Despite these near-perfect *trans*-periplanar relationships in the solid state, the desired eliminations have not been found to occur readily in these molecules. In order to obtain some information concerning the conformations of these systems in solution, the 360 MHz NMR spectra were measured. For either compound, a solution structure like that found in the crystal should ideally exhibit NMR couplings between a bridgehead hydrogen and the two neighboring α-protons of nearly identical magnitudes (Karplus, 1959, 1963). The presence of electronegative substituents modifies these expectations somewhat (Williams & Bhacca, 1964); nonetheless, the spectra of the majority of 4-pyranone derivatives are completely consistent with chair conformations in solution (Hirsch & Havinga, 1976). In contrast, in both (1) and (2) it is found that the *exo* α-protons couple strongly to the bridgehead hydrogen ($J = 4{\cdot}9$–$5{\cdot}3$ Hz) while the *endo* protons exhibit virtually no coupling at all [$J \sim 0$ Hz for (1), 0·8 Hz for (2)]. Similar spectroscopic results have also been obtained for certain other derivatives of this ring system and have been interpreted as resulting from a flattening of the pyran portion of the molecule (Cookson, Nye & Subrahmanyam, 1967; Vinter & Hoffmann, 1974). Such a conformational change in solution would have the effect of reducing the H(α-*endo*)—C—C—O dihedral angle well below the 180° value required for facile elimination, thus providing at least a partial explanation for the resistance shown by these molecules to this particular kind of reactivity. The uncertain effects of the additional functionality in (1) and (2) on their spectra cause us to question the validity of this interpretation in our systems, however. We see no obvious structural reason for either of these compounds to possess different conformations in solution *vs* the solid state. Moreover, both compounds also strongly resist normally facile elimination under acidic conditions. Thus other factors unrelated to the geometry for the simple *trans*-elimination mechanism must apparently be involved in the problem. These most likely include reversibility of the desired ring-opening process.

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Structure of Telluric Acid–Glycine (1:2) Monohydrate, $Te(OH)_6.2C_2H_5NO_2.H_2O$, at 120 K and Determination of Hydrogen Positions

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Abstract. $M_r = 406$, monoclinic, $P2_1/n$, $a = 7{\cdot}924\,(3)$, $b = 12{\cdot}154\,(5)$, $c = 7{\cdot}188\,(3)$ Å, $\beta = 110{\cdot}67\,(8)°$, $V = 648$ Å^3, $Z = 2$, D_x (at 120 K) $= 2{\cdot}08$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) $= 0{\cdot}71069$ Å, $\mu = 2{\cdot}45$ mm^{-1}, $F(000) = 392$. Final $R = 2{\cdot}0\%$ for 1942 independent observed reflections. The structure consists of slightly distorted $Te(OH)_6$ octahedra with the Te atom placed at the center of symmetry. The Te—OH distances range from 1·905 (2) to 1·929 (2) Å [average: 1·920 (2) Å]. Glycine species are found to be in the zwitterionic form. In addition, through the hydrogen bonds such as Te—OH···O—C, each glycine group is linked to either one or two corners of the $Te(OH)_6$ octahedra.

Introduction. The title compound was studied as part of a systematic investigation in our laboratory of adduct compounds of telluric acid with sulfates, phosphates, arseniates and NaF (Allmann, 1976; Allmann & Rius, 1978; Averbuch-Pouchot & Durif, 1981; Boudjada, Averbuch-Pouchot & Durif, 1981; Durif & Averbuch-Pouchot, 1981; Zilber, Durif & Averbuch-Pouchot, 1981).

The crystal structures of tellurate–phosphates and tellurate–sulphates have been characterized; structural information derived from these studies has shown that the characteristic feature of the atomic arrangements in these compounds is the coexistence of separate $Te(OH)_6$ molecules with tetrahedral PO_4 or SO_4 groups. Although in these studies the H-atom positions were not well established, there is strong evidence, based on short contacts between tellurate O atoms and phosphate O, that the hydrogen bonds such as Te—O—H···O—S are directly involved in the close packing of $Te(OH)_6$ and PO_4 or SO_4 groups. As observed in $Te(OH)_6$ crystals (Lindqvist & Lehmann, 1973), the tellurate O atoms may be both acceptors and donors of hydrogen bonds in tellurate–phosphates (or tellurate–sulphates).

This double role might confer on the $Te(OH)_6$ molecule the ability to form stable adduct compounds with a large variety of molecules. Recently an adduct of orthotelluric acid and urea was synthetized by Loub, Haase & Mergehenn (1979). The structure of this compound consists of infinite $[Te(OH)_6.2CO(NH_2)_2]$ layers parallel to the [100] direction. The positions of H atoms were not determined in this structure analysis but it was assumed that the $[Te(OH)_6.2CO(NH_2)_2]$ are linked together by hydrogen bonds (Te—O—H···O—C), and that within each layer the $Te(OH)_6$ molecules are hydrogen-bonded to neighboring $Te(OH)_6$ and urea molecules. In the present paper we describe the crystal structure of $Te(OH)_6.2NH_3CH_2CO_2.H_2O$ with special emphasis on the role of the hydrogen-bond network in the crystal cohesion. In order to locate the hydrogen positions with acceptable precision the crystal structure was determined using high-angle data at 120 K.

Experimental. Title compound prepared by crystallization from a concentrated aqueous solution of orthotelluric acid and glycine in a molar ratio 1:2. Transparent crystals obtained, in form of nearly regular prisms, some as large as 1 × 2 × 10 mm. Crystal selected for data collection shaped into sphere of diameter 0·22 mm. Automatic four-circle diffractometer equipped with monochromator and low-temperature gas-flow device; temperature of sample kept constant within ±5 K at approximately 120 K throughout experiments. Lattice parameters measured with 25 reflections collected on a computer-controlled diffractomer. About 3293 reflections in upper half of reflection sphere (h ±11, k 0–16, l ±10) measured in angular range $6 < 2\theta < 60°$ using Mo $K\bar{\alpha}$ radiation, 1942 considered observed with $I > 3\sigma(I)$. $R_{\rm int} \simeq 2\%$. ω-scan mode, scan speed $0{\cdot}03°\ s^{-1}$, scan width varied as $\Delta\omega = 1{\cdot}6° + 0{\cdot}1° \tan\theta$. Lorentz–polarization factors and a spherical absorption correction ($\mu R = 0{\cdot}27$) applied to raw data which were finally reduced to structure-factor amplitudes. Variation observed in intensities of three test reflections required correction <1%. Standard deviations associated with structure factors estimated by $\sigma^2(F^2) = \sigma^2_{\rm count} + (0{\cdot}011\ F^2)^2$ (factor 0·011 derived from observed variance of control reflections). Structure solved using three-dimensional Patterson map. Successive Fourier syntheses alternating with least-squares refinement progressively revealed all non-H-atom positions. R and R_w for all heavy atoms = 2·3 and 2·8% respectively; $w = 1/\sigma^2(F_o)$. A difference Fourier map based on non-H-atom coordinates clearly showed ten peaks ranging from 0·60 to 0·92 e Å^{-3}; these peaks correspond unambiguously to the ten H atoms in the asymmetric unit. Final least-squares refinement, with anisotropic temperature factors for heavy atoms and isotropic for H atoms, yielded final R and R_w = 2 and 2·3% respectively; S reduced from 1·2 to 1·02. In order to reduce asphericity bias of hydrogen positions, hydrogen parameters finally refined using high-order data with $\sin\theta/\lambda > 0{\cdot}50$ Å^{-1}. $(\Delta/\sigma)_{\rm max} \simeq 0{\cdot}6$. Final $\Delta\rho$ excursions $\simeq$ −0·2–0·2 e Å^{-3}. No correction for secondary extinction. All calculations (*SDP*, Enraf–Nonius, 1979) performed using form factors calculated by Doyle & Turner (1968) for O, N and C, and contracted spherical form factor (Stewart, Davidson & Simpson, 1965) for H atoms.

Discussion. Table 1 gives the positional and isotropic thermal parameters for all atoms.*

The intramolecular bond lengths and angles listed in Table 2 show that the glycine molecule is in the zwitterionic form $NH_3^+CH_2CO_2^-$. The covalent distances and angles (C—C, C—N and C—O) are in good agreement with the values observed in the molecular structure of α-glycine (Jönsson & Kvick, 1972).

The hydrogen-bond distances in Table 3 indicate that the dipolar glycine molecules are strongly attached to $Te(OH)_6$ by short O—H···O bonds: O(1)—H(9)···O(5), O(2)—H(1)···O(4) and O(3)—H(10)···O(5). Each $Te(OH)_6$ is bonded in this way to four glycine molecules, forming clusters of $Te(OH)_6$.(glycine)$_4$ (Fig. 1) which are linked together by hydrogen bonds [O(6)—H(8)···O(1), O(6)—H(4)···O(5)] to give a chain of $Te(OH)_6$.(glycine)$_4$ running along the [001] direction. Such a chain is bonded to four neighbors by hydrogen contacts such as O(3)—H(10)···O(5) (Fig. 2).

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* Lists of observed and calculated structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38804 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The Te atom placed at the center of symmetry is octahedrally coordinated by OH groups. The octahedron is slightly distorted with two long distances Te—O(1) and Te—O(3), respectively 1·927 (2) and 1·929 (2) Å, and one short distance Te—O(2) of 1·905 (2) Å. The deviation of the O atoms in the Te(OH)$_6$ group from ideal octahedral coordination around Te, already observed in monoclinic orthotelluric acid (Lindqvist & Lehmann, 1973), may be ascribed to the hydrogen-bonding network involving three O atoms coordinated to the Te atom. It is noted that O atoms O(1) and O(3) corresponding to two longer Te—O bonds are acceptors of rather strong hydrogen bonds, N—H''(N)···O(1), O*W*—H'(O*W*)···O(1) and N—H(N)···O(3) (respectively 2·963, 2·896 and 2·936 Å), whereas the O(2) atom corresponding to the shorter Te—O(2) bond is involved only in very weak hydrogen bonds, C(2)—H(C2)···O(2) and C(2)—H'(C2)···O(2) (Table 3).

Note added in proof: After submitting our manuscript we have learned that a paper on the same compound has been published by Andersen, Lindqvist & Moret (1983). The main structural features obtained independently by these authors at room temperature and in our analysis at low temperature are similar, except for the hydrogen positions which were not established previously. The differences in the bond lengths C(1)—O(5) [1·268 (5) (Andersen *et al.*), 1·274 (4) Å (this work)] and C(1)—O(4) [1·226 (5) (Andersen *et al.*), 1·249 (4) Å] may be ascribed to the shorter hydrogen bonds involving O(4) and O(5) observed in the previous work (*cf.* Table 3).

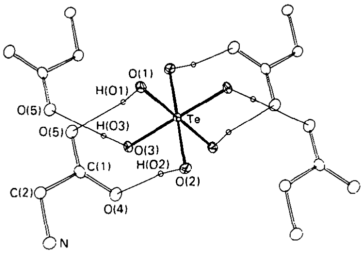

Fig. 1. Complex of Te(OH)$_6$.glycine and hydrogen bonds.

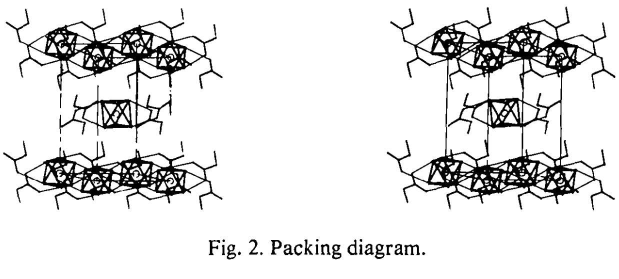

Fig. 2. Packing diagram.

Table 1. *Positional and isotropic thermal parameters with their e.s.d.'s in parentheses*

$$B_{eq}=\tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}/B_{iso}(Å^2)
Te	0	0	0	0·55 (3)
O(1)	−0·1106 (3)	0·0973 (2)	−0·2213 (3)	0·95 (3)
O(2)	0·2006 (2)	0·0979 (2)	0·0757 (3)	1·03 (3)
O(3)	−0·1066 (2)	0·0893 (2)	0·1511 (3)	0·96 (3)
O(4)	0·3726 (3)	0·1277 (2)	0·4590 (3)	1·38 (3)
O(5)	0·2450 (3)	0·9918 (2)	0·5765 (3)	1·33 (3)
O*W*	0·1266 (3)	0·2674 (2)	0·7306 (3)	1·68 (3)
N	0·5709 (3)	0·2040 (2)	0·8264 (3)	1·05 (4)
C(1)	0·3517 (3)	0·0730 (2)	0·5965 (4)	1·05 (4)
C(2)	0·4621 (3)	0·1027 (2)	0·8100 (4)	0·96 (4)
H(O2)	0·253 (5)	0·102 (3)	0·210 (5)	1·4 (7)
H(C2)	0·041 (4)	0·456 (3)	0·368 (5)	0·7 (6)
H(C3)	0·386 (5)	0·112 (3)	0·891 (5)	1·8 (8)
H(O*W*)	0·131 (7)	0·320 (5)	0·796 (7)	4·7 (13)
H(N)	0·002 (5)	0·233 (4)	0·261 (6)	2·6 (9)
H'(N)	0·104 (5)	0·276 (4)	0·459 (6)	2·3 (9)
H''(N)	0·169 (5)	0·302 (3)	0·296 (5)	2·0 (8)
H'(O*W*)	0·062 (6)	0·213 (4)	0·766 (7)	3·8 (11)
H(O1)	0·358 (5)	0·436 (3)	0·190 (5)	1·1 (7)
H(O3)	0·358 (6)	0·435 (4)	0·724 (6)	3·0 (10)

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

Te—O(1)	1·927 (2)	C(1)—O(4)	1·249 (4)
Te—O(2)	1·905 (2)	C(1)—O(5)	1·274 (4)
Te—O(3)	1·929 (2)	C(1)—C(2)	1·518 (3)
Average	1·920	C(2)—N	1·484 (4)
O(1)—Te—O(2)	86·74 (8)	C(1)—C(2)—H(C2)	109 (2)
O(1)—Te—O(3)	87·56 (8)	C(1)—C(2)—H(C3)	112 (2)
O(2)—Te—O(3)	88·56 (8)	N—C(2)—H(C2)	108 (2)
O(4)—C(1)—O(5)	126·2 (3)	H(C2)—C(2)—H(C3)	106 (3)
O(4)—C(1)—C(2)	118·9 (2)	N—C(2)—H(C3)	108 (2)
O(5)—C(1)—C(2)	114·9 (2)	H'(O*W*)—O*W*—H(O*W*)	109 (5)
C(1)—C(2)—N	112·9 (2)		

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)	0·72 (3)	2·628 (3)	167 (4)
O(2)—H(O2)···O(4)	0·91 (3)	2·632 (3)	171 (4)
O(3)—H(O3)···O(5)	0·74 (5)	2·743 (3)	176 (5)
O*W*—H(O*W*)···O(5)	0·78 (5)	3·067 (3)	152 (5)
O*W*—H'(O*W*)···O(1)	0·93 (5)	2·896 (3)	167 (5)
N—H(N)···O(3)	0·96 (4)	2·936 (3)	171 (4)
N—H'(N)···O(6)	0·93 (4)	2·802 (3)	164 (4)
N—H''(N)···O(1)	0·88 (4)	2·963 (3)	150 (4)
C(2)—H(C2)···O(2)	0·95 (3)	3·495 (4)	159 (3)
C(2)—H'(C2)···O(2)	0·98 (4)	3·277 (4)	169 (3)

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(1*R**,1′*R**,2*S**,2′*S**,3*R**,3′*R**,5*R**,5′*R**,6*S**,6′*S**)-3-Hydroxy-3′-trimethylsilyloxy[3,3′-bitricyclo[4.2.1.$0^{2,5}$]non-7-enyl]-4,4′-dione, $C_{21}H_{26}O_4Si$

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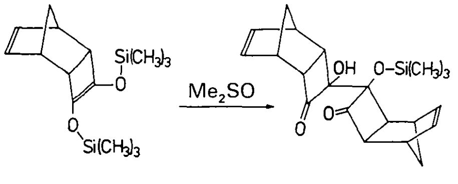
Abstract. $M_r = 370{\cdot}52$, triclinic, $P\bar{1}$, $Z = 2$, $a = 9{\cdot}041\ (2)$, $b = 10{\cdot}505\ (5)$, $c = 11{\cdot}546\ (3)$ Å, $\alpha = 76{\cdot}59\ (3)$, $\beta = 76{\cdot}09\ (2)$, $\gamma = 67{\cdot}54\ (3)°$, $V = 971{\cdot}5\ (5)$ Å^3, $D_x = 1{\cdot}266$ Mg m^{-3}, $F(000) = 396$, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}137$ mm^{-1}, room temperature, 3124 non-equivalent diffractometer data up to $\sin\theta/\lambda = 0{\cdot}58$ Å^{-1}, final $R(F) = 0{\cdot}045$, $R_w(F) = 0{\cdot}054$. The molecule consists of two *endo*-tricyclo[4.2.1.$0^{2,5}$]non-7-ene groups linked by a single C—C bond of 1·557 (2) Å. The norbornene groups show a bridging C—C—C angle of 93·6 (2)°. The molecules form dimers linked by hydrogen bonding.

Introduction. The title compound was obtained by an oxidative dimerization of *endo*-3,4-bis(trimethylsilyloxy)tricyclo[4.2.1.$0^{2,5}$]nona-3,7-diene with Me_2SO in acetic anhydride, accompanied by a partial hydrolysis (Bellinger, 1983). As a chemical analysis was not conclusive concerning the nature of the reaction product, an X-ray structure analysis was undertaken.



Experimental. Colorless crystals, recrystallization from diethyl ether. Crystal: 0·3 × 0·3 × 0·5 mm. Enraf–Nonius CAD-4 diffractometer. Cell constants from setting angles of 25 reflections with $6 < \theta \leq 12°$. One hemisphere up to $2\theta = 49°$, ω scan. Three standard reflections every 4000 s remained stable. 3846 reflections measured, 3281 unique, 3124 with $I > 0{\cdot}4\sigma(I)$ used. No absorption correction. Weighting scheme: $w(I) = 1/[\sigma^2(I) + (0{\cdot}03I)^2]$. Structure determination by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All H atoms from difference synthesis, H atoms included in refinement with fixed isotropic thermal parameters. Scattering factors and anomalous-dispersion factors from *International Tables for X-ray Crystallography* (1974). Extinction negligible. Calculations with SDP program system (Enraf–Nonius, 1981). Refinement on *F*. $(\Delta/\sigma)_{\max} = 0{\cdot}2$. Final difference synthesis featureless. $R(F) = 0{\cdot}045$, $R_w(F) = 0{\cdot}054$, $S = 2{\cdot}31$.

Discussion. The positional parameters are reported in Table 1.* Fig. 1 shows the bond distances and the numbering scheme of the atoms. The bond angles are

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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 38910 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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