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# CHEMISTRY

E. G. IPPOLITOV and P. A. KOZ' MIN

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**Abstract****Full Text**

# CHEMISTRY

**E. G. IPPOLITOV and P. A. KOZ' MIN**# X-RAY STUDY OF POTASSIUM AND RUBIDIUM OCTAFLUORORHEN-  
ATES*(Presented by Academician I. V. Tananaev, 24 VIII 1961)*

Potassium and rubidium octafluororhenates, recently synthesized by Nikolaev and Ippolitov <sup>(1)</sup>, proved sufficiently stable in the open air to permit a detailed X-ray study of them. X-ray spectra of these salts were obtained by the powder method, as well as oscillation radiographs and reciprocal-lattice radiographs.

(Figure: Fig. 1)

**Fig. 1**

To obtain X-ray spectra of potassium and rubidium octafluororhenates with filtered iron radiation, a URS-50-I ionization X-ray apparatus was used. Complete reproducibility of the spectra was observed on repeated exposures of the same specimen. The X-ray spectra obtained are shown in Fig. 1. The intensity of the reflections was determined by measuring the areas of the peaks, the intensity of the strongest reflection being taken as 100.

In the cited work <sup>(1)</sup> a method was also described for obtaining coarsely crystalline potassium octafluororhenate, containing crystals up to 0.2 mm in size. These crystals were stable for a week in the open air. By contrast, in the isolated state rapid decomposition occurred as a result of the catalytic influence of traces of hydrogen fluoride, which decomposes octafluororhenate with liberation of rhenium hexafluoride <sup>(2)</sup>. Owing to the very high stability of single crystals in the open air, it was possible to obtain oscillation radiographs along three mutually perpendicular directions and reciprocal-lattice radiographs along two directions of the zero, first, and second layer lines. The oscillation and reciprocal-lattice radiographs were taken repeatedly on different crystals using unfiltered copper radiation.

Proceeding from the symmetry of the rotation and reciprocal-lattice radiographs and from the extinction conditions determined from the reciprocal-lattice radiographs, the following space groups are possible:  $D_{2h}^{16} = Pnma$  and  $C_{2v}^9 = Pn2a$ . The periods of the unit cell of potassium octafluororhenate were determined from rotation radiographs (Fig. 2) and were found to be  $a = 8.70$  kX,

**Table 1****Results of indexing the X-ray spectra**

$K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$   $K_2RdF_8$

	intensity <sup>2</sup> $\theta hkl$	$\sin^2 \theta$	intensity <sup>2</sup> $\theta hkl$	$\sin^2 \theta$	intensity <sup>2</sup> $\theta hkl$	$\sin^2 \theta$	intensity <sup>2</sup> $\theta hkl$	$\sin^2 \theta$
	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.
3	0.019001	0.01824	0.030011	0.03072	0.325306	0.32566	0.322233	0.3210
17	0.033011	0.032700	0.032302	0.03353	0.330422	0.32846	0.331512	0.3296
100	0.036102	0.03639	0.041411	0.0419	117	0.3309	423	0.3310
21	0.045411	0.04509	0.044700	0.04527	0.334126	0.3337	503	0.3322
16	0.049200	0.04943	0.048701	0.05073	0.340207	0.3413	331	0.3331
4	0.056201	0.05545	0.060212	0.05865	0.358331	0.35777	0.339325	0.3408
7	0.065212	0.06296	0.062303	0.0612	423	0.35822	0.345415	0.3447
3	0.067603	0.06603	0.067202	0.0674	512	0.3594	226	0.3455
3	0.073202	0.07323	0.070210	0.07034	0.369325	0.36709	0.354008	0.3554
9	0.075810	0.07686	0.073013	0.0751	217	0.36805	0.367108	0.2484
22	0.080713	0.08068	0.075211	0.07582	0.387234	0.3853	035	0.6645
20	0.081811	0.08269	0.086813	0.08635	0.400424	0.3999	424	0.3669
13	0.092913	0.09274	0.088704	0.0888	135	0.40186	0.380333	0.3775
10	0.095004	0.09530	0.094803	0.09519	0.420118	0.4203	406	0.3380
7	0.103203	0.10306	0.100104	0.1001	521	0.42174	0.395514	0.3962
6	0.108020	0.1069	020	0.10042	0.439430	0.43820	0.403208	0.3400
	104	0.10775	0.111301	0.1072	416	0.4388	040	0.4401
6	0.130213	0.12976	0.120213	0.1202	235	0.4389	326	0.0416
	022	0.13075	0.125114	0.1252	522	0.4396	416	0.4558
5	0.135114	0.1344	302	0.12382	0.457505	0.4577	522	0.4049
	302	0.13504	0.130311	0.1323	218	0.45734	0.413601	0.4120
16	0.144122	0.14330	0.133122	0.13394	0.469602	0.4685	431	0.4121
	311	0.1439	204	0.1340	610	0.4714	334	0.4163
	204	0.14476	0.147220	0.1456	523	0.46931	0.445515	0.4461
2	0.157220	0.15630	0.158214	0.15914	0.489407	0.4895	240	0.4468
2	0.161105	0.16133	0.162123	0.1616	028	0.48816	0.476327	0.4770
	312	0.1617	015	0.16406	0.510019	0.5092	019	0.4747
	221	0.16239	0.170222	0.1678	327	0.5099	417	0.4777
3	0.167303	0.16484	0.173115	0.1751	524	0.51007	0.482426	0.4808
9	0.173123	0.17294	0.176313	0.17673	0.522506	0.5232	613	0.4815
	214	0.17147	0.185205	0.1839	119	0.5215	318	0.4819
4	0.180222	0.1802	401	0.1862	044	0.5229	506	0.4821
7	0.187115	0.18801	0.191024	0.18924	0.537228	0.53749	0.488119	0.4859
9	0.203024	0.2022	304	0.1904	144	0.5353	044	0.4904
	401	0.20367	0.198223	0.19552	0.548516	0.55008	0.506137	0.509
12	0.226106	0.2268	006	0.19983	0.565614	0.5667	516	0.507
7	0.242322	0.24192	0.210215	0.2090	525	0.5646	620	0.507
10	0.252403	0.2513	106	0.2111	336	0.56613	0.517614	0.5204
	224	0.25160	0.228322	0.22428	0.603506	0.6007	219	0.520
	116	0.25330	0.235224	0.2344	129	0.6017	531	0.514
5	0.270125	0.26829	0.245206	0.2450	623	0.60526	0.537244	0.5356
	323	0.2717	131	0.2427	533	0.6030	408	0.5358

$K_2ReF_8$ $Rb_2ReF_8$ $Rb_2ReF_8$ $ReF_8$								
3	0.277132	0.27675	0.255413	0.2557		3	0.544237	0.5430
	413	0.27803	0.269404	0.2694			605	0.5452
3	0.287315	0.2868	216	0.2701			435	0.5453
1	0.292230	0.29000	0.274315	0.2655		4	0.558623	0.5568
	216	0.29067	0.279033	0.2759			329	0.5572
	404	0.29298	0.288420	0.2810			701	0.5588
5	0.296033	0.2942	421	0.2866			418	0.5609
	231	0.2959	133	0.2871		13	0.572615	0.5703
6	0.305107	0.3042	501	0.2878				
	420	0.3046	324	0.2908				
	225	0.30526	0.297414	0.2945				
	133	0.3085	017	0.2971				
5	0.318017	0.31868	0.310117	0.3083				
	414	0.31927	0.313511	0.3129				
			126	0.3115				

$b = 5.91$  kX,  $c = 12.53$  kX. These data confirm the results of optical observations in polarized light, which showed that the crystal system of the crystals is rhombic <sup>(2)</sup>. On the basis of the calculated unit-cell periods of potassium octafluororhenate and the possible space groups, the spectra of  $K_2ReF_8$  were indexed within the range from 0 to 50° of the angle  $\theta$ . The indexing results are given in Table 1.

The external appearance of the X-ray spectrum of rubidium octafluororhenate indicated the probable isomorphism of this salt with potassium octafluororhenate. Proceeding

(Figure: Fig. 2)

**Fig. 2**

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From this assumption, the unit-cell periods of  $Rb_2ReF_8$  were calculated from the most intense and characteristic reflections. As was to be expected, the periods increased and became  $a = 9.10$  kX,  $b = 6.10$  kX,  $c = 12.97$  kX. Using these values of the periods, all the remaining reflections of the spectrum of rubidium octafluororhenate were indexed. The results of the indexing are given in Table 1.

Since the atomic factor of rubidium is twice as large as the atomic factor of potassium, the intensities of reflections with identical  $hkl$  for the rubidium and potassium octafluororhenates may differ greatly. Therefore some weak reflections present in the spectrum of the potassium salt are not observed in the spectrum of the rubidium salt, and conversely. This becomes especially noticeable beginning with the angle  $\theta = 30^\circ$ . It is possible that the unequal increase

of all the unit-cell parameters of rubidium octafluororhenate also plays a role here.

**Table 2**

**X-ray characteristics of potassium and rubidium octafluororhenates**

Formula	Crystal system	Fedorov group	Unit-cell periods in kX	Density, calculated, g/cm <sup>3</sup>	Density, determined, g/cm <sup>3</sup>	Number of molecules in the cell
K <sub>2</sub> ReF <sub>8</sub>	Orthorhombic	$D_{2h}^{16}$ $C_{2v}^9$	$Pnma$ $\frac{a}{8.70}$ $b =$ $5.91c =$ $12.53$	4.29	4.33 ± 0.03	4
Rb <sub>2</sub> ReF <sub>8</sub>	Orthorhombic	the same	$a =$ $9.10b =$ $6.10c =$ $12.97$	4.70	4.45 ± 0.04	4

On the basis of the obtained periods of the elementary-cell lattice, the densities of both salts were calculated. In Table 2 the results of these calculations are compared with the data obtained pycnometrically.

The results of the X-ray investigations confirm that potassium and rubidium octafluororhenates are individual compounds, isomorphous with one another. The investigation of the structure of potassium octafluororhenate is continuing.

Institute of General and Inorganic Chemistry  
named after N. S. Kurnakov  
Academy of Sciences of the USSR

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16 VIII 1961

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2. N. S. Nikolaev, E. G. Ippolitov, DAN, **136**, No. 1, 111 (1961).

*Note: Figure translations are in progress. See original paper for figures.*

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