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Structure of Potassium Amidoselenate $K^+.SeO_3NH_2^-$

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(*Dedicated to Professor K. Dostál on the occasion of his 70th birthday*)

Abstract

Potassium amidoselenate is composed of K^+ cations and $SeO_3NH_2^-$ anions. The anions are joined by N—H···O—Se hydrogen bonds. The average bond lengths and angles are Se—O 1.627 (6), Se—N 1.793 (3) Å, O—Se—O 111 (2) and O—Se—N 107 (4)°.

Comment

Although no X-ray structure determinations of simple amidoselenates are known, the structures of $K_3(NSeO_3)_3$ (Kocman & Rucklidge, 1974), Ag_3SeO_3N (Kocman & Szymanski, 1990) and $Ag_3SeO_3N.3NH_3.2H_2O$ (Žák, Růžička & Glowiak, 1991) have been reported. The chemistry of Se^{VI} compounds has been reviewed by Dostál (1977).

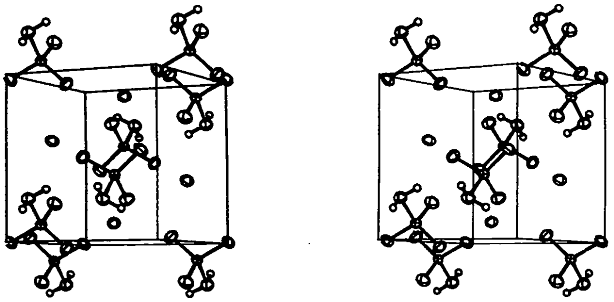

Fig. 1. Stereoview of the title compound.

Experimental

Crystal data

$K^+.SeO_3NH_2^-$
M_r = 182.1
Monoclinic
$P2_1/c$
a = 8.075 (2) Å
b = 7.306 (1) Å
c = 7.533 (2) Å
β = 96.65 (2)°
V = 441.4 (1) Å^3
Z = 4
D_x = 2.740 Mg m^{-3}
D_m = 2.706 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8.25–14.6°
μ = 9.23 mm^{-1}
T = 298 K
Regular prisms
0.22 mm (radius)
Colourless

Data collection

Kuma KM-4 diffractometer
θ-2θ scans
Absorption correction: spherical
$T_{\min}$ = 0.071, $T_{\max}$ = 0.117
3042 measured reflections
2719 independent reflections
1960 observed reflections [$|F_o|>3.92\sigma(F_o)$]
$R_{\rm int}$ = 0.025
$\theta_{\max}$ = 40°
h = −14 → 14
k = 0 → 13
l = 0 → 13
2 standard reflections monitored every 50 reflections intensity variations: 5, 3%

Refinement

Refinement on F
Final R = 0.065 (all), 0.035 (observed)
wR = 0.064 (all), 0.059 (observed)
S = 1.30
1960 reflections
55 parameters
H-atom parameters not refined
$w = [\sigma^2 + (0.04|F_o|)^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.02
$\Delta\rho_{\max}$ = 2.1 e Å^{-3}
$\Delta\rho_{\min}$ = −0.8 e Å^{-3}
Atomic scattering factors from *XTL* (Syntex, 1971)

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

$$B_{\rm eq} = \frac{8\pi^2}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
K	0.3530 (1)	−0.3751 (1)	0.1145 (1)	2.30 (2)
Se	0.25081 (3)	0.10687 (3)	0.09533 (3)	1.50 (1)
O(1)	0.2736 (4)	−0.0396 (3)	0.2593 (3)	2.80 (8)
O(2)	0.4106 (3)	0.2415 (3)	0.0967 (3)	2.50 (7)
O(3)	0.2050 (3)	−0.0020 (3)	−0.0935 (3)	2.30 (7)
N	0.0762 (3)	0.2566 (3)	0.1060 (4)	2.30 (8)
H(1)	−0.0203	0.1887	0.1251	3.5
H(2)	0.0938	0.3125	0.2188	3.5

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Se—O(1)	1.628 (2)	Se—N	1.793 (3)
Se—O(2)	1.620 (2)	H(1)···O(3$^{\rm i}$)	2.014 (2)
Se—O(3)	1.634 (2)	H(2)···O(3$^{\rm ii}$)	2.105 (2)
O(1)—Se—O(2)	112.0 (1)	O(2)—Se—O(3)	113.6 (1)
O)1)—Se—O(3)	109.6 (1)	O(2)—Se—N	105.0 (1)
O(1)—Se—N	112.8 (1)	O(3)—Se—N	103.7 (1)

Symmetry codes: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

The compound was prepared following the method of Dostál & Zbořilová (1962) and crystals suitable for X-ray work were obtained by recrystallization from water. The crystal chosen for the data collection was shaped into a sphere in order to reduce absorption effects. The density of the crystals was determined by pycnometry in decalin. During the data collection, the reference

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reflections ($\bar{2}20$ and $\bar{2}02$) showed decreases in intensity of 5 and 3%, respectively, but no corrections were made for this effect. Intensities were corrected for Lp and absorption and the structure was solved by direct methods. The H atoms were placed 1.0 Å from the N atoms and were not refined. The K, Se and O atoms were refined by anisotropic block-diagonal least squares on F_o, $w = 1/[\sigma^2(F_o) + (0.04\,F_o)^2]$; anomalous dispersion was taken into account for the K and Se atoms.

*SHELXS*86 (Sheldrick, 1986) was used for the structure solution and a PC version (Kowalski, 1988) of the *XTL* program system was used for the refinement (including atomic scattering factors for neutral atoms) and the molecular geometry calculations. The figures were drawn using *ORTEP* (Johnson, 1965). The calculations were performed on an Acer 1100/25 computer.

Fig. 1 shows a unit cell. The maximum residual electron density of 2.1 e Å^{-3} is associated with the Se atom and can be explained by the use of a neutral-atom scattering factor for Se.

Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55360 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1009]

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Structure of 4,4′-[2,6-Naphthylenebis(oxycarbonyl)]dibutyric Acid

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Abstract

The molecules of the title compound are bonded along extended rows through hydrogen bonds between carboxylic groups. The rows are parallel to the (3**a** + **c**) direction and belong to the t_i line repetition group (*i.e.* molecules along the rows are repeated by inversion centers). The packing of the rows shows a lateral matching of aromatic with aliphatic molecular sections.

Comment

The present study was undertaken as part of research into the synthesis and crystal structure analysis of α,ω-dicarboxylic acids containing a mesogenic core. These compounds may be considered as models for the crystal packing of semi-flexible mesogenic polymers (Centore, Roviello & Sirigu, 1989; Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1990; Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1992). The title compound is a model for semi-flexible polyesters based on 2,6-dihydroxynaphthalene and aliphatic dicarboxylic acids containing an even number of C atoms. The synthesis and phase characterization of these polymers has been reported by Watanabe, Ikeda & Krigbaum (1987). Unlike the analogous polyesters based on 4,4′-dihydroxybiphenyl which are smectogenic (Krigbaum, Watanabe & Ishikawa, 1983), the compounds containing a 2,6-naphthylenedioxy group are not mesogenic; only a monotropic liquid-crystal phase has been reported for one term of the series. On the other hand, random copolyesters in which only a small proportion of the 2,6-naphthylenedioxy groups have been replaced by 4,4′-biphenylenedioxy groups are mesogenic (Watanabe, Ikeda & Krigbaum, 1987). The crystal structure of a dicarboxylic acid (which is the model for semi-flexible polyesters based on 4,4′-dihydroxybiphenyl) has previously been reported (Centore, Ciajolo, Roviello, Sirigu & Tuzi, 1992). A complete structural analysis of the title compound was therefore undertaken in order to detect any significant differences in the crystal packing, in particular the lateral packing of aromatic moieties. The title compound was prepared by esterification of 2,6-dihydroxynaphthalene with glutaric anhydride. 4.000 g of 2,6-dihydroxynaphthalene, 11.410 g of glutaric anhydride and 27.600 g of potassium carbonate were refluxed in 200 ml of dioxane for 2 h. The reaction mixture was then poured into a large excess of water which was acidified with acetic acid. The solid residue was filtered, washed with water and crystallized once from ethanol/water and twice from ethanol giving pure crystals of the title compound (m.p. 465 K, yield 48%).

The conformation of the aliphatic chain is substantially *trans* planar with the largest deviation from planarity being observed for the torsion angle around the C4—C5 bond [C3—C4—C5—O3 = 21.5 (4)°]. The value of the torsion angle around the