The Crystal Structure of a Potassium Tellurate(VI)

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The crystal structure of $K_4[Te_2O_6(OH)_4](H_2O)_x$ with $x = 7.3$ has been determined. The crystals belong to the monoclinic space group $C2/c$ and there are four formula units in the unit cell, which has the dimensions $a = 15.370$ Å, $b = 6.920_0$ Å, $c = 18.241$ Å, and $\beta = 116.00_9°$.

The positions of the tellurium atoms were deduced from Patterson projections, those of the potassium atoms from Fourier projections, and those of the oxygen atoms from three-dimensional Fourier syntheses. The structure was refined by complete three-dimensional least squares calculations based on two independent series of reflexions. The final R value obtained was 0.080.

The tellurate anion, $Te_2O_6(OH)_4^{4-}$, is dimeric as indicated by the formula. Each tellurium atom is surrounded by a slightly distorted octahedron of oxygen atoms, two such octahedra having one edge in common. The Te—O distances in the anion range from 1.861—1.894 Å (Te—O) to 1.985—2.029 Å (Te—OH, Te—O_{bridge}). The structure is held together by electrostatic interaction between the potassium and the tellurate ions. The presence of hydrogen bonding is, moreover, indicated by a number of short O—O distances.

A detailed comparison is made with the isoelectronic periodate ion $I_2O_6(OH)_2^{4-}$.

An investigation of the crystal structures of some tellurium(IV) compounds was commenced in connection with a series of investigations of tetravalent metal ions.[1] The study of the crystal structures of some hexavalent tellurium compounds was, moreover, of interest for the purposes of comparison of the co-ordination polyhedra associated with tellurium(IV) and with tellurium(VI).

Before the commencement of this investigation previous work on the crystal structures of the tellurates comprised preliminary studies of $Te(OH)_6$,[2-5] K_2TeO_4,[6] and Hg_3TeO_6[7] only. During the course of the investigation, however, three papers on the crystal structures of the potassium tellurates $KTeO(OH)_5H_2O$,[8] $KTeO_2(OH)_3$,[9] and $KTeO_3(OH)$[10] were published. Their structures all contain six-co-ordinated tellurium atoms, as isolated octahedral $TeO(OH)_5^-$ ions,[8] as infinite linear complexes $(TeO_2(OH)_3^-)_n$ built up from TeO_6 octahedra sharing corners,[9] or as infinite linear complexes $(TeO_3(OH)^-)_n$ built up from TeO_6 octahedra sharing edges.[10]

Crystals of potassium tellurates were prepared by dissolving telluric acid in water and adding a solution of potassium hydroxide. Owing to the conditions under which the solutions were mixed, different potassium tellurates were obtained either as the pure substances or as mixtures. A tellurate with a K:Te ratio of 2:1 was chosen for this structural investigation.

PREPARATION OF THE CRYSTALS

10 ml of a saturated solution of telluric acid (BDH) was diluted with 30 ml of water and heated to approximately 80°C under continuous stirring and dropwise addition of 15 ml of 3.6 M potassium hydroxide solution. The resulting solution was then evaporated slowly, colourless, rod-shaped crystals of $K_4[Te_2O_6(OH)_4](H_2O)_x$ ($x \simeq 7$) being formed.

The addition of potassium hydroxide solutions of concentrations greater than 3.6 M led to contamination of the product with another crystalline potassium tellurate. It was also essential that the telluric acid solution was heated and stirred, otherwise an opaque, white precipitate, amorphous to X-rays was obtained. If telluric acid was added to the potassium hydroxide solution, instead of *vice versa*, an oily concentrated solution was formed, which gradually solidified to give a glassy product together with a small number of crystals.

Analysis

Since the crystals of $K_4[Te_2O_6(OH)_4](H_2O)_x$ ($x \simeq 7$) are very soluble in water, it was difficult to remove potassium hydroxide by washing with aqueous solvents. In order to decrease the adsorption of mother liquor during drying of the crystals, a rather coarse crystalline sample was prepared for the analysis.

The amount of *tellurium* was determined gravimetrically according to a method devised by Salaria.[11]

The *potassium* content was determined with an atomic absorption spectrometer, using standard solutions of known potassium concentration.

The *water* content was determined by Hartwig-Bendig's modification of Penfield's method as described by Kolthoff and Sandell.[12]

A measure of the *density* of the crystals was obtained by flotation experiments using mixtures of $CHBr_3$ and CCl_4.

The analysis gave the following results:

	Found		Calculated for $K_4[Te_2O_6(OH)_4](H_2O)_7$	Calculated for $K_4[Te_2O_6(OH)_4](H_2O)_{7\cdot3}$
% TeO_3	51.9 *		50.05	49.67
% K_2O	26.71	26.66	26.85	26.65
% H_2O	24.0_1	24.0_4	23.09	23.68
Density (g/cm³)	2.75	2.76	2.67	2.69

INTENSITY DATA

Intensity data corresponding to the layers $h0l-h4l$ and $hk0-hk11$ of the reciprocal lattice was obtained using $CuK\alpha$ radiation and multiple film Weissenberg techniques, six films being registered for each layer line. The intensities were estimated visually by comparison with an intensity scale, prepared by making timed exposures of chosen reflexions from the actual crystals. Each reflexion estimated appeared, as a rule, on at least two of the six films with an intensity falling within the most reliable part of the scale.

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* mean from eight determinations ($\sigma = \pm 1.0$)

UNIT CELL AND SPACE GROUP

The crystals of $K_4[Te_2O_6(OH)_4](H_2O)_x$ ($x \simeq 7$) are monoclinic, approximate cell dimensions, as determined from rotation and Weissenberg photographs, being $a = 15.3$ Å, $b = 6.90$ Å, $c = 18.2$ Å, and $\beta = 116°$. In order to determine more accurate cell dimensions, Guinier powder photographs were taken, using KCl as an internal standard (CuKα_1 radiation, $\lambda = 1.54050$ Å, $a_{KCl} = 6.2919_4$ Å at 20°C[13]). The reflexions were indexed using the Algol programme Xalg Powder,[14] and the cell constants were refined with the same programme.

The cell dimensions and their standard deviations were found to be:

$$a = 15.3705 \pm 0.0015 \text{ Å}$$
$$b = 6.9200 \pm 0.0006 \text{ Å}$$
$$c = 18.2407 \pm 0.0009 \text{ Å}$$
$$\beta = 116.009° \pm 0.006°$$

The observed and calculated values of $\sin^2\theta$ are listed in Table 1.

The volume of the unit cell as calculated from these cell dimensions is 1744 Å³, and hence, with an experimental density of 2.75 g/cm³ there are $4.11 \simeq 4$ formula units of $K_4[Te_2O_6(OH)_4](H_2O)_x$ ($x \simeq 7$) in the unit cell.

The systematically absent reflexions are:

$$hkl \text{ with } h + k = 2n + 1$$
$$h0l \text{ with } l = 2n + 1,$$

which is in accordance with the monoclinic space groups No. 9 — Cc and No. 15 — $C2/c$.[15]

DETERMINATION OF F_o FROM THE OBSERVED INTENSITY MATERIAL

Needle-shaped crystals of suitable size with well-defined edges were used to obtain the intensity data, the needle-axis corresponding to the crystallographic b axis. The crystal used to register the Weissenberg photographs $hk0-hk11$ had, however, to be cut so that it was of almost the same diameter in all directions.

In order to obtain as accurate F_o data as possible, the intensities were corrected for absorption, polarization, and Lorentz' effects. Both crystals were measured accurately and their faces indexed, the relevant absorption correction then being performed on a SAAB D21 computer using a programme written by Abrahamsson.[16] Such an absorption correction is necessary, since tellurium absorbs CuKα radiation strongly, the linear absorption coefficient for $K_4[Te_2O_6(OH)_4](H_2O)_x$ ($x \simeq 7$) being 364 cm⁻¹. The correction factors for the actual crystals used varied within the range 3—30.

Correction for Lorentz' and polarization effects was performed by the programme "General data reduction" (Abrahamsson and Larsson).[16]

The F_o values were brought on to an absolute scale by comparison with F_c only after an approximate structure had been devised.

Table 1. Powder photographs of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$ $CuK\alpha_1$ radiation. $\lambda(CuK\alpha_1) = 1.54050$ Å.

h k l	$10^5\sin^2\theta$ calc	$10^5\sin^2\theta$ obs	F calc	I obs
0 0 2	883	878	267	m
2 0 −2	1208	1203	295	s
2 0 0	1244	1238	270	m
1 1 0	1550	1544	288	s
1 1 −2	1973	1969	307	vs
1 1 1	2000	1998	85	vvw
1 1 −3	2847	2844	190	m
1 1 2	2893	2892	103	vvw
2 0 −4	2938	2934	125	w
2 0 2	3046	3040	187	vw
0 0 4	3532	3531⎱	270	vs
3 1 −2	3542	3531⎰	294	
4 0 −2	4020	4008	142	vw
3 1 0	4037	4040	142	vw
1 1 −4	4163	4159	120	vw
1 1 3	4226	4209	193	w
4 0 −4	4831	4817⎱	341	s
3 1 −4	4812	4817⎰	356	
0 2 0	4956	4957	129	vw
0 2 1	5177	5182	264	m
2 2 −2	6163	6157	220	vw
3 1 2	6299	6296	211	vw
2 0 4	6614	6613	281	w
2 2 1	6880	6883	217	vw
4 0 −6	7408	7402	291	m
5 1 −3	7552	7550	140	vvw
4 0 2	7696	7703	310	m
5 1 −4	7949	7955	220	w
5 1 −1	8084	8084	238	w
1 1 −6	8119	8112	245	vw
1 1 5	8218	8201	204	w
5 1 −5	8787	8783	196	vvw
5 1 0	9012	9008	437	s
4 2 −1	9232	9228	308	w
4 2 −4	9786	9797	181	vw
3 1 −7	10029	10033	209	vvw
1 1 6	10876	10875	346	s
4 2 1	11070	11071	281	w
2 2 4	11570	11571	112	vvw
5 1 2	12193	12190⎱	166	w
4 0 4	12183	12190⎰	207	
3 1 −8	12652	12657⎱	183	vw
4 2 2	12652	12657⎰	163	
3 3 −1	13480	13480	361	m
1 1 7	13976	13971⎱	168	w
3 3 0	13949	13971⎰	273	
1 1 −8	13841	13841	242	vvw
1 3 3	14138	14140⎱	241	w
7 1 −2	14140	14140⎰	261	
4 2 −7	14314	14307	320	vw
4 2 3	14675	14686	156	vvw

Table 1. Continued.

h k l	$10^5 \sin^2\theta$ calc	$10^5 \sin^2\theta$ obs	F calc	I obs
3 3 1	14859	14850	208	vw
1 3 −5	15832	15838	221	vw
8 0 −4	16078	16062	295	vvw
7 1 0	16474	16474	172	vw
2 2 −8	16652	16665	164	vvw
8 0 −2	17105	17105	416	m
5 3 −3	17464	17460	311	w
7 1 −8	17735	17746	185	vvw
4 0 −10	17860	17852	397	vvw
2 0 −10	18725	18708	430	m
6 2 −8	19249	19248⎫	134	vw
3 1 −10	19221	19248⎭	257	
4 2 −9	19541	19535	242	vvw
0 4 0	19823	19814	219	vvw
4 2 5	20045	20039⎫	367	s
0 4 −1	20044	20039⎭	181	
2 4 0	21067	21061	138	vvw
8 2 −5	21182	21173	346	vw
6 2 3	22272	22262	159	vvw
7 1 −10	22466	22476	218	vvw
0 2 9	22839	22827⎫	194	vw
8 2 −7	22804	22827⎭	235	
9 1 −2	23170	23159	215	vvw
2 4 −5	24288	24299	250	vw
10 0 −4	25433	25429	212	vw
4 4 1	25938	25927⎫	92	vw
1 1 10	25924	25927⎭	175	
5 3 −9	26466	26444⎫	214	
7 1 4	26440	26444⎪	184	w
2 4 4	26437	26444⎪	125	
4 0 8	26456	26444⎭	270	
8 2 1	26913	26909	228	vw
6 2 5	28561	28557	225	vvw
3 3 7	29592	29580	243	w
6 2 −12	31395	31395⎫	169	w
1 3 9	31412	31395⎭	287	
1 5 −2	31708	31710	181	w
5 1 8	32332	32352	268	vw
3 1 10	33007	33004⎫	203	w
5 3 −11	32998	33004⎭	119	
7 3 −11	35405	35379	293	vw

THE POSITIONS OF THE TELLURIUM ATOMS

In order to obtain the positions of the tellurium atoms, the Patterson projections $P(uvp)$ and $P(upw)$ and the generalized Patterson function $P_1(uw)$ were calculated, using the Fourier programme written by Larsson[16] for the computer SAAB D21. The highest peaks in $P(uvp)$ (*cf.* Fig. 1), discounting that at the origin, are situated at $u = 0$, $v = 0.15$, at $u = 0.18$, $v = 0$ and at $u = 0.18$, $v = 0.15$, and are probably due to Te—Te vectors. Their positions

Fig. 1. Patterson projection $P(uvp)$ of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$. The distance between two thin contours or between one thin and one thick contour within the maxima corresponds to 1/10 of that between two thick contours. The final vectors Te—Te (▲) and Te—K (■) are indicated.
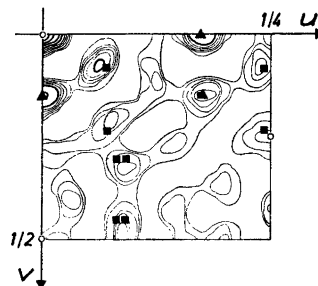
suggest an eight-fold tellurium position $C2/c$: $8(f)$ with $x_{Te} \simeq 0.090$ and $y_{Te} \simeq 0.075$ (x_{Te}, y_{Te}, and z_{Te} can arbitrarily be chosen within the limits $0 \leq x_{Te} \leq \frac{1}{4}$, $0 \leq y_{Te} \leq \frac{1}{4}$ and $0 \leq z_{Te} \leq \frac{1}{2}$). The z parameter of the tellurium atom was obtained from $P(upw)$ (*cf.* Fig. 2), which shows a large maximum on the line (0.18, w) at $w = 0.15$ and $w = 0.65$. Since the maximum $2x_{Te}, 2z_{Te}$ should fall on this line in $P(upw)$, z_{Te} is either $\simeq 0.075$ or $\simeq 0.325$.
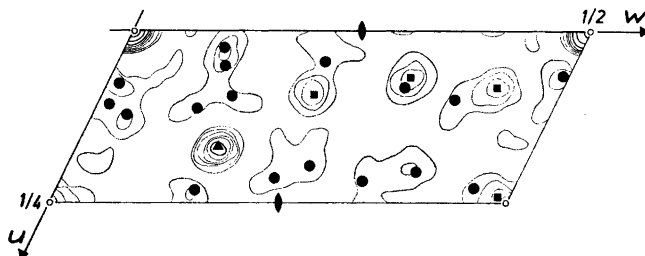
Fig. 2. Patterson projection $P(upw)$ of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$. The distance between two thin contours or between one thin and one thick contour within the maxima corresponds to 1/10 of that between two thick contours. The final vectors Te—Te (▲), Te—K (■), and Te—O (●) are indicated.

In order to decide, which of these two z_{Te} values is the correct one, the generalized Patterson function $P_1(uw)$ was calculated. Since the two types of interatomic vectors between atoms in $C2/c$: $8(f)$ containing $2x$ and $2z$ are $(2x,2y,2z)$ and $(2x,0,\frac{1}{2}+2z)$, $P_1(uw)$ was examined at the points $u = 0.18$, $w = 0.15$ and $u = 0.18$, $w = 0.65$. There are maxima at both points, but the former has a height of 213 arbitrary units, and the latter a height of 353 arbitrary units. This is possible only if $z \simeq 0.075$, which would give a ratio between the heights at the points $(0.18,0.15)$ and $(0.18,0.65)$ of $\cos(2\pi 2y_{Te})$, *i.e.* 0.588, which is in good agreement with the observed value of $213/353 = 0.603$.

We thus conclude:

8 Te in $C2/c$: $8(f)$ with $x_{Te} \simeq 0.090$ $y_{Te} \simeq 0.075$ $z_{Te} \simeq 0.075$

POSITIONS OF THE POTASSIUM AND OXYGEN ATOMS

The positions of the potassium atoms were deduced from Fourier projections $\varrho(xpz)$ (*cf.* Fig. 3) and $\varrho(xyp)$, the signs of the structure factors being
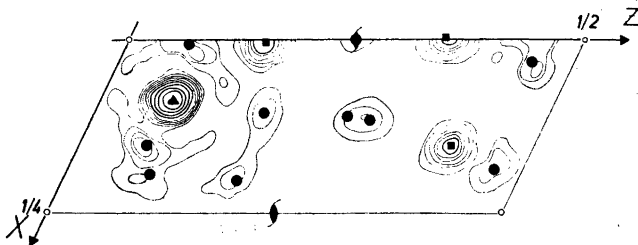
Fig. 3. Electron density projection $\varrho(xpz)$ of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$. The distance between two thin contours or between one thin and one thick contour within the maxima corresponds to 1/10 of that between two thick contours. The final positions of Te (▲). K (■), and O (●) are indicated.

obtained from the positions of the tellurium atoms. All the potassium atoms could be located as being situated in two eight-fold positions:

$$8 \text{ K}_1 \text{ in } C2/c: 8(f) \text{ with } x \simeq 0.155 \quad y \simeq 0.158 \quad z \simeq 0.408$$
$$8 \text{ K}_2 \text{ in } C2/c: 8(f) \text{ with } x \simeq 0.498 \quad y \simeq 0.131 \quad z \simeq 0.348$$

The positions of the oxygen atoms were obtained from "three-dimensional" electron density calculations, based on the $h0l-h4l$ reflexions, using the signs obtained by considering the contributions to the structure factors from tellurium and potassium. Eight oxygen positions $8(f)$, O_1-O_8, could be identified. There was, in addition, some indication as to a four-fold oxygen position, O_9. The preliminary parameters of these nine positions were thus as follows:

8 O_1 in $C2/c$: 8(f) with	$x \simeq 0.125$	$y \simeq 0.029$	$z \simeq 0.194$
8 O_2	—»—	$x \simeq 0.179$	$y \simeq 0.244$	$z \simeq 0.080$
8 O_3	—»—	$x \simeq 0.476$	$y \simeq 0.420$	$z \simeq 0.045$
8 O_4	—»—	$x \simeq 0.007$	$y \simeq 0.294$	$z \simeq 0.048$
8 O_5	—»—	$x \simeq 0.430$	$y \simeq 0.306$	$z \simeq 0.415$
8 O_6	—»—	$x \simeq 0.291$	$y \simeq 0.047$	$z \simeq 0.308$
8 O_7	—»—	$x \simeq 0.383$	$y \simeq 0.322$	$z \simeq 0.196$
8 O_8	—»—	$x \simeq 0.309$	$y \simeq 0.037$	$z \simeq 0.031$
4 O_9 in $C2/c$: 4(c) with	$x = 0$	$y \simeq 0.267$	$z = \frac{1}{4}$

REFINEMENT OF THE STRUCTURE

In order to refine the structural parameters, several different least squares calculations were performed. At first the reflexions from the [010] series were refined with Åsbrink and Bränden's programme [17] for the computer FACIT EDB. The co-ordinates and the corresponding isotropic temperature factors obtained from these calculations have been published in a preliminary note.[18]

The refinement was then continued on the computer SAAB D21 at Göteborgs Datacentral. The F_o of the layer lines were scaled separately for each photograph in the two series [010] and [001], using the least squares programme Xalg-LS written in Algol by Wengelin.[19] The R value of the b axis series then dropped to 0.127, and that of the c axis series to 0.094 ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$, the observed F_o only being taken into account).

Since there are about 1100 independent reflexions in each series it ought to be possible to extend the refinement to include anisotropic temperature factors. This was also performed using the programme SFLS written by Aleby[16] and Abrahamsson.[16] Hereby, the separate refinement of the two series of reflexions finally gave $R = 0.106$ (b axis series) and 0.083 (c axis series).

To check the refinement results, three-dimensional $F_o - F_c$ Fourier syntheses were calculated for both axes. They showed no peaks denser than about 1 electron/Å^3, except for one well-defined peak at $x \simeq 0.144$, $y \simeq 0.356$ and $z \simeq 0.277$, which had a maximum density of 2.3 electrons/Å^3 in both syntheses. This would correspond to a position O_{10} in $C2/c$: 8(f) filled to 25—30 % with oxygen atoms. The $O_9 - O_{10}$ distance would, however, be very short, about 1.6 Å, so that simultaneous existence of O_9 and O_{10} was not deemed possible. On the other hand the presence of a small minimum corresponding to the O_9 position in the difference syntheses might indicate that O_9 may not be completely filled either. The appearance of the difference syntheses might also suggest that the correct space group should be Cc and not $C2/c$ as assumed earlier. The calculations were thus continued with the eight-fold positions for Te, $K_1 - K_2$, and $O_1 - O_8$ each occupying two positions Cc: 4(a). A least squares refinement of this structure proposition, including O_9 as a position Cc: 4(a), gave, however, no significant deviation from the arrangement proposed when $C2/c$ was assumed to be the correct space group.

The void in the structure at the points (000, $\frac{1}{2}\frac{1}{2}0$) $\pm$ (0,0.27,$\frac{1}{4}$) may thus be filled by an oxygen atom distributed statistically over the positions $C2/c$: 4(c) (O_9) and $C2/c$: 8(f) (O_{10}). Additional support of this hypothesis is afforded by the analysis, which indicates a water content of more than 4.5 but less than 5.0 molecules of water per tellurium atom.

In the final stages of refinement, the data from both series were combined, a mean value being taken for those structure factors occurring in both series. The calculations were then performed on the computer CD 3600 using the programme LALS, written by Zalkin and modified in Uppsala.[20] In order to confirm the difference syntheses results, four calculations, differing only in the occupation numbers (N) for O_9 and O_{10}, were first performed, namely with $(N_9;N_{10}) = (1;0)$, (0.7;0.3), (0.5;0.5), and (0;1). The R value (cf. Fig. 4) as well as the temperature factors of O_9 and O_{10} suggest that the occupation numbers of O_9 and O_{10} are 0.70 and 0.30, respectively. The complete formula of this tellurate thus ought to be $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$.

The whole structure, including O_9 and O_{10} with occupation numbers 0.70 and 0.30, was then refined, using atomic form factors calculated from Slater-Dirac functions by Cromer and Waber[21] and the weighting function recommended by Cruickshank ($w = (a + F_o + cF_o^2 + dF_o^3)^{-1}$ with $a = 25$, $c = 0.0055$, and $d = 0$). After five cycles of refinement all parameter shifts were less

Table 2. Calculated and observed structure factors for $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$. (The columns are l, F_c and $|F_o|$, respectively).

0 0 L

l	Fc	Fo
0	1248	-
2	267	248
4	-270	286
6	-370	355
8	-354	361
10	55	59
12	251	221
14	144	145
16	-39	37
18	-151	155
20	-66	64

2 0 L

l	Fc	Fo
-20	-43	44
-18	77	86
-16	145	138
-14	169	152
-12	-22	23
-10	-430	378
-8	-223	192
-6	-5	18
-4	125	118
-2	295	259
0	270	249
2	-187	242
4	-281	338
6	-101	118
8	58	75
10	124	159
12	173	205
14	54	74
16	-132	179
18	-131	161

4 0 L

l	Fc	Fo
-22	-82	74
-20	46	46
-18	133	134
-16	116	103
-14	68	65
-12	-177	162
-10	-397	355
-8	35	40
-6	291	242
-4	341	287
-2	142	137
0	-186	186
2	-310	313
4	-207	238
6	219	252
8	270	303
10	11	25
12	-57	60
14	-137	180
16	-125	159
18	17	14

6 0 L

l	Fc	Fo
-22	45	49
-20	135	147
-18	56	59
-16	-68	70
-14	-104	104
-12	-75	79
-10	-110	89
-8	84	70
-6	133	116
-4	-116	118
-2	-20	34
0	-12	39
2	-109	117
4	60	73
6	211	225
8	196	206
10	-53	56
12	-114	135
14	-101	126
16	-52	72

8 0 L

l	Fc	Fo
-22	72	74
-20	132	153
-18	8	25
-16	-113	116
-14	-104	104
-12	-35	39
-10	149	123
-8	248	196
-6	80	69
-4	-295	276
-2	-416	383
0	-17	27
2	131	134
4	207	210
6	152	159

10 0 L

l	Fc	Fo
-22	34	43
-20	-26	22
-18	-129	132
-16	-84	83
-14	62	57
-12	161	155
-10	105	90
-8	19	26
-6	-87	77
-4	-212	204
-2	-114	114
0	217	211
2	125	130
4	78	87
6	1	20
8	-164	177
10	-115	136
12	32	34

12 0 L

l	Fc	Fo
-22	-36	36
-20	-97	98
-18	-93	105
-16	4	22
-14	211	198
-12	205	189
-10	-101	99
-8	-174	158
-6	-234	215
-4	-89	77
-2	202	210
0	224	222
2	22	31
4	-140	153
6	-127	143
8	-29	36
10	46	50

14 0 L

l	Fc	Fo
-20	-19	25
-18	46	51
-16	102	115
-14	111	127
-12	-14	22
-10	-149	155
-8	-49	70
-6	-6	24
-4	55	59
-2	136	141
0	72	79
2	-90	99
4	-155	165
6	-74	75

16 0 L

l	Fc	Fo
-20	47	43
-18	76	89
-16	71	97
-14	-7	20
-12	-132	157
-10	-139	148
-8	24	27
-6	167	172
-4	120	110
-2	18	24
0	-77	80
2	-151	151

18 0 L

l	Fc	Fo
-16	-33	35
-14	-56	56
-12	-68	72
-10	-5	17
-8	91	99
-6	96	99
-4	6	14
-2	-84	78

1 1 L

l	Fc	Fo
-21	-21	17
-20	-91	96
-19	42	49
-18	-32	37
-17	66	78
-16	88	99
-15	26	27
-14	111	105
-13	28	19
-12	71	71
-11	-74	76
-10	26	23
-9	-143	125
-8	-242	224
-7	-106	97
-6	-245	236
-5	191	196
-4	120	131
-3	190	219
-2	307	274
-1	6	16
0	288	252
1	-85	102
2	103	111
3	-193	220
4	-226	260
5	-204	233
6	-346	363
7	168	178
8	-167	177
9	192	212
10	175	179
11	53	56
12	172	156
13	-61	59
14	31	45
15	-71	77
16	-45	47
17	-52	55
18	-122	123
19	14	12
20	-67	52

3 1 L

l	Fc	Fo
-22	-67	56
-21	34	32
-20	-48	47
-19	100	113
-18	105	113
-17	65	71
-16	162	157
-15	-119	112
-14	16	25
-13	-106	107
-12	-156	150
-11	-56	54
-10	-257	221
-9	60	61
-8	-183	157
-7	209	190
-6	119	121
-5	128	119
-4	356	321
-3	-115	108
-2	294	273
-1	-206	200
0	-142	136
1	-61	76
2	-211	210
3	125	131
4	-16	37
5	-51	63
6	-9	20
7	70	84
8	169	192
9	59	60
10	203	213
11	-63	67
12	-20	29
13	-17	28
14	-107	108
15	4	25
16	-98	99
17	-17	21
18	-64	60

5 1 L

l	Fc	Fo
-21	33	37
-20	57	57
-19	24	29
-18	129	131
-17	-6	17
-16	60	60
-15	-78	71
-14	-62	60
-13	-56	51
-12	-166	166
-11	29	35
-10	-117	102
-9	25	29
-8	64	65
-7	94	89
-6	56	58
-5	196	190
-4	220	224
-3	-140	139
-2	11	17
-1	-238	240
0	-437	420
1	-82	85
2	-166	171
3	56	67
4	25	48
5	122	134
6	131	137
7	119	127
8	268	247
9	-20	26
10	71	77
11	-126	125
12	-121	131
13	-53	52
14	-145	139
15	61	55
16	-52	50
17	54	46

7 1 L

l	Fc	Fo
-22	69	71
-21	10	18
-20	94	96
-19	-48	46
-18	23	30
-17	-75	75
-16	-62	58
-15	-42	42
-14	-183	175
-13	86	75
-12	-92	88
-11	172	159
-10	218	203
-9	22	23
-8	185	158
-7	-108	98
-6	76	78
-5	-27	37
-4	-16	20
-3	-144	146
-2	-261	258
-1	20	20
0	-172	168
1	142	141
2	86	88
3	34	40
4	184	187
5	-45	53
6	131	132
7	-52	53
8	-4	25
9	-42	50
10	-70	80
11	-13	22
12	-118	121
13	23	23
14	-63	61
15	74	63

9 1 L

l	Fc	Fo
-22	76	72
-21	-41	39
-20	-20	34
-19	-20	24
-18	-52	51
-17	-23	32
-16	-79	76
-15	-25	25
-14	-107	105
-13	103	95
-12	47	46
-11	75	74
-10	204	191
-9	-11	23
-8	72	77
-7	-130	117
-6	-71	72
-5	-68	73
-4	-203	207
-3	-23	23
-2	-215	201
-1	110	114
0	-38	49
1	179	172
2	195	190
3	3	23
4	234	225
5	-188	177
6	28	25
7	-92	98
8	-114	114
9	-7	23
10	-86	98
11	74	80
12	-40	43
13	83	66

11 1 L

l	Fc	Fo
-22	11	20
-21	-59	68
-20	-94	98
-19	17	23
-18	-89	96
-17	84	85
-16	-19	27
-15	45	46
-14	103	94
-13	24	33
-12	176	164
-11	-101	95
-10	122	115
-9	-144	134
-8	-107	106
-7	7	27
-6	-211	193
-5	152	134
-4	-83	75
-3	103	98
-2	30	36
-1	-2	26
0	85	87
1	-70	67
2	106	108
3	-77	82
4	-7	22
5	-78	86
6	-120	119
7	41	51
8	-89	93
9	62	70
10	15	17
11	20	14

13 1 L

l	Fc	Fo
-22	-56	50
-21	-12	17
-20	-95	109
-19	34	37
-18	-24	24
-17	45	48
-16	67	68
-15	-19	27
-14	96	94
-13	-16	28
-12	78	82
-11	-49	51
-10	-36	33
-9	-63	63
-8	-146	142
-7	44	46
-6	-116	114
-5	-52	51
-4	60	61
-3	49	48
-2	169	158
-1	-21	29
0	86	96
1	-78	83
2	9	21
3	-74	77
4	-103	110
5	-30	39
6	-138	146
7	68	74
8	-1	15

15 1 L

l	Fc	Fo
-20	-21	15
-19	45	57
-18	83	95
-17	-12	22
-16	98	115
-15	-65	75
-14	22	25
-13	-53	66
-12	-87	93
-11	5	26
-10	-96	99
-9	32	41
-8	-46	51
-7	43	48
-6	47	49
-5	61	58
-4	135	127
-3	-42	47
-2	54	55
-1	47	52
0	-83	94
1	0	18
2	-87	79
3	-9	14
4	-65	65
5	14	13

17 1 L

l	Fc	Fo
-18	57	68
-17	-17	17
-16	30	44
-15	-58	74
-14	-67	76
-13	-29	34
-12	-90	102
-11	5	21
-10	-6	21
-9	47	46
-8	49	53
-7	54	65
-6	114	122
-5	21	36
-4	94	97
-3	-71	73
-2	-53	60
-1	-70	72
0	-123	117

19 1 L

l	Fc	Fo
-14	-65	82
-13	46	49
-12	-12	16
-11	43	43
-10	76	70
-9	4	12
-8	73	68
-7	-23	21
-6	28	28

0 2 L

l	Fc	Fo
0	129	133
1	-264	232
2	83	93
3	-344	332
4	13	22
5	9	24
6	12	27
7	83	86
8	-106	102
9	194	181
10	-18	20
11	147	138
12	41	41
13	-68	60
14	36	45
15	-165	162
16	5	26
17	-79	76
18	-49	50
19	24	28
20	-54	45

2 2 L

l	Fc	Fo
-21	40	36
-20	-44	44
-19	91	94
-18	13	21
-17	60	68
-16	50	53
-15	21	26
-14	64	65
-13	-111	110
-12	51	53
-11	-183	177
-10	-106	104
-9	66	66
-8	-164	151
-7	-55	49
-6	-55	49
-5	-87	108
-4	-76	98
-3	129	140
-2	-62	68
-1	-62	68
0	66	65
1	-217	228
2	-85	96
3	-46	49
4	115	110
5	90	102
6	-123	104
7	167	178
8	74	74
9	99	109
10	147	152
11	54	58
12	57	61
13	-74	76
14	-1	27
15	-162	152
16	-82	80
17	-23	19
18	-53	54

4 2 L

l	Fc	Fo
-21	93	92
-20	18	22
-19	72	75
-18	64	69
-17	21	25
-16	49	47
-15	-63	64
-14	8	25
-13	-190	153
-12	-94	87
-11	-62	64
-10	-80	79
-9	242	228
-8	-33	33
-7	320	265
-6	117	115
-5	-5	15
-4	181	182
-3	-263	264
-2	92	89
-1	-308	279
0	-71	70
1	-281	284
2	-163	164
3	156	160
4	-115	123
5	367	362
6	-3	52
7	108	109
8	109	109
9	-100	103
10	81	90
11	-120	129
12	-25	34
13	-79	84
14	-77	80
15	5	25
16	-75	67
17	94	81

6 2 L

l	Fc	Fo
-22	15	19
-21	54	67
-20	68	80
-19	-78	84
-18	75	77
-17	-131	126
-16	-9	25
-15	-31	37
-14	-99	87
-13	30	27
-12	-169	145
-11	136	128
-10	-36	36
-9	223	199
-8	134	124
-7	24	24
-6	175	168
-5	-203	185
-4	50	48
-3	21	26
-2	-178	164
-1	-57	60
0	-205	210
1	-17	19
2	-17	18
3	159	161
4	123	135
5	225	228
6	198	190
7	-37	38
8	76	76
9	-164	165
10	-39	49
11	-91	94
12	-84	87
13	-14	23
14	-53	51
15	63	62
16	0	15

8 2 L

l	Fc	Fo
-22	55	57
-21	-2	16
-20	41	44
-19	-129	139
-18	-29	24
-17	-119	130
-16	-79	92
-15	55	55
-14	-38	36
-13	151	132
-12	33	33
-11	187	174
-10	121	121
-9	97	98
-8	99	102
-7	-235	210
-6	-39	37
-5	-346	315
-4	-115	116
-3	-74	70
-2	-150	148
-1	222	224
0	-89	89
1	228	216
2	66	70
3	41	44
4	59	62
5	-55	57
6	84	82
7	-211	203
8	-22	24
9	-102	110
10	-69	76
11	112	118
12	-37	36
13	108	86
14	-4	18

10 2 L

l	Fc	Fo
-22	31	37
-21	-77	65
-20	-29	26
-19	-61	77
-18	-66	71
-17	54	45
-16	-49	60
-15	-5	26
-14	132	135
-13	88	89
-12	107	102
-11	-46	53
-10	106	104
-9	-67	74
-8	-2	25
-7	-68	79
-6	-40	48
-5	-106	106
-4	-134	129
-3	140	141
-2	-22	27
-1	163	152
0	82	83
1	-11	23
2	88	82
3	-60	59
4	51	52
5	-108	111
6	-37	34
7	-120	122
8	-84	91
9	10	20
10	-30	40
11	120	98

12 2 L

l	Fc	Fo
-21	-48	60
-20	-43	50
-19	-16	25
-18	-23	25
-17	85	90
-16	18	26
-15	120	125
-14	52	59
-13	-29	26
-12	53	64
-11	-166	168
-10	-2	27
-9	-98	91
-8	-66	61
-7	28	50
-6	-62	63
-5	106	96
-4	-65	60
-3	205	189
-2	45	43
-1	38	39
0	65	58
1	-172	165
2	30	34
3	-129	130
4	-12	20
5	-15	20
6	-69	73
7	60	72
8	31	35
9	81	71

This page consists of a dense multi-column crystallographic structure-factor listing (columns of h, F_o, F_c grouped under reflection indices). The tabulated numerical data are reproduced below by column groups.

14 2 L

h	Fo	Fc
-21	46	39
-20	-29	39
-19	41	50
-18	-1	<22
-17	26	38
-16	49	61
-15	3	<25
-14	19	102
-13	-99	114
-12	4	<27
-11	-66	65
-10	-52	53
-9	38	43
-8	-63	63
-7	71	65
-6	-10	<23
-5	59	54
-4	86	86
-3	37	42
-2	67	70
-1	-31	30
0	-8	28
1	-125	129
2	-71	72
3	-56	57
4	-89	99
5	61	66
6	-13	<15

16 2 L

h	Fo	Fc
-19	31	33
-18	39	46
-17	-30	28
-16	16	22
-15	-69	78
-14	-16	<20
-13	-96	120
-12	-57	70
-11	-5	<22
-10	-38	44
-9	140	147
-8	15	<25
-7	98	108
-6	59	66
-5	-10	<20
-4	59	63
-3	-80	83
-2	10	<18
-1	-85	92
0	-48	48
1	-25	33
2	-46	45
3	69	50

18 2 L

h	Fo	Fc
-15	-29	33
-14	-52	54
-13	-9	<15
-12	-33	41
-11	48	46
-10	17	22
-9	78	82
-8	55	61
-7	2	<15
-6	49	50
-5	-80	68
-4	-13	16
-3	-56	43

1 3 L

h	Fo	Fc
-20	-15	17
-19	84	82
-18	-13	<19
-17	135	134
-16	7	<25
-15	53	53
-14	52	53
-13	-54	58
-12	49	46
-11	-115	110
-10	-83	93
-9	-188	189
-8	-10	17
-7	-17	21
-6	59	67
-5	221	217
-4	13	18
-3	118	118
-2	31	36
-1	-57	57
0	29	28
1	-163	178
2	-99	109
3	-241	262
4	-38	48
5	-77	85
6	46	60
7	177	184
8	39	39
9	287	254
10	-71	70

(column 2)

h	Fo	Fc
11	61	61
12	-34	32
13	-96	90
14	13	<25
15	-120	115
16	6	<25
17	-90	94
18	19	26
19	25	20

3 3 L

h	Fo	Fc
-21	42	32
-20	13	<22
-19	127	123
-18	-33	33
-17	59	62
-16	-27	28
-15	-97	95
-14	25	<26
-13	-138	121
-12	78	68
-11	-79	81
-10	-37	47
-9	56	64
-8	-78	84
-7	246	243
-6	-64	71
-5	210	210
-4	-119	126
-3	-44	53
-2	131	141
-1	-361	345
0	273	260
1	-208	207
2	5	<20
3	26	29
4	-61	72
5	147	167
6	-60	70
7	243	247
8	-12	<23
9	102	103
10	36	37
11	-165	159
12	24	30
13	-141	136
14	15	<27
15	-45	49
16	-44	42
17	34	35

5 3 L

h	Fo	Fc
-21	72	65
-20	41	48
-19	50	63
-18	35	40
-17	-78	76
-16	-61	60
-15	-148	145
-14	14	<27
-13	-88	85
-12	-58	55
-11	119	117
-10	-89	93
-9	214	214
-8	47	52
-7	122	114
-6	43	44
-5	-26	33
-4	-48	52
-3	-311	308
-2	-78	81
-1	-376	365
0	18	20
1	110	109
2	8	<21
3	226	230
4	-12	<23
5	193	204
6	23	26
7	95	98
8	-24	31
9	-131	126
10	-39	45
11	-167	172
12	37	41
13	-34	40
14	43	46
15	83	77
16	-3	<17

7 3 L

h	Fo	Fc
-21	6	<24
-20	11	<24
-19	-85	91
-18	-9	<26
-17	-115	124
-16	0	<27
-15	-65	73
-14	33	42
-13	134	126
-12	-38	32
-11	293	267

(column 3)

h	Fo	Fc
-10	-56	63
-9	60	58
-8	33	35
-7	-105	105
-6	68	69
-5	-202	192
-4	64	65
-3	-223	231
-2	-31	37
-1	26	36
0	-7	<27
1	214	207
2	-34	28
3	129	126
4	-29	27
5	-40	42
6	87	84
7	-135	133
8	11	<25
9	-102	107
10	-69	71
11	-37	37
12	-13	<21
13	78	71
14	-6	<18

9 3 L

h	Fo	Fc
-21	-92	96
-20	9	<18
-19	-88	95
-18	-32	38
-17	-28	35
-16	-32	37
-15	58	65
-14	30	33
-13	166	151
-12	64	54
-11	116	115
-10	-34	48
-9	-105	114
-8	-41	45
-7	-159	154
-6	-64	74
-5	-83	94
-4	-72	78
-3	33	46
-2	60	65
-1	152	157
0	106	101
1	172	161
2	2	<25
3	-6	<25
4	-36	36
5	-174	177
6	-8	<25
7	-134	137
8	9	<24
9	-8	<22
10	-2	<20
11	66	60
12	22	18

11 3 L

h	Fo	Fc
-21	-89	93
-20	21	23
-19	-4	<20
-18	22	<21
-17	97	84
-16	5	<25
-15	97	105
-14	-4	<25
-13	58	58
-12	5	28
-11	-55	56
-10	-21	<28
-9	-173	168
-8	30	31
-7	-87	87
-6	29	<29
-5	90	86
-4	-74	70
-3	134	127
-2	-12	<26
-1	86	86
0	58	54
1	4	<24
2	3	<24
3	-143	146
4	1	<23
5	-142	148
6	-34	32
7	-2	<20
8	-34	45
9	105	93

13 3 L

h	Fo	Fc
-20	4	<20
-19	71	81
-18	-2	<23
-17	33	42
-16	22	<25
-15	21	24
-14	20	27

(column 4)

h	Fo	Fc
-13	-50	50
-12	-25	34
-11	-115	116
-10	-52	57
-9	-86	86
-8	1	<26
-7	73	72
-6	51	58
-5	182	163
-4	22	20
-3	121	112
-2	11	<24
-1	-63	66
0	7	<24
1	-123	119
2	-37	42
3	-123	125
4	-2	<18
5	-29	33
6	30	35
7	111	85

15 3 L

h	Fo	Fc
-19	80	73
-18	-17	24
-17	11	<18
-16	-11	<20
-15	-94	107
-14	42	55
-13	-119	132
-12	15	<23
-11	-28	40
-10	-30	31
-9	62	69
-8	-16	28
-7	134	130
-6	-24	34
-5	98	100
-4	-1	<20
-3	-60	65
-2	24	28
-1	-136	142
0	21	22
1	-49	49
2	-21	21
3	21	23

17 3 L

h	Fo	Fc
-15	-99	101
-14	0	<18
-13	-24	29
-12	-7	<19
-11	86	97
-10	0	19
-9	84	81
-8	24	32
-7	53	58
-6	2	<16
-5	-37	35
-4	-22	22
-3	-123	113
-2	0	<14
-1	-65	45

0 4 L

h	Fo	Fc
0	-219	203
1	-182	158
2	-53	61
3	-165	184
4	126	135
5	-134	146
6	86	90
7	-25	36
8	191	177
9	65	69
10	22	27
11	132	122
12	-94	92
13	19	<27
14	-84	85
15	-84	85
16	26	34
17	-125	115
18	36	31

2 4 L

h	Fo	Fc
-19	96	72
-18	-22	20
-17	89	81
-16	-27	27
-15	25	<25
-14	-52	46
-13	-119	113
-12	23	22
-11	-216	191
-10	102	108
-9	-44	49
-8	20	25
-7	127	139
-6	-61	68
-5	250	239
-4	0	<16
-3	117	125

(column 5)

h	Fo	Fc
-2	-54	53
-1	-169	162
0	-138	128
1	-294	276
2	10	<18
3	-120	138
4	125	142
5	122	143
6	21	22
7	258	251
8	8	<25
9	154	146
10	-15	<24
11	-38	36
12	-80	81
13	-131	130
14	-35	43
15	-99	105
16	67	67
17	-3	<20

4 4 L

h	Fo	Fc
-20	-23	18
-19	62	54
-18	-36	32
-17	-27	32
-16	-37	37
-15	-81	77
-14	-47	45
-13	-82	84
-12	64	68
-11	7	<25
-10	166	154
-9	157	152
-8	55	54
-7	104	115
-6	-131	141
-5	38	50
-4	-81	83
-3	-142	150
-2	-44	44
-1	-222	239
0	34	47
1	-92	104
2	168	163
3	18	28
4	89	99
5	156	163
6	-149	145
7	123	121
8	-120	111
9	-26	29
10	-14	<25
11	-79	86
12	28	28
13	-87	88
14	46	51
15	-1	<22
16	51	40

6 4 L

h	Fo	Fc
-20	-56	44
-19	-4	<17
-18	-23	20
-17	-92	79
-16	42	44
-15	-143	115
-14	24	<26
-13	-12	<27
-12	29	<28
-11	124	125
-10	26	<28
-9	193	178
-8	-75	80
-7	62	68
-6	-99	110
-5	-159	158
-4	70	74
-3	-171	185
-2	46	56
-1	-70	77
0	8	<24
1	126	131
2	59	62
3	184	179
4	4	<24
5	34	41
6	-87	87
7	-78	78
8	-46	45
9	-142	150
10	34	33
11	-48	47
12	34	29
13	53	48
14	20	28

8 4 L

h	Fo	Fc
-20	-47	38
-19	-85	62
-18	4	<21
-17	-48	42
-16	80	75

(column 6)

h	Fo	Fc
-15	28	29
-14	39	35
-13	91	81
-12	11	<26
-11	97	94
-10	-34	47
-9	-24	26
-8	-107	98
-7	-88	89
-6	-48	50
-5	-99	105
-4	134	138
-3	-57	63
-2	92	97
-1	88	94
0	-47	51
1	85	91
2	-59	59
3	45	45
4	-58	61
5	-38	37
6	-60	60
7	-108	115
8	35	38
9	-63	62
10	77	77
11	38	36
12	8	<18

10 4 L

h	Fo	Fc
-20	1	<20
-19	-68	56
-18	40	33
-17	18	<23
-16	28	28
-15	100	83
-14	-50	41
-13	122	98
-12	-40	33
-11	36	<27
-10	5	<27
-9	-122	122
-8	-19	<30
-7	-116	108
-6	4	<30
-5	-14	25
-4	87	87
-3	112	116
-2	47	42
-1	181	168
0	-31	38
1	0	<27
2	-22	27
3	-111	114
4	-28	25
5	-145	150
6	-44	47
7	-79	86
8	34	36
9	66	59
10	55	47

12 4 L

h	Fo	Fc
-19	33	24
-18	48	42
-17	60	56
-16	24	<24
-15	24	<26
-14	-85	80
-13	-31	<28
-12	-69	63
-11	-120	118
-10	3	<31
-9	-67	61
-8	45	48
-7	-15	28
-6	58	57
-5	107	96
-4	47	40
-3	146	139
-2	-60	65
-1	46	42
0	-109	115
1	-58	61
2	-10	<22
3	-126	131
4	-17	<20
5	111	116
6	93	94
7	114	106
8	-72	72
9	12	<28
10	-147	151
11	-62	59
12	-25	-
13	-93	-
14	74	-
15	-52	-

14 4 L

h	Fo	Fc
-18	-7	<18
-17	63	50
-16	-35	<20
-15	-14	<22
-14	-42	40
-13	-110	94
-12	11	<23
-11	-120	103
-10	65	62
-9	13	<25
-8	34	75

(column 7)

h	Fo	Fc
-7	123	111
-6	-5	<24
-5	136	113
-4	-3	<22
-3	26	30
-2	-56	58
-1	-110	110
0	-52	62
1	-124	124
2	39	41
3	-42	42
4	61	49

16 4 L

h	Fo	Fc
-15	-82	59
-14	-15	<15
-13	-58	47
-12	54	48
-11	16	<16
-10	64	55
-9	82	72
-8	-30	32
-7	95	84
-6	-78	74
-5	25	<20
-4	-23	21
-3	-59	56
-2	0	<17
-1	-76	57

1 5 L

h	Fo	Fc
-17	103	-
-16	-77	-
-15	74	-
-14	-96	-
-13	-58	-
-12	-58	-
-11	-140	142
-10	50	62
-9	-62	60
-8	159	150
-7	83	99
-6	165	156
-5	110	116
-4	-56	71
-3	52	62
-2	-181	185
-1	-67	63
0	-121	116
1	-211	201
2	-64	65
3	-92	98
4	62	75
5	105	111
6	180	175
7	95	92
8	46	58
9	62	62
10	-84	91
11	27	-
12	-101	-
13	-48	-
14	-31	-
15	-55	-
16	30	-

3 5 L

h	Fo	Fc
-18	-58	-
-17	5	-
-16	-136	-
-15	-12	-
-14	-36	-
-13	-51	-
-12	49	-
-11	-57	64
-10	102	108
-9	67	60
-8	143	140
-7	98	99
-6	13	<24
-5	36	50
-4	-162	177
-3	21	24
-2	-166	166
-1	-78	87
0	-17	<20
1	-159	161
2	122	127
3	-7	<18
4	104	109
5	111	116
6	93	94
7	114	106
8	-72	72
9	12	<28
10	-147	151
11	-62	59
12	-25	-
13	-93	-
14	74	-
15	-52	-

(column 8)

5 5 L

h	Fo	Fc
-18	-114	-
-17	-73	-
-16	-81	-
-15	-56	-
-14	85	-
-13	-23	-
-12	164	-
-11	49	57
-10	65	67
-9	143	144
-8	-25	35
-7	-24	25
-6	-101	119
-5	-118	121
-4	-173	179
-3	-98	103
-2	29	<20
-1	-31	38
0	152	157
1	59	60
2	95	92
3	76	75
4	-27	25
5	75	58
6	-82	89
7	6	<26
8	-101	101
9	-72	79
10	-43	41
11	-11	-
12	79	-
13	-8	-

7 5 L

h	Fo	Fc
-18	-52	-
-17	-26	-
-16	35	-
-15	-7	-
-14	9	-
-13	9	-
-12	81	-
-11	60	76
-10	-73	88
-9	94	111
-8	-109	119
-7	32	<30
-6	-34	35
-5	-94	108
-4	14	33
-3	-126	132
-2	142	143
-1	-19	<20
0	142	136
1	32	38
2	-38	40
3	92	87
4	-135	130
5	55	59
6	-94	95
7	-99	102
8	-18	<25
9	-105	107
10	50	56
11	-3	-

9 5 L

h	Fo	Fc
-18	49	-
-17	-32	-
-16	81	-
-15	64	-
-14	54	-
-13	101	-
-12	-91	-
-11	32	<35
-10	-135	148
-9	-34	41
-8	-48	54
-7	73	79
-6	95	95
-5	-69	69
-4	113	120
-3	66	67
-2	72	64
-1	111	116
0	8	<20
1	63	<20
2	-132	129
3	-12	<22
4	-40	36
5	30	30
6	-30	30
7	-59	62
8	68	65
9	-9	-

11 5 L

h	Fo	Fc
-18	65	-
-17	22	-
-16	2	-
-15	89	-
-14	-41	-
-13	58	-

Table 3. Atomic co-ordinates, expressed as fractions of the cell edges, and anisotropic thermal parameters for $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$. (The temperature factor is $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$). All atoms occupy the general position $8f$ in $C2/c$ except O_9 which is situated in $4c$).

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Te	0.08640	0.0805	0.07773	0.00170	0.0081	0.00146	−0.0003	0.00053	−0.0005
K_1	0.1542	0.1542	0.4078	0.0038	0.0137	0.0027	0.0010	0.0014	0.0006
K_2	0.4962	0.1218	0.3488	0.0032	0.0126	0.0026	−0.0006	0.0013	0.0016
O_1	0.1083	0.051	0.1858	0.0039	0.020	0.0017	−0.004	0.0011	−0.001
O_2	0.1945	0.234	0.0912	0.0029	0.011	0.0032	−0.004	0.0017	−0.002
O_3	0.4634	0.423	0.0427	0.0026	0.013	0.0015	−0.004	0.0003	−0.004
O_4	0.0066	0.314	0.0673	0.0030	0.008	0.0034	0.004	0.0015	0.000
O_5	0.3474	0.334	0.4261	0.0042	0.008	0.0026	−0.004	0.0023	0.001
O_6	0.297	0.061	0.3108	0.0061	0.018	0.0028	−0.008	0.001	−0.002
O_7	0.3825	0.316	0.1963	0.0027	0.020	0.0032	0.002	0.0014	−0.003
O_8	0.3128	0.033	0.0314	0.0036	0.011	0.0029	0.000	0.0021	0.001
O_9	0	0.284	1/4	0.004	0.003	0.0026	0	0.003	0
O_{10}	0.105	0.343	0.270	0.008	0.02	0.004	0.015	0.004	−0.007

Table 4. Standard deviations of the atomic parameters.

Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	$\sigma(\beta_{11})$	$\sigma(\beta_{22})$	$\sigma(\beta_{33})$	$\sigma(\beta_{12})$	$\sigma(\beta_{13})$	$\sigma(\beta_{23})$
Te	0.00005	0.0001	0.00005	0.00005	0.0002	0.00004	0.0001	0.00006	0.0001
K_1	0.0002	0.0005	0.0002	0.0002	0.0007	0.0001	0.0006	0.0002	0.0005
K_2	0.0002	0.0005	0.0002	0.0002	0.0007	0.0001	0.0005	0.0002	0.0005
O_1	0.0008	0.002	0.0006	0.0006	0.003	0.0004	0.002	0.0008	0.002
O_2	0.0007	0.002	0.0007	0.0005	0.002	0.0004	0.002	0.0008	0.002
O_3	0.0007	0.001	0.0006	0.0005	0.002	0.0003	0.002	0.0007	0.001
O_4	0.0007	0.001	0.0007	0.0005	0.002	0.0004	0.002	0.0008	0.002
O_5	0.0008	0.001	0.0006	0.0006	0.002	0.0004	0.002	0.0008	0.002
O_6	0.001	0.002	0.0007	0.0008	0.003	0.0005	0.002	0.001	0.002
O_7	0.0007	0.002	0.0007	0.0005	0.003	0.0004	0.002	0.0008	0.002
O_8	0.0008	0.002	0.0004	0.0005	0.002	0.0004	0.002	0.0008	0.002
O_9		0.004		0.001	0.006	0.0008		0.002	
O_{10}	0.004	0.006	0.003	0.003	0.01	0.002	0.009	0.004	0.008

than 2 % of the standard deviations and the final R value was 0.080. The resulting set of parameters is given in Table 3 and the corresponding standard deviations in Table 4. The calculated and observed structure factors are listed in Table 2. The structure factors which could not be obtained from the films were computed using the programme SFGEN written by Ingvarsson,[22] this calculation being based on the co-ordinates of Table 3, but with isotropic temperature factors taken from the earlier, preliminary structure proposition.[18]

A final difference Fourier was then calculated, which showed good agreement with the least squares refinement results.
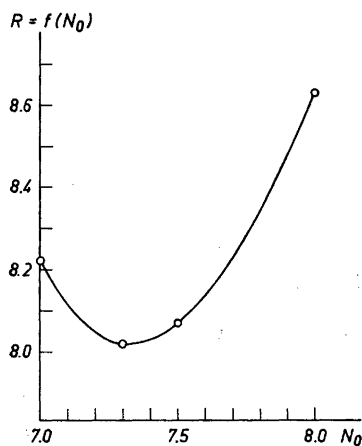
Fig. 4. The reliability index R as a function of the number (N_0) of water molecules in the stoichiometric formula $K_4[Te_2O_6(OH)_4](H_2O)_{N_0}$.
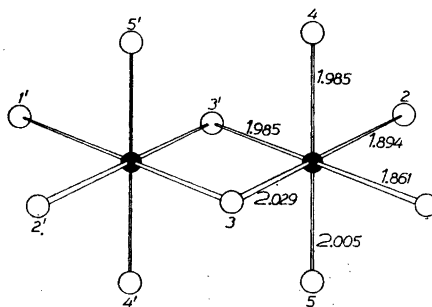
Fig. 5. A schematic drawing of the dimeric tellurate ion $Te_2O_6(OH)_4^{4-}$.

THE POSITIONS OF THE HYDROGEN ATOMS

The hydrogen atoms are presumably situated so that they form hydrogen bonds, connecting oxygen atoms belonging to different Te$-$O polyhedra, or connecting Te$-$O polyhedra with oxygen atoms situated outside the tellurate ions. The hydrogen bonds may perhaps also be located within a K$-$O polyhedron but not within a Te$-$O polyhedron. The distance between two oxygen atoms forming a hydrogen bond is expected to be 2.5$-$2.9 Å.

Since there are 74.4 hydrogen atoms in the unit cell (assuming the formula to be $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$), there should be at least 10 eightfold hydrogen atom positions, some of which being only partially filled, thus corresponding to at least 10 independent O$-$O distances of 2.5$-$2.9 Å. With the aid of the programme DISTAN,[20] written for the computer CD 3600, the interatomic distances in the structure were calculated. It was then found that there were only 12 independent O$-$O distances less than 3.14 Å (cf. Table 5) fulfilling the condition stated at the beginning of this paragraph. Since the O_9 and O_{10} positions are, however, not occupied simultaneously, these O$-$O distances correspond to 74.4 hydrogen atoms. By considering the values listed in Table 5 and the fact that O_1-O_5 are in close contact with Te (cf. Table 6), the most plausible arrangement of the hydrogen atoms is when O_6-O_{10} all correspond to water molecules and O_4-O_5 to hydroxide groups.

Table 5. Possible hydrogen bonds in the structure of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$.

$O_6 - O_5$	2.67 ± 0.02 Å		$O_1-O_6-O_2$	103.6 ± 0.5°
$O_6 - O_1$	2.80 ± 0.02 Å		$O_1-O_6-O_5$	119.8 ± 0.6°
$O_6 - O_2$	2.86 ± 0.02 Å		$O_2-O_6-O_5$	98.7 ± 0.5°
$O_7 - O_1$	2.65 ± 0.02 Å		$O_1-O_7-O_2$	111.1 ± 0.5°
$O_7 - O_2$	2.74 ± 0.02 Å		$O_2-O_8-O_2$	102.8 ± 0.4°
$O_8 - O_2$	2.72 ± 0.02 Å		$O_1-O_9-O_1$	113 ± 1°
$O_8 - O_2$	2.86 ± 0.02 Å			
$O_9 - O_1$	2×(2.90 ± 0.02) Å			
$O_{10}-O_1$	2.55 ± 0.05 Å			
$O_{10}-O_6$	2.94 ± 0.05 Å			
$O_4 - O_5'$	2.85 ± 0.02 Å			

Table 6. Distances in the dimeric tellurate ion $Te_2O_6(OH)_4^{4-}$. (cf. Table 7).

Te$-$Te'	3.119 ± 0.002 Å
Te$-$O$_1$	1.861 ± 0.010 Å
Te$-$O$_2$	1.894 ± 0.010 Å
Te$-$O$_3'$	1.985 ± 0.009 Å
Te$-$O$_3$	2.029 ± 0.010 Å
Te$-$O$_4$	1.985 ± 0.010 Å
Te$-$O$_5$	2.005 ± 0.010 Å

O_3-O_3'	2.527 ± 0.019 Å		$O_2 - O_4$	2.781 ± 0.014 Å
O_3-O_1	2.733 ± 0.014 Å		$O_3 - O_4$	2.776 ± 0.014 Å
O_1-O_2	2.887 ± 0.015 Å		$O_3'-O_5$	2.689 ± 0.014 Å
O_2-O_3'	2.798 ± 0.014 Å		$O_1 - O_5$	2.850 ± 0.015 Å
$O_3'-O_4$	2.779 ± 0.014 Å		$O_2 - O_5$	2.830 ± 0.014 Å
O_1-O_4	2.729 ± 0.015 Å		$O_3 - O_5$	2.776 ± 0.014 Å
	$O_4-O_5' = O_4'-O_5$	2.851 ± 0.015 Å		

Table 7. Comparison of the anions $I_2O_8(OH)_2^{4-}$ and $Te_2O_6(OH)_4^{4-}$. Indices given as in Fig. 5 of this paper.

Bond	$I_2O_8(OH)_2^{4-}$	$Te_2O_6(OH)_4^{4-}$
M—O	1.793 ± 0.016 Å	1.861 ± 0.010 Å
	1.810 ± 0.015 Å	1.894 ± 0.010 Å
	1.818 ± 0.010 Å	
M—O$_{bridge}$	1.992 ± 0.012 Å	1.985 ± 0.009 Å
	2.017 ± 0.007 Å	2.029 ± 0.010 Å
M—OH	1.980 ± 0.016 Å	1.985 ± 0.010 Å
		2.005 ± 0.010 Å

Angle		
O_3—M—O_3' (bridge)	77.6 ± 0.4°	78.0 ± 0.3°
O_3—M—O_1	89.6 ± 0.5°	89.2 ± 0.4°
O_1—M—O_2	100.0 ± 0.7°	100.5 ± 0.5°
O_2—M—O_3'	92.0 ± 0.6°	92.3 ± 0.4°
O_3—M—O_4	88.0 ± 0.5°	87.5 ± 0.4°
O_1—M—O_4	97.1 ± 0.7°	90.4 ± 0.5°
O_2—M—O_4	97.6 ± 0.6°	91.6 ± 0.4°
O_3'—M—O_4	88.2 ± 0.6°	88.9 ± 0.4°
O_3—M—O_5	86.2 ± 0.6°	86.9 ± 0.4°
O_1—M—O_5	89.6 ± 0.7°	94.9 ± 0.5°
O_2—M—O_5	87.0 ± 0.7°	93.0 ± 0.4°
O_3'—M—O_5	83.9 ± 0.6°	84.7 ± 0.4°

DISCUSSION

The structure of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$ is built up from dimeric $Te_2O_6(OH)_4^{4-}$ ions, K^+ ions and water molecules, which are held together by electrostatic interaction and by hydrogen bonds. A schematic drawing of the $Te_2O_6(OH)_4^{4-}$ ion is given in Fig. 5 and of the structure in Fig. 6.

The bond distances and bond angles within the $Te_2O_6(OH)_4^{4-}$ ion are given in Tables 6 and 7 (*cf.* also Fig. 5). As is seen, the ion consists of two Te—O octahedra having one edge in common. The Te—O distances within these octahedra can be divided into two groups, one comprising Te—O distances of 1.985—2.029 Å (Te—O_3, Te—O_3', Te—O_4, Te—O_5) and the other comprising Te—O distances of 1.861—1.894 Å (Te—O_1, Te—O_2). It is interesting to note that the oxygen atoms belonging to the first group are co-ordinated to two other atoms (O_3 and O_3' to two Te atoms, O_4 and O_5 to one Te and one H atom), whereas those belonging to the second group are closely bond to one Te atom only but participate, in addition, in weak hydrogen bonding to water molecules. This is in accordance with the fact that the Te—O_1 and Te—O_2 distances are significantly shorter than the Te—O_3, Te—O_3', Te—O_4, and Te—O_5 distances. A similar but less pronounced tendency was found in the structure of $KTeO(OH)_5H_2O$, in which Raman [8] determined the Te—O bond length to be 1.83 Å and the Te—OH bonds to be 1.90—1.95 Å (standard deviation 0.02—0.03 Å) in the monomeric $TeO(OH)_5^-$ ion. These distances may be compared with our values Te—O = 1.861—1.894 Å (standard deviation
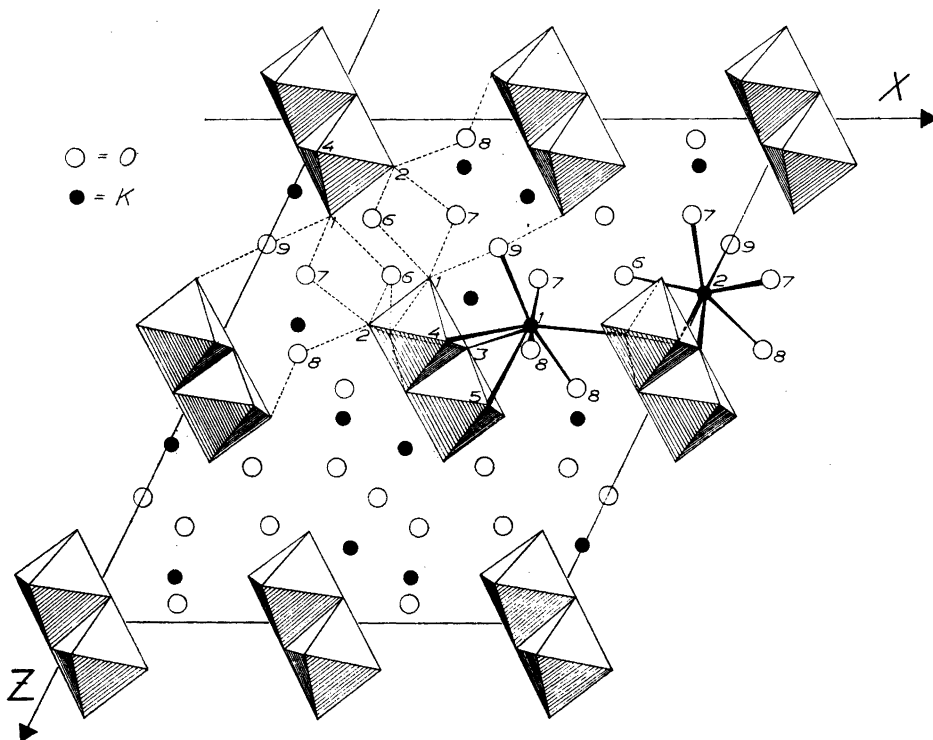
Fig. 6. A schematic picture of the structure of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$, projected on to the xz plane. The O_{10} position has been omitted.

0.010 Å), Te—OH = 1.985—2.005 Å (standard deviation 0.010 Å), and Te—O_{bridge} = 1.985—2.029 Å (standard deviation 0.009—0.010 Å).

In the structures of $KTeO_2(OH)_3$[9] and $KTeO_3(OH)$,[10] the Te—O distances are reported to be 1.84 Å ($KTeO_3(OH)$) and 1.89 Å ($KTeO_2(OH)_3$), the Te—OH distances 1.84 Å ($KTeO_3(OH)$) and 1.89 Å ($KTeO_2(OH)_3$) and the Te—O_{bridge} distances 1.96—2.01 Å ($KTeO_3(OH)$) and 1.84 Å ($KTeO_2(OH)_3$). The accuracy of the distances in $KTeO_2(OH)_3$ is, however, low, the standard deviations of the oxygen positions being 0.2 Å or more, since only an idealized structure is given. A detailed comparison with the distances in $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$ cannot therefore be made. The Te—O_{bridge} distances in the $KTeO_3(OH)$ structure are in good agreement with our value for $Te_2O_6(OH)_4^{4-}$. The value reported for Te—OH in $KTeO_3(OH)$, is, however, lower than the Te—O_{bridge} value, lower than the Te—OH distances found in other structures, and even, in fact, almost the same as that which has been found for Te—O in $KTeO(OH)_5H_2O$. The accuracy in the positional parameters of $KTeO_3(OH)$ is not, however, stated, so that it is not possible to say if this difference is significant or not.

A structure determination of $K_4[I_2O_8(OH)_2](H_2O)_8$ has recently been published.[23] This compound has been shown to contain the dimeric anion $I_2O_8(OH)_2^{4-}$ which is isoelectronic with the dimeric tellurate ion described in this paper. Both ions are built up in the same way and their bond distances and bond angles are very similar. As is seen in Table 7 the $M-OH$ and $M-O_{bridge}$ distances fall within the standard deviations. The $M-O$ distances are, however, significantly different, the $I-O$ distance being 0.07 Å shorter than that of $Te-O$. This is also to be expected in this case, since I^{VII} and Te^{VI} are isoelectronic. Likewise, most of the $O-M-O$ angles fall within the standard deviations, but the four angles O_1-M-O_4, O_1-M-O_5, O_2-M-O_4, and O_2-M-O_5 are significantly different in the two anions (cf. Table 7). This deviation may be explained by the bending of the $M-O_1$ and $M-O_2$ bonds away from the $M-O_4$ bond, O_4 being a hydroxide group in the tellurate ion but an oxygen atom in the periodate ion. The $M-O_4$ bond is also, therefore, shorter in $I_2O_8(OH)_2^{4-}$ (1.810 Å) than in $Te_2O_6(OH)_4^{4-}$ (1.985 Å).

Anions of the same general appearance as $Te_2O_6(OH)_4^{4-}$ and $I_2O_8(OH)_2^{4-}$ have been found in the crystal structures of $[Al_2(OH)_2(H_2O)_8](SO_4)_2(H_2O)_2$ and the corresponding selenate.[24] A detailed comparison between the bond distances of the tellurate and periodate ions with those of the $Al_2(OH)_2(H_2O)_8^{4+}$ ions is not, however, profitable, since the electronic configurations of Te^{VI} and I^{VII} are different from that of Al^{III}.

In order to study the condensation products of the tellurates, it is suitable to start from the corresponding acid. Orthotelluric acid, H_6TeO_6, is built up from discrete molecules $Te(OH)_6$.[2-5] The first anion formed from this acid is $TeO(OH)_5^-$, which is present in $KTeO(OH)_5H_2O$,[8] as the monomeric anion $TeO(OH)_5^-$. It can condense to $[TeO_2(OH)_3]_n^{n-}$ and $[TeO_3(OH)]_n^{n-}$, which have been found in the structures of $KTeO_2(OH)_3$[9] and $KTeO_3(OH)$.[10] These ions are built up from TeO_6-octahedra sharing corners ($KTeO_2(OH)_3$) or edges ($KTeO_3(OH)$) to form linear polymeric anions. The $O-O$ distances in the double oxygen bridges of $[TeO_3(OH)]_n^{n-}$ are 2.52 Å (no standard deviation given).

The second dissociation step of orthotelluric acid should give rise to an anion of the type $TeO_2(OH)_4^{2-}$. This anion is, however, not yet known but the ion described in this paper, $[TeO_3(OH)_2]_2^{4-}$, is a dimeric, condensed form of $TeO_2(OH)_4^{2-}$. The $O-O$ distance in the double oxygen bridge of $Te_2O_6(OH)_4^{4-}$, 2.527 ± 0.019 Å, is in good agreement with the value 2.512 ± 0.015 Å found in the anion $I_2O_8(OH)_2^{4-}$ and with the corresponding distance 2.52 Å in $[TeO_3(OH)]_n^{n-}$.

The completely condensed form corresponding to the $TeO_2(OH)_4^{2-}$ ion and based on a six-fold co-ordination, should have the formula $[TeO_4]_n^{2n-}$. Such ions have not yet been described, but may be present in some slightly soluble "metatellurates" (cf. the slightly soluble uranates, some of which are built up from UO_2O_4 octahedra sharing corners to form sheets of composition $[UO_2O_2]_n^{2n-}$).[25-27]

Only a very preliminary structure determination showing a monomeric, tetrahedral TeO_4^{2-} has been published.[6] Nor have the polymeric forms $Te_nO_{3n+1}^{2-}$ yet been shown to exist in tellurates.

The co-ordination of potassium in the structure of $K_4[Te_2O_6(OH)_4](H_2O)_{7.3}$ is eight-fold for K_1 and seven-fold for K_2 (*cf.* Table 8). The contact distances are within the range $2.629-3.099$ Å with a mean value of 2.910 Å for K_1 and 2.800 Å for K_2, which is in agreement with the values reported in the *International Tables*.[28]

Table 8. Distances in the potassium — oxygen polyhedra (Å).

K_1-O_3	2.843 ± 0.010 Å	K_2-O_3	2.748 ± 0.010 Å
K_1-O_4	2.918 ± 0.011 Å	K_2-O_4	2.636 ± 0.011 Å
K_1-O_5	3.043 ± 0.011 Å	K_2-O_6	2.854 ± 0.014 Å
K_1-O_5	3.099 ± 0.011 Å	K_2-O_7	2.702 ± 0.012 Å
K_1-O_7	2.908 ± 0.013 Å	K_2-O_7	2.888 ± 0.012 Å
K_1-O_8	2.804 ± 0.011 Å	K_2-O_8	2.854 ± 0.011 Å
K_1-O_8	2.807 ± 0.011 Å	K_2-O_9	2.969 ± 0.021 Å
K_1-O_9	2.954 ± 0.009 Å	or K_2-O_{10}	2.810 ± 0.045 Å
or K_1-O_{10}	2.629 ± 0.044 Å		

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