tives even though the total data have not been measured. A judgment of s^2 could be made from those data used to solve the structure and an estimate of the confidence region could then also be made.

All this might not suffice, however, to give the individual investigator the confidence needed to abandon the work even if there is strong indication that a refined structure is not feasible.

The cases which are not extreme are those in which there may be apparent convergence but some large correlations and error estimates. The calculation of confidence regions should be worthwhile in such cases, although with a *large* number of correlated parameters, it is doubtful that it will be extremely useful. It is especially important however to note by examination of the correlation matrix which of the parameters appear to be correlated and which of them do not.

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Several books and a paper by Box & Coutie (1956) were useful in this work. The books are: Acton (1959), Anderson (1958), Scheffé (1959) and Wilks (1946).

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Potassium Osmyl Chloride — Refinement of the Crystal Structure*

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The crystal structure of $K_2OsO_2Cl_4$, first reported by Hoard & Grenko (1934), has been refined by least-squares and Fourier calculations using three dimensional data. The crystals are tetragonal, $I4/mmm$, with $a_0 = 6{\cdot}991 \pm 0{\cdot}004$ and $c_0 = 8{\cdot}752 \pm 0{\cdot}006$ Å. The two primary bond lengths in the $OsO_2Cl_4^{--}$ ion were refined to: Os–O: $1{\cdot}750 \pm 0{\cdot}022$ Å and Os–Cl: $2{\cdot}379 \pm 0{\cdot}005$ Å.

Introduction

The crystal structure of potassium osmyl chloride, $K_2OsO_2Cl_4$, was first determined by Hoard & Grenko (1934) (referred as H.&G. in remaining text). At that time the unit cell dimensions, space group and approximate values for the variable parameters of oxygen and chlorine were established, as well as the location of the osmium and potassium atoms in special positions. In conjunction with an extensive study of the

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vibrational spectra of complexes containing linear inorganic oxygen–metal–oxygen groups (Jones, 1958, 1959) an accurate measure of the oxygen–osmium bond length was needed and thus the refinement of this structure was undertaken.

The uranyl ion, UO_2^{++}, is the best known member of this type of inorganic linear O–*M*–O group. A number of other actinides, neptunium, plutonium and americium, are known to form such linear arrangements. Osmium, ruthenium and rhenium are the few non-actinide elements which form these complexes. They probably never exist as free triatomic ions but are always coordinated with from two to six ligands. The type of ligand coordinated to the central metal atom has a very strong influence on the metal to oxygen bond length. For example, in various uranyl complexes the metal to oxygen bond length ranges from 1·65 to 1·95 Å (Zachariasen & Plettinger, 1959; Zachariasen, 1954).

Experimental

Potassium osmyl chloride was prepared from potassium osmate, $K_2OsO_2(OH)_4$, by treatment with dilute HCl as described by Wintrebert (1903). Small tetragonal bipyramids were obtained on recrystallization from dilute hydrochloric acid solution. The compound has been described as unstable in air, but the dry, recrystallized material was found to be stable in the relatively dry New Mexico atmosphere.

A Cu $K\alpha$ ($\lambda = 1{\cdot}5418$ Å) powder photograph gave unit cell dimensions in agreement with those published by H.&G., that is:

	Hoard & Grenko	This work
a_0	6·99	6·991 ± 0·004 Å
c_0	8·75	8·752 ± 0·006 Å
$Z=2$	Obs. density 3·4 g.cm.$^{-3}$	Calc. density 3·42 g.cm.$^{-3}$

Optically, $K_2OsO_2Cl_4$ crystals are uniaxial negative with $\eta_0 = 1{\cdot}757 \pm 0{\cdot}005$ and $\eta_e = 1{\cdot}665 \pm 0{\cdot}005$ (5893 Å). The normally dark red-amber crystals are dichroic in thin sections displaying a pale amber color in the ε direction and darker red-amber color in the o direction.

As shown by H.&G. the diffraction symmetry is characteristic of the following tetragonal body-centered space groups: $I422$, $I4mm$, $I\bar{4}m2$, $I\bar{4}2m$, and $I4/mmm$. Of these five the only centrosymmetric group is $I4/mmm$. Stereochemical considerations of packing for the $OsO_2Cl_4^{--}$ ion by H.&G. indicated $I4/mmm$ to be the most likely group and subsequently there has been found no evidence of non-centrosymmetry in this structure.

The diffraction data were collected using Mo $K\alpha$ radiation and a scintillation counter detector mounted on a Weissenberg camera (Evans, 1953). An approximately octahedral specimen was mounted and then rounded to a sphere of 0·03 mm. radius by solvent attrition (methanol–HCl–water solution). The crystal was slightly elongated about the rotation axis. Essentially all the data detectable by the scintillation counter were recorded, yielding 340 independent reflections from more than 2000 observations of the eight zones $0kl$ to $7kl$. The intensity data were corrected for background, for the usual Lorentz-polarization effects, and for absorption assuming a spherical specimen with μ of 190 cm.$^{-1}$ and thus a μr of 0·6. Absorption-factor tables of Evans & Ekstein (1952) were used. From a plot of ratios of related intensities from the various layers, an empirical cylindrical-type upper layer absorption correction curve was obtained and this additional correction was applied to the seven upper layers. All symmetry-related reflections from the various layers showed internal consistency and were averaged in the final compilation of the 340 unique reflections.

Refinement of the parameters

The position parameters for all four atoms in the space group, $I4/mmm$, were established by H.&G. to be:

$(0, 0, 0;\ \tfrac{1}{2}, \tfrac{1}{2}, \tfrac{1}{2})+$

Os	(*a*)	$0, 0, 0;$	
K	(*d*)	$0, \tfrac{1}{2}, \tfrac{1}{4};\ \tfrac{1}{2}, 0, \tfrac{1}{4};$	
O	(*e*)	$0, 0, z;\ 0, 0, \bar{z};$	$z = 0{\cdot}212;$
Cl	(*h*)	$x, x, 0;\ \bar{x}, \bar{x}, 0;\ x, \bar{x}, 0;\ \bar{x}, x, 0;$	$x = 0{\cdot}230.$

With these initial values a least-squares refinement was carried out with isotropic individual atom temperature factors using the 340 observed reflections, not weighted. All computations were carried out on an IBM 704. The refinement led to

$$z_O = 0{\cdot}199,\ x_{Cl} = 0{\cdot}230 \text{ and } R = 0{\cdot}0765\,.$$

The standard deviation of the Os–O bond length was ± 0·03 Å. Only one reflection, (022), had a negative calculated structure factor.

A three-dimensional F_o Fourier synthesis (Fig. 1) was calculated and several unidentified peaks were found of nearly the same magnitude as the oxygen peak. However, the calculation of a difference Fourier wherein the osmium and potassium contributions were removed showed the three peaks (of approximately 10 e.Å^{-3}) to be due to heavy-atom effects. The difference map (Fig. 1) also indicated anisotropic thermal parameters for K^+.

At this point in the refinement the least-squares routine was improved to include anisotropic temperature factors of the form:

$$\exp -(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)\,.$$

The special positions of the atoms Os, K and O require the cross terms to be zero and $B_{11} = B_{22}$ while for Cl $B_{13} = B_{23} = 0$ and $B_{11} = B_{22}$. The least-squares refinement program uses the full matrix solution of the normal equations and full inverse of the matrix

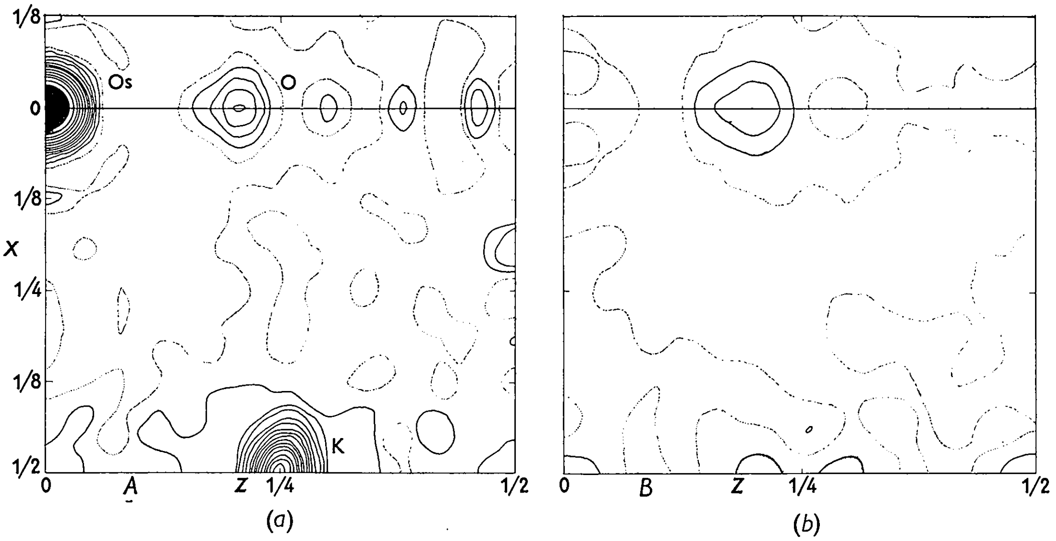

Fig. 1. Electron density functions, sections at $x=0$, (a) F_o Fourier section and (b) $[F_o-F_c$ (Os and K$^+$)] difference Fourier section. The zero contour is dotted and the contour scale is approximately 5 e.Å^{-3}. Os at (0, 0), K$^+$ at ($\frac{1}{2}$, $\frac{1}{4}$), and O at (0, 0·2).

Table 1. *Parameters of* O *and* Cl, *R*, *Wtd. R and ESD of unit-wt. observation as determined by least-squares refinement*

	x_{Cl}	z_{O}	R	Wtd. R	ESD of unit-wt. observation
Anisotropic temp. factors	0·2406 ± 0·0005	0·1999 ± 0·0022	0·0579	0·0442	2·940
Isotropic temp. factors	0·2409 ± 0·0007	0·2013 ± 0·0023	0·0785	0·0550	3·662

in solving for the standard deviations. With only twelve variables less than three minutes was required for each cycle for 340 reflections.

Before calculating with anisotropic thermal factors, the $F_o{}^2(hkl)$ data were assigned weights based on counting-rate errors. The scattering factor curve for osmium was improved by replacing the Thomas–Fermi factor with the more recent Thomas–Umeda (1957) scattering factor curve. In a few cycles the refinement settled on the parameters shown in Table 1. A complete (F_o-F_c) Fourier synthesis was then computed and all spurious peaks above 1 e.Å^{-3} had been removed by application of anisotropic temperature factors. In all of these calculations only the observed reflections were employed with no special provision for the unobserved ones. With the final parameters a structure factor calculation was computed for all reflections allowed by the space group within the Mo sphere. No calculated structure factor for an accessible unobserved reflection exceeded the minimum value for that region of the sphere. The F_o, F_c data are presented in Table 3.

Table 2. *Anisotropic temperature factors*

A. Final thermal parameters × 10^4 after the anisotropic least-squares refinement

	B_{11}	B_{12}	B_{33}
Os	76 ± 2	(00)	52 ± 2
K$^+$	274 ± 12	(00)	77 ± 9
O	117 ± 23	(00)	136 ± 34
Cl	137 ± 11	−46 ± 27	171 ± 8

B. B_i (Å^2) and root-mean-square amplitudes in Å along ellipsoid axes

	Crystal axis direction of ellipsoid axis	B_i	Root-mean-square amplitude
Os	X	1·49	0·14
	Z	1·58	0·14
K$^+$	X	5·36	0·26
	Z	2·34	0·17
O	X	2·29	0·17
	Z	4·16	0·23
Cl	X, X	3·12	0·20
	$X, \bar{X}$	2·22	0·17
	Z	5·24	0·26

The anisotropic B's (in Å^2) and root-mean-square amplitudes are given in Table 2. The direction cosines of the ellipsoid axes are prescribed by symmetry of the special positions in the space group $I4/mmm$. The values in Table 2 were computed with an IBM 704 program by Larson (1960).

While a least-squares cycle of refinement was being computed, the following factors were also calculated and then printed at the end of the cycle.

$$R=\Sigma||F_o|-|F_c||/\Sigma|F_o|\,.$$

$$\text{Wtd. } R=\{\Sigma w(|F_o|-|F_c|)^2/\Sigma wF_o{}^2\}^{\frac{1}{2}}.$$

EDS of unit-wt. observation

$$=\{\Sigma w(|F_o|-|F_c|)^2/(m-n)\}^{\frac{1}{2}}.$$

Table 3. *Final observed and calculated structure factors for* $K_2OsO_2Cl_4$

hkl	F_o	F_c
002	169	167
004	226	229
006	108	98
008	102	103
0,0,10	50	47
0,0,12	44	46
0,0,14	22	19
0,0,16	22	19
013	112	110
015	115	112
017	74	72
019	56	57
0,1,11	40	41
0,1,13	25	27
0,1,15	22	19
020	115	106
022	49	−31
024	91	86
026	30	25
028	58	58
0,2,10	29	27
0,2,12	33	36
031	107	106
033	86	80
035	86	87
037	58	59
039	48	49
0,3,11	31	36
0,3,13	27	25
0,3,15	23	17
040	208	204
042	113	113
044	151	148
046	79	80
048	76	77
0,4,10	39	40
0,4,12	34	36
0,4,14	21	18
051	102	102
053	86	83
055	89	80
057	57	56
059	44	45
0,5,11	30	32
0,5,13	22	22
060	62	56
064	47	48
066	40	28
068	38	36
0,6,10	22	24
071	49	49
073	41	42
075	39	45
077	36	34
079	29	29
0,7,11	18	23
0,7,13	19	16
080	82	79
082	64	59
084	66	63
086	42	44
088	42	37
0,8,10	26	25
091	50	49
093	48	43
095	39	40
097	34	30
099	24	24
0,10,0	29	27
0,10,4	22	24
0,10,6	23	19
0,10,8	23	18
0,12,0	30	26
0,12,2	28	22
0,12,4	22	22
0,12,6	24	18
112	166	165
114	70	72
116	115	115
118	40	42
1,1,10	61	62
1,1,12	22	24
1,1,14	28	28
121	127	128
123	92	93
125	97	98
127	64	65
129	52	53
1,2,11	34	39
1,2,13	24	26
1,2,15	20	18
130	93	93
132	128	128
134	66	67
136	96	97
138	33	40
1,3,10	53	54
1,3,14	29	25
141	103	104
143	84	83
145	82	84
147	57	58
149	44	47
1,4,11	32	34
1,4,13	24	23
150	70	76
152	90	92
154	54	59
156	71	73
158	33	37
1,5,10	41	43
161	67	70
163	57	59
165	55	59
167	43	43
169	34	36
1,6,11	25	27
170	49	56
172	56	60
174	43	46
176	48	50
178	27	30
1,7,10	24	31
181	46	51
183	43	44
185	42	43
187	35	33
189	31	27
1,8,11	29	20
1,8,13	36	14
190	36	40
192	38	40
194	34	34
196	29	33
198	29	23
1,9,10	22	21
1,10,1	28	32
1,10,3	28	28
1,10,5	32	28
1,11,0	23	26
1,11,2	23	24
1,11,4	22	22
1,11,6	23	21
1,12,3	23	18
220	261	268
222	131	133
224	179	181
226	86	89
228	86	89
2,2,10	41	43
2,2,12	36	41
231	117	122
233	95	95
235	91	94
237	60	64
239	47	51
2,3,11	33	36
2,3,13	20	24
2,3,15	20	17
240	70	73
242	18	8
244	63	61
246	28	27
248	45	44
2,4,10	22	25
2,4,12	29	28
251	66	69
253	57	56
255	61	60
257	44	44
259	39	38
2,5,11	28	28
260	111	121
262	81	83
264	94	94
266	57	60
268	53	53
2,6,10	29	32
2,6,12	25	26
271	63	70
273	61	59
275	51	56
277	41	41
279	30	33
2,7,11	22	24
280	31	35
282	18	20
284	30	32
286	21	24
288	25	25
291	28	31
293	26	27
295	26	29
297	22	23
2,10,0	46	44
2,10,2	41	36
2,10,4	38	37
2,10,6	28	28
2,10,8	26	23
2,11,1	32	30
2,11,3	30	26
2,11,5	28	25
2,11,7	23	19
330	105	85
332	107	109
334	57	65
336	90	84
338	33	39
3,3,10	46	48
3,3,12	23	22
3,3,14	25	22
341	76	79
343	64	64
345	67	67
347	50	48
349	41	40
3,4,11	31	30
3,4,13	21	21
350	62	66
352	87	77
354	52	53
356	63	63
358	32	34
3,5,10	36	38
361	66	70
363	60	60
365	55	58
367	41	42
369	32	34
3,6,11	25	25
3,6,13	25	17
370	44	54
372	52	56
374	39	44
376	42	46
378	26	28
3,7,10	28	28
381	40	42
383	36	36
385	36	37
387	28	28
389	26	24
3,8,11	22	18
3,8,13	24	13
390	41	35
392	36	34
394	30	30
396	33	29
398	25	20
3,9,10	25	19
3,10,1	34	31
3,10,3	30	28
3,10,5	28	27
440	148	142
442	97	93
444	114	110
446	67	67
448	68	60
4,4,10	30	35
4,4,12	31	29
451	80	79
453	70	67
455	63	64
457	48	46
459	38	37
4,5,11	28	27
4,5,13	20	18
460	38	41
462	21	19
464	37	37
466	24	25
468	32	29
4,6,10	22	21
471	36	40
473	34	35
475	33	37
477	27	28
479	25	25
4,7,11	20	19

Table 3 (*cont.*)

hkl	F_o	F_c
480	66	64
482	54	50
484	55	52
486	38	38
488	31	31
491	41	41
493	40	36
495	32	34
497	27	25
499	21	20
550	65	59
552	64	64
554	44	48
556	51	52
558	29	31
5,5,10	25	31
561	44	45
563	38	39
565	38	40
567	29	31
569	25	26
570	39	41
572	44	41
574	34	35
576	35	35
578	22	23
5,7,10	22	23
581	42	42
583	18	36
585	35	35
587	22	26
590	28	33
592	27	31
594	19	28
596	20	26
660	75	72
662	57	56
664	58	59
666	43	42
668	33	34
6,6,10	19	23
671	46	46
673	41	40
675	36	38
677	27	28
679	23	23
680	18	24
684	20	22
6,10,0	28	30
6,10,2	26	26
6,10,4	22	25
6,10,6	21	20
770	35	36
772	33	35
774	26	30
776	26	28
781	27	24
783	23	22
785	23	22
787	23	18
790	26	22

Sum delta squared $=\Sigma(\Delta)^2=\Sigma(|F_o|-|F_c|)^2$.
Sum wtd. delta squared
$=\Sigma$ wtd. $(\Delta)^2=\Sigma w(|F_o|-|F_c|)^2$.
Sum delta $=\Sigma\Delta=\Sigma\big||F_o|-|F_c|\big|$.

Estimated standard deviation of unit-wt. observation, known also as the error-of-fit function, has unity as the ideal value. Deviation from unity has been ascribed variously to an incorrect model, to structural parameters not properly optimized, or to a weighting scheme which does not reflect the true errors of the observation. For any given model of the structure where the standard deviations of the $I_0(hkl)$ data are used to determine the least-squares weighting it is possible to 'scale' those standard deviations by a constant factor and thus cause the ESD of unit-wt. observations to approach unity. This may not be a legitimate use of this function but it does indicate the standard deviations in the data which are considered compatible with the model and the data set being refined. However, no ESD of unit-wt. observation given in this paper was so scaled.

The bulk of the least-squares refinements were carried out using the following atomic scattering factor curves:

f_{Os^0} — Thomas & Umeda (1957).
f_{O^0} — Berghuis *et al.* (1955).
f_{Cl^-} — Freeman (1959).
f_{K^+} — Freeman (1959).

Computation of a few least-squares refinement cycles was carried out employing combinations of f curves for O^-, O^{--}, and Cl^0. None of the results fell outside the standard deviations obtained with the f curves mentioned above.

Weighting

The weight, w, used in the least-squares refinements was determined from standard deviations by the relation $\sqrt{w}=1/\sigma$.

The statistical counting-rate error as described by Friedlander & Kennedy (1955) was assigned as follows:

Background count $=C_b=N_b\pm\sqrt{N_b}$.
Total count $=C_t=N_t\pm\sqrt{N_t}$.
Net count $=C_t-C_{bkgd}=C_{\text{net}}\pm(N_b+N_t)^{\frac{1}{2}}$.

The count for each piece of data was a 'fixed time' count of 1·2 minutes. This equation for net count does not explicitly include effects due to absorption, extinction or any other experimental errors but tends to merge them as a single effect. The standard deviation was assigned to the I_0 of each reflection and was properly scaled to give σ_{Fo} for each unique reflection employed in the least-squares refinements.

The factor of $1/\sqrt{n}$ was not employed for standard deviations of symmetry-related reflections observed n times because these observations were averaged to minimize absorption errors in the assumption of a spherical specimen.

Least-squares calculations also were carried out with unit weighting of the F_o and F_o^2 in order to compare the results of this approach with that of statistical counting-error weighting. Use of unit weight for F_o implies that every intensity was collected with a standard deviation such that the corrected and scaled F_o has a $\sigma_{F_o}=1{\cdot}00$. Experimental standard deviations are usually determined from measurements of I_0 from which one obtains F_o^2 and $\sigma_{F_o^2}$. In calculating the corresponding F_o and σ_{F_o} one uses the relation: $\sigma_{F_o}=\sigma_{F_o^2}/(2F_o^2)^{\frac{1}{2}}$. Consequently if one assigns unity to all σ_{F_o}, this means one is assuming $\sigma_{F_o^2}=(2F_o^2)^{\frac{1}{2}}$ or the standard deviation of a measured intensity has been assigned as proportional to the square root of the magnitude of the intensity. The converse relation holds true if one assumes $\sigma_{F_o^2}=1{\cdot}00$.

Interestingly these refinements gave essentially the same oxygen and chlorine parameters as counting-error weighting but slightly different R factors:

Unit-wt. on F_o:
$x_{\mathrm{Cl}}=0{\cdot}2409\pm0{\cdot}0006$ $R=0{\cdot}0559$
$z_{\mathrm{O}}=0{\cdot}1982\pm0{\cdot}0027$ Wtd. $R=0{\cdot}0448$

Unit-wt. on F_o^2:

$$x_{\text{Cl}} = 0{\cdot}2406 \pm 0{\cdot}0005 \qquad R = 0{\cdot}0600$$
$$z_{\text{O}} = 0{\cdot}1975 \pm 0{\cdot}0019 \qquad \text{Wtd. } R = 0{\cdot}0347\,.$$

However, the unit weighting of the F_o^2 or F_o is not considered the best approach because the standard deviations of I_0 must in reality vary over the range of magnitudes measured and the weighting scheme can seriously affect final results.

Oxygen contribution

Though the least-squares refinement had settled upon $z_{\text{O}} = 0{\cdot}1999$, it was not known how great an effect would be created by moving the oxygen atom and holding it fixed on either side of the minimum. This calculation was carried out starting with oxygen at 0·1908 and moving it by increments of 0·0023 to 0·2046. Aside from the fixed change imposed on oxygen the only variables which showed small changes were standard deviation of oxygen parameter, oxygen temperature factors, R, wtd. R and ESD of unit-wt. observation. There is presented in Fig. 2 a plot of the value of R and of wtd. R versus oxygen position. The minimum in wtd. R occurs at 1·745 Å from the origin while the minimum in R is lower, around 1·730 Å.

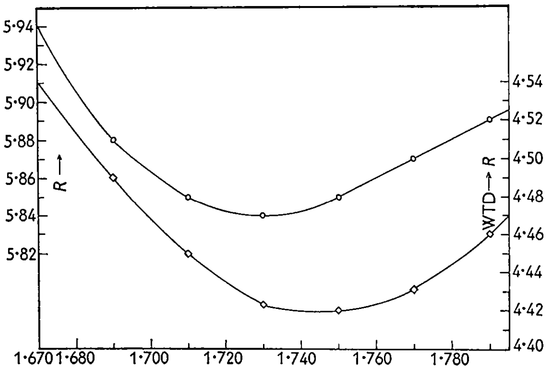

Fig. 2. Plot of variation in R and Wtd. R as the oxygen position is varied relative to the osmium atom.

○ = R plot. ◇ = Wtd. R plot.

In another cycle of refinement the oxygen contribution was held to zero to see if the absence of oxygen would have any effect on the other variables. Small changes did occur in x_{Cl} as well as in the temperature factors. The R and wtd. R shifted upward to

$$R = 0{\cdot}0837, \text{ wtd. } R = 0{\cdot}0986\,.$$

Discussion

The general features of the $K_2OsO_2Cl_4$ structure were well described by Hoard & Grenko (1934). In Fig. 3 is seen the osmyl tetrachloride ion in which is apparent the four-fold O–Os–O axis. At the time of H.&G.'s work, McCullough's (1936) report on K_2OsCl_6 had not appeared and the closest related structure was K_2PtCl_6. Both K_2OsCl_6 and K_2PtCl_6 have face-centered cubic CsF_2 type structures. H.&G. showed that $K_2OsO_2Cl_4$ could be considered as face-centered tetragonal with cell dimensions of $a_0 = 9{\cdot}90$ and $c_0 = 8{\cdot}75$ Å. This compares favorably with the cubic cell of 9·75 Å for the two hexachlorides.

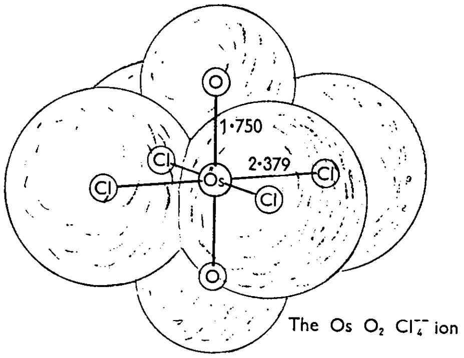

Fig. 3. The $OsO_2Cl_4^{--}$ complex ion. Bond lengths indicated are in Å.

Hepworth & Robinson (1957) have prepared and crystallographically examined $Cs_2RuO_2Cl_4$, Cs_2RuCl_6, $Cs_2OsO_2Cl_4$ and Cs_2OsCl_6. They find the cesium ruthenyl and osmyl chlorides to be cubic with $a_0 = 10{\cdot}07$ Å and the hexachlorides also cubic with $a_0 = 10{\cdot}22$ Å. They suggested that in the cesium compounds the oxygens of the linear O–Os–O group randomly occupy the three axial positions in the nearly octahedral $OsO_2Cl_4^{--}$ ion in contrast to the specific four-fold axis orientation in the potassium compound.

A packing diagram of the larger face-centered cell is included in H.&G.'s paper and there is a packing diagram of the body-centered tetragonal cell in Fig. 4. The pertinent interatomic distances in the refined structure are given in Table 4.

The potassium ion is coordinated to eight chlorines equidistant at 3·30 Å. There is weak K^+–O interaction over a distance of 3·55 Å. The only reported osmium–

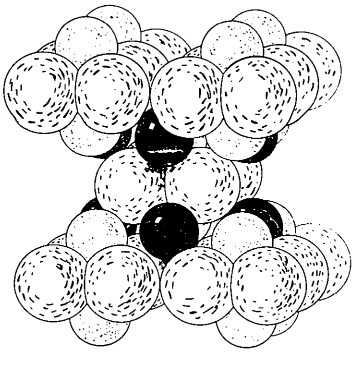

Fig. 4. Packing diagram of the $K_2OsO_2Cl_4$ structure. Black spheres are K^+, dotted spheres are O and flecked spheres are Cl atoms.

oxygen bond length is 1·66 Å in OsO_4 with a different type of bonding than in $K_2OsO_2Cl_4$. Pauling's (1960) covalent radii lead to an Os–O bond distance in the range 1·95 to 2·07 Å, larger than the 1·750 Å found here. From K_2OsCl_6 a value of 2·36 Å was obtained for the Os–Cl bond and Pauling (1960) predicts 2·10 to 2·32 Å. The value of 2·379 Å for Os–Cl in $K_2OsO_2Cl_4$ indicates a weaker binding which correlates with a stronger Os–O bond.

Table 4. *Pertinent interatomic distances*

A. Interatomic distances within the complex ion	
Os–O	1·750 ± 0·022 Å
Os–Cl	2·379 ± 0·005
O–Cl	2·950 ± 0·022
Cl–Cl	3·365 ± 0·006
B. Intermolecular contact distances	
K^+–O	3·524 ± 0·022 Å
K^+–Cl	3·303 ± 0·005
O_1–O_2	3·489 ± 0·025 (1)
O–Cl_2	3·675 ± 0·022
Cl_1–Cl_2	3·628 ± 0·006 (2)

(1) To oxygen in nearest neighbor $OsO_2Cl_4^{--}$ ion at body-centered position.
(2) Nearest neighbors in *xx* plane.

In a recent paper on the compound, $K_2OsO_2(OH)_4$, Symons & Lott (1960) discuss the probable structure of this material from crystal field and magnetochemical considerations and conclude that the Os–O bonds should be stronger and shorter than Os–OH (or, also, Os–Cl) bonds. We now have a measurement of the Os–O bond in which case the ligand is Cl. Analysis of the structure of $K_2OsO_2(OH)_4$ is currently being carried out in this laboratory. It will be interesting to have data from other osmyl compounds in order to establish a correlation between bond length and bond strength similar to the work done by Zachariasen (1954).

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Die Kristallstruktur des Kupferhydroxids, $Cu(OH)_2$

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The crystal structure of copper hydroxide $Cu(OH)_2$ has been determined by powder methods (Guinier films and counter goniometer measurements). The unit-cell dimensions are

$$a = 2{\cdot}949,\ b = 10{\cdot}59,\ c = 5{\cdot}256\ \text{Å};$$

space group $Cmcm(D_{2h}^{17})$. The structure is related to that of γ-FeOOH (lepidocrocite), EO_4 type. Chains of distorted octahedra form corrugated layers. The copper and each of the two oxygens of the formula occupy crystallographic positions of the same type, 4(*c*), with different values of the parameters.

In einer kurzen Mitteilung (Oswald & Jaggi, 1960) haben wir über die Elementarzelle des Kupferhydroxids berichtet, die nach der Methode de Wolff (1957) aus Guinier-Diagrammen ermittelt wurde. In der Zwischenzeit ist es uns gelungen, aus Aufnahmen mit dem Zählrohr-Goniometer die Struktur zu ermitteln.