

The Crystal Structure of Telluric Acid, $\text{Te}(\text{OH})_6$ (*mon*)

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The crystal structure of the monoclinic modification of telluric acid, $\text{Te}(\text{OH})_6$, has been determined by single crystal X-ray methods. The space group is $P2_1/n$ with $a=6.495$, $b=9.320$, $c=11.393$ Å, $\beta=133.88^\circ$, and $Z=4$.

The tellurium atoms were located through symmetry considerations, while the positions of the oxygen atoms and an indication as to the hydrogen positions were obtained from three-dimensional Fourier summations. The structure was refined to an R value of 0.066 using full matrix least squares calculations.

The structure contains discrete $\text{Te}(\text{OH})_6$ octahedra firmly connected by hydrogen bonds, each molecule being involved in twelve such bonds. The six independent Te—O bonds have lengths between 1.905 Å and 1.925 Å with a mean value of 1.916 Å.

An X-ray structure analysis of the cubic phase and a neutron diffraction study of the monoclinic phase of $\text{Te}(\text{OH})_6$ are in progress.

As pointed out by Wells in his survey of inorganic crystal structures,¹ the only pure hydroxy-acid whose structure is known is boric acid, $\text{B}(\text{OH})_3$.² For telluric acid, $\text{Te}(\text{OH})_6$, no definite crystal structure has been devised, but a number of papers³⁻¹⁰ have been published on the cell parameters and space groups of the different phases of this acid. Several of these studies contain, however, contradictory results.

The aim of this investigation has been to determine the arrangement of the oxygen atoms in telluric acid. This information is also considered to be of importance when discussing Te—O bonds in structures of different tellurates.

In the present paper a study of the monoclinic modification of telluric acid is presented. An investigation of the cubic phase is in progress.

EXPERIMENTAL

Colourless, rod-shaped crystals of monoclinic telluric acid were obtained by re-crystallization of an aqueous solution of commercial (BDH) telluric acid.

Preliminary X-ray studies showed that the monoclinic crystals had a great tendency to form twins. A single crystal suitable for the X-ray analysis could, however, be obtained by cutting out a well-shaped piece from an aggregate of large crystals (*cf.* Table 1). This

Table 1. Dimensions of the crystal used for the intensity measurements, given as indices of the boundary planes and the distance to these planes from an internal origin.

<i>h</i>	<i>k</i>	<i>l</i>	<i>d</i> (mm)
0	1	0	0.019
0	-1	0	0.019
2	-1	0	0.050
-2	1	0	0.050
0	0	-1	0.045
-1	0	2	0.070
1	0	-2	0.070

crystal was rotated around the *c*-axis, and 10 layer lines were collected using the multiple-film, equi-inclination Weissenberg method with six films for each layer line. All odd zones, as well as every second reflection on the even zones were very weak, and in order to decrease the exposure times, unfiltered Cu radiation was used.

A total of 589 independent reflections were estimated visually by comparing the $\text{CuK}\alpha$ spots (the β spots were not measured) with an intensity strip prepared from a suitable reflection of the actual crystal. The film factors were refined by means of the least squares program *SCALE*,¹¹ using the weighting scheme $w = \{3.5 + [(I - 8)/6.3]^2\}^{-1}$. Correction for *Lp* and absorption effects ($\mu = 610 \text{ cm}^{-1}$)¹² were performed using the program *DATA P2*.¹¹ The absorption correction was not, however, applied until an approximate structure had been devised.

Approximate values of the scale factors for the 10 layer lines were obtained from Weissenberg data from another crystal rotated about $[\bar{1}04]$.

UNIT CELL AND SPACE GROUP

Preliminary cell dimensions, determined from the Weissenberg and rotation photographs, were used to index a Guinier powder photograph, taken with $\text{CuK}\alpha_1$ radiation, using lead nitrate as internal standard ($a = 7.8566 \text{ \AA}$ at 21°C).¹² The subsequent refinement of the cell parameters were performed with a least squares, linear regression method,¹¹ and the result is given in Table 2. The observed and calculated $\sin^2\theta$ values are listed in Table 3.

Assuming four formula units of $\text{Te}(\text{OH})_6$ in the unit cell, a calculated density of 3.070 g.cm^{-3} is obtained, which is in good agreement with the experimental value of $3.06 \pm 0.01 \text{ g.cm}^{-3}$.⁹

The only systematic absences occurred when $h + l = 2n + 1$ in $h0l$, and when $k = 2n + 1$ in $0k0$, unambiguously indicating the space group to be $P2_1/n$. For

Table 2. Cell dimensions and space group of $\text{Te}(\text{OH})_6$ (*mon*).

	Gossner and Kraus, 1934	Aviness and Petit, 1968	Bayer, 1968	Present work	Present work transformed
<i>a</i>	5.70 Å	6.494 ± 0.006	6.494 ± 0.003	6.495 ± 0.001	6.495 ± 0.001
<i>b</i>	9.30	9.318 ± 0.004	9.322 ± 0.002	9.320 ± 0.001	9.320 ± 0.001
<i>c</i>	9.74	8.333 ± 0.005	8.333 ± 0.003	11.393 ± 0.001	8.331 ± 0.001
β	104.50°	99.68 ± 0.03	99.72 ± 0.05	133.88 ± 0.01	99.69 ± 0.01
Space group	$P2_1/c$	<i>Cc</i> or $C2/c$	$P2_1$ or $P2_1/m$	$P2_1/n$	$P2_1/c$

Table 3. Powder data for $\text{Te}(\text{OH})_6$ (mon). $\text{CuK}\alpha_1$ radiation, $\lambda=1.5405 \text{ \AA}$.

hkl	$10^5 \sin^2 \theta$ (obs)	$10^5 \sin^2 \theta$ (calc)	d (calc)	I (obs)
-1 1 2	2629	2630	4.7493	vvs
0 2 0	2732	2732	4.6601	vs
1 1 0	3387	3390	4.1834	vvs
0 0 2	3519	3519	4.1060	vs
-1 1 3	4887	4890	3.4833	vvw
0 0 2	6251	6251	3.0808	s
-2 0 4	7790	7790	2.7598	w
-1 3 2	8095	8094	2.7074	m
-2 2 2	8520	8522	2.6385	vs
1 3 0	8854	8854	2.5886	vs
-1 1 4	8908	8909	2.5806	vs
2 0 0	10832	10829	2.3407	vw
0 4 0	10926	10928	2.3301	vw
1 1 2	11193	11188	2.3028	w
0 4 1	11800	11807	2.2416	vvw
1 3 1	11873	11873	2.2354	vvw
2 2 0	13565	13561	2.0917	s
0 0 4	14075	14076	2.0530	m
-1 3 4	14372	14373	2.0317	w
0 4 2	14447	14447	2.0265	w
-3 1 2	15724	15730	1.9421	s
1 3 2	16646	16652	1.8876	s
-2 4 2	16721	16718	1.8838	s
0 2 4	16814	16808	1.8788	s
-2 4 4	18723	18717	1.7804	m
-3 3 4	18919	18915	1.7711	m
-1 5 2	19026	19022	1.7661	m
-2 2 6	19560	19559	1.7416	m
1 2 3	19774	19775	1.7321	m
-3 3 2	21188	21194	1.6731	m
2 4 0	21759	21756	1.6514	m
-1 1 6	22226	22225	1.6338	m
2 0 2	22901	22906	1.6094	vvw
-3 3 6	23662	23673	1.5831	m
0 6 0	24594	24587	1.5534	vw
0 4 4	25016	25004	1.5404	m
-1 5 4	25304	25300	1.5313	vw
2 2 2	25631	25638	1.5212	w
-4 2 6	26378	26372	1.4999	w
1 5 2	27573	27580	1.4667	w
-2 4 6	27741	27755	1.4621	w
0 6 2	28100	28106	1.4529	vw
-4 0 2	29717	29719	1.4129	vw
-3 5 4	29838	29842	1.4100	vw
-3 1 8	30021	30006	1.4061	vw
-2 6 2	30377	30377	1.3975	vvw
3 3 0	30527	30512	1.3944	vvw
-4 0 8	31169	31158	1.3799	vw
1 3 4	31500	31488	1.3727	w
-3 5 2	32124	32122	1.3590	vw
-4 2 8	33878	33890	1.3231	vvw
-4 4 4	34080	34088	1.3193	vvw
0 2 6	34388	34404	1.3132	vw
-1 7 2	35415	35413	1.2943	m

computational reasons, no transformation to the conventional setting of $P2_1/c$ was performed.

The cell dimensions and the space group of monoclinic telluric acid have been reported in three previous papers (*cf.* Table 2), but none of these are quite in accordance with the present results. Gossner and Kraus⁴ have obtained the same symmetry as in this investigation, but as pointed out by Bayer⁸ and by Aviness and Petit,¹⁰ their choice of unit cell does not seem to be in accordance with this symmetry. Both Bayer, and Aviness and Petit have obtained cell dimensions in good agreement with the present study, but they have reported a different symmetry. Aviness and Petit were unable to detect any weak reflections with $h+k=2n+1$ in their X-ray diagrammes, and these authors therefore suggested $C2/c$ or Cc . Bayer deduced the space group to be either $P2_1/m$ or $P2_1$. His own data do, however, not contain any reflection of type $h0l$ with either $h=2n+1$ or $l=2n+1$.

It is possible that the different results listed in Table 2 are due to investigation of different phases of monoclinic telluric acid, but it seems much more plausible that the discrepancies result from the non-observance of all weak reflections.

DETERMINATION AND REFINEMENT OF THE STRUCTURE

The reflections of the monoclinic phase of telluric acid fall into two groups which differ considerably in intensity. All reflections with $l=2n$ and $h+k=2n$ are always very strong. In the space group $P2_1/n$ there is only one type of special position, *i.e.* the centre of symmetry.* Atoms which occupy these positions contribute only to reflections with $h+k+l=2n$. Two of the four tellurium atoms ought therefore to be situated in the site $2(a)$ (Te_1), while the other two should occupy $2(c)$ (Te_2), which is shifted from $2(a)$ by the translation $c/2$. This arrangement accounts for the fact that only reflections with $h+k=2n$ and $l=2n$ are very strong.

A three-dimensional Fourier summation was calculated, using the 200 strong reflections only. The signs of these strong $|F_o|$ values ought certainly to be determined by the tellurium positions, *i.e.* all of them are positive. The resulting Fourier maps were, of course, rather poorly resolved with respect to the oxygen atoms, and since the tellurium arrangement implies a body centering, ambiguities also occurred. Nevertheless, assuming the oxygen coordination around tellurium to be octahedral and a Te—O bond distance of about 2 Å, six fourfold peaks could be selected to represent the oxygen atoms, resulting in a reasonable packing in the structure. A preliminary isotropic refinement, using all 589 observed reflections yielded an R value of 0.11 ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$).

In the next step, the intensities were corrected for absorption effects, using the crystal dimensions listed in Table 1. This lowered the R factor to about

* Positions in $P2_1/n$:
 $4(e): \pm(x,y,z), \pm(1/2+x, 1/2-y, 1/2+z);$ $2(b): (0, 1/2, 0), (1/2, 0, 1/2);$
 $2(d): (1/2, 0, 0), (0, 1/2, 1/2);$ $2(a): (0, 0, 0), (1/2, 1/2, 1/2);$
 $2(c): (0, 0, 1/2), (1/2, 1/2, 0).$

0.09. A correction for secondary extinction was then carried out according to Zachariasen,^{13,14} using the computing method described by Åsbrink and Werner¹⁵ (cf. also note given by Åsbrink and Norrby¹⁶). The C value in Zachariasen's formula was calculated for about 40 of the strongest reflections giving an average value of 0.0065. In the subsequent refinement $C=0.0050$ and $C=0.0080$ were also tested, but the original value, $C=0.0065$, proved to be superior, and improved agreement between the strong $|F_o|$ and $|F_c|$ values was obtained. A few of the strongest reflections could, however, not be corrected for all possible errors in this way. This was obviously the case for 020, and this reflection was therefore excluded in subsequent cycles of refinement.

In the previous cycles of refinement the inter-layer scale factors had been included, but all weak reflections with $l=2n$ had systematically too small $|F_c|$ values. This effect can be traced back to the visual measurement, which has a tendency to give too low film factors when small, intense spots are estimated. Since most of the strong reflections were not weak enough to be measured until on the fourth of the six films, while the weak ones were all measured on the first film, this effect occurred systematically and thus became apparent.

To correct for this scaling error, all weak reflections from the even zones were picked out and assigned a single scale factor, and the refinement was continued with the weak reflections only. Once acceptable scale factors were obtained, the oxygen parameters were refined anisotropically with an over-all scale factor. The resulting R value was 0.102.

From a difference Fourier map based on the weak reflections, all six independent hydrogen positions could be deduced. Some spurious peaks of approximately the same electron densities ($0.4-0.5 e/\text{\AA}^3$) were excluded for geometrical reasons. In the final stages of refinement based on the weak reflections, the hydrogen parameters were included, each hydrogen atom being assigned a constant isotropic temperature factor of 4.5\AA^2 . One of the hydrogen atoms was shifted to an unrealistic position, while the other five appeared to have enough influence on the weak reflections to be able to be successfully refined.

The final R value obtained for the 389 weak reflections was 0.091, and if 246 reflections which were too weak to be observed ($I_{\text{unobs}} = 2/3 \cdot I_{\text{obs}} (\text{min})$, I_{unobs} being corrected in the same way as I_{obs}) were included, an R value of 0.15 was obtained.

Finally all strong and weak observed reflections were used in the refinement, keeping the hydrogen parameters constant. The R factor converged to 0.066 (0.083 including unobserved reflections), and the corresponding atomic parameters and structure factors are given in Tables 4 and 5, respectively. Atomic scattering factors due to Cromer and Waber,¹⁷ corrected for the real part of the anomalous scattering,¹² were used for the tellurium atoms, while those devised by Doyle and Turner¹⁸ and by Stewart *et al.*¹⁹ were used for the oxygen and hydrogen atoms, respectively. The weighting function $w = (a + |F_o| + c|F_o|^2 + d|F_o|^3)^{-1}$, as recommended by Cruickshank, was used with $a=10.0$, $c=0.045$, and $d=0.0008$.

Table 4. Final atomic parameters. The anisotropic temperature factor is $\exp[-2\pi(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + hka^*b^*U_{12} + hla^*c^*U_{13} + klb^*c^*U_{23})]$. Standard deviations given in parentheses.

Atom	x/a	y/b	z/c	$B \text{ \AA}^2$		
Te ₁	0	0	0			
Te ₂	0	0	1/2			
O ₁	0.7291(10)	0.1544(5)	0.8928(6)			
O ₂	0.1255(11)	0.0482(7)	0.2037(6)			
O ₃	0.7739(10)	0.3744(5)	0.5373(7)			
O ₄	0.6895(9)	0.0957(5)	0.4544(6)			
O ₅	0.3241(10)	0.4509(7)	0.7845(6)			
O ₆	0.7168(10)	0.3243(5)	0.0774(6)			
H ₁	0.199(20)	0.282(12)	0.457(14)	4.5		
H ₂	0.268(23)	0.118(12)	0.275(13)	4.5		
H ₃	0.499(23)	0.084(12)	0.130(14)	4.5		
H ₅	0.994(20)	0.080(14)	0.264(11)	4.5		
H ₆	0.883(24)	0.346(12)	0.192(14)	4.5		
	$U_{11} \times 10^4$	$U_{22} \times 10^4$	$U_{33} \times 10^4$	$U_{12} \times 10^4$	$U_{13} \times 10^4$	$U_{23} \times 10^4$
Te ₁	103(8)	133(9)	84(11)	1(3)	111(15)	7(3)
Te ₂	99(8)	130(9)	81(11)	-10(3)	100(15)	6(3)
O ₁	194(23)	259(25)	166(26)	98(37)	195(42)	-7(42)
O ₂	219(26)	377(32)	174(27)	-63(41)	299(48)	-110(48)
O ₃	183(25)	242(23)	264(31)	6(38)	200(44)	-130(43)
O ₄	142(22)	293(25)	192(27)	-1(38)	197(42)	-53(41)
O ₅	146(24)	426(33)	133(25)	-19(47)	174(42)	-95(53)
O ₆	228(24)	198(23)	229(26)	59(38)	272(43)	-72(41)

In Table 4 it is noticed that the standard deviations for U_{12} and U_{23} of the tellurium atoms are relatively low. These parameters are dependent on the weak reflections only, and the influence this has on the *e.s.d.*'s has recently been discussed by Åsbrink and Norrby.¹⁶ In their case, the number of weak reflections was less than 10 % of the total number, which caused increased *e.s.d.*'s in the U_{12} and U_{23} terms, but in the present study, in which about 60 % of the reflections are weak, the opposite would, according to Åsbrink and Norrby, be expected to occur.

The Fourier and least squares calculations were carried out with the programs *DRF*¹¹ and *LALS*,¹¹ respectively, all calculations being performed on an IBM 360/65 computer.

DISCUSSION

The structure of the monoclinic modification of telluric acid is built up from discrete $\text{Te}(\text{OH})_6$ octahedra, each molecule being involved in twelve hydrogen bonds. Schematic pictures of the two independent tellurium-oxygen octahedra and of the hydrogen bonding net in the structure are shown in Figs. 1 and 2, respectively.

Table 5. Observed and calculated structure factors. The columns are k , $10|F_o|$ and $10F_c$, respectively. (* indicates unobserved reflections).

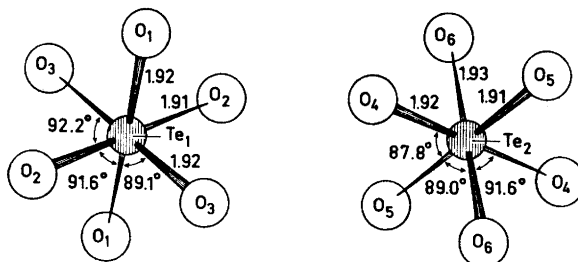
0 K 0	2 207 -178	5 983 901	7 881 785	6 58 -55	8 70 75	-2 K 5
2 3361 2138*	3 152 158	6 27 -10*	8 19 42*	7 133 149	9 857 817	1 111 102
4 1046 1083	4 245 234	7 745 742		8 69 -71	10 59 52	2 415 401
6 1138 1289	5 33 12*			9 25 -11*	11 638 566	3 69 63
8 843 707	6 142 -150	-4 K 2	3 1314 980	10 82 82		4 283 325
10 715 585	7 142 -150	0 1421 1458	1 55 51		0 K 4	5 88 -97
	8 29 16*	1 91 -90	2 904 867	2 K 3	0 1625 1640	6 72 58
1 K 0	9 26 -23*	2 1271 1130	3 71 84	1 31 -33*	1 79 -65	7 32 -8*
1 2109 1999	10 62 58	3 87 -84	4 868 793	2 94 86	2 1677 1577	8 30 23*
2 24 -1*	11 83 77	4 918 886	5 49 55	3 101 -93	3 97 87	9 27 -7*
3 1874 1794		5 74 -56	6 726 719	4 224 226	4 1361 1284	10 45 45
4 176 187	-1 K 1	6 57 871		5 74 58	5 102 111	
5 1673 1422	3 249 238	7 29 -23*	-6 K 3	6 59 -44	5 929 307	-1 K 5
5 39 11*	4 70 -59	8 852 787	2 87 -84	7 32 29*	7 67 -53	0 98 -81
7 975 1311	5 30 28	9 37 51	3 134 130	8 105 -90	8 797 880	1 415 426
8 19 -39*	6 27 -21*		4 78 69	9 44 -50	9 28 -26*	2 23 -13*
9 726 768	7 270 -306	-3 K 2	5 26 -5*		10 594 733	3 275 260
10 31 -19*	8 60 55	1 1645 1525	5 81 92	3 K 3		4 36 4*
11 612 528	9 25 -21*	2 23 1*		0 35 26*		5 89 -72
	10 53 -26	3 1859 1686	-5 K 3	1 35 -11*	1 K 4	6 34 22*
2 K 0	11 99 91	4 69 -97	0 159 174	2 35 -5*	1 1325 1234	7 100 106
0 714 723		5 1035 987	1 141 151	3 37 -30*	2 105 -112	8 61 67
1 154 -149	3 K 1	6 36 -29*	2 73 75	4 37 -20*	3 1515 1523	9 35 -34*
2 1550 1523	1 12 15*	7 816 795	3 128 137	5 85 -79	4 105 98	9 27 17*
3 88 -84	2 201 -212	8 73 -53	4 53 -56	6 30 33*	5 1131 1103	10 39 -31
4 1668 1607	3 74 75	9 777 784	5 38 14*	7 47 -47	6 125 108	
5 45 -1*	4 555 -565	10 49 -58	5 35 -38*		7 782 777	0 K 5
6 1112 980	5 50 -40			4 K 3	8 100 -51*	1 205 -16*
7 44 -19*	6 162 -166	-2 K 2	-4 K 3	1 59 50	9 652 648	2 215 -235
8 815 878	7 134 149	3 1331 1386	1 28 12*	2 75 -77	2 K 4	3 32 15*
9 35 36*	8 28 15*	1 67 55	2 207 223	3 84 87	3 1159 1244	4 41 29*
10 733 725	9 75 17*	2 1726 1855	3 131 -133	4 134 -124	1 81 87	5 87 82
	10 56 -48	3 81 -80	4 104 -95		2 1061 1084	6 15 -34*
	11 104 -111	4 1939 1799	5 63 48	-6 K 4	3 41 6*	8 30 33*
3 K 0		5 32 -49*	6 57 -43	0 864 935	4 951 921	9 24 10*
1 1101 1179	1 K 1	6 1180 1125	7 75 73	1 37 -38*	5 44 -53*	10 18 -57*
2 40 -23*	0 15 -13*	7 28 3*	8 44 38	2 860 834	6 1020 838	
3 1105 1031	1 43 -20	8 723 787	9 21 -12*	7 75 6	7 75 6	1 K 5
4 105 -109	2 109 118	9 52 -55		4 888 925	8 625 553	0 166 176
5 969 880	3 483 -503	10 665 662	-3 K 3	5 34 -23*		1 183 -185
6 55 -50	4 78 74	11 15 13*	3 211 -194	6 736 728	3 K 4	2 35 30*
7 915 921	5 306 -290	-1 K 2	1 357 -354		1 902 942	3 136 -132
8 37 -17*	6 88 -101	1 2075 2153	2 116 -122	-5 K 4	2 100 112	4 41 -12*
9 803 803	7 125 133	2 22 -3*	3 404 -399	1 1195 1170	3 713 697	5 43 -21*
	8 62 -51	3 1375 1104	4 77 72	2 71 -64	4 35 -23*	5 43 0*
4 K 0	9 43 -36	4 134 -124	5 65 -52	3 999 964	5 764 700	7 130 -12*
0 1053 1123	10 53 46	5 1463 1413	6 37 28*	4 78 -71	6 42 -47	8 83 -88
1 85 79	11 77 -57	6 46 -37	7 53 -23	5 945 915		9 49 -42
2 874 861		7 1174 1191	8 33 -40*	6 119 -116		
3 49 -15*	2 K 1	8 68 58	9 26 -2*	7 836 781	-7 K 5	1 104 -88
4 834 752	1 23 -14*	9 729 778	10 57 47	5 23 -5*	2 29 6*	1 37 35*
5 177 -78	2 90 76	10 32 31	-2 K 3	-4 K 4	3 59 -37	2 38 43*
6 1074 885	3 27 17*	11 675 683	1 48 -65	0 1408 1354	4 53 58	3 39 5*
7 35 -7*	4 147 163		2 464 -307	1 28 -24*		4 39 -15*
8 709 715	5 57 58	0 K 2	3 72 72	2 1508 1353	-6 K 5	6 154 -148
	6 70 -42	1 13 -1*	4 32 15*	3 64 -52	1 75 -72	7 21 18*
1 710 742	7 83 -91	2 1482 1312	5 75 -72	4 1012 995	2 82 -77	
2 42 -43*	8 98 -92	3 104 -78	6 105 -175	5 31 -38*	3 33 -15*	0 3 K 5
3 797 532	9 44 -38	4 1362 1351	7 70 -63	6 40 29*	4 98 -9*	0 51 -53
4 39 -18*	10 30 23	5 27 -13*	8 151 -166	7 35 12*	5 36 -23*	1 31 16*
5 846 793		6 1287 1220	9 27 13*	8 938 866	6 28 -7*	2 30 23*
	-5 K 1	7 57 -66	10 84 75	9 24 14*		3 29 -35*
0 45 -43	0 29 -15*	8 1343 1358	11 52 51	10 654 563	-5 K 5	4 25 3*
1 33 -22*	1 30 -20*	9 25 12*			0 54 39	1 189 194
2 33 37	2 174 175	10 866 821	-1 K 3	-3 K 4	2 100 -93	-7 K 6
3 33 0*	3 72 -75		3 5 7*	1 1222 1055	3 38 26*	1 780 790
4 32 -11*	4 72 -75	1 K 2	1 395 355	2 75 65	4 103 -127	2 33 -52*
5 33 3*	5 145 143	1 891 920	2 26 13*	3 1611 1459	5 217 -228	3 701 559
6 88 -81	6 131 125	2 158 -143	3 302 299	4 91 78	6 40 29*	4 29 10*
7 48 -49	7 65 -52	3 1568 1614	4 95 -87	5 1051 1107	7 57 -52	5 602 631
	8 48 45	4 77 87	5 138 -150	6 112 120	8 24 39*	
	9 35 43	5 1475 1247	6 28 25*	7 783 303		-6 K 6
-4 K 1		6 33 -5*	7 109 -107	8 60 -53	-4 K 5	0 833 815
1 137 148	1 47 -51	7 956 979	8 89 98	9 872 853	1 93 -89	1 88 22*
2 207 242	2 34 -25*	8 71 -71	9 27 1*	10 84 -81	2 91 -74	2 806 766
3 83 -81	3 56 -50	9 845 886	10 69 -63		3 70 -55	3 132 96
4 42 44	4 34 13*	10 19 -11*	11 38 28		4 67 -41	4 938 814
5 37 -27*	5 65 -70	2 K 2		-2 K 4	5 74 89	5 66 41
6 37 31*	6 94 84	0 1054 1018	0 K 3	0 872 1130	6 94 814	6 914 814
7 62 58	7 42 41	1 87 -87	1 93 -93	1 73 75	7 23 -38*	7 23 -38*
8 22 20*		2 1386 1252	2 224 241	2 1106 1030		
9 15 -24*		3 118 -114	3 25 -13*	3 32 5*	8 29 0*	
	5 K 1	4 124 -109	4 124 -109	4 1379 1352	9 24 14	-5 K 5
-3 K 1	3 89 -93	5 35 -4*	5 35 -4*	5 31 -18*	10 17 -6*	1 1014 888
0 109 111	1 48 44	6 1261 1226	6 208 220	6 1296 1387		2 59 53
1 111 101	2 32 24*	7 35 -16*	7 31 39*	7 53 48	0 39 -32	3 1035 961
2 26 -8*	3 24 9*	8 1105 933	9 27 42*	8 859 882	1 378 -410	4 40 23*
3 33 27	4 21 36*	9 73 25*	10 71 -73	9 29 -2*	2 75 69	5 874 834
4 59 69		10 615 538	11 40 -34	10 705 705	3 120 -112	6 39 -1*
5 101 100	-6 K 2			11 17 3*	4 70 73	7 935 824
6 92 79	2 661 680		1 K 3	-1 K 4	5 274 320	8 78 59
7 209 235	3 82 81	1 1295 1343	0 49 -42	1 1721 1865	6 34 -35*	9 720 711
8 64 -65	4 444 718	2 151 172	1 43 -34	2 65 61	7 33 54*	-4 K 6
9 49 41		3 927 889	2 47 -31	3 1193 1231	8 59 54	3 1205 1291
10 43 -40	-5 K 2	4 32 31*	3 35 -16*	4 85 58	9 38 34	1 25 11*
	1 887 857	5 874 822	5 35 5*	5 1162 1165	10 21 9*	2 1275 1225
0 -17 K 0	3 775 797	6 107 -106	4 33 17*	6 107 -106		3 32 10*
1 18 32*	4 77 71	7 1345 1113	5 237 250	7 1345 1113		4 1065 1029

Table 5. Continued.

5 82 72	3 35 17*	5 34 44*	5 33 13*	-5 K 8	3 977 960	-4 K 9
6 760 775	4 1087 1093	6 30 5*	7 25 -23*	1 1206 1167	4 43 57*	1 83 -57
7 33 40*	5 127 131	7 35 26	8 22 5*	2 72 -73	5 752 748	2 341 -349
8 945 865	6 837 739	8 40 -37	9 17 -32*	3 1111 1144	6 37 -18*	3 46 -39
9 65 -38	7 33 -55*	9 25 34	0 K 7	4 97 -115	7 752 775	4 87 -83
10 654 714	8 795 786	-4 K 7	1 75 78	5 803 773	8 80 -74	5 22 13*
-3 K 6	9 84 -87	1 19 23*	2 233 -248	5 53 -48	0 K 8	6 25 -15
1 1192 1385	1 K 6	2 197 -186	3 63 -71	7 712 579	0 743 815	7 45 59
2 24 15*	1 751 802	3 37 28	4 33 -5*	8 25 -4*	1 39 -27*	9 45 -57
3 1338 1289	2 65 -49	4 212 233	5 36 5*	-4 K 8	2 875 870	-3 K 9
4 95 -3*	3 922 910	5 64 57	6 90 69	3 1329 1468	3 42 -35*	0 11 -30*
5 1122 1243	4 92 -93	6 162 156	7 23 -14*	1 24 -35*	4 883 852	1 69 59
6 64 51	5 985 922	7 25 4*	8 17 -13*	2 1152 1265	5 41 36*	2 55 51
7 808 895	6 36 9*	8 23 -11*	0 K 7	3 105 -8*	6 816 772	3 60 -51
8 66 -65	7 812 781	9 18 -31*	0 29 15*	4 1055 989	7 22 1*	4 24 -6*
9 671 543	8 19 6*	-3 K 7	1 30 12*	5 38 13*	1 K 8	5 115 -121
10 74 -72	2 K 5	3 7 9*	2 33 -33*	6 748 800	1 797 826	7 22 22
-2 K 6	0 832 875	1 159 -182	3 105 112	7 32 -11*	2 60 50	8 16 14*
0 1246 1845	1 70 71	2 67 57	4 30 -36*	8 709 570	3 748 762	-2 K 9
1 39 45	2 759 733	3 134 -122	5 92 77	9 95 -10*	4 75 -75	1 45 -45
2 1363 1436	3 34 -33*	4 29 -25	5 23 11*	-3 K 8	-7 K 9	2 259 255
3 33 -18*	4 728 682	5 73 57	2 K 7	1 1050 1150	0 45 53	3 53 51
4 1294 1255	5 126 -141	6 43 -43	1 102 -110	2 57 52	1 49 32	4 38 28
5 121 -137	-7 K 7	7 25 -1*	2 135 137	3 1088 1017	2 57 49	5 26 9*
6 1023 1172	0 114 -113	8 23 2*	3 23 14*	4 34 -31*	3 24 -14*	6 22 -7*
7 32 2*	1 98 -83	9 20 3*	-7 K 8	5 1006 1131	4 23 32*	7 26 -28
8 716 702	2 28 -30*	10 14 -11*	1 777 737	6 34 -22*	-6 K 9	8 44 39
9 68 71	3 111 -103	-2 K 7	2 38 18*	7 793 843	1 52 55	-1 K 9
10 571 531	4 26 27*	1 15 -14*	3 664 531	8 29 1*	2 150 151	0 24 11*
-1 K 6	-6 K 7	2 259 252	4 72 89	9 536 549	3 27 -10*	1 40 -42
1 1701 1662	1 86 89	3 52 56	5 697 673	-2 K 8	0 1000 989	2 66 -69
2 25 1*	2 132 133	4 224 -212	6 189 -21*	1 26 2*	1 26 2*	3 53 45
3 1460 1293	3 37 3*	5 45 -52	7 26 -10*	2 1051 986	6 20 -48*	4 27 -19*
4 36 37*	4 70 -59	6 189 -21*	8 23 -28*	3 35 3*	3 35 3*	5 177 158
5 1060 928	5 32 -23*	9 19 21*	-1 K 7	4 969 978	4 969 978	6 68 62
6 71 -49	6 25 16*	0 63 -56	0 161 168	5 82 -87	5 82 -87	7 73 74
7 781 824	7 19 -27*	1 44 32	1 225 201	6 840 905	6 840 905	0 K 9
8 30 5*	8 43 42	2 24 -16*	2 50 49	7 45 -26	7 45 -26	1 33 30
9 780 745	0 K 5	3 154 -143	3 242 253	8 794 764	8 794 764	2 79 -70
10 43 42	1 91 -92	4 51 51	4 32 5*	9 77 85	9 77 85	3 84 -73
0 K 5	2 50 49	5 173 -184	5 173 184	-1 K 8	6 50 46	4 96 82
0 1068 989	3 242 253	6 809 745	7 45 -47	1 1015 935	7 105 -132	5 17 -12*
1 91 -92	4 32 5*	7 45 -47	8 794 764	2 84 -81		
2 1302 1207						

Table 6. Distances (Å) and angles (°) within the two tellurium-oxygen coordination polyhedra. Standard deviations in parentheses.

Te ₁ -O ₁	1.918(5)	O ₁ -Te ₁ -O ₂	91.6(2)
Te ₁ -O ₂	1.905(5)	O ₁ -Te ₁ -O ₃	89.1(2)
Te ₁ -O ₃	1.916(5)	O ₂ -Te ₁ -O ₃	92.2(2)
Te ₂ -O ₄	1.918(4)	O ₄ -Te ₂ -O ₅	87.8(2)
Te ₂ -O ₅	1.911(5)	O ₄ -Te ₂ -O ₆	91.6(2)
Te ₂ -O ₆	1.925(5)	O ₅ -Te ₂ -O ₆	89.0(2)

Fig. 1. The two independent Te(OH)₆ octahedra.

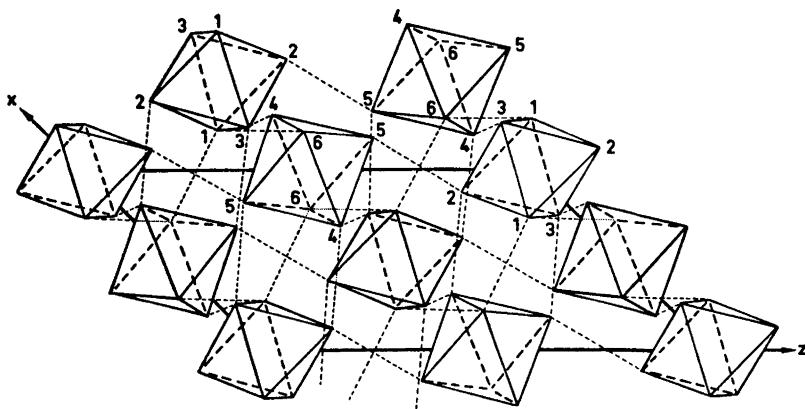


Fig. 2. A schematic picture of the structure of $\text{Te}(\text{OH})_6$ (*mon*), projected along the *b*-axis. The hydrogen bonds indicated as dotted lines.

The oxygen coordination around tellurium is very nearly a regular octahedron (*cf.* Fig. 1 and Table 6), none of the $\text{Te}-\text{OH}$ bonds being significantly different from the mean value 1.196 Å and those angles, which according to the *e.s.d.*'s deviate significantly from regularity, do not differ by more than $1-2^\circ$.

The $\text{Te}-\text{OH}$ bond lengths in telluric acid are shorter than in most of the tellurates which have been investigated. The tellurium-oxygen bond distances in a number of tellurates are given in Table 7, and, with the exception of $\text{KTeO}_2(\text{OH})_3$ ²¹ and $\text{KTeO}_3(\text{OH})$ ²² this table shows that there is a significant tendency to weaker bonds the higher the negative charge on the anion. The bond distances found in $\text{KTeO}_2(\text{OH})_3$ and $\text{KTeO}(\text{OH})$ seem to be relatively uncertain, and this might be one reason why they deviate from the trend in Table 7. It is also possible that the bonding in these two structures, which

Table 7. Average bond lengths (Å) in compounds containing TeO_6 octahedra. Three different kinds of bonds are recognized: "single" bonds in $\text{Te}-\text{O}-\text{Te}$ bridges and in $\text{Te}-\text{OH}$, and "double" bonds $\text{Te}=\text{O}$. The tabulated standard deviations refer to those given for the individual bonds in the original papers.

Compound	Reference	$\text{Te}-\text{OH}$	$\text{Te}-\text{O}_{\text{br.}}$	$\text{Te}=\text{O}$	E.s.d.
$\text{Te}(\text{OH})_6$ (<i>mon</i>)		1.916	—	—	0.005
$\text{KTeO}(\text{OH})_5 \cdot \text{H}_2\text{O}$	20	1.93	—	1.83	0.01–0.02
$\text{KTeO}_2(\text{OH})_3$	21	1.89	1.84	1.89	0.1
$\text{KTeO}_3(\text{OH})$	22	1.84	1.99	1.84	—
$\text{Ag}_2\text{TeO}_2(\text{OH})_4$	23	1.98	—	1.85	0.05–0.09
$\text{K}_4[\text{Te}_2\text{O}_8(\text{OH})_4] \cdot 7.3\text{H}_2\text{O}$	24	2.00	2.03	1.88	0.01
$\text{Na}_2\text{K}_4[\text{Te}_2\text{O}_8(\text{OH})_2] \cdot 14\text{H}_2\text{O}$	25	2.03	2.04	1.90	0.01–0.02
Hg_5TeO_6	26	—	—	1.98	—

Table 8. Hydrogen bond distances (Å) and Te—O—H angles (°) in Te(OH)₆ (*mon.*). Standard deviations in parentheses.

	Te—O—H	O—H	O···H	O—O
Te ₁ —O ₁ —H ₁ ···O ₆	120(6)	1.07(11)	1.63(11)	2.681(7)
Te ₁ —O ₂ —H ₂ ···O ₄	123(6)	0.95(11)	1.99(11)	2.696(7)
Te ₁ —O ₃ —H ₃ ···O ₅	114(6)	1.12(11)	1.59(11)	2.694(7)
Te ₂ —O ₄ —H ₄ ···O ₃	—	—	—	2.687(7)
Te ₂ —O ₅ —H ₅ ···O ₂	117(4)	1.31(10)	1.44(9)	2.678(7)
Te ₂ —O ₆ —H ₆ ···O ₁	103(7)	0.98(11)	1.78(11)	2.728(7)

contain infinite chains, is somewhat different from that in the other compounds which contain discrete polyhedra.

A corresponding effect to that shown in Table 7 is also apparent in the similar iodine compounds, the I—OH bonds being 1.90, 1.90 and 1.89 Å in the neutral oxy-acids α -HIO₃, HI₃O₈ and H₅IO₆, respectively,²⁷ while, in a periodic salt like K₄[I₂O₈(OH)₂](H₂O)₈,²⁸ it is as long as 1.98 Å.

The observed Me—OH bond lengthening in the anions can crudely be considered to be a result of increasing electrostatic repulsion as the negative charge increases. A more accurate description of the effect ought, however, to be possible from a calculation of the energies of probable molecular orbitals. The highly symmetric coordination figure of telluric acid would certainly allow π -bond contributions to the Te—OH bonds. In the tellurates, which contain more localized π -bond character in the Te—O “double” bonds, the π -bond order in the Te—OH orbitals would probably be lower. The successive removal of protons from the tellurates would probably also cause the introduction of more electrons in the Te 5s, 5p and 5d orbital system. Electrons might therefore be placed in previously unoccupied non-bonding or anti-bonding orbitals, thus decreasing the π -bond order even for the Te—O “double” bonds. A discussion of likely molecular orbitals for Te(OH)₆ will be profitable after more reliable hydrogen positions have been obtained. A neutron diffraction study is therefore planned.

Although the precision in the hydrogen positions is rather low, those five hydrogen atoms which could be refined fit reasonably into the hydrogen bonding net (*cf.* Table 8). Each oxygen atom is connected to two others in neighbouring molecules (*cf.* Fig. 2), and the six independent O···H—O distances range from 2.678 Å to 2.728 Å, with a mean value of 2.694 Å. On the average, the hydrogen bonding seems to be of the same strength in Te(OH)₆ as in H₅IO₆,²⁹ which has two relatively short O···H—O interactions of about 2.60 Å, and three longer of about 2.78 Å. In B(OH)₃ the hydrogen bonds form layers through the structure,² and, as in Te(OH)₆, each oxygen takes part in two hydrogen bonds, with a slightly longer average O···H—O distance of 2.720 Å.

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