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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{e}^- \text{O}) = 0.002 \text{ \AA}$
 R factor = 0.024
 wR factor = 0.058
Data-to-parameter ratio = 17.2

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Ammonium hydrogen selenate(IV)

The title compound, $\text{NH}_4(\text{HSeO}_3)$, is isostructural with KHSeO_3 and contains tetrahedral NH_4^+ cations and trigonal–pyramidal HSeO_3^- anions, the latter containing two short Se–O bonds and one long Se–OH bond. The crystal packing is consolidated by a network of weak hydrogen bonds, with donor–acceptor distances ($\text{N} \cdots \text{O}$ and $\text{O} \cdots \text{O}$) lying between 2.80 and 3.07 Å.

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Comment

The formation conditions, thermal behaviour and vibrational spectra of several ammonium selenate(IV) compounds have been reported, including $(\text{NH}_4)_2\text{SeO}_3$ and its monohydrate $(\text{NH}_4)_2\text{SeO}_3 \cdot \text{H}_2\text{O}$, $\text{NH}_4\text{H}_3(\text{SeO}_3)_2$, $(\text{NH}_4)_2\text{Se}_2\text{O}_5$, as well as $\text{NH}_4(\text{HSeO}_3)$ (Micka *et al.*, 1985). From these reported compounds, the crystal structures of only $\text{NH}_4\text{H}_3(\text{SeO}_3)_2$ (Tellgren & Liminga, 1974; Gorbatyii *et al.*, 1976) and $(\text{NH}_4)_2\text{Se}_2\text{O}_5$ (Chomnilpan, 1980; Makarova *et al.*, 1990) have been determined and refined from single-crystal data. The structure of the title compound, (I), is reported here.

Compound (I) contains tetrahedral NH_4^+ cations and trigonal–pyramidal HSeO_3^- anions. It is isostructural with KHSeO_3 (Sarin *et al.*, 1984) although a different cell setting with all angles $> 90^\circ$ was used for the potassium compound ($a = 5.003 \text{ \AA}$, $b = 5.726 \text{ \AA}$, $c = 6.729 \text{ \AA}$; $\alpha = 108.95^\circ$, $\beta = 107.31^\circ$, $\gamma = 91.27^\circ$). The anions in (I) are associated as inversion-symmetry generated dimers *via* weak hydrogen bonding between the hydroxy group and O3 (Fig. 1 and Table 1). The structure of (I) is held together by an intricate network of weak hydrogen

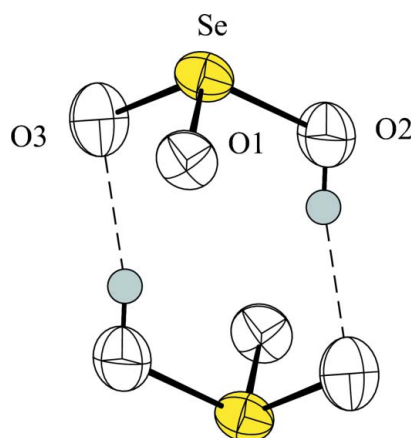
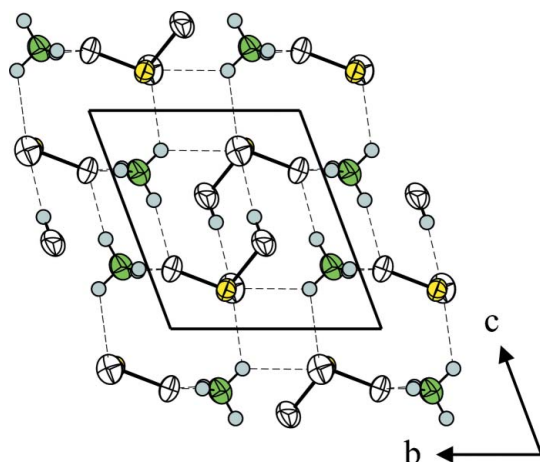


Figure 1
Detail of (I), showing the hydrogen-bonded (HSeO_3^-)₂ dimer with displacement parameters drawn at the 64% probability level for non-H atoms. The unlabelled atoms are generated by the symmetry operation $(-x, 1 - y, 1 - z)$.

**Figure 2**

Projection of the structure of (I) along [100]. Displacement parameters are drawn at the 64% probability level (arbitrary spheres for the H atoms). Colour key: Se yellow, N green, O white, H grey. Hydrogen bonds are indicated by dashed lines.

bonds between the NH_4^+ cations and the $(\text{HSeO}_3^-)_2$ dimers, with $\text{N}\cdots\text{O}$ donor–acceptor distances ranging between 2.888 (9) and 3.065 (3) Å (Fig. 2 and Table 1). A similar bond-length distribution is observed for the eight-coordinate K atom in the isostructural potassium compound. The K–O distances range between 2.78 and 3.10 Å, with an average of 2.91 Å, if a cut-off limit for the K–O distances of 3.15 Å is chosen.

The HSeO_3 group in (I) exhibits its characteristic bond-length distribution, with two short Se–O bonds, with an average length of 1.671 Å, and one long Se–OH bond of 1.761 (2) Å. This behaviour has also been observed for other hydrogenselenate(IV) anions MHSeO_3 (M = alkali metal), where M = Li (1.660 versus 1.782 Å; Eichhorn & Kek, 1997), Na (1.678 and 1.681 Å versus 1.764 and 1.719 Å; Chomnilpan *et al.*, 1981), K (1.662 versus 1.784 Å; Sarin *et al.*, 1984), Rb (1.670 versus 1.781 Å; Bannova *et al.*, 1987) and Cs (1.653 versus 1.804; Micka *et al.*, 1988).

Experimental

SeO_2 (Merck, p.A.) was dissolved in an excess of 25 wt% ammonia solution (Merck, p.A.). The hot solution was allowed to cool to room temperature and was left for several days. The resulting crystalline product was then recrystallized from a dilute ammonia solution, from which, after evaporation, colourless single crystals of (I) were isolated. X-ray powder diffraction of the bulk material revealed (I) as a single phase.

Crystal data

$\text{NH}_4(\text{HSeO}_3)$
 $M_r = 146.01$
 Triclinic, $P\bar{1}$
 $a = 5.0660$ (11) Å
 $b = 5.9587$ (12) Å
 $c = 6.9337$ (14) Å
 $\alpha = 70.473$ (4)°
 $\beta = 72.124$ (4)°
 $\gamma = 89.791$ (4)°
 $V = 186.60$ (7) Å³

$Z = 2$
 $D_x = 2.599$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1344 reflections
 $\theta = 3.3$ – 30.0 °
 $\mu = 9.89$ mm⁻¹
 $T = 293$ (2) K
 Lath, colourless
 $0.08 \times 0.05 \times 0.02$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2002)
 $T_{\min} = 0.490$, $T_{\max} = 0.827$
 2107 measured reflections

1066 independent reflections
 1004 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$
 $\theta_{\max} = 30.0$ °
 $h = -7 \rightarrow 6$
 $k = -8 \rightarrow 8$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.058$
 $S = 1.04$
 1066 reflections
 62 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0368P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.68$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Se–O1	1.6678 (17)	Se–O2	1.761 (2)
Se–O3	1.6747 (19)		
O1–Se–O3	103.79 (10)	O3–Se–O2	100.93 (10)
O1–Se–O2	103.97 (10)		

Table 2

Hydrogen-bond geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N–H1 ⁱ ⋯O1 ⁱ	0.895 (19)	2.05 (2)	2.934 (3)	170 (5)
N–H2 ⁱⁱ ⋯O3 ⁱⁱ	0.896 (19)	2.26 (4)	2.963 (3)	135 (4)
N–H2 ⁱⁱⁱ ⋯O3 ⁱⁱⁱ	0.896 (19)	2.51 (4)	3.065 (3)	121 (4)
N–H3 ^{iv} ⋯O1	0.900 (19)	2.02 (2)	2.891 (3)	164 (5)
N–H4 ^{iv} ⋯O1 ^{iv}	0.907 (19)	1.99 (2)	2.889 (3)	174 (5)
O2–H5 ^{iv} ⋯O3 ⁱⁱ	0.844 (19)	1.96 (2)	2.795 (3)	169 (5)

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

The H atoms were located in a difference Fourier analysis. Their positions were refined with distance restraints of N–H \simeq 0.90 (2) Å and O–H \simeq 0.85 (2) Å, and a common isotropic displacement parameter was used for all H atoms.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ATOMS (Dowty, 2004); software used to prepare material for publication: SHELXL97.

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