

## The Crystal Structure of Rubidium Dichromate, $\text{Rb}_2\text{Cr}_2\text{O}_7$ ( $C2/c$ )

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Rubidium dichromate,  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ), is isostructural with ammonium dichromate,  $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ . The structure parameters have been refined by full matrix least squares refinement with the application of a three-dimensional set of X-ray data obtained with use of a single crystal and Ni-filtered  $\text{CuK}\alpha$  radiation. The structure is monoclinic,  $a = 13.332 \pm 2$ ,  $b = 7.552 \pm 1$ ,  $c = 7.734 \pm 1$  Å,  $\beta = 92.04 \pm .01^\circ$ ,  $V = 778.1$  Å<sup>3</sup>, space group  $C2/c$ . The unit cell contains four formula units of  $\text{Rb}_2\text{Cr}_2\text{O}_7$ . The final  $R$  index is 0.083. The Cr-O (terminal) distances range from 1.609 to  $1.627 \pm 14$  Å; the Cr-O (bridging) distance is  $1.774 \pm 10$  Å. The O-Cr-O bond angles at the chromium atoms range from 108.1 to  $112.1 \pm .8^\circ$ ; the Cr-O-Cr bond angle at the bridging atom is  $122.9 \pm 1.2^\circ$ .

In 1951 the crystal structure of  $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ , space group  $C2/c$ , was determined by Byström and Wilhelmi.<sup>1</sup> This was the first structure determination of a compound with the stoichiometric formula  $A_2M_2O_7$  in which the  $A$  atom is a large cation (alkali or alkaline earth metal) and  $M$  is a small group V or VI atom in a high formal oxidation state. Structures of this type of compound contain  $M_2O_7$  groups in which two  $MO_4$  tetrahedra share one corner. Since then several new types of  $A_2M_2O_7$  structures have been reported. Different packings of the  $M_2O_7$  units give rise to several modifications. In a recent paper Brown and Calvo<sup>2</sup> have outlined a scheme to visualize the relation between the different structure types.

Rubidium dichromate seems to exist in at least four different forms: (1) one quenchable high temperature form of unknown structure, (2) one triclinic ( $P\bar{1}$ ), (3) a monoclinic form of space group  $P2_1/n$ , and (4) a monoclinic form of space group  $C2/c$ . The crystal structures of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $P\bar{1}$ )<sup>3</sup> and  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $P2_1/n$ )<sup>4</sup> have been determined recently. This paper reports on a refinement of the  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ) structure.

## EXPERIMENTAL

The starting materials used were chromium trioxide ("Analar", Analytical Reagent, Hopkin and Williams Ltd.) and rubidium carbonate (Merck, analytical grade). Solutions of rubidium dichromate were prepared by slow addition of rubidium carbonate to hot solutions of chromium trioxide in water. (Mole ratio  $\text{Rb}_2\text{CO}_3:\text{CrO}_3 = 1:3$ .) Crystals were obtained by slow evaporation of the solvent at room temperature. The major product was  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $P2_1/n$ ) but variable, minor amounts of the triclinic and the new monoclinic form,  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ) were also present.

The single crystal used for collecting Weissenberg data, was selected from a sample, containing an excess of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $P2_1/n$ ). The orange-red crystal had the following approximate dimensions: 0.08 (along the unique ( $b$ ) axis)  $\times$  0.07  $\times$  0.01 mm<sup>3</sup>. The photographs  $h0l-h4l$  were taken with  $\text{CuK}\alpha$  radiation and multiple-film technique (3 films). Intensities were estimated visually for 455 symmetrically independent reflections by comparison with a calibrated scale. Lorentz and polarization effects were accounted for. Correction for absorption was made by applying the linear absorption coefficient  $\mu = 402.2$  cm<sup>-1</sup>. Mass absorption coefficients were taken from Ref. 5 and the scattering factors for the neutral atoms from Ref. 6.

Several methods were tried to crystallize the  $C2/c$  modification as a single phase from water solutions by varying the pH, temperature, initial Rb:Cr ratio as well as adding minor amounts of ammonium dichromate to a saturated rubidium dichromate solution. The last method gave a fairly pure product of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ). The powder pattern obtained with a Hägg-Guinier camera ( $\text{CuK}\alpha$ , (20°C) = 1.54050 Å) indicated the presence of this modification. No extra lines were observable. Potassium chloride,  $a$  (20°C) = 6.2919 Å was used as an internal standard for the derivation of lattice parameters.

The following cell dimensions were derived:  $a = 13.332 \pm 2$  Å,  $b = 7.552 \pm 1$  Å,  $c = 7.734 \pm 1$  Å,  $\beta = 92.04 \pm .01^\circ$ ,  $V = 778.1$  Å<sup>3</sup>.

Observed and calculated  $\sin^2\theta$  values and intensities are given in Table 1.

Table 1. Powder pattern of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ). Reflections systematically absent in space group  $C2/c$  have been omitted. The intensities were calculated from the expression  $P(F_c)^2/1000$ , where  $P$  is the number of equivalent reflections.

$hkl$	$10^3 \sin^2\theta_{\text{obs}}$	$10^3 \sin^2\theta_{\text{calc}}$	$I_{\text{obs}}$	$I_{\text{calc}}$
2 0 0		01337		0
1 1 0		01375		0
1 1 $\bar{1}$		02327		0
1 1 1	02408	02410	vw	11
0 0 2	03971	03974	w	22
3 1 0	04047	04050	w	28
0 2 0	04169	04163	m	62
3 1 $\bar{1}$	04922	04920	st	81
2 0 $\bar{2}$	05160	05147}	vvst	244}
0 2 1		05157}		320}
3 1 1		05166		0
1 1 $\bar{2}$		05267		2
4 0 0	05344	05349	vst	248
1 1 2		05431		3
2 0 2	05475	05475	vvst	284
2 2 0		05501		3
2 2 $\bar{1}$	06412	06412	vst	157
2 2 1	06579	06576	st	127
3 1 $\bar{2}$	07777	07777	m	48
0 2 2	08139	08137	vvw	14
3 1 $\bar{2}$	08266	08270	w	34
4 0 $\bar{2}$	08994	08994	vw	30

Table 1. Continued.

2 2 $\bar{2}$		09310		5
5 1 0		09309		0
4 2 0	09512	09512	m	96
2 2 2	09630	09638	w	117
4 0 2		09651		8
1 3 0	09697	09702	vw	30
5 1 $\bar{1}$	10187	10187	w	28
1 1 $\bar{3}$		10192		34
4 2 $\bar{1}$	10339	10341	st	224
1 1 3	10435	10439	w	45
5 1 $\bar{1}$	10600	10598	m	125
1 3 $\bar{1}$		10654		9
4 2 1	10673	10670	w	71
1 3 1		10736		1
6 0 0		12035		17
3 3 0	12379	12376	st	95
3 1 $\bar{3}$		12620		5
5 1 2		12961		18
0 2 3	13101	13104	vw	41
4 2 $\bar{2}$		13157		4
3 3 $\bar{1}$		13246		5
3 1 3		13360		4
3 3 $\bar{1}$	13492	13493	w	53
1 3 $\bar{2}$		13593		20
1 3 2		13757		21
5 1 2		13783		33
4 2 $\bar{2}$	13815	13815	vw	49
2 2 $\bar{3}$	14193	14194	st	334
2 2 $\bar{3}$	14686	14688	m	240
6 0 $\bar{2}$	15510	15516	vw	30
0 0 4	15893	15894	w	56
3 3 $\bar{2}$	16095	16103	vw	40
6 2 0		16199		6
6 0 2	16502	16502	m	115
3 3 2		16596		0
0 4 0	16641	16653	vwvw	44
2 0 4	16908	16903	w	41
6 2 $\bar{1}$		16945		32
1 1 4		17105		8
7 1 0		17422		35
1 1 4	17432	17434	m	18
6 2 1		17438		88
2 0 4		17560		14
0 4 1	17637	17646	w	62
5 1 $\bar{3}$	17722	17723	m	108
5 3 0		17725		41
4 2 $\bar{3}$		17960		26
2 4 0		17990		0

Addition of ammonium dichromate in amounts varying from 10 to 50 mol% did not give rise to a detectable variation of the cell dimensions of the rubidium dichromate thus indicating no observable solubility of ammonium dichromate in rubidium dichromate. According to Ref. 7 the solubility of  $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$  and  $\text{Rb}_2\text{Cr}_2\text{O}_7$  (monocl.) in 100 g water is 30.8 g (15°C) and 5.42 g (18°C), respectively.

The cell parameters as well as all subsequent calculations were carried out on the IBM 360/75 computer with the application of the programs given in Table 2.

Table 2. Computer programs used for the crystallographic calculations. All programs are written in FORTRAN IV.

Program name and function. Computer.	Authors.
1. DATAP2. Lp- and absorption corrections. Preparative calculations for extinction correction according to Zachariasen's 1963-formula. IBM 360/75.	P. Coppens, L. Leiserowitz and D. Rabinovich, Rehovoth, Israel. Modified by O. Olofsson and M. Elfström, Uppsala, Sweden. Inclusion of calculations for extinction correction by B. G. Brandt and S. Åsbrink, Stockholm, Sweden. Further modifications by B. G. Brandt and A. G. Nord, Stockholm, Sweden.
2. DRF. Fourier summations and structure factor calculations. IBM 360/75.	A. Zalkin, Berkeley, USA. Modified by R. Liminga and J.-O. Lundgren, Uppsala, Sweden. Further modified by O. Lindgren, Göteborg and by A. G. Nord and B. G. Brandt, Stockholm, Sweden.
3. LALS. Full matrix least squares refinement of positional and thermal parameters and of scale factors. IBM 360/75.	P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Los Angeles, USA. Modified by A. Zalkin, Berkeley, USA and by J.-O. Lundgren, R. Liminga and C.-I. Brändén, Uppsala, Sweden. Further modified by O. Lindgren, Göteborg and by B. G. Brandt and A. G. Nord, Stockholm, Sweden.
4. DISTAN. Calculation of interatomic distances and bond angles with estimated standard deviations. IBM 360/75.	A. Zalkin, Berkeley, USA. Modified by A. G. Nord and B. G. Brandt, Stockholm, Sweden.
5. DATA. Reflexion data handling including storing on disk, correction of erroneous reflexions or inclusion of new ones in a data set stored on disk; index transformation. IBM 360/75.	B. G. Brandt, Stockholm, Sweden.
6. LIST. Editing of structure factor tables. IBM 360/75.	I. Carlbom, Stockholm, Sweden.
7. POWDER. Generation of $\sin^2\theta$ values. Indexing of powder lines from preliminary cell constants. Refinement of cell constants. IBM 360/75.	O. Lindqvist and F. Wengelin, Göteborg, Sweden. Modified by B. G. Brandt and A. G. Nord, Stockholm, Sweden.

#### REFINEMENT AND DESCRIPTION OF THE STRUCTURE

On the Weissenberg photographs the following reflections were missing:

$$\begin{aligned} hkl & \text{ for } h+k=2n+1 \\ h0l & \text{ for } l=2n+1 \end{aligned}$$

which is characteristic for space groups  $Cc$  (No. 9) and  $C2/c$  (No. 15). The similarity in the cell dimensions and the possible space groups indicated the

Table 3. Weight analysis used in the last cycle of the refinement of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  (C2/c).  
 $w$  = weighting factor.  $\Delta = |F_o| - |F_c|$ .

Interval $\sin \theta$	$\overline{w\Delta^2}$	Number of independent reflections	Interval $F_o$	$\overline{w\Delta^2}$	Number of independent reflections
0.00–0.46	0.97	89	0–16	2.08	43
0.46–0.58	0.47	71	16–22	1.32	45
0.58–0.67	0.85	63	22–30	1.67	45
0.67–0.74	1.15	54	30–38	1.14	46
0.74–0.79	0.93	35	38–46	0.92	45
0.79–0.84	0.94	46	46–58	0.58	46
0.84–0.89	1.32	26	58–73	0.45	45
0.89–0.93	1.65	35	73–97	0.53	46
0.93–0.97	1.29	26	97–123	0.86	45
0.97–1.00	2.24	7	123–358	0.55	46

Table 4. The crystal structure of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  (C2/c).

Space group: C2/c (No. 15).

Unit cell dimensions:

$a = 13.332 \pm 2 \text{ \AA}$ ,  $b = 7.552 \pm 1 \text{ \AA}$ ,  $c = 7.734 \pm 1 \text{ \AA}$ ,  $\beta = 92.04 \pm .01^\circ$ ,  $V = 778.1 \text{ \AA}^3$ .

Cell content: 4  $\text{Rb}_2\text{Cr}_2\text{O}_7$ .

8 Rb, 8 Cr, 8 O(1)–8 O(3) in 8(f):  $\pm(x, y, z; \frac{1}{2} + x, \frac{1}{2} + y, z; \bar{x}, y, \frac{1}{2} - z; \frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$

4 O(1) in 4(e):  $0, y, \frac{1}{4}; 0, \bar{y}, \frac{3}{4}; \frac{1}{2}, \frac{1}{2} + y, \frac{1}{4}; \frac{1}{2}, \frac{1}{2} - y, \frac{3}{4}$

Final coordinates, isotropic temperature factors, and standard deviations resulting from the least-squares refinement.

Atom	$x \pm \sigma(x)$	$y \pm \sigma(y)$	$z \pm \sigma(z)$	$B \pm \sigma(B) \text{ \AA}^2$
Rb	0.3561 $\pm$ 1	0.6527 $\pm$ 3	0.6532 $\pm$ 2	2.69 $\pm$ 6
Cr	0.3988 $\pm$ 2	0.1734 $\pm$ 4	0.6428 $\pm$ 3	2.07 $\pm$ 7
O(1)	0.4421 $\pm$ 8	0.3052 $\pm$ 19	0.4986 $\pm$ 13	2.93 $\pm$ 20
O(2)	0.3220 $\pm$ 9	0.0271 $\pm$ 20	0.5582 $\pm$ 15	4.09 $\pm$ 25
O(3)	0.3424 $\pm$ 8	0.2902 $\pm$ 18	0.7837 $\pm$ 13	2.96 $\pm$ 20
O(4)	1/2	0.0612 $\pm$ 28	3/4	2.93 $\pm$ 26

compound to be isostructural with  $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ .<sup>1</sup> Thus, the structure refinement was started with the assumption that the space group was C2/c, and the coordinates given in Ref. 1 were used as a starting point for the subsequent work. These were converted to correspond to the same origin of coordinates used in the papers describing the isostructural  $\text{K}_2\text{S}_2\text{O}_7$ ,<sup>8</sup>  $\text{K}_2\text{HN}(\text{SO}_3)_2$ ,<sup>9</sup> and  $\text{K}_2\text{H}_2\text{C}(\text{SO}_3)_2$ .<sup>10</sup> The transformation is  $x = 1/2 - x'$ ,  $y = y'$ ,  $z = 1/2 + z'$  where  $x'$ ,  $y'$ , and  $z'$  are the coordinates given in Ref. 1. Thus the following values were used:

	$x$	$y$	$z$
Rb	0.328	0.667	0.667
Cr	0.400	0.175	0.639
O(1)	0.450	0.300	0.500
O(2)	0.322	0.000	0.570
O(3)	0.356	0.311	0.778
O(4)	1/2	0.042	3/4



Table 5. Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	3	-7	21.2	-16.4	14	4	1	15.5	16.2	2	4	-3	174.8	-166.7	8	4	-5	25.2	-26.1
5	3	-7	19.6	18.4	2	4	-1	86.6	93.8	4	4	-3	59.9	-53.5	10	4	-5	0.0 *	-1.0
7	3	-7	14.9	-13.1	4	4	-1	123.0	-134.1	6	4	-3	79.3	79.5	12	4	-5	16.2	-15.0
9	3	-7	15.1	-12.5	6	4	-1	130.2	-121.6	8	4	-3	23.6	18.0	0	4	6	81.6	-73.1
1	3	8	31.2	-22.5	8	4	-1	37.8	38.0	10	4	-3	0.0 *	-5.7	2	4	6	64.3	57.1
3	3	8	0.0 *	-3.8	12	4	-1	0.0 *	-3.9	12	4	-3	19.4	20.3	4	4	6	53.4	50.1
5	3	8	42.2	34.9	10	4	-1	34.0	24.6	14	4	-3	16.3	-12.0	6	4	6	61.6	-51.2
7	3	8	34.5	-36.2	14	4	-1	16.5	20.1	0	4	4	114.3	107.7	8	4	6	14.8	-19.2
1	3	-8	52.4	41.6	0	4	2	45.4	55.1	2	4	4	75.0	74.2	10	4	6	27.4	28.5
3	3	-8	14.0	-13.2	2	4	2	146.2	-141.6	4	4	4	105.0	-111.7	2	4	-6	39.8	-29.7
5	3	-8	90.6	-73.2	4	4	2	12.7	-11.7	6	4	4	13.0	-15.2	4	4	-6	76.4	65.3
7	3	-8	28.6	20.1	6	4	2	84.0	101.2	8	4	4	64.0	63.9	6	4	-6	0.0 *	-3.5
1	3	5	11.7	12.9	8	4	2	32.8	-29.8	10	4	4	27.4	-18.8	8	4	-6	25.2	-33.1
3	3	9	0.0 *	-6.8	10	4	2	33.4	-39.2	12	4	4	10.8	-20.1	10	4	-6	14.7	17.1
1	3	-9	0.0 *	2.8	12	4	2	38.9	32.0	2	4	-4	91.6	-94.9	0	4	7	29.4	-22.5
3	3	9	11.7	13.0	14	4	2	10.4 *	-1.7	4	4	-4	52.6	-46.1	2	4	7	68.1	66.7
5	3	-9	0.0 *	-5.0	2	4	-2	130.0	127.5	6	4	-4	69.0	68.6	4	4	7	20.2	11.4
2	4	C	0.0 *	4.4	4	4	-2	55.6	-56.6	8	4	-4	0.0 *	-1.8	6	4	7	64.8	-60.2
4	4	0	119.0	119.9	6	4	-2	75.7	-74.5	10	4	-4	31.6	-34.9	8	4	7	30.0	-21.6
6	4	0	20.6	-22.0	8	4	-2	46.8	49.7	12	4	-4	25.5	26.6	2	4	-7	73.3	69.7
8	4	0	57.6	-70.4	10	4	-2	11.7	14.2	14	4	-4	21.7	11.0	4	4	-7	13.1	-12.5
10	4	C	38.8	37.8	12	4	-2	40.3	-39.6	0	4	5	137.6	147.1	6	4	-7	42.9	-37.4
12	4	C	0.0 *	10.4	14	4	-2	25.1	18.5	2	4	5	C.0 *	-2.9	8	4	-7	0.0 *	0.9
14	4	0	33.3	-31.2	C	4	3	73.4	-76.4	4	4	5	133.9	-126.8	0	4	8	21.0	-16.6
C	4	1	105.7	-124.0	2	4	3	228.6	-199.6	6	4	5	C.0 *	3.2	2	4	8	48.0	-42.2
2	4	1	95.7	90.1	4	4	3	33.0	32.6	8	4	5	64.9	59.5	4	4	8	30.3	21.2
4	4	1	122.6	117.8	6	4	3	94.8	77.5	10	4	5	0.0 *	3.0	6	4	8	23.1	28.1
6	4	1	59.7	-55.4	8	4	3	38.7	-38.0	12	4	5	0.0 *	1.1	2	4	-8	57.9	47.4
8	4	1	68.2	-57.0	10	4	3	28.5	-18.7	2	4	-5	10.4	-7.4	4	4	-8	0.0 *	3.1
10	4	1	29.2	23.4	12	4	3	0.0 *	4.5	4	4	-5	70.1	68.5	6	4	-8	39.3	-32.2
12	4	1	14.6	12.6	14	4	3	5.4	-14.8	6	4	-5	17.6	-19.3					

There were included 452 observed reflections in the full matrix least squares refinement (see Table 3).

The structure factors were weighted according to Cruickshank's formula

$$w = (a + |F_o| + c|F_c|^2)^{-1}$$

with  $a = 3.1$ ,  $c = 0.03$ . The final  $R$  value was 0.083. Extinction correction was not necessary. The weight analysis obtained in the last cycle is given in Table

Table 6. Distances (Å) and angles (°) with estimated standard deviations in  $\text{Rb}_2\text{Cr}_2\text{O}_7$  (C2/c). The numbering of the atoms refers to Fig. 1. The designation +b and +c refers to atoms translated +b and +c, respectively. The standard errors are those obtained by least squares refinement.

#### Dichromate ion

Distances:

Cr—Cr	3.117 ± 4	O(1)—O(2)	2.691 ± 19	O(3)—O(4)	2.741 ± 18
Cr—O(1)	1.617 ± 12	O(1)—O(3)	2.617 ± 15	O(1)—O(3')	3.279 ± 15
Cr—O(2)	1.627 ± 14	O(1)—O(4)	2.768 ± 18	O(1)—O(1')	4.132 ± 21
Cr—O(3)	1.609 ± 12	O(2)—O(3)	2.651 ± 18		
Cr—O(4)	1.774 ± 10	O(2)—O(4)	2.765 ± 12		

Angles

Cr—O(4)—Cr	122.9 ± 1.2	O(2)—Cr—O(3)	110.0 ± 0.6
O(1)—Cr—O(2)	112.1 ± 0.6	O(2)—Cr—O(4)	108.7 ± 0.8
O(1)—Cr—O(3)	108.4 ± 0.7	O(3)—Cr—O(4)	108.1 ± 0.5
O(1)—Cr—O(4)	109.4 ± 0.5		

#### Distances Rb—O

Rb—O(1)III(+c)	2.887 ± 11	Rb—O(1)II	2.990 ± 11
Rb—O(3)II	2.889 ± 10	Rb—O(1)IV	3.120 ± 13
Rb—O(3)III	2.900 ± 11	Rb—O(2)I	3.143 ± 13
Rb—O(3)IV	2.926 ± 14	Rb—O(2)III	3.449 ± 12
Rb—O(2)IV(+b)	2.952 ± 15	Rb—O(4)IV(+b)	3.695 ± 17

3 and the final parameters in Table 4. A three-dimensional ( $F_o - F_c$ ) synthesis, computed in sections 0.3 Å apart along the  $b$  axis showed maxima and minima of about 15 % of the height of an oxygen atom. The observed and calculated structure factors are listed in Table 5.

In Table 6 the interatomic distances and bond angles are given together with the standard deviations. The arrangement of the atoms is shown in Fig. 1. There are four crystallographically non-equivalent oxygen atoms, one rubidium and one chromium atom. Each chromium is surrounded by a somewhat distorted tetrahedron of four oxygen atoms. Two tetrahedra share the O(4) atom forming a  $\text{Cr}_2\text{O}_7^{2-}$  ion in an almost eclipsed conformation. The Cr—O (bridging) distance is  $1.774 \pm 10$  Å and the Cr—O (terminal) distances range from 1.609 to  $1.627 \pm 14$  Å (Fig. 2). The bond angles within the chromate tetrahedra range from 108.1 to  $112.1 \pm 8^\circ$  and the bridging angle is  $122.9 \pm 1.2^\circ$ . These values are in good agreement with those given for  $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ),<sup>1</sup>  $\text{K}_2\text{Cr}_2\text{O}_7$  ( $PI$ ),<sup>11</sup> and  $\text{Ag}_2\text{Cr}_2\text{O}_7$  ( $PI$ ),<sup>12</sup> each of which represents a different type of structures. The rubidium atoms are surrounded by an irregular polyhedron formed by eight oxygen atoms (Fig. 1); the distances range from 2.89

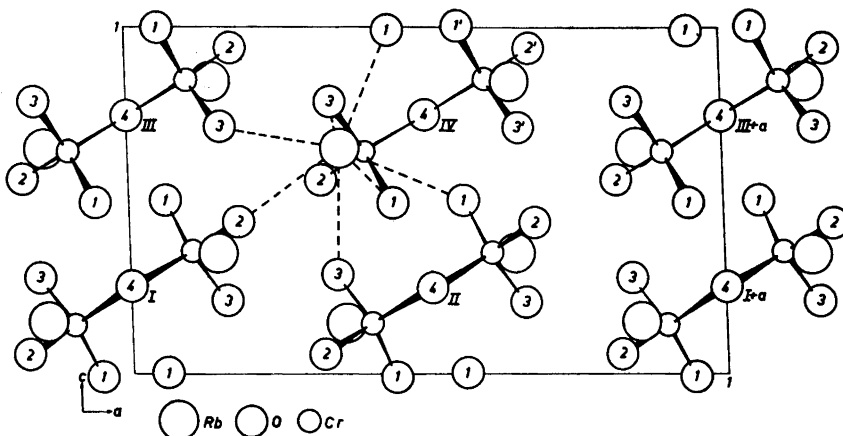


Fig. 1. Projection of the structure of  $\text{Rb}_2\text{Cr}_2\text{O}_7$  ( $C2/c$ ) along  $[010]$ . Rb—O bonds are shown as broken lines.

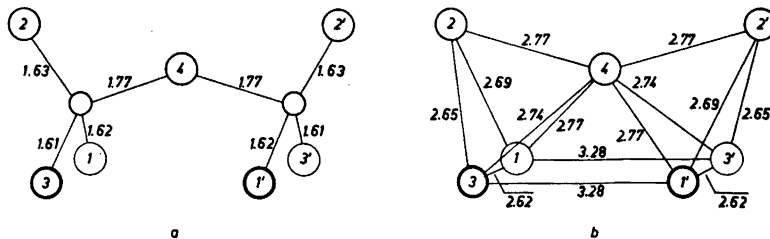


Fig. 2 a: Chromium-oxygen distances within the dichromate ion. b: Oxygen-oxygen distances within the dichromate ion.



to 3.14 Å (Table 6). The closest distance outside this range is 3.45 Å. Attempts to refine the structure on the basis of the lower symmetry,  $Cc$ , were also made. However, very large standard deviations and anomalies in the temperature factors indicated  $C2/c$  to be the most correct space group.

A more detailed discussion of the structure and its relation to the structure of  $Rb_2Cr_2O_7$  ( $P2_1/n$ ) will be given in a forthcoming paper.<sup>4</sup>

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#### REFERENCES

1. Byström, A. and Wilhelmi, K.-A. *Acta Chem. Scand.* **5** (1951) 1003.
2. Brown, I. D. and Calvo, C. *J. Solid State Chem.* **1** (1970) 173.
3. Panagiotopoulos, N. and Brown, I. D. *Can. J. Chem.* **48** (1970) 537.
4. Löfgren, P. *Acta Chem. Scand.* **25** (1971) 44.
5. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1959, Vol. III, p. 162.
6. Hanson, H. P., Herman, F., Lea, J. D. and Skillman, S. *Acta Cryst.* **17** (1964) 1040.
7. *Handbook of Chem. and Phys.* **48** (1967–68).
8. Lynton, H. and Truter, M. R. *J. Chem. Soc.* **1960** 5112.
9. Cruickshank, D. W. J. and Jones, D. W. *Acta Cryst.* **16** (1963) 877.
10. Truter, M. R. *J. Chem. Soc.* **1962** 3393.
11. Brandon, J. K. and Brown, I. D. *Can. J. Chem.* **46** (1968) 933.
12. Grønbaek Hazell, R. *Acta Cryst.* **A 25** (1969) 116.

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