Diglycine–Telluric Acid Monohydrate, $Te(OH)_6.2NH_2CH_2COOH.H_2O$

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Abstract. $M_r = 397.8$, monoclinic, $P2_1/n$, $a = 7.117$ (4), $b = 12.201$ (5), $c = 7.936$ (4) Å, $\beta = 110.46$ (4)°, $Z = 2$, $V = 645.6$ (5) Å³, $D_x = 2.046$ (2) Mg m⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 2.46$ mm⁻¹, $T = 293$ K. 1816 diffractometer reflections gave a final $R = 0.029$. The structure is built up from telluric acid, glycine and water molecules connected by hydrogen bonds. The Te–O distances in the $Te(OH)_6$ group range from 1.895 (3) to 1.919 (3) Å.

Introduction. The interesting physical properties of the triglycine sulphate (TGS), *e.g.* pyroelectricity, are associated with the asymmetric structure arising when H_2SO_4 is combined with glycine. We have tried to find other compounds with corresponding properties by changing the acid.

Experimental. Crystals obtained by evaporating an aqueous solution of telluric acid and glycine are stable in air at ambient temperature, but decompose forming an anhydrous phase at 343 K; colourless prisms, $0.09 \times 0.16 \times 0.15$ mm, Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$, lattice parameters from settings of 15 reflections with $6.4 < 2\theta < 17.5°$, 2875 reflections (*hkl* and *hk̄l*: *h* 0–10, *k* 0–19, *l* 1̄2–12) with $2\theta < 70°$, 1059 unobserved, 2600 independent, of which 1693 have $I > 3\sigma(I)$, 1059 unobserved; intensities from profile analysis of $\omega/2\theta$ scans (*cf.* Lindqvist & Ljungström, 1979) according to Lehmann & Larsen (1974), 2θ scan intervals 2.0–3.0°, standard reflections 1̄41 and 1̄12 stable, systematic absences: *h0l* for *h+l* odd and 0*k*0 for *k* odd, Lp correction (XTL *Operation Manual*, 1973), absorption ignored; Patterson methods (XTL *Operation Manual*, 1973), H not located, anisotropic block-diagonal least-squares refinement (Lindgren, 1977) of positional and thermal parameters and a scale factor minimizing $\sum w(|F_o| - |F_c|)^2$, final $R = 0.029$ (0.038 after isotropic refinement), $wR =$

0.034,* weights $w = 1/\sigma^2(|F_o|)$ from counting statistics gave acceptable weight analysis, ratio of least-squares shift to error below 1%, $F(000) = 392$, scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates and equivalent isotropic temperature factors are in Table 1.

Bond distances and angles within the telluric acid and glycine molecules are listed in Table 2 and a stereoscopic view of the structure is shown in Fig. 1. The dimensions of the octahedral $Te(OH)_6$ molecule are in good agreement with those found in monoclinic $Te(OH)_6$ (Lindqvist, 1969; Lindqvist & Lehmann, 1973), in which the Te–O bond distances all are close to 1.910 Å and the O–Te–O angles deviate by 1.5–2.5° from 90°. For cubic telluric acid diverging results have been reported, *i.e.* an X-ray refinement indicating a centrosymmetric $Te(OH)_6$ octahedron with

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38175 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å²) *according to Hamilton* (1959)

W denotes the O atom of the water molecule.

	x	y	z	B_{eq}
Te	0.0	0.0	0.0	1.176 (4)
O(1)	0.2236 (4)	0.0953 (2)	0.1116 (4)	1.67 (5)
O(2)	−0.1509 (4)	0.0877 (2)	0.1068 (4)	1.70 (5)
O(3)	−0.0738 (4)	0.0978 (2)	−0.1980 (4)	1.76 (5)
W	0.7678 (6)	0.2313 (3)	0.3759 (5)	2.97 (8)
N	0.6749 (5)	0.2959 (3)	−0.0719 (4)	1.84 (6)
C(1)	0.6906 (6)	0.3969 (3)	0.0384 (5)	1.67 (7)
C(2)	0.9032 (6)	0.4250 (3)	0.1475 (5)	1.64 (7)
O(4)	0.9234 (5)	0.5055 (3)	0.2536 (4)	2.37 (6)
O(5)	1.0410 (5)	0.3722 (3)	0.1284 (4)	2.44 (6)

Table 2. *Distances* (Å) *and angles* (°)

Te–O(1)	1·919 (3)	N–C(1)	1·493 (5)
Te–O(2)	1·912 (3)	C(1)–C(2)	1·497 (6)
Te–O(3)	1·895 (3)	C(2)–O(4)	1·268 (5)
		C(2)–O(5)	1·226 (5)
O(1)–Te–O(2)	87·7 (1)	N–C(1)–C(2)	112·4 (4)
O(1)–Te–O(3)	86·9 (2)	C(1)–C(2)–O(4)	114·7 (4)
O(2)–Te–O(3)	88·8 (1)	C(1)–C(2)–O(5)	119·9 (4)
		O(4)–C(2)–O(5)	125·4 (4)
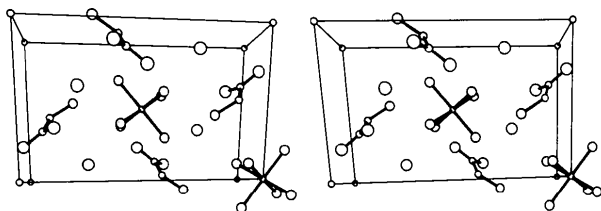
Fig. 1. Stereoscopic picture of the structure. Right-handed system with *b* on the horizontal line and *c* vertical.

Table 3. *Possible hydrogen bonds* (Å)

All distances below 3·2 Å are listed.

Donor	Acceptor		Donor	Acceptor	
O(1)	O(4^i)	2·604 (4)	W^{ii}	O(1)	2·912 (5)
O(2^{iii})	O(4^i)	2·738 (4)	W	O(2^{iii})	2·971 (5)
O(3^v)	O(5^{vi})	2·628 (5)	W^{iv}	O(3^v)	3·099 (5)
N^{ii}	O(1)	2·967 (5)			
N	O(2^{iii})	2·962 (5)			
N^{iv}	W	2·799 (5)			
N^{iv}	O(2^{iii})	3·124 (5)			

Relevant angles (°)

Te–O(1)–O(4^i)	117·4 (2)	O(1^{iv})–N^{vii}–O(2^v)	129·5 (2)
Te–O(2)–O(4^i)	124·2 (2)	O(1^{iv})–N^{vii}–W^{vi}	110·4 (1)
Te^v–O(3^v)–O(5^{vi})	117·3 (2)	O(2^v)–N^{vii}–W^{vi}	102·0 (2)
		O(2^v)–W–O(1^{iv})	169·0 (3)

Symmetry code: (i) 1·5−*x*, *y*−0·5,0·5−*z*; (ii) *x*−0·5,0·5−*y*, *z*−0·5; (iii) 1+*x*,*y*,*z*; (iv) 0·5+*x*,0·5+*y*,0·5+*z*; (v) 1+*x*,*y*,1+*z*; (vi) *x*−0·5,0·5−*y*,0·5+*z*; (vii) *x*,*y*,1+*z*.

Te–O bond distances of 1·913 (2) Å (Falck & Lindqvist, 1978) and a neutron refinement resulting in a non-centrosymmetric octahedron with three short [1·81 (1) Å] and three long [1·98 (1) Å] Te–O bond distances (Mullica, Korp, Milligan, Beall & Bernal, 1980). The conflicting results may be due to disorder in cubic telluric acid at room temperature.

The glycine molecule in Te(OH)$_6$.gly$_2$.H$_2$O shows small but significant differences from the stable modifications of crystalline glycine. Both α-glycine (Jönsson & Kvick, 1972; Power, Turner & Moore, 1976) and γ-glycine (Kvick, Canning, Koetzle & Williams, 1980) have been refined from neutron data at room temperature. The C–N distances are 1·476 (1) and 1·473 (1) Å and the C–C bonds are 1·526 (1) and 1·531 (1) Å, in the α and γ forms, respectively. Also the two C–C–O angles (*cf.* Table 2) differ from α-glycine [117·5 (1) and 117·1 (1)°] and γ-glycine [117·7 (1) and 116·5 (1)°]. These differences are probably induced by the hydrogen bonds, as is the significant difference between C(2)–O(4) and C(2)–O(5) in Te(OH)$_6$.gly$_2$.H$_2$O. O(4) is connected to two tellurate OH groups while O(5) participates only in one such hydrogen bond (*cf.* Table 3). A similar situation occurs in glycine–trimesic acid monohydrate (Herbstein, Kapon, Maor & Reisner, 1981) which has C–O bonds of 1·275 (4) and 1·232 (2) Å, the more distant oxygen being involved in a strong hydrogen bond to the trimesic acid. However, the C–N and C–C bonds in the glycine–trimesic acid structure compare well with α- and γ-glycine. In TGS (Itoh & Mitsui, 1973) the three glycine molecules are all different having C–C and C–N bond distances of 1·527 (7) and 1·478 (8) Å (glycine I), 1·544 (8) and 1·540 (7) Å (glycine II), and 1·494 (7) and 1·460 (6) Å (glycine III). The deviations of glycine (II) and (III) in TGS from α- and γ-glycine

are of the same order of magnitude as in Te(OH)$_6$.gly$_2$.H$_2$O, but owing to differences in hydrogen bonding, the deviations are not identical.

The small differences between Te–O(1) and Te–O(2) on one hand and Te–O(3) on the other are probably also an effect of hydrogen bonding, since both O(1) and O(2) are involved in interactions with the amino group of the glycine molecule, while O(3) is not.

The hydrogen bonding in Te(OH)$_6$.gly$_2$.H$_2$O could not be evaluated in detail, since it was not possible to determine the positions of the H atoms with sufficient accuracy. Probable hydrogen bonds are suggested in Table 3, but there are other possibilities, especially with regard to the water molecule. A neutron diffraction study is planned, and should resolve this question as well as the detailed influence of hydrogen bonding on the Te(OH)$_6$ symmetry.

References

FALCK, L. & LINDQVIST, O. (1978). *Acta Cryst.* B34, 3145–3146.
HAMILTON, W. C. (1959). *Acta Cryst.* 12, 609–610.
HERBSTEIN, F. H., KAPON, M., MAOR, I. & REISNER, G. M. (1981). *Acta Cryst.* B37, 136–140.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
ITOH, K. & MITSUI, T. (1973). *Ferroelectrics*, 5, 235–251.
JÖNSSON, P. G. & KVICK, Å. (1972). *Acta Cryst.* B28, 1827–1833.
KVICK, Å., CANNING, W. M., KOETZLE, T. F. & WILLIAMS, J. B. (1980). *Acta Cryst.* B36, 115–120.
LEHMANN, M..S. & LARSEN, F. K. (1974). *Acta Cryst.* A30, 580–584.
LINDGREN, O. (1977). Thesis. Univ. of Göteborg.
LINDQVIST, O. (1969). *Acta Chem. Scand.* 23, 3062–3070.
LINDQVIST, O. & LEHMANN, M. S. (1973). *Acta Chem. Scand.* 27, 85–95.
LINDQVIST, O. & LJUNGSTRÖM, E. (1979). *J. Appl. Cryst.* 12, 134.
MULLICA, D. F., KORP, J. D., MILLIGAN, W. O., BEALL, G. W. & BERNAL, I. (1980). *Acta Cryst.* B36, 2565–2570.
POWER, L. F., TURNER, K. E. & MOORE, F. H. (1976). *Acta Cryst.* B32, 11–16.
XTL *Operation Manual* (1973). Syntex Analytical Instruments, Cupertino, California 95015, USA.