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Compounds of glycine with metal sulfates and thiosulfates: glycine cobalt sulfate pentahydrate, glycine sodium thiosulfate dihydrate and glycine potassium thiosulfate

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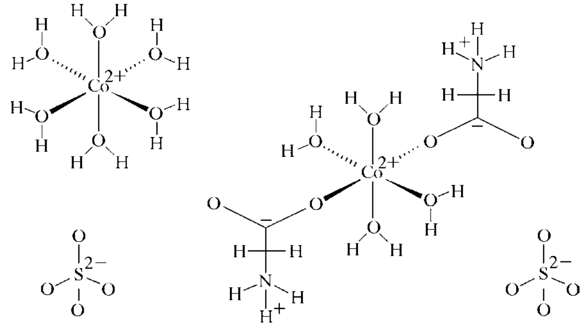
In the crystal structures of the title compounds, hexaaquacobalt(II) tetraaquadiglycinatocobalt(II) bis(sulfate), $[Co(H_2O)_6][Co(C_2H_5NO_2)_2(H_2O)_4](SO_4)_2$, (I), poly[diaqua-$\mu_3$-glycinato-di-$\mu_4$-thiosulfato-tetrasodium(I)], $[Na_4(C_2H_5NO_2)(S_2O_3)_2(H_2O)_2]_n$, (II), and poly[$\mu_2$-glycinato-$\mu_4$-thiosulfato-dipotassium(I)], $[K_2(C_2H_5NO_2)(S_2O_3)]_n$, (III), all atoms are located on general positions, except the Co atoms in (I), which are located on inversion centres. In (I), hydrogen bonds play an important role, while the alkali thiosulfate compounds are characterized by three-dimensional frameworks of polyhedra. Relations to other compounds of glycine and metal sulfates are commented on.

Comment

During our studies of new compounds of glycine with inorganic salts (Fleck & Bohatý, 2005*a*,*b*,*c*), we have examined the group of glycine metal sulfates and thiosulfates. Two such compounds we have reported previously, namely glycine lithium sulfate, $[Li_2(C_2H_5NO_2)(SO_4)]_n$, and glycine zinc sulfate trihydrate, $[Zn(C_2H_5NO_2)(SO_4)(H_2O)_3]_n$ (Fleck & Bohatý, 2004). Only two members of this group have been described in the literature previous to our studies. These are glycinium ammonium sulfate, $C_2H_6NO_2^+\cdot NH_4^+\cdot SO_4^{2-}$ (Vilminot *et al.*, 1974), and glycine nickel sulfate hexahydrate, $[Ni(C_2H_5NO_2)(H_2O)_5]SO_4\cdot H_2O$ (Peterková *et al.*, 1991). No glycine thiosulfate compounds have been reported to date. In the present paper, we present three new members of this group.

The crystal structure of glycine cobalt sulfate pentahydrate, (I) (Fig. 1), is characterized by two different isolated $[CoO_6]$ octahedra. Atoms Co1 and Co2 are both located on inversion centres; for both atoms, the coordination is more or less octahedral. While all the ligands around atom Co1 are O atoms of water molecules (2 × O*W*1, 2 × O*W*2 and 2 × O*W*3), with Co—O distances of between 2.0487 (10) and 2.1333 (11) Å, atom Co2 is surrounded by the O atoms of water molecules (2 × O*W*4 and 2 × O*W*5) as well as O atoms of the carboxylate group of the glycine molecule (atom O2), with Co—O distances of between 2.0358 (11) and 2.1404 (10) Å. Thus, two glycine molecules are attached to each Co2 octahedron, forming a $[Co(H_2O)_4(C_2H_5NO_2)_2]$ cluster. The sulfate tetrahedra are located in the interstices between these isolated groups. Consequently, the crystal structure can be considered as being composed of isolated units (Co1–water octahedra, Co2–water–glycine clusters and sulfate tetrahedra) that are connected to each other only by hydrogen bonds (Fig. 2 and Table 1).



(I)

In glycine sodium sulfate dihydrate, (II) (Fig. 3), there are four crystallographically different sodium positions. The Na atoms are coordinated by O and (in the case of atoms Na1 and Na4) S atoms; the coordination polyhedra are irregular. The Na—O distances (Table 2) range from 2.3359 (16) to 2.4765 (14) Å for Na1, from 2.3331 (12) to 2.9590 (19) Å for Na2, from 2.3526 (12) to 2.6736 (16) Å for Na3 and from 2.3883 (17) to 2.6930 (15) Å for Na4. The Na—S distances are 2.8990 (10) Å for Na1 and 3.0135 (10) Å for Na4. Bond-valence sums have been calculated and show that Na1 is slightly oversaturated, while the values for the other Na atoms are very close to the expected value of 1. The polyhedra share common corners and edges, thus forming sheets parallel to

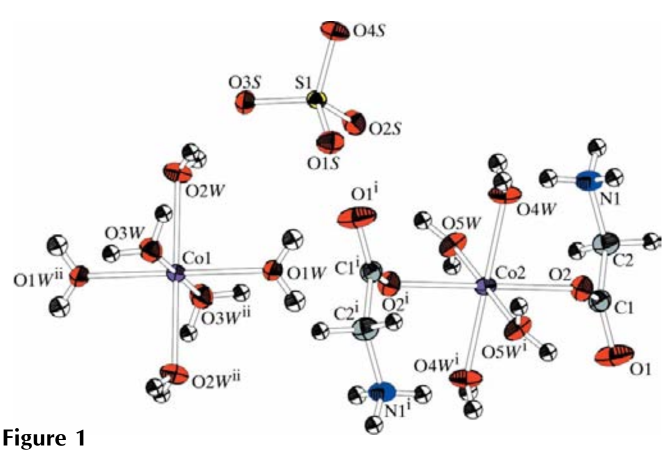
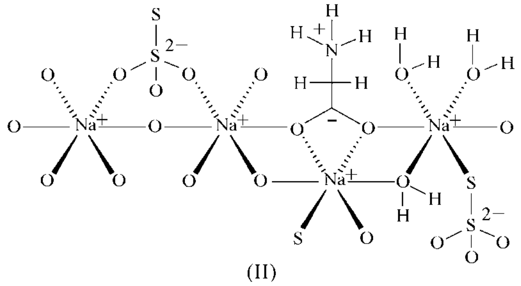


Figure 1
The connectivity in (I), shown with displacement ellipsoids at the 50% probability level for all non-H atoms and at the 20% level for all H atoms (*DIAMOND*; Bergerhoff *et al.*, 1997). [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 1, -y, -z$.]

(100). These sheets are connected by thiosulfate groups into a three-dimensional framework. It is interesting to note that this framework leaves channels parallel to [010] in which there is virtually no electron density (the negligible electron density remaining after the refinement is mostly close to the O and S atoms). The glycine molecules are connected to the Na cations *via* the carboxylate O atoms and reach into the channels but do not fill them (Fig. 4). The remaining channels are rather large (diameter approximately 3.5 Å). Details of the hydrogen bonding are given in Table 3.



(II)

The main structural elements of glycine potassium thiosulfate, (III) (Fig. 5), are the coordination polyhedra of the two crystallographically different K atoms. Atom K1 is eight-coordinate; the polyhedron resembles an irregular hexagonal bipyramid. All ligands are O atoms, with K—O distances of between 2.6225 (11) and 3.2050 (15) Å. In the case of atom K2, there is a sixfold coordination in the form of a distorted trigonal prism, the ligands being four O atoms and two S atoms, with K—O distances of between 2.6940 (12) and 2.8732 (12) Å, and K—S distances of 3.1527 (9) and 3.1887 (10) Å. The calculation of bond-valence sums yields values slightly greater than the ideal value of 1 for both K atoms. The [K1O$_8$] bipyramids share common faces and thus form a chain parallel to [001]. By sharing common corners, these chains are further connected to layers parallel to (010). These layers are again connected by the [K2O$_4$S$_2$] prisms to form a three-dimensional framework. The terminal O and S atoms of thiosulfate groups and glycine molecules act as ligands for this polyhedral framework. As in the case of (II), empty channels remain in the structure (parallel to [100]),

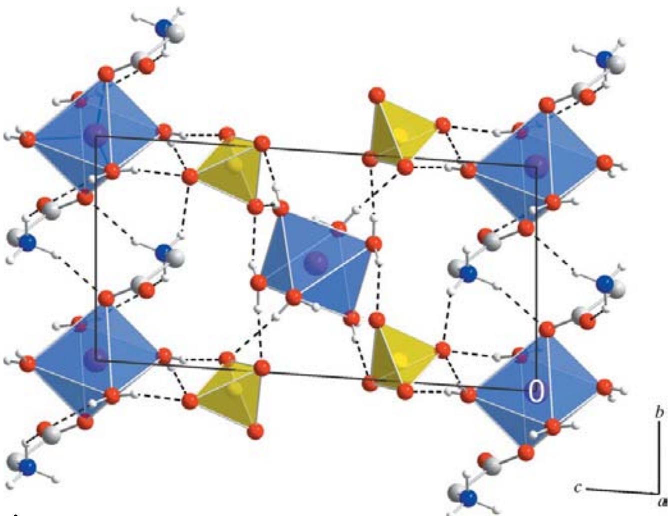


Figure 2
A packing diagram of the structure of (I), viewed along [010]. Co—O polyhedra are larger and sulfate tetrahedra are smaller; the glycine molecules are shown in a ball-and-stick representation.

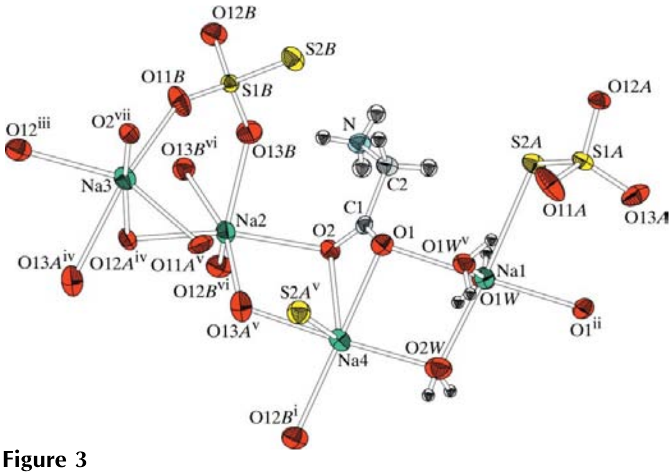


Figure 3
The connectivity in (II), shown as in Fig. 1. [Symmetry codes: (i) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x, -y, -z$; (iv) $x, -y + 1, z - \frac{1}{2}$; (v) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (vi) $-x, -y + 1, -z$; (vii) $x, -y - 1, z$.]

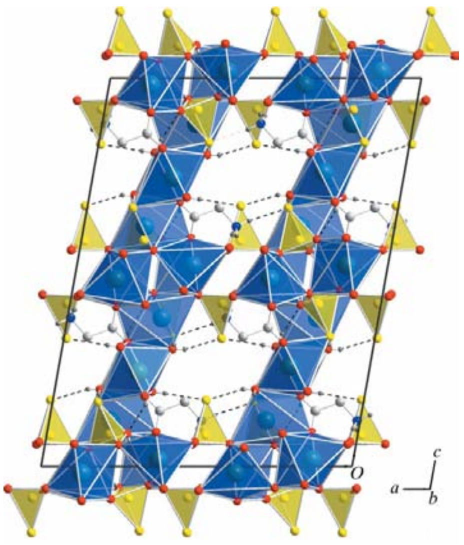


Figure 4
A packing diagram of the structure of (II), viewed along [010]; the polyhedra are as in Fig. 2. Note the channels in the viewing direction.

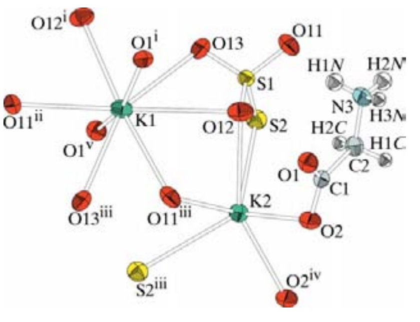
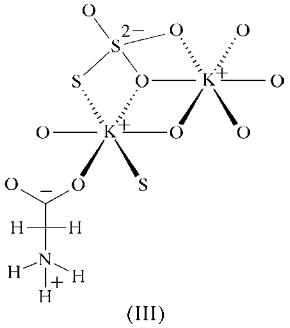


Figure 5
The connectivity in (III), shown as in Fig. 1. [Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $x + 1, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x + 1, y, z$; (iv) $-x + 1, -y + 1, -z + 1$; (v) $x, y, z - 1$.]

although they are smaller in diameter (about 2.9 Å) (Fig. 6). As in (II), there is no significant electron density remaining in the channels. Details of the hydrogen bonding are given in Table 4.



(III)

As is usually the case for compounds of glycine with inorganic salts, the glycine molecules exist in the zwitterionic form, $[H_3N^+CH_2COO^-]$, in all three compounds. However, there are examples of glycinium sulfates, for example triglycine sulfate (Matthias *et al.*, 1956), which has been studied intensively because of its interesting physical properties, such as ferroelectricity. The conformations of the glycine molecules are similar in all three title compounds. The molecules are nearly planar (as far as the non-H atoms are concerned); the O1—C1—C2—N3 torsion angles are −178.92 (3), 174.23 (2) and 175.01 (1)° in compounds (I), (II) and (III), respectively. These angles are in good agreement with the values usually found in salts of glycine and inorganic compounds; perfectly planar molecules are very rare (as in glycine magnesium dichloride tetrahydrate; Fleck & Bohatý, 2005*b*). In addition, all the distances within the carboxylate groups are within the range of the values usually found [the C—O1 and C—O2 distances for compounds (I), (II) and (III) are 1.2328 (15) and 1.2714 (14), 1.2467 (18) and 1.2574 (19), and 1.2483 (16) and 1.2506 (17) Å, respectively]. The role of the glycine molecule as a ligand is different in all three compounds. The molecule acts as a monodentate ligand in (I), as a bidentate ligand in (II) and as a bridging ligand in (III).

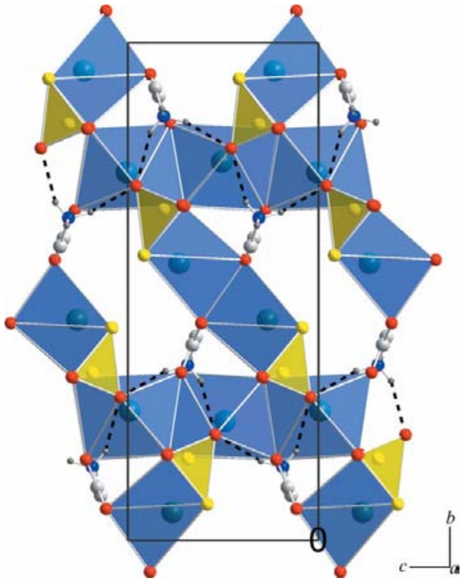


Figure 6
A packing diagram of the structure of (II), viewed along [100]; the polyhedra are as in Fig. 2. Note the channels in the viewing direction.

Remarkably, all of the reported glycine metal sulfate and thiosulfate compounds are structurally different. This can be deduced from a quick comparison of the unit-cell parameters (see Table 5). When examining the atomic arrangements of these compounds, the structural diversity becomes obvious: glycine lithium sulfate (Fleck & Bohatý, 2004) is mainly composed of tetrahedral sheets (made of SO_4 and LiO_4 tetrahedra). The structure of glycine zinc sulfate trihydrate (Fleck & Bohatý, 2004) is made up of [O3SOZnO5] clusters. These clusters are linked by glycine molecules into zigzag chains. In glycinium ammonium sulfate (Vilminot *et al.*, 1974), all building units (glycinium ions, ammonium ions and sulfate groups) are isolated, connected by ionic and hydrogen bonds. The connectivity in glycine nickel sulfate hexahydrate (Peterková *et al.*, 1991) is again different; the Ni cations are octahedrally coordinated by five O atoms from water molecules and one from a glycine molecule. Thus, isolated $[Ni(C_2H_5NO_2)(H_2O)_5]$ clusters exist, which are connected by ionic and hydrogen bonds to the sulfate tetrahedra and by hydrogen bonds to the non-coordinating water molecules.

Experimental

For the synthesis of each of the title compounds, a stoichiometric mixture of glycine and the respective metal sulfate or thiosulfate was dissolved in water. The solutions were evaporated slowly at a temperature of approximately 295 K over a period of several weeks. The syntheses yielded small crystals up to a size of several millimetres.

Compound (I)

Crystal data

$[Co(H_2O)_6][Co(C_2H_5NO_2)_2(H_2O)_4](SO_4)_2$
$M_r = 640.28$
Triclinic, $P\bar{1}$
$a = 5.970$ (1) Å
$b = 6.775$ (1) Å
$c = 13.335$ (3) Å
$\alpha = 85.23$ (3)°
$\beta = 83.31$ (3)°
$\gamma = 83.22$ (3)°
$V = 530.62$ (17) Å^3
$Z = 1$
$D_x = 2.004$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2894 reflections
$\theta = 4.9$–29.8°
$\mu = 1.87$ mm^{-1}
$T = 293$ (2) K
Fragment, red
0.40 × 0.35 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (Otwinowski & Minor, 1997)
$T_{min} = 0.479$, $T_{max} = 0.571$
5847 measured reflections
3191 independent reflections
3036 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 30.5$°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.052$
$S = 1.06$
3191 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 0.2124P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.076 (4)

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W1···O2S	0.76	2.02	2.7605 (16)	167
O1W—H2W1···O3S^{iii}	0.78	1.97	2.7329 (14)	168
O2W—H1W2···O4S^{iv}	0.79	1.96	2.7438 (15)	170
O2W—H2W2···O3S	0.84	1.97	2.7843 (15)	164
O3W—H1W3···O2S^{v}	0.84	1.90	2.7311 (13)	175
O3W—H2W3···O3S	0.82	2.04	2.8101 (15)	157
O4W—H1W4···O1^{vi}	0.81	1.85	2.6543 (16)	170
O4W—H2W4···O1S^{vii}	0.82	1.95	2.7531 (16)	170
O5W—H1W5···O4S^{iii}	0.81	1.91	2.7075 (15)	172
O5W—H2W5···O1S	0.79	1.99	2.7666 (14)	172
N1—H1N···O2^{vi}	0.92	2.03	2.9042 (16)	159
N1—H2N···O1^{vi}	0.85	2.28	2.8725 (17)	128
N1—H2N···O5W	0.85	2.38	3.0966 (15)	142
N1—H3N···O1S^{ix}	0.85	2.13	2.8887 (16)	149

Symmetry codes: (iii) $x+1, y, z$; (iv) $-x, -y+1, -z$; (v) $x, y-1, z$; (vi) $x-1, y, z$; (vii) $-x, -y+1, -z+1$; (viii) $-x+1, -y+2, -z+1$; (ix) $x, y+1, z$.

Compound (II)

Crystal data

$[Na_4(C_2H_5NO_2)(S_2O_3)_2(H_2O)_2]$
$M_r = 427.30$
Monoclinic, $C2/c$
$a = 17.775$ (4) Å
$b = 7.311$ (1) Å
$c = 22.595$ (5) Å
$\beta = 100.10$ (3)°
$V = 2890.8$ (10) Å^3
$Z = 8$

$D_x = 1.964$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2906 reflections
$\theta = 4.9$–26.8°
$\mu = 0.82$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.20 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (Otwinowski & Minor, 1997)
$T_{min} = 0.852$, $T_{max} = 0.936$
10482 measured reflections

3292 independent reflections
2966 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$; $\theta_{max} = 27.5°$
$h = -20 \rightarrow 22$
$k = -8 \rightarrow 9$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.065$
$S = 1.04$
3292 reflections
227 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 3.48P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0013 (2)

Table 2
Selected geometric parameters (Å) for (II).

Na1—O2W	2.3359 (16)	Na3—O12B^{iii}	2.5442 (15)
Na1—O1W	2.4095 (15)	Na3—O13A^{iv}	2.6736 (16)
Na1—O1^{ii}	2.4185 (13)	Na4—O2W	2.3883 (17)
Na1—O1W^{v}	2.4308 (15)	Na4—O1	2.4196 (15)
Na1—O1	2.4765 (14)	Na4—O13A^{v}	2.4281 (15)
Na1—S2A	2.8990 (10)	Na4—O12B^{i}	2.4820 (16)
Na2—O12A^{iv}	2.3331 (12)	Na4—O2	2.6930 (15)
Na2—O13B	2.3803 (15)	Na4—S2A^{v}	3.0135 (10)
Na2—O13A^{v}	2.4233 (15)	S1A—O11A	1.4656 (13)
Na2—O2	2.4689 (13)	S1A—O12A	1.4676 (12)
Na2—O12B^{vi}	2.4692 (14)	S1A—O13A	1.4721 (13)
Na2—O13B^{vi}	2.9590 (19)	S1A—S2A	2.0142 (8)
Na3—O2^{viii}	2.3526 (12)	S1B—O11B	1.4649 (12)
Na3—O11B	2.3633 (15)	S1B—O13B	1.4662 (12)
Na3—O12A^{iv}	2.4285 (13)	S1B—O12B	1.4767 (13)
Na3—O11A^{v}	2.4735 (16)	S1B—S2B	1.9945 (7)

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

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1N···O11B^{ix}	0.84 (3)	2.01 (3)	2.843 (2)	170 (2)
N3—H2N···S2A^{x}	0.82 (3)	2.41 (3)	3.2268 (17)	174 (2)
N3—H3N···O13B	0.87 (2)	1.98 (2)	2.804 (2)	159 (2)
O1W—H1W1···O11A^{ii}	0.83 (2)	1.98 (3)	2.8069 (19)	173 (2)
O1W—H2W1···S2B^{ix}	0.76 (3)	2.46 (3)	3.2191 (16)	179 (3)
O2W—H1W2···S2B^{i}	0.74 (3)	2.48 (3)	3.2007 (16)	162 (3)
O2W—H2W2···S2A^{ii}	0.86 (4)	2.34 (4)	3.1940 (16)	171 (3)

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

Compound (III)

Crystal data

$[K_2(C_2H_5NO_2)(S_2O_3)]$
$M_r = 265.39$
Monoclinic, $P2_1/c$
$a = 5.630$ (1) Å
$b = 20.244$ (4) Å
$c = 7.762$ (2) Å
$\beta = 94.33$ (3)°
$V = 882.1$ (3) Å^3
$Z = 4$

$D_x = 1.998$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 904 reflections
$\theta = 4.1$–27.5°
$\mu = 1.53$ mm^{-1}
$T = 293$ (2) K
Sphere, colourless
0.10 × 0.08 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (Otwinowski & Minor, 1997)
$T_{min} = 0.862$, $T_{max} = 0.941$
5037 measured reflections

2564 independent reflections
2251 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$; $\theta_{max} = 30.0°$
$h = -7 \rightarrow 7$
$k = -28 \rightarrow 28$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.058$
$S = 1.10$
2564 reflections
130 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2 + 0.267P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0115 (11)

Table 4
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1N···O13^{vi}	0.84 (2)	2.28 (2)	3.059 (2)	154 (2)
N3—H2N···O2^{vii}	0.89 (2)	2.26 (2)	3.0114 (16)	142 (2)
N3—H3N···O13^{viii}	0.93 (2)	2.15 (2)	3.044 (2)	161 (2)

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

Table 5
Overview of the stoichiometries, symmetries and unit-cell parameters (Å, °) of glycine metal sulfate and thiosulfate compounds.

	a	b	c	α	β	γ		Ref
Glycine $NH_4H(SO_4)$	8.26	10.07	8.63	90	92.66	90	$P2_1/c$	(*a*)
Glycine $Li_2(SO_4)$	16.42	5.00	7.65	90	90	90	$Pna2_1$	(*b*)
Glycine $Co(SO_4)\cdot 5H_2O$	5.57	6.78	13.34	85.20	83.30	83.21	$P\overline{1}$	(*c*)
Glycine $Ni(SO_4)\cdot 6H_2O$	5.73	12.30	17.01	90	97.92	90	$P2_1/c$	(*d*)
Glycine $Zn(SO_4)\cdot 3H_2O$	8.44	8.28	12.52	90	90	90	$Pca2_1$	(*b*)
Glycine $K_2(S_2O_3)$	5.63	20.24	7.76	90	94.33	90	$P2_1/c$	(*c*)
Glycine $Na_4(S_2O_3)_2\cdot 2H_2O$	17.78	7.31	22.60	90	100.10	90	$C2_1/c$	(*c*)

References: (*a*) Vilminot *et al.* (1974); (*b*) Fleck & Bohatý (2004); (*c*) this work; (*d*) Peterková *et al.* (1991).

For all three compounds, data collection: *COLLECT* (Nonius, 2003); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Version 2.1; Bergerhoff *et al.*, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG3001). Services for accessing these data are described at the back of the journal.

References

Bergerhoff, G., Berndt, M. & Brandenburg, K. (1997). *J. Res. NIST*, **101**, 221–225.

Fleck, M. & Bohatý, L. (2004). *Acta Cryst.* C**60**, m291–m295.

Fleck, M. & Bohatý, L. (2005*a*). *Acta Cryst.* C**61**, m412–m416.

Fleck, M. & Bohatý, L. (2005*b*). *Acta Cryst.* E**61**, m1887–m1889.

Fleck, M. & Bohatý, L. (2005*c*). *Acta Cryst.* E**61**, m1890–m1893.

Matthias, B. T., Miller, C. E. & Remeika, J. P. (1956). *Phys. Rev.* **104**, 849–850.

Nonius (2003). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Peterková, J., Podlahová, J., Loub, J. & Micka, Z. (1991). *Acta Cryst.* C**47**, 2664–2666.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Vilminot, S., Philippot, E. & Cot, L. (1974). *Acta Cryst.* B**30**, 2602–2606.