.0363 (2)
O1	0.0764 (4)	0.41077 (6)	0.4152 (2)	0.0431 (4)
O2	−0.1877 (4)	0.45104 (5)	0.9691 (2)	0.0394 (4)
N	−0.0849 (4)	0.44494 (6)	0.6722 (2)	0.0327 (4)
C1	0.0729 (5)	0.37709 (7)	0.8715 (2)	0.0296 (4)
C2	0.1350 (5)	0.34549 (8)	1.0199 (3)	0.0373 (5)
C3	0.2702 (6)	0.29941 (8)	0.9905 (3)	0.0435 (5)
C4	0.3404 (6)	0.28653 (8)	0.8207 (3)	0.0442 (5)
C5	0.2803 (5)	0.31898 (7)	0.6718 (3)	0.0376 (5)
C6	0.1455 (4)	0.36453 (7)	0.7015 (2)	0.0293 (4)
C7	0.0472 (5)	0.40839 (7)	0.5788 (3)	0.0310 (4)
C8	−0.0791 (5)	0.42793 (7)	0.8475 (3)	0.0294 (4)

Table 2. *Selected geometric parameters (Å, °)*

O1—C7	1.235 (2)	K···O1	2.736 (2)
O2—C8	1.236 (2)	K···O1^{ii}	2.769 (2)
N—C8	1.367 (2)	K···O2^{iii}	2.839 (2)
N—C7	1.367 (3)	K···N^{iii}	2.935 (2)
C1—C8	1.499 (3)	K···O2^{iv}	3.086 (2)
C6—C7	1.497 (3)	K···N^{v}	3.175 (2)
K···O2^{i}	2.683 (2)	K···N^{ii}	3.199 (2)
C8—N—C7	107.1 (2)	O2—C8—N	124.7 (2)
O1—C7—N	124.9 (2)	O2—C8—C1	124.6 (2)
O1—C7—C6	124.6 (2)	N—C8—C1	110.7 (2)
N—C7—C6	110.5 (2)		

Symmetry codes: (i) $1+x, y, z-1$; (ii) $1+x, y, z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y, z-1$; (v) $1-x, 1-y, 1-z$.

The title crystal was twinned systematically by reflection in the (001) plane. Only reflections belonging to one twin fraction were measured, which corresponded to 50% of the crystal volume. The $0kl$ reflections, for which both twin reflections coincided, were rescaled. Due to the very short a axis, no other reflections were affected by overlap of twin reflections. Equivalent reflections were averaged [$R(F)_{int} = 0.018$]. H centres were refined using a riding model, with isotropic displacement parameters of $U_{iso}(H) = 1.2U_{eq}(C)$ and C—H distances of 0.93 Å.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-centre coordinates and complete geometry have been deposited with the IUCr (Reference: CF1066). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(μ_3-2-methyl-5-thienyl)tetrakis(diethyl ether-*O*)-*tetrahedro*-tetralithium

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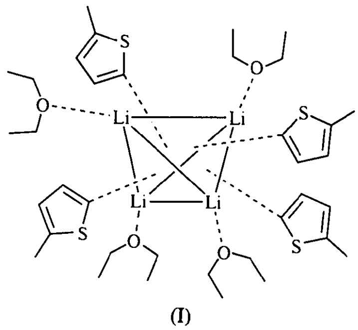
Abstract

The lithium 2-methylthiophene complex, $[Li(C_5H_5S)(C_4H_{10}O)]_4$, crystallizes as a tetramer on a $\bar{4}$ symmetry site. The four Li atoms form a slightly distorted tetrahedron. The thiophene carbanions are each coordinated to a face of the lithium tetrahedron. One diethyl ether is also coordinated to each Li atom.

Comment

The crystal structure of the diethyl ether adduct of tetrameric lithium and 5-methylthiophene, the title compound (I), was determined as part of a study of the reaction mechanism of the lithium–iodine exchange reaction (Whipple, 1991; Reich, Green & Phillips, 1989, 1991; Bailey & Patricia, 1988).



(I)

The lithium reagent was prepared by metallation of 2-methylthiophene with *n*-butyllithium in ether at 273 K, and was recrystallized twice from ether at 253 K. The title compound, (I), crystallizes as discrete tetrameric units with $\bar{4}$ site symmetry. The four Li atoms form a slightly distorted tetrahedron. The twofold and non-twofold related Li—Li distances are 2.69 (2) and 2.75 (2) Å, respectively. Although these Li—Li distances are longer than usually seen, they are comparable with those found in the phenyllithium tetramer (Hope & Power, 1983), the (2,6-dimethoxyphenyl)-lithium tetramer (Harder *et al.*, 1988) and the *tert*-butylethynyllithium–thf tetramer (Geissler *et al.*, 1987). The thiophene carbanion C5 bonds to a face of the lithium tetrahedron, as is common in structures of this type.

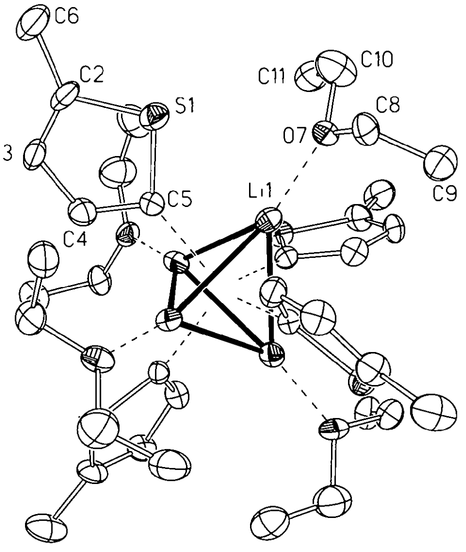


Fig. 1. Tetramer unit drawn with 30% probability displacement ellipsoids. H atoms have been removed for clarity.