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1-Carbamoylguanidinium hydrogenselenite

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The title compound, $C_2H_7N_4O^+ \cdot HSeO_3^-$, contains a network of 1-carbamoylguanidinium cations and hydrogenselenite anions. The crystal packing is controlled by $N-H\cdots O$ [$d_{av}(H\cdots O) = 1.89$ Å, $\theta_{av}(N-H\cdots O) = 167^\circ$ and $d_{av}(N\cdots O) = 2.760$ (1) Å] and $O-H\cdots O$ [$d_{av}(H\cdots O) = 1.86$ Å, $\theta_{av}(O-H\cdots O) = 175^\circ$ and $d_{av}(O\cdots O) = 2.712$ (1) Å] hydrogen bonds, resulting in a layered structure.

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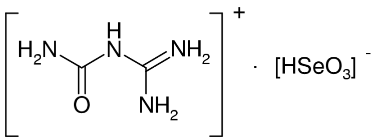
Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(Se–O) = 0.002 Å
R factor = 0.031
wR factor = 0.078
Data-to-parameter ratio = 25.9

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

Comment

The title compound, $(C_2H_7N_4O)(HSeO_3)$, (I) (Fig. 1), contains a hydrogen-bonded network of $(C_2H_7N_4O)^+$ (1-carbamoylguanidinium or guanylurea) cations and hydrogenselenite anions. It complements simple salt-like guanylurea compounds, including $(C_2H_7N_4O)(ClO_4)$ (Begley *et al.*, 1985), $(C_2H_7N_4O)(H_2PO_4)$ (Zaman & Darlow, 1986), $(C_2H_7N_4O)Cl\cdot 0.5H_2O$ (Scoponi *et al.*, 1991) and $(C_2H_7N_4O)(CH_4PO_3)\cdot H_2O$ (Brauer & Kottsieper, 2003).



(I)

In (I), the $(C_2H_7N_4O)^+$ cation has normal geometrical parameters (Begley *et al.*, 1985), with $d_{av}(N-C) = 1.343$ (3) Å, indicating significant delocalization of electrons over the non-H-atom skeleton (Scoponi *et al.*, 1991) and is almost planar (for the non-H atoms, the root-mean-square deviation from the least-squares plane = 0.031 Å). A non-linear ($\theta = 129^\circ$) intramolecular $N3-H5\cdots O4$ hydrogen bond is present, which is typical for $(C_2H_7N_4O)^+$ (Bremner & Harrison, 2002). The $(HSeO_3)^-$ hydrogenselenite group shows its standard (Verma, 1999) pyramidal geometry [$d_{av}(Se-O) = 1.699$ (2) Å and $\theta_{av}(O-Se-O) = 102.2$ (1)°], with the protonated Se—O3 vertex showing its expected lengthening relative to the other Se—O bonds.

The component species in (I) interact by means of a network of $N-H\cdots O$ and $O-H\cdots O$ hydrogen bonds (see Table 2 for symmetry codes). The guanylurea cations form hydrogen-bonded chains (*via* $N4-H7\cdots O4^{v}$ bonds) cross-linked by the hydrogenselenite groups to form hydrogen-bonded layers (Fig. 2) in the (011) plane. There are various intermolecular hydrogen-bonding motifs including $N-H\cdots O_S$, and bifurcated $N-H\cdots(O_S,O_S)$ and $N-H\cdots(O_S,O_G)$ (S = selenite, G = guanylurea) bonds. Based on the $H\cdots O$

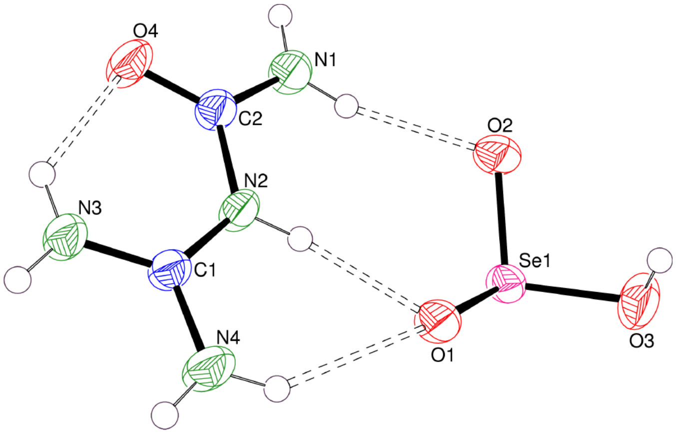


Figure 1
The asymmetric unit of (I) (50% displacement ellipsoids). H atoms are drawn as small spheres of arbitrary radius and hydrogen bonds are indicated by dashed lines.

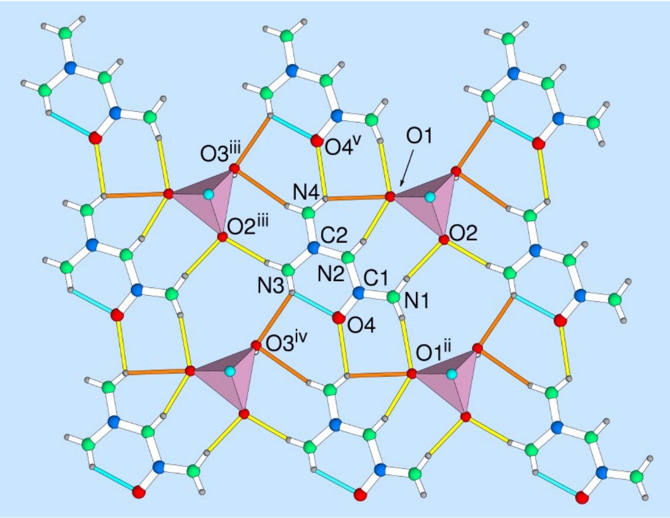


Figure 2
Detail of a (011) hydrogen-bonded sheet in (I) with the selenite groups represented by $HSeO_3E$ pseudo-tetrahedra (dummy atom E placed 1.0 Å from Se). Colour key: $[HSeO_3E]^-$ groups pink, O atoms red, C atoms blue, N atoms green, H atoms grey, E dummy atoms light blue (all radii arbitrary). The H$\cdots$O portion of the intramolecular hydrogen bond is highlighted in light blue. The H$\cdots$O portions of the N—H$\cdots$O hydrogen bonds with 1.90 Å $< d(\text{H}\cdots\text{O}) <$ 2.30 Å and 2.30 Å $< d(\text{H}\cdots\text{O}) <$ 2.55 Å are highlighted in yellow and orange, respectively. Symmetry labels as in Table 2.

separations, these N—H$\cdots$O bonds vary in strength from fairly strong (1.98 Å) to very weak (2.53 Å). Atoms O1 and O2 accept three hydrogen bonds each, and atoms O3 and O4 accept two each.

The strong, short, inter-selenite O3—H8$\cdots$O2^{i} hydrogen bonds help to fuse the layers into double sheets (Fig. 3); when considered in isolation, pairs of $(HSeO_3)^-$ units form unusual, inversion-symmetry generated, dimers by way of two such bonds (Fig. 3). Pseudo-π–π-stacking interactions between adjacent guanylurea moieties [$d(\text{N2}\cdots\text{C4}^{i})$ = 3.295 (3) Å and $d(\text{N3}\cdots\text{N1}^{i})$ = 3.437 (3) Å] may also provide some coherence between the layers. The double sheets stack normal to (011), with bonding between the double sheets controlled by van der Waals forces.

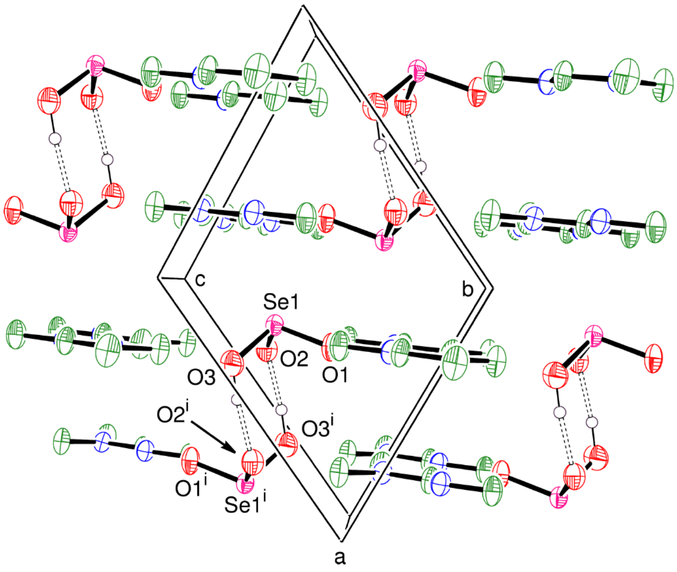


Figure 3
[010] projection of (I) showing the inter-selenite connectivity by way of hydrogen bonds. Colour key as in Fig. 1 and symmetry code as in Table 2. Guanylurea H atoms have been omitted for clarity.

Experimental

5 ml of 0.1 *M* 'H_2SeO_3' (dissolved SeO_2) and 5 ml of 0.1 *M* cyanoguanidine ($C_2H_4N_4$) were mixed, resulting in a clear solution. Rod- and block-shaped crystals of (I) grew as the water evaporated over the course of a few days. The cyanoguanidine was transformed to guanylurea by slow acid hydrolysis.

Crystal data

$C_2H_7N_4O^+\cdot HSeO_3^-$
M_r = 231.08
Triclinic, $P\overline{1}$
a = 6.7643 (4) Å
b = 7.9045 (5) Å
c = 8.2612 (5) Å
α = 63.021 (1)°
β = 81.414 (1)°
γ = 72.200 (1)°
V = 374.77 (4) Å^3
Z = 2
D_x = 2.048 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2368 reflections
θ = 2.8–32.5°
μ = 4.99 mm^{-1}
T = 293 (2) K
Block, colourless
0.45 × 0.35 × 0.30 mm

Data collection

Bruker SMART1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{\min}$ = 0.143, $T_{\max}$ = 0.224
3843 measured reflections
2591 independent reflections
2259 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
$\theta_{\max}$ = 32.5°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 7$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.078
S = 1.01
2591 reflections
100 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 1.00 e Å^{-3}
$\Delta\rho_{\min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å).

Se1—O1	1.6523 (15)	N2—C1	1.359 (3)
Se1—O2	1.6807 (16)	N2—C2	1.396 (3)
Se1—O3	1.7626 (17)	N3—C1	1.308 (3)
O4—C2	1.227 (3)	N4—C1	1.323 (3)
N1—C2	1.328 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H8···O2^{i}	0.98	1.71	2.685 (3)	173
N1—H1···O1^{ii}	0.86	2.15	2.990 (3)	167
N1—H2···O2	0.86	2.06	2.903 (3)	165
N2—H3···O1	0.86	1.99	2.824 (2)	163
N3—H4···O2^{iii}	0.86	1.98	2.826 (2)	169
N3—H5···O4	0.86	2.02	2.646 (3)	129
N3—H5···O3^{iv}	0.86	2.48	3.132 (2)	133
N4—H6···O3^{iii}	0.86	2.53	3.372 (3)	165
N4—H7···O4^{v}	0.86	2.23	2.774 (2)	122
N4—H7···O1	0.86	2.48	3.200 (3)	142

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

Atom H8 was found in a difference map and refined as riding, starting in its as-found position. H atoms bonded to nitrogen were placed in calculated positions [d(N—H) = 0.86 Å] and refined as riding. The constraint $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom) was applied in all cases.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *ATOMS* (Shape Software, 1999); software used to prepare material for publication: *SHELXL*97.

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