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Diglycine Selenate

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Abstract. $(NH_2CH_2COOH)_2.H_2SeO_4$, orthorhombic, *Pbca*, $a=18{\cdot}163$ (5), $b=11{\cdot}149$ (3), $c=10{\cdot}004$ (3) Å, $Z=8$. The crystal structure is built of layers, parallel to the *xz* plane, of glycine ions $[NH_3CH_2COOH]^+$ and selenate groups SeO_4^{2-} bound together by numerous hydrogen bonds. This accounts for the perfect (001) cleavage and the less perfect (100) cleavage.

Introduction. Crystals of diglycine selenate (DGSe) grown from aqueous solution were kindly provided by Mr J. Baran of Wrocław University. Most of the crystals were of prismatic habit and proved to be twinned and not suitable for X-ray investigation. We succeeded, however, in finding a small single crystal of DGSe which grew on the face of a larger one. A fragment in the shape of a cube of dimension 0·1 mm was cleaved and used to collect intensity data. Weissenberg photographs established the space group [Hilczer, Szczepańska, Baran, Olejnik & Łukaszewicz, 1973). A Syntex $P2_1$ diffractometer and Mo radiation with a graphite monochromator were used for lattice-parameter and intensity measurements. Intensities were measured by the 2θ–ω scan technique. The 1038 reflexions with $I>1{\cdot}92\sigma(I)$ were used in the analysis. The crystal structure was solved by Patterson and Fourier methods. The calculations were performed on a NOVA minicomputer with programs supplied by Syntex. The block-diagonal refinement brought the R index from 0·12 down to 0·052. At this stage a difference synthesis revealed the positions of the hydrogen atoms. They were included in the refinement with constant isotropic temperature factors, and a few cycles of refinement yielded a final R value 0·041 (observed reflexions only). The positional parameters of the non-hydrogen atoms with anisotropic temperature factors are presented in Table 1. The positional parameters of the hydrogen atoms are given in Table 2.*

Discussion. The arrangement of the molecules in projection on the (010) plane is shown in Fig. 1. The crystal structure consists of glycine ions and SeO_4^{2-} groups

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30948 (20 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final structure parameters of the non-hydrogen atoms*

Anisotropic thermal parameters (in $Å^2/8\pi^2$) are in the form $\exp[-\frac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)]$. Positional parameters are $\times 10^4$ for Se, $\times 10^5$ for the other atoms.

Selenate group	*x/a*	*y/b*	*z/c*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Se	18736 (3)	3067 (6)	15018 (7)	1·99 (2)	1·53 (2)	1·31 (2)	0·22 (3)	0·13 (3)	0·30 (3)
O(1)	1695 (3)	33 (4)	−74 (5)	5·0 (2)	2·4 (2)	0·9 (2)	1·2 (2)	0·1 (2)	0·4 (2)
O(2)	1143 (2)	1030 (4)	2126 (4)	2·2 (2)	2·6 (2)	1·1 (2)	0·6 (2)	0·0 (2)	0·7 (2)
O(3)	1955 (2)	−965 (3)	2312 (4)	3·2 (2)	1·5 (2)	1·4 (2)	−0·1 (2)	−0·2 (2)	0·9 (2)
O(4)	2607 (2)	1126 (4)	1698 (5)	2·2 (2)	3·2 (2)	3·5 (3)	−0·5 (2)	−0·4 (2)	1·5 (3)
Glycine I									
O(5)	576 (2)	−1753 (4)	−1938 (5)	3·4 (2)	5·7 (3)	1·6 (2)	1·6 (2)	−0·2 (2)	1·2 (2)
O(6)	−183 (2)	−1625 (4)	−199 (5)	2·5 (2)	5·3 (3)	2·2 (3)	1·2 (2)	0·2 (1)	1·2 (3)
C(1)	444 (3)	−1911 (5)	−780 (7)	2·4 (3)	1·9 (3)	1·6 (4)	−0·2 (3)	−0·6 (3)	0·1 (3)
C(2)	958 (3)	−2522 (6)	176 (6)	2·4 (3)	2·0 (2)	1·1 (3)	0·4 (3)	0·1 (3)	0·6 (3)
N(1)	1711 (3)	−2535 (5)	−394 (5)	2·5 (2)	2·9 (2)	1·1 (3)	0·7 (2)	−0·6 (2)	0·3 (3)
Glycine II									
O(7)	1721 (2)	3226 (4)	58 (5)	2·7 (2)	3·6 (2)	1·0 (2)	−1·2 (2)	−0·3 (2)	0·1 (2)
O(8)	787 (3)	4477 (4)	−426 (5)	4·6 (2)	4·2 (3)	1·1 (2)	−2·1 (2)	−0·2 (2)	−0·2 (2)
C(3)	1227 (3)	3877 (7)	388 (7)	2·7 (3)	1·8 (3)	1·4 (3)	0·8 (3)	−0·3 (3)	1·0 (3)
C(4)	1048 (3)	4151 (6)	1855 (7)	2·8 (3)	2·3 (3)	1·3 (3)	−0·1 (3)	−0·1 (3)	−0·8 (3)
N(2)	1551 (3)	3510 (5)	2709 (6)	2·7 (2)	2·7 (2)	1·0 (3)	0·2 (2)	0·7 (2)	−0·2 (2)

Table 2. *Positional parameters of the hydrogen atoms* ($B=5{\cdot}0$ Å^2)

All values are $\times 10^3$.

	x/a	*y/b*	*z/c*
Glycine I			
H(1)	72 (3)	−349 (5)	29 (7)
H(2)	94 (3)	−202 (5)	95 (6)
H(3)	39 (3)	134 (5)	64 (7)
H(4)	177 (3)	−300 (5)	−86 (7)
H(5)	188 (3)	−154 (5)	−49 (7)
H(6)	204 (3)	−266 (6)	15 (7)
Glycine II			
H(7)	55 (3)	392 (5)	211 (6)
H(8)	100 (3)	525 (5)	190 (7)
H(9)	80 (3)	446 (5)	−107 (6)
H(10)	151 (3)	347 (5)	339 (6)
H(11)	136 (3)	250 (6)	271 (7)
H(12)	199 (3)	348 (5)	243 (7)

Table 3. *Hydrogen-bond lengths* (Å) *and angles* (°)

X–H···Y	X···Y	X–H	H···Y	X–H···Y
O(6)–H(3)—O(2)	2·683	0·66	2·05	160·4
O(8)–H(9)—O(2)	2·596	0·65	1·99	157·6
N(1)–H(4)—O(3)	2·874	0·71	2·19	164·5
N(1)–H(5)—O(1)	2·881	1·15	1·83	149·0
N(1)–H(6)—O(4)	2·854	0·82	2·15	143·1
N(2)–H(10)–O(1)	2·761	0·69	2·29	126·5
N(2)–H(11)–O(2)	2·921	1·18	1·78	160·3
N(2)–H(12)–O(3)	2·804	0·85	2·02	154·0

connected by the hydrogen bonds listed in Table 3. Interatomic distances in the glycine molecules are shown in Fig. 2. In both molecules there are carboxyl groups with C–O equal to 1·20 Å and C–O(H) equal to 1·32 Å. In spite of this, one of the molecules is almost planar and in the other the nitrogen group sticks out from the best-fit plane by 0·38 Å (Table 4). This seems to confirm the opinion of Warkusz & Łukaszewicz (1973) that the planarity of glycine molecules does not depend on the ionic state of glycine (whether it is a glycine ion or zwitterion).

Table 4. *Analysis of the planarity of glycine*

Equations of the planes

I $0{\cdot}3881X+0{\cdot}8912Y-0{\cdot}23512=4{\cdot}1336$
II $0{\cdot}6350X-0{\cdot}7724Y-0{\cdot}01612=0{\cdot}3737$

Atomic deviations

O(5)	0·006 Å	O(7)	0·008 Å
O(6)	0·005	O(8)	0·002
C(1)	−0·015	C(3)	0·010
C(2)	0·004	C(4)	−0·013
N(1)	−0·380*	N(2)	−0·006

* Not included in the calculation of the plane

The geometry of the SeO_4^{2-} group is shown in Fig. 3. There is so far very little information in the literature on the crystal chemistry of selenates. In DGSe the SeO_4^{2-} group is slightly deformed with one Se–O bond

Table 5. *Bond distances* (Å) *and angles* (°) *in the* SeO_4^{2-} *group*

Se–O(1)	1·638 (5)	O(1)–Se–O(2)	106·96 (22)
Se–O(2)	1·673 (4)	O(1)–Se–O(3)	109·41 (22)
Se–O(3)	1·640 (4)	O(1)–Se–O(4)	112·52 (22)
Se–O(4)	1·628 (4)	O(2)–Se–O(3)	107·72 (20)
		O(2)–Se–O(4)	109·48 (21)
		O(3)–Se–O(4)	110·57 (21)

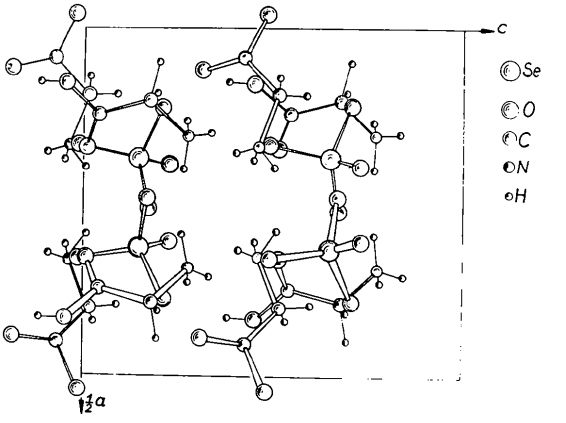

Fig. 1. The crystal structure of DGSe: projection on the (010) plane.

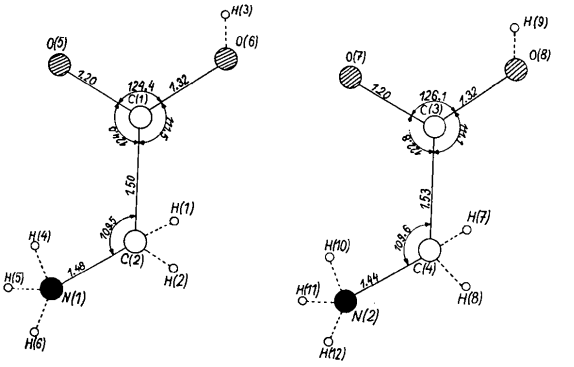

Fig. 2. Bond distances (Å) and angles (°) in the glycine ion.

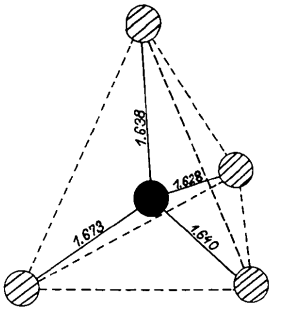

Fig. 3. Arrangement of oxygen atoms around Se. The bond lengths in Å are given.

significantly longer than the others (Table 5). The Se–O bond lengths seem to be correlated with the number of hydrogen bonds formed by the corresponding oxygen. The shortest Se–O(4) bond equal to 1·628 Å results probably from the fact that O(4) forms one hydrogen bond only. The oxygens O(1) and O(3), with intermediate bond distances (1·638 and 1·640 Å), form two hydrogen bonds each, while the Se–O(2) distance, equal to 1·673 Å, takes part in three hydrogen bonds.

Our preliminary results on the crystal structure of diglycine sulphate indicate that $(NH_3CH_2COOH)_2SeO_4$ and $(NH_3CH_2COOH)_2SO_4$ are isomorphous.

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p-Bis(trimethylsilyl)benzene

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Abstract. $C_{12}Si_2H_{22}$ (PBTMSB). M.W. 222·5, space group $P2_1/n$, monoclinic, $a=6{\cdot}608$ (16), $b=10{\cdot}683$ (8), $c=10{\cdot}520$ (8) Å, $\beta=95{\cdot}6$ (3)° (from oscillation and Weissenberg photographs), $Z=2$, $D_x=0{\cdot}9932$, $D_m=0{\cdot}97$ (2) g cm^{-3} (by flotation), $\mu_l=18{\cdot}4$ cm^{-1} for Cu $K\alpha$ radiation. The molecules are located on centres of inversion and form layers almost parallel to $(\bar{1}01)$.

Introduction. The analysis of PBTMSB was carried out because it is a monomeric model of polymers of the polysilphenylene type. The sample was prepared and recrystallized from ethanol by B. Zelei in the Laboratory of Inorganic Chemistry of the Hungarian Academy of Sciences. The compound forms needles (needle axis *a*) and volatilizes rapidly; the specimen (0·7 × 0·3 × 0·4 mm) was therefore sealed in a thin-walled glass tube for the exposures. Weissenberg photographs of the 0*kl*–4*kl* and *hk*0–*hk*4 layers were taken with unfiltered Cu radiation. 1221 independent reflexions were measured by visual estimation (339 with zero intensity). No absorption corrections were made. The structure was solved by direct methods. The program *LSAM* of Main, Woolfson & Germain (1968) produced four sets of signs for 149 reflexions with $E>1{\cdot}55$. An *E* map with the signs of the best set revealed the positions of all non-hydrogen atoms. A structure-factor calculation with an overall temperature factor $B=3{\cdot}05$ Å^2 given by the Wilson plot resulted in $R=$

Table 1. *Final atomic parameters with standard deviations in parentheses*

Fractional coordinates (× 10^4) of the non-hydrogen atoms

	x/a	*y/b*	*z/c*
Si	1154 (3)	2154 (2)	7228 (2)
C(2)	537 (11)	914 (6)	5967 (6)
C(3)	1963 (12)	432 (6)	5206 (6)
C(4)	1440 (12)	−459 (6)	4287 (6)
C(5)	3901 (16)	2417 (9)	7495 (10)
C(6)	160 (17)	1597 (11)	8750 (8)
C(7)	−165 (18)	3677 (9)	6730 (10)

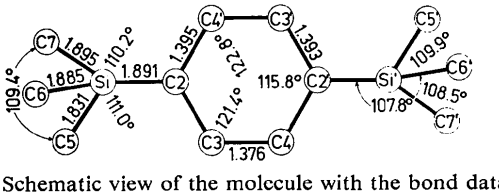

Fig. 1. Schematic view of the molecule with the bond data for the non-hydrogen atoms.

Table 1 (*cont.*)

Thermal parameters of the non-hydrogen atoms

The B_{ij} coefficients are given by $\exp[-10^{-4}(h^2B_{11}+hkB_{12}+hlB_{13}+k^2B_{22}+klB_{23}+l^2B_{33})]$

	B_{11}	B_{12}	B_{13}	B_{22}	B_{23}	B_{33}
Si	268 (6)	11 (5)	−11 (5)	73 (1)	−50 (2)	67 (1)
C(2)	264 (20)	−5 (17)	47 (16)	62 (5)	4 (8)	56 (4)
C(3)	187 (18)	−18 (18)	22 (18)	75 (5)	−42 (10)	85 (5)
C(4)	204 (20)	−4 (18)	82 (18)	72 (5)	−40 (10)	89 (6)
C(5)	331 (31)	16 (26)	30 (32)	122 (8)	−114 (15)	168 (11)
C(6)	432 (32)	−44 (36)	35 (24)	200 (12)	−82 (16)	78 (6)
C(7)	522 (39)	−100 (31)	−124 (36)	107 (8)	68 (16)	164 (11)