Title
THE CRYSTAL STRUCTURE OF POTASSIUM BROMORHENATE

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D. H. Templeton and Carol H. Dauben

November 16, 1950

Berkeley, California

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THE CRYSTAL STRUCTURE OF POTASSIUM BROMORHENATE

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November 16, 1950

ABSTRACT

K_2ReBr_6 has a face-centered cubic lattice with $\underline{a} = 10.445 \pm 0.005$ Å. It is isostructural with K_2PtCl_6. The parameter for the bromine atoms has been determined to be $x = 0.242 \pm 0.004$, corresponding to a Re-Br distance of 2.53 Å.

THE CRYSTAL STRUCTURE OF POTASSIUM BROMORHENATE

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The compound K_2ReBr_6 seems to have been first prepared by Krauss and Steinfeld[1] who reported no structure determination. We have shown by x-ray diffraction that its structure is the K_2PtCl_6 type,[2] as are those of K_2ReCl_6[3] and K_2ReF_6.[4]

(1) F. Krauss and H. Steinfeld, Ber., 64, 2552 (1931).

(2) J 1_1 type, Strukturbericht, 1, 429 (1937); 3, 121 (1937).

(3) B. Aminoff, Z. Kristallographie, 94, 246 (1936).

(4) O. Ruff and W. Kwasnik, Z. anorg. Chem., 219, 78 (1934).

Dr. Z Zimmerman Hugus of this laboratory prepared single crystals of potassium bromorhenate by dissolving ReO_2 in hydrobromic acid and crystallizing the potassium salt. These crystals were well formed cubes, very dark red in color.

The powder diffraction pattern, taken with copper Kα x-rays (λ = 1.5418 Å) correspond to a face-centered cubic lattice with a = 10.445 ± 0.005 Å. With four molecules to the unit cell, the density is calculated to be 4.34 g/cm^{-3}. The intensities of most of the reflections were estimated visually from a rotation photograph taken about the [100] axis. Weissenberg photographs were used to obtain several reflections absent or not resolved in the rotation pattern. The magnitudes of 95 independent structure factors (all that are permitted by the space group up to $h^2 + k^2 + l^2 = 180$) were calculated from these data after correction for Lorenz, polarization, and velocity factors.

No correction was made for absorption other than to average the results for equivalent reflections.

Preliminary calculations showed these data to be consistent with the structure:

Space group O_h^5 -- Fm3m; Z = 4

4 Re in 4(a): (000) + F. C.

8 K in 8(c): $\pm(\frac{111}{444})$ + F. C.

24 Br in 24(e): $\pm$(x00; 0x0; 00x) + F. C.

with the parameter x approximately 0.25. Signs of the structure factors were calculated on this basis; only seven were negative. An optical Fourier summation of the electron density made according to Huggins[5] for a section

(5) M. L. Huggins, J. Am. Chem. Soc., 63, 66 (1941).

through a rhenium atom and four bromine atoms showed the series to be well convergent with well-resolved peaks for the atoms. A numerical summation of the same series along the edge of the unit cell showed the bromine atom to be at x = 0.242 $\pm$ 0.004. The probable error was estimated from the rate of change of a few structure factors of large indices with change in x.

The percentage discrepancy, $100\ (\Sigma\,|\,|F_{obs}|-|F_{calc}|\,|)/\Sigma|F_{obs}|$, is 25% for this value of the parameter x, which is as good as was expected because of the crude visual estimates and the neglect of absorption corrections. In view of the simplicity of the structure, further refinement of the observations was not deemed necessary.

The bromine-rhenium distance in the bromorhenate ion is 2.53 $\pm$ 0.04 Å. If the covalent radius of bromine is taken[6] as 1.14 Å, the octahedral radius

(6) L. Pauling, "Nature of the Chemical Bond," Cornell University Press, Ithaca, New York, 1942, p. 165.

of rhenium is 1.39 Å, in good agreement with the value 1.38 Å calculated in the same way from K_2ReCl_6.

We wish to thank Dr. Hugus for making available the crystals. This research was supported by the U. S. Atomic Energy Commission.

APPENDIX

As the crystal is face-centered, it is possible to sum over half the face with indices of the body-centered tetragonal cell, thereby allowing the use of more terms than would otherwise be possible with the Huggins masks. The axes were shifted to place the rhenium at the center of the base. The resulting optical summation is shown in Fig. 1.

Fig. 2 shows the result of the numerical summation, along a cell edge from one rhenium to another.

In Table I are tabulated the observed magnitudes of the structure factors, and the values calculated for $x = 0.242$.

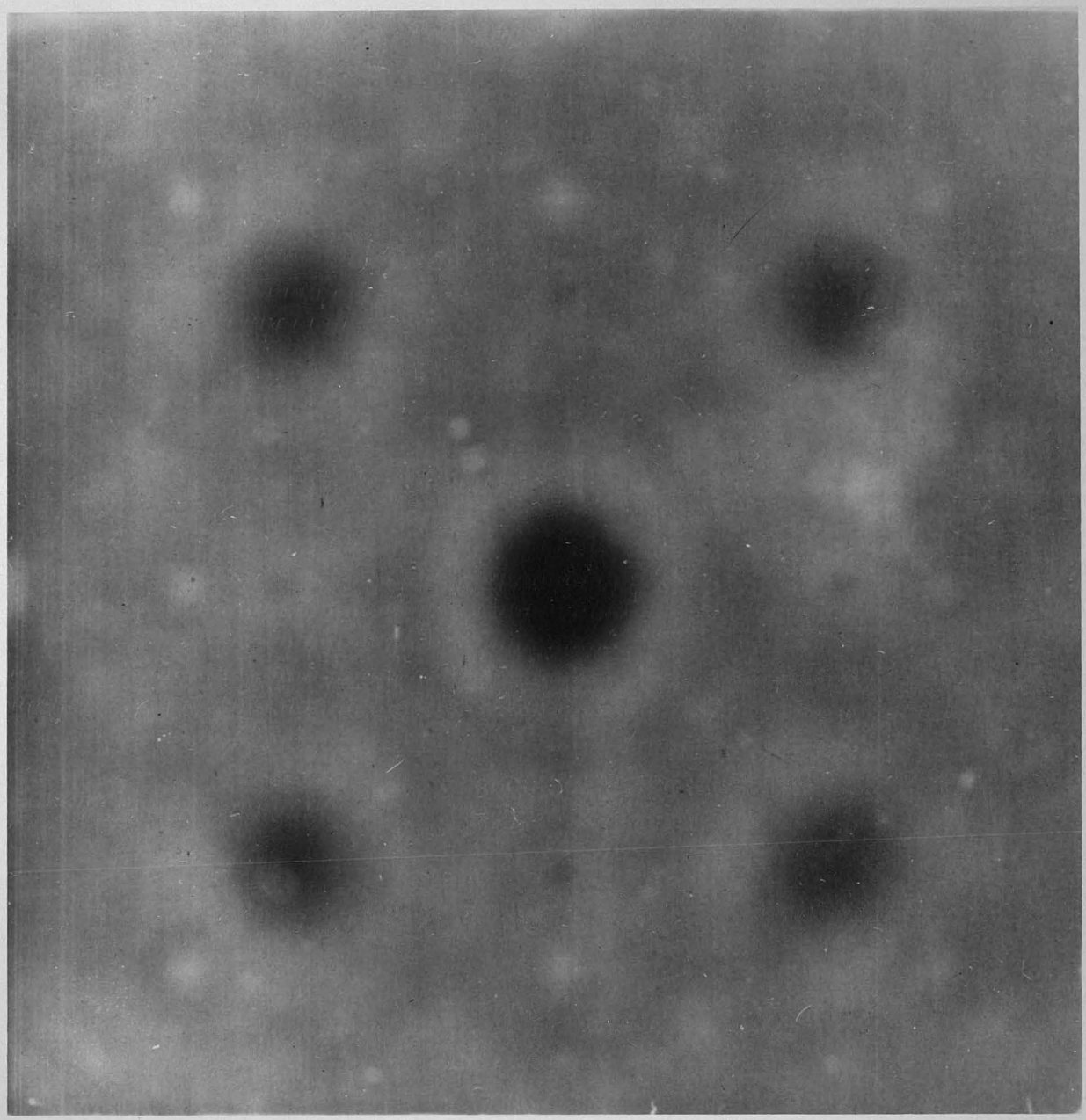

FIG. 1

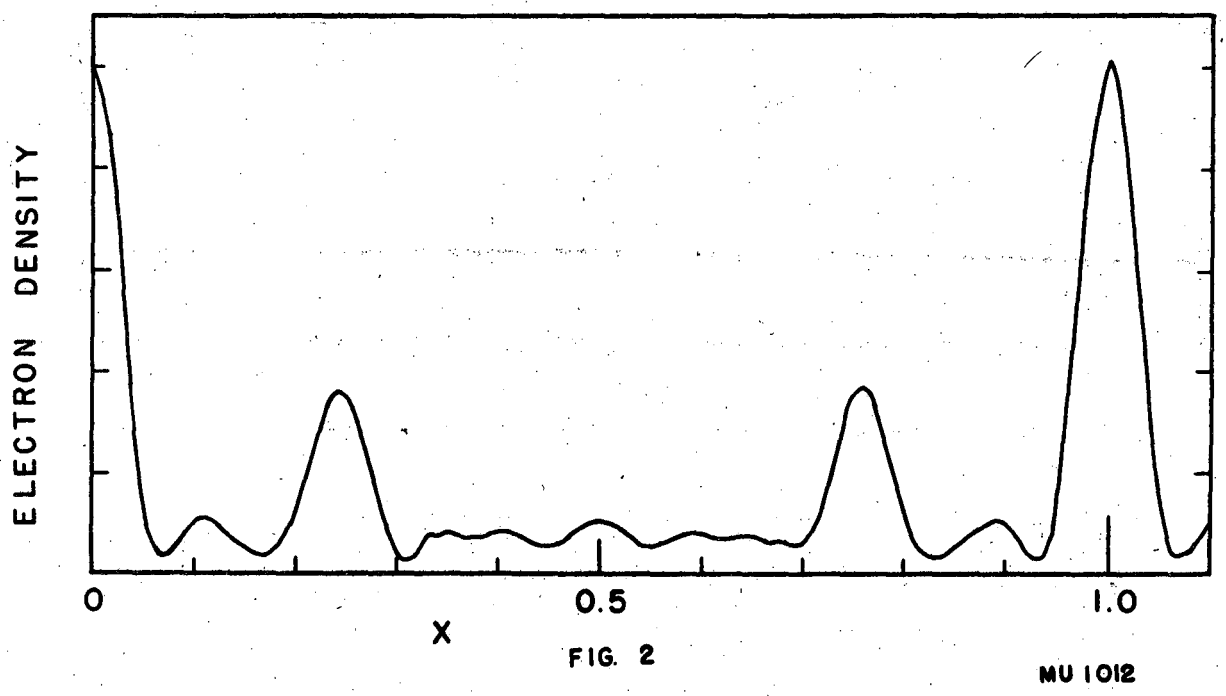

FIG. 2

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Table I

Observed and Calculated Values of the Structure Factor

$h_2^2 + k^2 + l^2$	h,k,l	F_{obs}	$F_{calc.}$
3	111	26	32
4	200	43	52
8	220	9	15
11	311	23	25
12	222	37	-53
16	400	74	100
19	331	28	30
20	420	34	34
24	422	13	13
27	511	36	30
	333	17	14
32	440	59	86
35	531	30	25
36	600	42	32
	442	40	30
40	620	6	13
43	533	22	20
44	622	43	-38
48	444	61	78
51	711	23	16
	551	32	29
52	640	39	29
56	642	11	12
59	731	16	12

Table I (Cont.)

$h_2^2 + k^2 + l^2$	h,k,l	F_{obs}	$F_{calc.}$
59	553	30	25
64	800	69	72
67	733	12	9
68	820	45	27
	644	42	27
72	822	13	10
	660	0	12
75	751	30	18
	555	32	30
76	662	26	-32
80	840	58	67
83	911	39	26
	753	19	14
84	842	34	24
88	664	8	11
91	931	32	22
96	844	46	64
99	933	31	19
	771	7	8
	755	20	19
100	10,0,0	33	26
	8,6,0	27	24
104	10,2,0	13	11
	8,6,2	9	9

Table I (Cont.)

$h_2^2 + k^2 + l^2$	h,k,l	F_{obs}	$F_{calc.}$
107	9,5,1	34	26
	7,7,3	8	5
108	10,2,2	29	-28
	6,6,6	23	-28
115	9,5,3	28	23
116	10,4,0	27	24
	8,6,4	24	22
120	10,4,2	10	11
123	11,1,1	12	10
	7,7,5	16	18
128	8,8,0	34	58
131	11,3,1	7	8
	9,7,1	19	18
	9,5,5	29	28
132	10,4,4	21	23
	8,8,2	33	20
136	10,6,0	11	11
	8,6,6	8	9
139	11,3,3	7	5
	9,7,3	20	15
140	10,6,2	10	-25
144	12,0,0	43	56
	8,8,4	47	56
147	11,5,1	11	18
	7,7,7	0	3

Table I (Cont.)

$h_2^2 + k^2 + l^2$	h,k,l	F_{obs}	$F_{calc.}$
148	12,2,0	27	20
152	12,2,2	14	7
	10,6,4	9	11
155	11,5,3	9	10
	9,7,5	20	21
160	12,4,0	38	53
163	9,9,1	30	26
164	12,4,2	26	19
	10,8,0	26	21
	8,8,6	27	20
168	10,8,2	12	9
171	13,1,1	25	23
	11,7,1	5	5
	11,5,5	13	14
	9,9,3	27	23
172	10,6,6	11	-23
176	12,4,4	31	51
179	13,3,1	26	20
	11,7,3	5	3
	9,7,7	16	11
180	12,6,0	20	18
	10,8,4	24	20