

Fig. 3. Stereo packing diagram of the molecules.

expected value in related structures and C(11a)—C(17) is shorter than the expected value. The C(8)—C(16) bond [ $C(sp^2_{\text{aryl}})$ — $C(sp^3_{\text{methyl}})$ ] is unusually long. The large value may be due to the fact that this bond involves a methyl C atom which has high thermal vibration ( $B_{\text{eq}} = 7.3 \text{ \AA}^2$ ). Such lengthening is not unusual with the presence of methyl C atoms. In fact lengths of 1.586 and 1.592 Å for C( $sp^3$ )—C( $sp^3$ ) bonds have been observed (Pajunen & Pajunen, 1979) as compared with the C—C single-bond distance of 1.544 Å (Pauling, 1960).

A stereoview of the molecular packing is shown in Fig. 3. It was obtained with projection down the  $a$  axis with  $4^\circ$ ,  $20^\circ$  and  $5^\circ$  rotations around the plotter  $x$ ,  $y$  and  $z$  directions using the molecular plotting program of Radhakrishnan (1982). Packing results from normal van der Waals contacts and the crystal is thus a molecular crystal.

*Acta Cryst.* (1987). **C43**, 907–909

## Neutron Refinement of Telluric Acid–Glycine (1/2) Monohydrate

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(Received 16 January 1986; accepted 29 September 1986)

**Abstract.**  $\text{Te}(\text{OH})_6 \cdot 2\text{C}_2\text{H}_5\text{NO}_2 \cdot \text{H}_2\text{O}$ ,  $M_r = 397.8$ , monoclinic,  $P2_1/n$ ,  $a = 7.981$  (3),  $b = 12.286$  (5),  $c = 7.154$  (2) Å,  $\beta = 110.51$  (3)°,  $V = 657 \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 2.01 \text{ g cm}^{-3}$ ,  $\lambda = 1.18 \text{ \AA}$ ,  $\mu = 2.5 \text{ cm}^{-1}$ , room temperature, final  $wR = 0.05$  for 1214 independent reflections. The H-atom positions confirm the zwitterion form of the glycine species and the role of H bonds in linking the glycine molecule to the  $\text{Te}(\text{OH})_6$  group. Te—O distances and O—Te—O angles indicate substan-

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tial distortion of  $\text{Te}(\text{OH})_6$  groups from ideal octahedral coordination.

**Introduction.** The crystal structure of  $\alpha$ -glycine–telluric acid has been recently and independently determined at room temperature and at 120 K by X-ray diffraction studies (Anderson, Lindqvist & Moret, 1983; Tran Qui, Vicat & Durif, 1984). In this structure, despite the presence of the heavy Te atom, the H-atom positions in

the unit cell were successfully located from difference Fourier maps. The precision of the H-atom parameters was fairly acceptable. In order to gain more precise information about the H-bonding network in  $\alpha$ -glycine–telluric acid we decided to reinvestigate the crystal structure of this compound by neutron diffraction.

**Experimental.** Crystal obtained by slow evaporation in air of a saturated aqueous solution of glycine and telluric acid. After about three weeks single crystals had formed. A crystal with well-developed faces,  $0.8 \times 0.6 \times 1.1$  mm, was selected for intensity measurements.

Data collection carried out on an automatic four-circle diffractometer installed at SILOE Reactor, DRF, CEA.  $\omega$ -scan technique with incident beam wavelength of 1.18 Å. A total of 2300 reflections with  $h-8 \rightarrow 8$ ,  $k 0 \rightarrow 13$ ,  $l 0 \rightarrow 7$  and  $\sin\theta/\lambda \leq 0.55 \text{ \AA}^{-1}$  recorded. No significant variation in a standard reflection, 413, measured every 50 reflections. Unit-cell parameters used for data reduction and subsequent calculations taken from a previous X-ray study (Tran Qui *et al.*, 1984). Structure-factor amplitudes obtained from the reflection profiles by determining the points of division between the background and the peak so that  $\sigma(I)/I$  was minimized (Lehmann & Larsen, 1974);  $\sigma(I)$  is the standard deviation based on counting statistics of the integrated intensity.

An absorption correction was applied to  $F^2$  factors using a Gaussian integration algorithm. Finally the symmetry-related reflections based on  $P2_1/n$ , were averaged and 712 reflections having  $F^2 > 2\sigma(F^2)$  were kept for structure refinement.

Conventional full-matrix least-squares refinement using the *SHELXTL* program (Sheldrick, 1983). Initial positional parameters of non-H atoms (Te, O, N, C) were those given by Tran Qui *et al.* (1984). Neutron-scattering amplitudes (Boucherle, Ravot & Schweitzer, 1982; Lindqvist & Lehmann, 1973): Te 5.80, O 5.80, N 9.40, C 6.65, H -3.72 fm. A difference Fourier map at this stage showed ten H-atom positions. Subsequent least-squares refinement allowed the location of the remaining H atoms to be determined. Final refinement, which included a correction for secondary extinction, gave  $R = 0.05$ .\*

**Discussion.** Positional and anisotropic thermal parameters are given in Table 1. Table 2 contains the interatomic distances and angles. Fig. 1 is a view of the complex showing the H bonds.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43364 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

All distances and angles involving heavy atoms (Te, O, C, N) and in particular the H bonds listed in Table 3 are in good agreement with values determined by previous X-ray investigation; the O–H, C–H and

Table 1. Positional parameters and  $B_{eq}$  values with their *e.s.d.*'s in parentheses

	$B_{eq} = \frac{2}{3}\pi^2 \sum_i U_{ii}$			
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
Te	0	0	0	0.87 (21)
O(1)	-0.1112 (5)	0.0949 (3)	-0.2228 (7)	1.36 (19)
O(2)	0.1992 (6)	0.0981 (3)	0.0751 (9)	1.48 (20)
O(3)	-0.1066 (6)	0.0879 (3)	0.1513 (7)	1.40 (18)
O(4)	0.3720 (6)	0.1285 (4)	0.4580 (6)	2.09 (19)
O(5)	0.2445 (6)	0.9956 (3)	0.5766 (6)	1.88 (18)
O <sub>w</sub>	0.1251 (7)	0.2682 (6)	0.7325 (8)	2.6 (3)
N	0.5712 (4)	0.2035 (2)	0.8236 (5)	1.59 (15)
C(1)	0.3519 (4)	0.0746 (3)	0.5967 (5)	1.15 (14)
C(2)	0.4623 (5)	0.1042 (3)	0.8090 (5)	1.26 (16)
H(O1)	0.3457 (10)	0.4461 (6)	0.1451 (12)	2.2 (3)
H(2N)	0.1775 (15)	0.3113 (7)	0.2860 (14)	3.5 (4)
H(1N)	0.1184 (11)	0.2728 (7)	0.4713 (15)	3.1 (4)
H(O2)	0.2583 (11)	0.1043 (5)	0.2211 (16)	2.2 (4)
H(O3)	0.3469 (11)	0.4530 (6)	0.7413 (13)	2.5 (3)
H1(C2)	0.3754 (11)	0.1148 (7)	0.8940 (13)	3.2 (4)
H(O <sub>w</sub> )	0.1362 (17)	0.3323 (11)	0.8077 (19)	4.9 (6)
H1(O <sub>w</sub> )	0.0582 (16)	0.2157 (9)	0.7743 (17)	4.7 (6)
H(C2)	0.0540 (12)	0.4622 (7)	0.3731 (13)	3.4 (4)
H(N)	-0.0029 (13)	0.2338 (7)	0.2398 (14)	3.5 (4)

Table 2. Bond distances (Å) and angles (°) with their *e.s.d.*'s in parentheses

Te–O(1)	1.923 (4)	C(1)–O(4)	1.249 (6)
Te–O(2)	1.916 (4)	C(1)–O(5)	1.269 (6)
Te–O(3)	1.925 (5)	C(1)–C(2)	1.509 (4)
		C(2)–N	1.480 (5)
O(1)–Te–O(2)	87.0 (2)	C(1)–C(2)–H(C2)	108.3 (6)
O(1)–Te–O(3)	87.7 (2)	C(1)–C(2)–H1(C2)	109.4 (6)
O(2)–Te–O(3)	88.6 (2)	N–C(2)–H1(C2)	109.8 (6)
		N–C(2)–H(C2)	107.3 (6)
		H(C2)–C(2)–H1(C2)	108.9 (7)
		H(O <sub>w</sub> )–O <sub>w</sub> –H1(O <sub>w</sub> )	109.8 (1.3)
O(4)–C(1)–O(5)	125.8 (4)	C(2)–N–H(N)	111.9 (6)
O(4)–C(1)–C(2)	118.6 (4)	C(2)–N–H1(N)	106.4 (6)
O(5)–C(1)–C(2)	115.5 (3)	C(2)–N–H2(N)	111.5 (7)
C(2)–C(2)–N	113.0 (3)	H(N)–N–H1(N)	108.4 (8)
		H(N)–N–H2(N)	110.6 (8)
		H1(N)–N–H2(N)	106.8 (8)

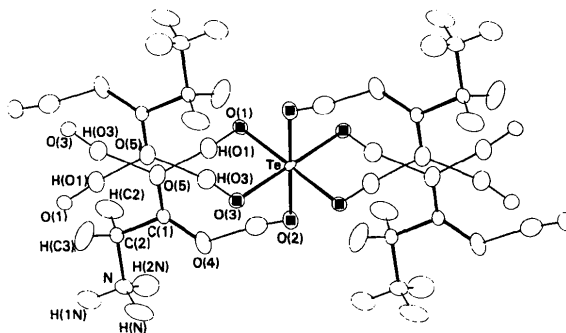


Fig. 1.  $\text{Te}(\text{OH})_6$ -glycine complex and hydrogen bonds.

N—H bond lengths observed by neutron diffraction in this work are, as expected, significantly larger than those given by X-rays. Thus the previous discussion on the role of H in the telluric acid–glycine structure is supported by our present results.

The Te—O distances O—Te—O angles indicate substantial distortion of Te(OH)<sub>6</sub> groups from ideal octahedral coordination. As shown by X-rays, O(1) and O(3) participate in two longer Te—O bonds and are acceptors and donors of strong H bonds, N—H2(N)…O(1), Ow—H1(Ow)…O(1) and N—H(N)…O(3), whereas the O(2) atoms involved in the shorter Te—O(2) distances are acceptors of relatively weak H bonds (Table 3). The six O atoms in Te(OH)<sub>6</sub>, all involved in H bonds, are both H-bond acceptors and donors. As donors they link the telluric acid group to four glycine molecules through strong bonds, O(1)—H(O1)…O(5), O(2)—H(O2)…O(4) and O(3)—H(O3)…O(5), and as H-bond acceptors they receive the electron excess from the water molecule, CH<sub>2</sub> and NH<sub>3</sub> groups.

In addition the C—H and N—H bonds observed in the glycine skeleton confirm the zwitterion form of this molecule; the bond lengths and bond angles characterizing the glycine molecule are, within e.s.d.'s, in good agreement with the values observed by neutron diffraction in the structure of α-glycine (Jönsson & Kvick, 1972).

Table 3. *Hydrogen-bond distances (Å) and angles (°) with their e.s.d.'s in parentheses*

X—H…Y	X—H	X…Y	X—H…Y
O(1)—H(O1)…O(5)	1.018 (9)	2.624 (6)	171.0 (8)
O(2)—H(O2)…O(4)	0.987 (12)	2.626 (7)	172.2 (9)
O(3)—H(O3)…O(5)	0.986 (11)	2.748 (7)	166.5 (8)
Ow—H(Ow)…O(5)	0.940 (15)	3.123 (8)	154.2 (1.2)
Ow—H1(Ow)…O(1)	0.950 (15)	2.935 (8)	163.3 (1.2)
N—H(N)…O(3)	1.027 (9)	2.979 (4)	162.8 (8)
N—H1(N)…O(w)	1.032 (10)	2.825 (7)	156.0 (8)
N—H2(N)…O(1)	0.992 (13)	2.984 (6)	153.7 (9)
C(2)—H1(C2)…O(2)	1.078 (11)	3.549 (8)	167.7 (7)
C(2)—H(C2)…O(2)	1.084 (9)	3.292 (8)	160.3 (7)

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*Acta Cryst.* (1987). **C43**, 909–912

## Structure of 13,13'-[1,4-Phenylenebis(methylene)]bis-1,4,7,10-tetraoxa-13-azacyclopentadecane Dihydrochloride, a Bicyclic Macrocycle Containing Two [15]-NO<sub>4</sub> Subunits Linked by an Aromatic Group

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(Received 8 July 1986; accepted 1 December 1986)

**Abstract.** C<sub>28</sub>H<sub>50</sub>N<sub>2</sub>O<sub>8</sub><sup>2+</sup>.2Cl<sup>-</sup>, M<sub>r</sub> = 613.6, monoclinic, P2<sub>1</sub>/c, a = 9.873 (3), b = 10.823 (3), c = 15.823 (9) Å, β = 108.26 (4)°, V = 1606 (5) Å<sup>3</sup>, Z = 2, D<sub>x</sub> = 1.269 g cm<sup>-3</sup>, λ(Mo Kα) = 0.71073 Å, μ =

2.468 cm<sup>-1</sup>, F(000) = 660, T = 295 (1) K, final R = 0.034 for 1249 observed reflections. The title compound contains two identical monoaza tetraoxa 15-membered macrocycles linked by a *p*-xylylene bridge.

0108-2701/87/050909-04\$01.50

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