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Short Communications

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The crystal structures of caesium, ammonium and potassium pertechnetates.

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The crystal structures of caesium, ammonium and potassium pertechnetates have been determined from powder photographs. The preparation of these compounds is reported by Boyd (1959). He states that the ammonium and potassium salts are isomorphous with the corresponding perrhenates and have the tetragonal $CaWO_4$ structure.

The structures of ammonium and potassium pertechnetate were confirmed as tetragonal, space group $I4_1/a$, $CaWO_4$ type. The lattice parameters were found to be $a_0 = 5{\cdot}790$, $c_0 = 13{\cdot}310$ Å for NH_4TcO_4 and $a_0 = 5{\cdot}654$, $c_0 = 13{\cdot}030$ Å for $KTcO_4$.

The structure of $CsTcO_4$ was found to be orthorhombic with lattice parameters

$$a_0 = 5{\cdot}718,\ b_0 = 5{\cdot}918,\ c_0 = 14{\cdot}304\ \text{Å}\ .$$

The space group is *Pnma* and the metal atoms are in special position (*c*) $\pm(x, \frac{1}{4}, z;\ x+\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-z)$ with $x = 0{\cdot}03$, $z = \frac{1}{8}$ for the 4 Cs ions and $x = 0{\cdot}97$, $z = \frac{5}{8}$ for the 4 Tc atoms (the Tc atom being at the centre of the tetrahedral TcO_4^- ion). The structure was found to be isomorphous with caesium perrhenate and caesium periodate. Table 1 shows the agreement between observed and calculated intensities for the first twenty lines of the pattern, neglecting the contribution of the oxygen atoms. The values of 0·03 and 0·97 for the *x* parameter are not very accurate because changes in the parameter have little effect on the calculated intensities. Table 2 illustrates the effect of varying *x* from 0 to 0·05 for Cs and from 0·95 to 1·0 for Tc, on the intensities of several lines.

Table 1. *Observed and calculated intensities for* $CsTcO_4$

hkl	*d* (obs.)	*d* (calc.)	*I* (obs.)	*I* (calc.)
111	3·959 Å	3·953 Å	15	0·28
004	3·576	3·576	100	100
113	3·121	3·127	5	5
020	2·959	2·959	60	62
200	2·859	2·859	50	49
022	2·736	2·734	5	0
121	2·582	2·585	10	1·4
203	2·460	2·459	1	0·09
115	2·353	2·348	5	3
024	2·285	2·280	60	60
204	2·236	2·233	50	49
116	2·067	2·062	80	86
221	2·038	2·035	5	6
125	1·937	1·935	1	0·65
223	1·893	1·891	1	0·09
026	1·850	1·856	1	0
132	1·804	1·804	50	56
224	1·787	1·783	50	49
312	1·758	1·759	40	38
313	1·697	1·698	5	7

We should like to thank J. R. Embleton for preparation of the compounds and useful discussions.

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Table 2. *Variation of line intensities with x parameter*

		I (calc.)					
hkl	*I* (obs.)	$x_{Cs} = 0$ $x_{Tc} = 1$	$x_{Cs} = 0{\cdot}01$ $x_{Tc} = 0{\cdot}99$	$x_{Cs} = 0{\cdot}02$ $x_{Tc} = 0{\cdot}98$	$x_{Cs} = 0{\cdot}03$ $x_{Tc} = 0{\cdot}97$	$x_{Cs} = 0{\cdot}04$ $x_{Tc} = 0{\cdot}96$	$x_{Cs} = 0{\cdot}05$ $x_{Tc} = 0{\cdot}95$
116	80	89	89	88	86	84	81
132	50	58	58	57	56	55	53
224	50	56	55	53	49	43	38
200	50	56	55	53	49	43	37
312	40	54	52	46	38	29	19
313	5	0	0·85	3	7	11	16