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The Crystal Structure of Rubidium Dichromate, $Rb_2Cr_2O_7$ ($P2_1/n$)

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The crystal structure of $Rb_2Cr_2O_7$ ($P2_1/n$) has been determined and refined by full matrix least squares calculations on a three-dimensional set of photographic data obtained from a single crystal with the use of Ni-filtered Cu$K\alpha$ radiation. The structure is monoclinic, $a = 13.714 \pm 4$, $b = 7.602 \pm 2$, $c = 7.701 \pm 2$ Å, $\beta = 93.35 \pm 0.02°$, $V = 801.5$ Å^3, space group $P2_1/n$. The unit cell contains four formula units of $Rb_2Cr_2O_7$. The final R index is 0.085. The structure contains $Cr_2O_7^{2-}$ complexes in which two CrO_4 tetrahedra share one corner. The Cr – O (terminal) distances range from 1.580 to 1.617 ± 18 Å; the two Cr – O (bridging) distances are 1.795 and 1.796 ± 14 Å. The O – Cr – O bond angles range from 108.3 to 110.5°; the Cr – O – Cr angle at the bridging atom is $122.9 \pm 0.9°$. The structure of $Rb_2Cr_2O_7$ ($P2_1/n$) is closely related to that of $Rb_2Cr_2O_7$ ($C2/c$). Bond distances and angles correspond closely to those observed in other dichromates.

According to earlier reports,[1] only two modifications of rubidium dichromate are obtainable from water solutions, *viz.* $Rb_2Cr_2O_7$ ($P2_1/n$) and $Rb_2Cr_2O_7$ ($P\bar{1}$). Later on Wilhelmi [2] observed that crystals of two monoclinic modifications of rubidium dichromate were formed from a saturated rubidium dichromate solution after slow evaporation of the solvent at room temperature. The major product was $Rb_2Cr_2O_7$ ($P2_1/n$) and $Rb_2Cr_2O_7$ ($C2/c$) was present in minor quantities.

The crystal structure of the triclinic modification has been determined by Panagiotopoulos and Brown.[3] Recently, Löfgren and Waltersson [4] have published a refinement of the structure of the new modification $Rb_2Cr_2O_7$ ($C2/c$). The present paper reports the structure determination of $Rb_2Cr_2O_7$ ($P2_1/n$).

EXPERIMENTAL

Rubidium dichromate ($P2_1/n$) was prepared by slow addition of rubidium carbonate (Merck) to a hot solution of chromium trioxide (Hopkin & Williams "Analar") in water. (Mole ratio $Rb_2CO_3:CrO_3 = 1:3$.) The solvent was then allowed to evaporate at room

Table 1. Powder pattern of $Rb_2Cr_2O_7$ ($P2_1/n$). Reflections systematically absent in space group $P2_1/n$ have been omitted. The intensities were calculated from the expression $P(F_c)^2/1000$ where P is the number of equivalent reflections.

$h\,k\,l$	$10^5\sin^2\theta_{obs}$	$10^5\sin^2\theta_{calc}$	I_{obs}	I_{calc}
$\bar{1}\,0\,1$	–	1255	–	0
$2\,0\,0$	–	1266	–	1
$1\,1\,0$	–	1343	–	0
$1\,0\,1$	–	1386	–	0
$0\,1\,1$	2034	2030	vvw	5
$\bar{1}\,1\,1$	–	2281	–	1
$2\,1\,0$	–	2293	–	0
$1\,1\,1$	–	2413	–	1
$\bar{2}\,1\,1$	–	3165	–	3
$2\,1\,1$	3430	3428	vvw	7
$\bar{3}\,0\,1$	–	3655	–	1
$3\,1\,0$	3883	3875	w	16
$0\,0\,2$	4017	4015	w	25
$3\,0\,1$	–	4050	–	1
$0\,2\,0$	4109	4107	m	57
$1\,2\,0$	4428	4423	w	13
$\bar{3}\,1\,1$	–	4682	–	1
$\bar{2}\,0\,2$	5017	5018	st	100
$0\,1\,2$	–	5042	–	3
$4\,0\,0$	5069	5065	st	105
$3\,1\,1$		5077		54
$0\,2\,1$	5124	5110	st	77
$\bar{1}\,1\,2$	–	5227	–	3
$\bar{1}\,2\,1$	5367	5361	m	41
$2\,2\,0$	–	5373	–	3
$1\,1\,2$	–	5490	–	0
$1\,2\,1$	–	5493	–	2
$2\,0\,2$	5543	5545	m	95
$\bar{2}\,1\,2$	6037	6045	w	62
$4\,1\,0$	6091	6091	vvw	16
$\bar{2}\,2\,1$	6251	6245	vw	17
$2\,2\,1$	6514	6508	w	37
$2\,1\,2$	–	6571	–	7
$\bar{4}\,1\,1$	–	6832	–	7
$3\,2\,0$	–	6955	–	5
$4\,1\,1$	–	7358	–	5
$\bar{3}\,1\,2$	–	7496	–	5
$3\,2\,1$	–	7762	–	1
$0\,2\,2$	8149	8122	m	14
$3\,2\,1$		8156		38
$3\,1\,2$	8297	8285	w	28
$\bar{1}\,2\,2$		8307		15
$\bar{4}\,0\,2$	8551	8553	vvw	12
$1\,2\,2$	–	8570	–	0
$\bar{5}\,0\,1$	–	8588	–	8
$5\,1\,0$	–	8940	–	2
$\bar{2}\,2\,2$	9116	9125	w	36
$\bar{1}\,0\,3$	–	9153	–	2
$4\,2\,0$	9170	9171	m	55
$5\,0\,1$	–	9246	–	0
$1\,0\,3$	–	9548	–	3
$1\,3\,0$	–	9556	–	3
$\bar{4}\,1\,2$	–	9580	–	8

Table 1. Continued.

$\bar{4}$ 0 2 $\bar{5}$ 1 1	9615	9606 9615	m	20 39
$\bar{2}$ 2 2	–	9651	–	8
$\bar{4}$ 2 1	–	9912	–	1
0 1 3	–	10061	–	2
$\bar{1}$ 1 3	10169	10180	vvw	11
0 3 1	–	10243	–	4
5 1 1	10279	10273	vvw	14
$\bar{4}$ 2 1	10435	10438	w	41
$\bar{1}$ 3 1	–	10494	–	3
2 3 0	10527	10506	w	54
$\bar{1}$ 1 3 $\bar{3}$ 2 2	10565	10575 10576	vw	34 7
1 3 1	–	10626	–	0
$\bar{4}$ 1 2	–	10633	–	0
$\bar{2}$ 1 3	10938	10932	vvw	12
3 0 3	–	11291	–	1
3 2 2	11367	11365	vvw	12
$\bar{2}$ 3 1	–	11378	–	3
6 0 0	–	11395	–	5
2 3 1	–	11641	–	1
2 1 3	–	11722	–	2
5 2 0	–	12020	–	3
3 3 0	12088	12088	vvw	23
$\bar{5}$ 1 2	12301	12297	vvw	16
$\bar{3}$ 1 3	–	12318	–	2
6 1 0	–	12422	–	1
3 0 3	–	12475	–	0
$\bar{4}$ 2 2 $\bar{5}$ 2 1	12699	12660 12695	w	40 62
3 3 1	–	12895	–	10
$\bar{6}$ 1 1	–	13031	–	10
0 2 3	–	13141	–	6
0 3 2 $\bar{1}$ 2 3	13250	13255 13260	vvw	16 8
3 3 1	–	13290	–	0
$\bar{5}$ 2 1	–	13353	–	4
$\bar{1}$ 3 2	–	13440	–	4
3 1 3	–	13502	–	1
5 1 2	–	13613	–	5
1 2 3	–	13655	–	10
1 3 2	–	13703	–	0
4 2 2	–	13713	–	5
6 1 1	–	13820	–	5
$\bar{2}$ 2 3	14027	14012	vvw	33
$\bar{2}$ 3 2	–	14258	–	7
$\bar{4}$ 3 0	–	14304	–	18
$\bar{4}$ 1 3	–	14336	–	0
$\bar{6}$ 0 2	14637	14621	vvw	24
2 3 2 2 2 3	14814	14784 14802	m	32 84

temperature. Prismatic, orange-red crystals selected from the samples were studied with single-crystal X-ray techniques. The Weissenberg photographs $h0l-h4l$ and $0kl$ were taken with Ni-filtered Cu$K\alpha$ radiation. The prism axis of the crystal (which is also the unique b axis) was 0.19 mm long, and the cross section of the prism was 0.02×0.07 mm^2. Multiple-film technique (3 films) was used and the intensities were estimated visually by comparison with a calibrated scale. Lorentz, polarization, and absorption corrections were applied to the intensities. The linear absorption coefficient, $\mu = 402.2$ cm^{-1}, was calculated from the mass absorption coefficients given in Ref. 5. A total of 858 symmetrically independent reflections were measured. The scattering factors for the neutral atoms, corrected for anomalous dispersion (real part only) were taken from Ref. 6.

Accurate cell parameters were obtained from a powder photograph taken in the Hägg-Guinier focusing camera of 80 mm diameter. Cu$K\alpha$ radiation, $\lambda(20°C) = 1.54050$ Å,

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.

was used and potassium chloride, $a(20°C) = 6.2119$ Å, was added to the sample as an internal standard. The powder was prepared from a carefully selected sample. The cell parameters obtained from a least squares refinement were: $a = 13.714 \pm 4$, $b = 7.602 \pm 2$, $c = 7.701 \pm 2$ Å, $\beta = 93.35 \pm .02°$, $V = 801.5$ Å^3. The powder data are listed in Table 1. The unit cell corresponds closely to the values given by Klement and Schwab [1] and hence it seems reasonable to assume that the cell contains four formula units of $Rb_2Cr_2O_7$.

All calculations were carried out on the IBM 360/75 computer. The programs used are described in Table 2.

DETERMINATION OF THE STRUCTURE

The reflections of zero intensity fit the following scheme:

$$h0l \text{ for } h+l=2n+1$$
$$0k0 \text{ for } k=2n+1$$

which is characteristic for space group $P2_1/n$. (No. 14 in an alternative orientation.)

The point sets of space group $P2_1/n$ are:

2(*a*):	0,0,0;	$\frac{1}{2},\frac{1}{2},\frac{1}{2}$
2(*b*):	$\frac{1}{2}$,0,0;	$0,\frac{1}{2},\frac{1}{2}$
2(*c*):	$0,\frac{1}{2}$,0;	$\frac{1}{2}$,0,$\frac{1}{2}$
2(*d*):	0,0,$\frac{1}{2}$;	$\frac{1}{2},\frac{1}{2}$,0
4(*e*):	$\pm(x,y,z$;	$\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$

From three-dimensional Patterson sections perpendicular to the *b* axis tentative coordinates of the eight rubidium and the eight chromium atoms were derived and then tested and refined by the least-squares method. The positions of the twenty-eight oxygen atoms were then found by means of successive electron density sections. All atoms were accommodated in general point set 4(*e*). The trial coordinates for each atom were then refined by least squares calculations. The structure factors were weighted according to Cruickshank's formula

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

Table 3. Weight analysis used in last cycle of the refinement of $Rb_2Cr_2O_7$ ($P2_1/n$). w = weighting factor. $\Delta = ||F_o| - |F_c||$.

Interval sin θ	$\overline{w\Delta^2}$	Number of independent reflections	Interval $\|F_o\|$	$\overline{w\Delta^2}$	Number of independent reflections
0.00 – 0.46	1.36	151	0 – 16	0.73	83
0.46 – 0.58	0.92	139	16 – 19	0.86	85
0.58 – 0.67	0.93	122	19 – 25	0.79	85
0.67 – 0.74	0.78	87	25 – 29	0.79	84
0.74 – 0.79	0.95	75	29 – 35	1.01	85
0.79 – 0.84	0.84	66	35 – 43	0.82	85
0.84 – 0.89	0.97	60	43 – 53	1.34	84
0.89 – 0.93	0.66	58	53 – 65	0.87	85
0.93 – 0.97	1.07	55	65 – 88	1.20	85
0.97 – 1.00	1.44	33	88 – 200	1.59	85

with $a=19.0$, $c=0.01$. The final R value was 0.085. Of the 858 independent reflections, 11 strong, low-angle reflections obviously affected by extinction were omitted from the final least squares cycle. The results are given in Tables 3 (weight analysis), 4 (final values), and 5 (observed and calculated structure factors). A three-dimensional (F_o-F_c) synthesis computed in sections 0.3 Å apart along the b axis showed maxima of a height corresponding to about 15 % of the average oxygen atom. The most negative regions (magnitude 25 % of oxygen) were near the rubidium atoms.

Table 4. The crystal structure of $Rb_2Cr_2O_7$ ($P2_1/n$).

Space group: $P2_1/n$
Unit cell dimensions: $a=13.714\pm4$ Å
$b=7.602\pm2$ Å, $c=7.701\pm2$ Å
$\beta=93.35\pm.02$, $V=801.5$ Å^3

Cell content: 4 $Rb_2Cr_2O_7$
All atoms in 4(*e*): $\pm(x,y,z;\ \frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$
Final coordinates, isotropic temperature factors and standard deviations resulting from the least-squares refinement.

	$x\pm\sigma(x)$	$y\pm\sigma(y)$	$z\pm\sigma(z)$	$B\pm\sigma(B)$ Å^2
Rb(1)	0.3696± 1	0.7275± 4	0.3061± 2	2.66± 5
Rb(2)	0.6582± 1	0.6213± 3	0.1443± 2	2.34± 4
Cr(1)	0.3872± 2	0.2172± 5	0.3821± 3	1.91± 6
Cr(2)	0.5792± 2	0.1650± 5	0.1815± 3	1.89± 6
O(4)	0.4771± 9	0.0791±22	0.2901±15	2.66±23
O(11)	0.4357±10	0.3321±24	0.5374±17	3.31±26
O(21)	0.3033±10	0.0958±25	0.4532±17	3.60±28
O(31)	0.3410±10	0.3425±24	0.2330±17	3.37±27
O(12)	0.5418± 9	0.2911±22	0.0258±15	2.58±23
O(22)	0.6388±11	0.0058±26	0.1091±19	4.04±31
O(32)	0.6480± 9	0.2788±23	0.3171±15	2.85±24

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The arrangement of the atoms is shown in Fig. 1. The structure is composed of $Cr_2O_7^{2-}$ and Rb^+ ions. Two CrO_4 tetrahedra sharing one oxygen atom form a $Cr_2O_7^{2-}$ complex in a nearly eclipsed conformation. The bridging oxygen O(4) lies almost on the line joining two of the terminal oxygens O(21) and O(22). According to Ref. 7 these three oxygens will be referred to as the backbone of the ion and the remaining four oxygens as the feet. Distances and angles within the dichromate complex, and the estimated standard deviations are given in Table 6. The Cr – O (terminal) distances range from 1.580 to 1.617±18 Å, mean value 1.60 Å. The two Cr – O (bridging) distances are 1.795 and 1.796±14 Å. The Cr – O (terminal) bond angles within the chromate tetrahedra range from 108.3 to 110.2±0.9°. The mean value 109.5° is very nearly the same as that of a regular tetrahedron. The Cr – O(bridge) – Cr angle is 122.9±0.9°. The oxygen-oxygen distances within a $Cr_2O_7^{2-}$ complex comprise three distance ranges. The distances between the terminal oxygens range from 2.601 to 2.620±24 Å, the distances from the bridging oxygen

Table 5. Observed and calculated structure factors for $Rb_2Cr_4O_7$ ($P2_1/n$). Reflections given zero weight and reflections excluded from the refinement because of assumed secondary extinction are indicated by one and two asterisks, respectively.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	0.0*	24.3	-9	0	7	86.6	65.5	7	1	2	77.3	77.5	13	1	5	33.2	38.0
4	0	0	261.8**	-324.8	-7	0	7	45.7	-44.1	8	1	2	20.0	-21.2	14	1	5	10.0	11.8
6	0	0	77.9	68.6	-5	0	7	53.7	-51.2	9	1	2	27.7	-28.9	-14	1	6	15.9	-17.1
8	0	0	66.9	63.8	-3	0	7	13.2	13.4	10	1	2	18.4	16.4	-13	1	6	0.0*	8.4
10	0	0	65.7	-65.9	-1	0	7	50.2	-52.9	11	1	2	40.7	-43.8	-12	1	6	0.0*	11.8
12	0	0	0.0*	0.5	1	0	7	25.4	-28.8	12	1	2	0.0*	-0.8	-11	1	6	12.7	-14.2
14	0	0	27.2	28.1	3	0	7	87.7	90.6	13	1	2	0.0*	4.6	-10	1	6	33.6	29.6
16	0	0	0.0*	-6.3	5	0	7	18.0	17.3	14	1	2	58.7	-57.6	-9	1	6	0.0*	8.4
-17	0	1	44.7	44.8	7	0	7	47.0	-52.7	15	1	2	0.0*	7.6	-8	1	6	74.9	-62.3
-15	0	1	36.9	-34.8	9	0	7	0.0*	-5.2	16	1	2	0.0*	-3.0	-7	1	6	0.0*	-3.0
-13	0	1	105.1	-96.3	11	0	7	0.0*	5.2	17	1	2	0.0*	-17.6	-6	1	6	21.8	-19.5
-11	0	1	106.9	112.3	-10	0	8	34.8	-28.4	-16	1	3	15.9	16.0	-5	1	6	22.6	-22.1
-9	0	1	157.4	161.6	-8	0	8	31.1	23.1	-15	1	3	14.8	9.8	-4	1	6	132.6	138.2
-7	0	1	101.3	-94.5	-6	0	8	40.5	33.4	-14	1	3	17.2	14.2	-3	1	6	38.3	37.8
-5	0	1	96.3	-88.5	-4	0	8	16.1	-17.5	-13	1	3	19.1	-14.8	-2	1	6	37.5	40.3
-3	0	1	12.0	9.2	-2	0	8	0.0*	-0.2	-12	1	3	25.2	-25.6	-1	1	6	16.6	18.3
-1	0	1	0.0*	10.1	0	0	8	15.8	-16.1	-11	1	3	0.0*	0.2	0	1	6	131.5	-134.5
1	0	1	0.0*	8.5	2	0	8	27.1	-29.6	-10	1	3	17.6	14.3	1	1	6	33.7	-37.2
3	0	1	26.2	-21.8	4	0	8	23.6	25.0	-9	1	3	32.4	-29.1	2	1	6	49.6	-54.9
5	0	1	0.0*	-8.7	6	0	8	0.0*	10.9	-8	1	3	0.0*	-2.7	3	1	6	24.5	27.5
7	0	1	76.6	76.7	8	0	8	17.4	-18.3	-7	1	3	35.2	-31.7	4	1	6	53.9	60.7
9	0	1	14.7	16.2	-7	0	9	22.7	-18.4	-6	1	3	69.7	-74.5	5	1	6	0.0*	-4.4
11	0	1	126.6	-132.5	-5	0	9	24.6	20.1	-5	1	3	72.0	73.3	6	1	6	21.3	20.4
13	0	1	21.2	-20.7	-3	0	9	0.0*	-13.7	-4	1	3	10.5	-7.3	7	1	6	54.1	-58.9
15	0	1	70.7	78.9	-1	0	9	0.0*	-4.9	-3	1	3	31.7	-28.0	8	1	6	0.0*	-11.9
17	0	1	0.0*	1.1	1	0	9	54.9	54.3	-2	1	3	73.3	75.4	9	1	6	0.0*	13.5
-16	0	2	0.0*	0.7	3	0	9	0.0*	-1.3	-1	1	3	83.4	-73.6	10	1	6	0.0*	0.9
-14	0	2	18.2	-16.8	5	0	9	50.1	-57.6	0	1	3	30.3	-27.0	11	1	6	29.5	33.2
-12	0	2	22.4	-25.6	1	1	0	0.0*	7.6	1	1	3	111.1	125.2	12	1	6	13.8	10.5
-10	0	2	54.5	47.2	2	1	0	0.0*	-13.6	2	1	3	31.1	-31.5	13	1	6	0.0*	-11.5
-8	0	2	26.6	-23.8	3	1	0	94.2	86.1	3	1	3	24.6	-26.5	-13	1	7	45.9	44.9
-6	0	2	152.5	-157.5	4	1	0	94.2	-88.6	4	1	3	34.3	32.9	-12	1	7	16.5	12.0
-4	0	2	101.3	108.5	5	1	0	29.5	-28.3	5	1	3	152.5	-176.3	-11	1	7	0.0*	-7.9
-2	0	2	233.6	321.0	6	1	0	28.9	23.7	6	1	3	58.1	-58.1	-10	1	7	19.3	-16.4
0	0	2	140.2	-158.9	7	1	0	69.1	-73.1	7	1	3	73.2	72.0	-9	1	7	47.6	-43.5
2	0	2	226.6	308.3	8	1	0	27.1	27.1	8	1	3	0.0*	-11.0	-8	1	7	15.3	16.3
4	0	2	144.5	145.2	9	1	0	62.4	62.1	9	1	3	155.1	157.7	-7	1	7	0.0*	-9.4
6	0	2	82.4	83.4	10	1	0	33.6	-29.2	10	1	3	44.0	46.8	-6	1	7	31.6	29.1
8	0	2	87.8	-86.6	11	1	0	23.7	23.2	11	1	3	44.4	-44.2	-5	1	7	23.6	26.4
10	0	2	0.0*	9.7	12	1	0	33.7	34.7	12	1	3	0.0*	-9.3	-4	1	7	49.2	-49.9
12	0	2	0.0*	4.0	13	1	0	30.1	-26.8	13	1	3	76.1	-78.7	-3	1	7	20.6	20.6
14	0	2	38.9	-41.3	14	1	0	34.9	35.6	14	1	3	0.0*	2.2	-2	1	7	24.9	-23.9
16	0	2	0.0*	-2.0	15	1	0	19.6	18.6	15	1	3	14.2	14.2	-1	1	7	18.1	-20.0
-15	0	3	48.5	-43.8	16	1	0	49.4	-47.8	16	1	3	15.8	20.7	0	1	7	32.0	34.7
-13	0	3	105.5	104.6	17	1	0	0.0*	0.7	-16	1	4	0.0*	7.1	1	1	7	12.9	-16.2
-11	0	3	110.4	102.6	-17	1	1	0.0*	-2.9	-15	1	4	17.8	-19.5	2	1	7	0.0*	-6.1
-9	0	3	199.5	-176.7	-16	1	1	28.6	-28.1	-14	1	4	21.5	20.3	3	1	7	58.2	60.5
-7	0	3	144.0	-128.9	-15	1	1	0.0*	3.3	-13	1	4	18.4	13.5	4	1	7	0.0*	-13.3
-5	0	3	107.3	104.0	-14	1	1	0.0*	9.2	-12	1	4	25.2	-19.6	5	1	7	22.8	23.0
-3	0	3	42.9	-37.5	-13	1	1	0.0*	-9.6	-11	1	4	0.0*	-0.3	6	1	7	0.0*	13.1
-1	0	3	51.0	-48.0	-12	1	1	0.0*	10.6	-10	1	4	52.5	46.4	7	1	7	73.4	-76.4
1	0	3	48.5	49.4	-11	1	1	29.1	27.3	-9	1	4	0.0*	-11.2	8	1	7	16.4	-13.8
3	0	3	0.0*	-8.0	-10	1	1	0.0*	-7.0	-8	1	4	67.3	60.2	9	1	7	17.3	-19.3
5	0	3	0.0*	-12.4	-9	1	1	72.2	68.3	-7	1	4	41.8	36.5	10	1	7	15.2	-20.9
7	0	3	14.4	-12.8	-8	1	1	54.5	51.4	-6	1	4	114.1	-110.2	11	1	7	39.5	49.5
9	0	3	39.3	-39.0	-7	1	1	92.9	-78.8	-5	1	4	21.8	-16.8	-10	1	8	34.4	-30.2
11	0	3	26.1	25.3	-6	1	1	76.4	66.6	-4	1	4	103.6	-100.6	-9	1	8	0.0*	1.1
13	0	3	59.6	66.1	-5	1	1	139.1	-138.1	-3	1	4	50.3	-49.1	-8	1	8	0.0*	6.6
15	0	3	0.0*	-7.0	-4	1	1	57.7	-57.2	-2	1	4	101.9	102.4	-7	1	8	0.0*	-10.0
-14	0	4	0.0*	0.9	-3	1	1	28.8	23.8	-1	1	4	40.6	37.1	-6	1	8	100.1	85.1
-12	0	4	0.0*	9.2	-2	1	1	34.4	-24.6	0	1	4	81.2	85.1	-5	1	8	18.5	17.1
-10	0	4	0.0*	10.7	-1	1	1	0.0*	26.1	1	1	4	23.9	-23.6	-4	1	8	27.3	-23.6
-8	0	4	51.0	-40.6	0	1	1	0.0*	46.9	2	1	4	80.9	-82.6	-3	1	8	0.0*	3.9
-6	0	4	27.6	28.0	1	1	1	0.0*	19.6	3	1	4	13.7	-11.5	-2	1	8	107.9	-110.5
-4	0	4	62.0	65.2	2	1	1	64.5	-55.9	4	1	4	57.6	-62.1	-1	1	8	31.6	-31.4
-2	0	4	102.3	-98.0	3	1	1	140.4	**162.1	5	1	4	71.2	83.1	0	1	8	12.7	14.0
0	0	4	57.5	-95.9	4	1	1	57.5	47.6	6	1	4	13.1	9.5	1	1	8	0.0*	5.2
2	0	4	144.6	163.8	5	1	1	100.1	83.1	7	1	4	18.2	14.5	2	1	8	61.0	60.7
4	0	4	122.7	137.5	6	1	1	49.0	46.0	8	1	4	20.6	18.0	3	1	8	0.0*	10.2
6	0	4	72.2	-77.5	7	1	1	136.7	-139.2	9	1	4	46.8	-48.9	4	1	8	0.0*	-14.9
8	0	4	17.1	-15.1	8	1	1	60.4	-58.0	10	1	4	0.0*	-0.6	5	1	8	23.4	-26.1
10	0	4	0.0*	8.1	9	1	1	36.1	-35.2	11	1	4	0.0*	5.9	6	1	8	19.2	-18.0
12	0	4	47.1	-52.6	10	1	1	12.2	-12.7	12	1	4	27.8	-27.5	7	1	8	15.4	16.8
14	0	4	0.0*	12.1	11	1	1	102.1	97.0	13	1	4	24.3	26.8	8	1	8	0.0*	5.7
-15	0	5	58.3	62.9	12	1	1	0.0*	5.8	14	1	4	16.7	16.0	9	1	8	18.8	28.3
-13	0	5	21.2	19.0	13	1	1	0.0*	6.9	15	1	4	C.0*	-6.7	-7	1	9	24.8	24.1
-11	0	5	135.9	-119.1	14	1	1	27.8	-27.5	-15	1	5	38.3	-40.0	-6	1	9	25.7	-19.9
-9	0	5	0.0*	-4.9	15	1	1	38.1	-35.2	-14	1	5	26.7	-26.5	-5	1	9	0.0*	4.6
-7	0	5	150.6	145.3	16	1	1	C.0*	C.0	-13	1	5	0.0*	-13.8	-4	1	9	0.0*	-7.2
-5	0	5	32.9	-30.9	17	1	1	0.0*	-0.3	-12	1	5	0.0*	4.9	-3	1	9	0.0*	-9.9
-3	0	5	58.7	-61.4	-17	1	2	19.5	22.1	-11	1	5	31.6	30.0	-2	1	9	39.6	40.2
-1	0	5	68.7	73.2	-16	1	2	55.2	-52.1	-10	1	5	0.0*	6.6	-1	1	9	0.0*	-8.5
1	0	5	52.1	-58.5	-15	1	2	0.0*	-11.3	-9	1	5	24.2	22.8	0	1	9	0.0*	4.3
3	0	5	41.9	-45.9	-14	1	2	C.0*	-7.4	-8	1	5	46.0	-39.7	1	1	9	0.0*	11.8
5	0	5	71.4	79.3	-13	1	2	0.0*	-8.8	-7	1	5	0.0*	-5.4	2	1	9	19.6	-19.0
7	0	5	27.6	28.1	-12	1	2	34.8	35.4	-6	1	5	45.3	49.5	3	1	9	0.0*	1.5
9	0	5	0.0*	-2.3	-11	1	2	27.5	25.2	-5	1	5	42.0	-43.8	4	1	9	0.0*	7.5
11	0	5	0.0*	10.6	-10	1	2	27.7	-20.8	-4	1	5	74.9	77.5	5	1	9	25.0	-29.4
13	0	5	14.1	-17.3	-9	1	2	46.6	-38.4	-3	1	5	15.1	-15.7	0	2	0	0.0*	-245.3
-14	0	6	0.0*	-C.3	-8	1	2	64.2	57.0	-2	1	5	28.3	-32.2	1	2	0	0.0*	-83.2
-12	0	6	25.8	19.1	-7	1	2	0.0*	-2.3	-1	1	5	55.8	60.7	2	2	0	51.8	-40.3
-10	0	6	0.0*	-8.5	-6	1	2	145.1	142.4	0	1	5	23.9	-25.6	3	2	0	56.2	-51.6
-8	0	6	27.8	-25.0	-5	1	2	86.C	88.6	1	1	5	53.1	-54.6	4	2	0	164.2	175.5
-6	0	6	34.9	32.4	-4	1	2	64.5	-63.1	2	1	5	26.8	29.7	5	2	0	51.6	43.1
-4	0	6	17.9	-17.2	-3	1	2	97.7	-88.5	3	1	5	71.0	-78.8	6	2	0	45.8	42.8
-2	0	6	16.9	-16.0	-2	1	2	150.4	**172.5	4	1	5	16.9	-17.7	7	2	0	57.1	-53.1
0	0	6	89.0	92.9	-1	1	2	41.9	-35.2	5	1	5	122.7	136.9	8	2	0	78.5	-73.4
2	0	6	21.4	24.3	C	1	2	41.1	38.0	6	1	5	0.0*	-7.8	9	2	0	27.4	29.1
4	0	6	73.5	-84.6	1	1	2	0.0*	-2.2	7	1	5	96.4	101.2	10	2	0	0.0*	4.5
6	0	6	0.0*	-8.2	2	1	2	56.2	55.1	8	1	5	40.5	44.3	11	2	0	106.5	116.6
8	0	6	0.0*	13.5	3	1	2	104.8	-115.6	9	1	5	87.0	-90.4	12	2	0	45.7	42.9
10	0	6	41.5	-46.4	4	1	2	C.0*	-3.8	10	1	5	13.9	-9.3	13	2	0	24.2	-24.0
12	0	6	18.7	19.4	5	1	2	52.1	-49.7	11	1	5	46.9	-57.4	14	2	0	0.0*	-4.8
-11	0	7	40.2	35.6	6	1	2	0.0*	4.5	12	1	5	16.4	-19.5	15	2	0	42.7	-49.5

Table 5. Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
16	2	0	26.5	-24.1	-13	2	4	26.4	24.7	8	2	7	0.0 *	9.5	6	3	2	134.6	145.3
17	2	0	14.1	16.1	-12	2	4	25.8	-24.0	9	2	7	18.4	19.0	7	3	2	109.6	-119.5
-17	2	1	18.0	-19.6	-11	2	4	77.3	64.7	10	2	7	13.2	-16.7	8	3	2	37.5	-33.5
-16	2	1	18.8	-17.0	-10	2	4	57.4	52.0	11	2	7	10.6	-12.6	9	3	2	78.0	-69.3
-15	2	1	24.0	21.6	-9	2	4	67.6	-58.8	-9	2	8	49.7	47.6	10	3	2	80.8	-86.9
-14	2	1	0.0 *	-5.7	-8	2	4	C.C *	6.8	-8	2	8	0.0 *	3.3	11	3	2	41.2	41.7
-13	2	1	48.6	49.4	-7	2	4	87.7	-86.7	-7	2	8	0.0 *	-2.3	12	3	2	27.5	26.5
-12	2	1	43.7	39.8	-6	2	4	77.8	-77.1	-6	2	8	C.0 *	-10.9	13	3	2	19.5	16.1
-11	2	1	25.4	-24.3	-5	2	4	25.7	26.7	-5	2	8	41.1	-36.2	14	3	2	26.8	28.8
-10	2	1	0.0 *	-5.6	-4	2	4	10.6	-10.5	-4	2	8	25.8	-26.5	15	3	2	0.0 *	-5.0
-9	2	1	112.3	-106.9	-3	2	4	25.0	-28.7	-3	2	8	0.0 *	15.1	16	3	2	14.7	-18.6
-8	2	1	30.9	-25.5	-2	2	4	84.7	82.9	-2	2	8	0.0 *	11.3	-16	3	3	12.7	-13.6
-7	2	1	17.2	13.3	-1	2	4	11.4	6.8	-1	2	8	0.0 *	-11.2	-15	3	3	0.0 *	-4.2
-6	2	1	62.8	51.7	C	2	4	14.7	13.2	0	2	8	27.9	24.8	-14	3	3	23.2	-19.8
-5	2	1	165.5	184.2	1	2	4	84.4	101.5	1	2	8	36.1	-38.5	-13	3	3	26.6	-25.4
-4	2	1	25.7	-27.3	2	2	4	64.6	-68.6	2	2	8	13.3	-14.0	-12	3	3	19.7	15.6
-3	2	1	26.7	22.9	3	2	4	28.9	-27.8	3	2	8	45.2	47.9	-11	3	3	61.3	48.5
-2	2	1	85.2	-98.6	4	2	4	31.3	-27.7	4	2	8	13.7	-14.6	-10	3	3	22.7	-23.3
-1	2	1	124.9 *	-148.0	5	2	4	48.9	-55.6	5	2	8	34.5	33.3	-9	3	3	19.9	17.0
0	2	1	143.5 *	207.5	6	2	4	22.4	22.7	6	2	8	22.8	20.6	-8	3	3	0.0 *	-3.2
1	2	1	38.6	36.1	7	2	4	13.9	12.4	7	2	8	32.5	-41.2	-7	3	3	74.8	-75.9
2	2	1	124.0 *	143.1	8	2	4	28.6	27.3	8	2	8	C.0 *	8.2	-6	3	3	91.4	98.0
3	2	1	132.5	145.3	9	2	4	14.2	-15.7	-6	2	9	30.9	32.6	-5	3	3	59.5	57.4
4	2	1	140.4	-151.1	10	2	4	0.0 *	13.6	-5	2	9	0.0 *	4.2	-4	3	3	12.2	-10.2
5	2	1	53.2	-49.1	11	2	4	11.7	-16.2	-4	2	9	0.0 *	-11.7	-3	3	3	75.0	76.3
6	2	1	31.6	-31.0	12	2	4	0.0 *	13.1	-3	2	9	0.0 *	12.9	-2	3	3	123.0	-148.7
7	2	1	129.1	-122.7	13	2	4	34.7	39.1	-2	2	9	0.0 *	-9.6	-1	3	3	89.5	-89.2
8	2	1	21.5	20.5	14	2	4	0.0 *	3.0	-1	2	9	0.0 *	-4.6	0	3	3	10.0	-10.3
9	2	1	28.4	28.8	15	2	4	14.4	17.2	0	2	9	0.0 *	10.1	1	3	3	64.7	-63.8
10	2	1	29.1	-34.9	-15	2	5	32.6	-37.2	1	2	9	25.5	-22.2	2	3	3	51.9	59.2
11	2	1	67.3	64.5	-14	2	5	13.1	-12.7	2	2	9	31.2	-26.4	3	3	3	25.9	18.8
12	2	1	25.1	23.2	-13	2	5	0.0 *	-7.0	3	2	9	0.0 *	2.9	4	3	3	0.0 *	-7.7
13	2	1	17.5	-16.1	-12	2	5	31.0	-27.6	4	2	9	0.0 *	-8.6	5	3	3	23.8	22.5
14	2	1	38.1	39.3	-11	2	5	62.0	56.2	5	2	9	24.7	28.8	6	3	3	33.7	28.8
15	2	1	33.1	-35.2	-10	2	5	52.0	43.5	1	3	0	49.7	38.4	7	3	3	27.4	-24.0
16	2	1	11.2	-11.0	-9	2	5	0.0 *	16.4	2	3	0	153.2	147.7	8	3	3	22.9	21.1
-16	2	2	22.0	19.6	-8	2	5	C.C *	10.3	3	3	0	117.8	-100.7	9	3	3	51.9	-46.6
-15	2	2	0.0 *	4.8	-7	2	5	53.4	-55.4	4	3	0	93.4	-85.0	10	3	3	19.8	-17.1
-14	2	2	49.6	49.3	-6	2	5	40.9	-41.3	5	3	0	69.3	61.7	11	3	3	0.0 *	-9.9
-13	2	2	84.3	-83.0	-5	2	5	17.0	-14.1	6	3	0	34.3	-27.7	12	3	3	0.0 *	-0.7
-12	2	2	31.1	-28.6	-4	2	5	46.7	44.2	7	3	0	119.3	115.3	13	3	3	40.7	39.8
-11	2	2	0.0 *	11.5	-3	2	5	54.7	57.4	8	3	0	108.9	109.1	14	3	3	16.7	-14.7
-10	2	2	59.9	-51.4	-2	2	5	11.2	-8.8	9	3	0	60.4	-59.1	15	3	3	0.0 *	10.2
-9	2	2	124.7	112.6	-1	2	5	30.2	32.0	10	3	0	16.0	14.3	-15	3	4	17.9	21.6
-8	2	2	92.8	84.3	0	2	5	90.7	-91.5	11	3	0	54.0	-56.0	-14	3	4	24.9	21.3
-7	2	2	44.3	-36.5	1	2	5	11.2	-11.5	12	3	0	43.2	-48.4	-13	3	4	18.6	-14.7
-6	2	2	95.7	87.3	2	2	5	36.6	37.7	13	3	0	16.7	15.2	-12	3	4	17.4	-12.6
-5	2	2	0.0 *	-1.7	3	2	5	17.1	15.4	14	3	0	9.5	9.1	-11	3	4	0.0 *	-12.6
-4	2	2	130.3	-149.7	4	2	5	79.0	84.6	15	3	0	0.0 *	-5.9	-10	3	4	21.8	-20.2
-3	2	2	65.7	62.5	5	2	5	12.1	7.0	16	3	0	22.5	22.7	-9	3	4	0.0 *	-9.7
-2	2	2	111.5 *	-142.1	6	2	5	35.8	-33.5	-16	3	1	39.2	38.8	-8	3	4	14.7	-11.2
-1	2	2	83.0	-90.0	7	2	5	37.5	-31.3	-15	3	1	39.0	36.2	-7	3	4	41.4	-34.6
0	2	2	85.3	88.0	8	2	5	0.0 *	-8.0	-14	3	1	0.0 *	9.6	-6	3	4	16.5	-13.6
1	2	2	8.9 *	3.7	9	2	5	14.0	-13.6	-13	3	1	0.0 *	-2.8	-5	3	4	22.5	17.4
2	2	2	61.4	67.6	10	2	5	22.3	24.7	-12	3	1	0.0 *	-0.7	-4	3	4	21.9	22.6
3	2	2	83.9	80.8	11	2	5	12.2	13.5	-11	3	1	49.9	-44.3	-3	3	4	53.5	51.1
4	2	2	53.9	-51.6	12	2	5	32.7	-38.2	-10	3	1	0.0 *	0.6	-2	3	4	13.9	-12.1
5	2	2	22.9	-17.0	13	2	5	0.0 *	2.6	-9	3	1	50.9	45.3	-1	3	4	0.0 *	3.9
6	2	2	62.9	-62.5	-13	2	6	28.0	25.5	-8	3	1	70.7	-68.8	0	3	4	76.3	-80.2
7	2	2	0.0 *	-3.7	-12	2	6	35.1	30.4	-7	3	1	0.0 *	11.8	1	3	4	25.9	31.3
8	2	2	0.0 *	3.5	-11	2	6	59.7	-45.8	-6	3	1	39.4	-41.3	2	3	4	15.8	10.4
9	2	2	44.0	45.8	-10	2	6	0.0 *	3.0	-5	3	1	36.5	-27.3	3	3	4	15.4	10.3
10	2	2	29.2	28.9	-9	2	6	59.1	-47.6	-4	3	1	87.4	93.1	4	3	4	99.7	111.9
11	2	2	45.3	-44.4	-8	2	6	30.0	-27.0	-3	3	1	67.9	64.0	5	3	4	71.6	-83.4
12	2	2	0.0 *	1.0	-7	2	6	52.3	50.4	-2	3	1	35.1	39.8	6	3	4	41.0	-36.4
13	2	2	52.5	-52.9	-6	2	6	12.7	17.4	-1	3	1	31.3	33.9	7	3	4	28.2	-22.1
14	2	2	0.0 *	-1.9	-5	2	6	31.1	25.7	0	3	1	40.9	-39.0	8	3	4	75.7	-88.9
15	2	2	29.1	31.4	-4	2	6	29.9	30.7	1	3	1	10.3	-10.8	9	3	4	60.3	70.9
16	2	2	8.6	8.1	-3	2	6	15.9	-15.1	2	3	1	28.5	24.8	10	3	4	54.1	55.5
-16	2	3	0.0 *	-1.0	-2	2	6	26.2	-27.2	3	3	1	0.0 *	-1.4	11	3	4	21.6	16.0
-15	2	3	18.1	16.6	-1	2	6	54.5	60.8	4	3	1	35.8	-35.7	12	3	4	42.2	43.4
-14	2	3	34.9	31.3	0	2	6	39.9	-42.8	5	3	1	0.0 *	2.9	13	3	4	31.8	-34.1
-13	2	3	53.7	-53.7	1	2	6	12.8	-10.0	6	3	1	64.3	-64.7	14	3	4	28.2	-33.1
-12	2	3	28.6	-22.4	2	2	6	13.1	11.6	7	3	1	0.0 *	3.6	-14	3	5	28.5	26.7
-11	2	3	67.9	-65.1	3	2	6	58.6	-65.9	8	3	1	23.1	22.5	-13	3	5	27.8	30.0
-10	2	3	49.0	-45.8	4	2	6	20.0	19.0	9	3	1	33.7	33.2	-12	3	5	0.0 *	-1.7
-9	2	3	56.9	53.6	5	2	6	37.4	40.8	10	3	1	24.5	23.3	-11	3	5	22.6	-17.4
-8	2	3	48.0	42.4	6	2	6	15.5	-10.6	11	3	1	19.9	-16.7	-10	3	5	0.0 *	7.0
-7	2	3	114.7	110.7	7	2	6	29.3	29.6	12	3	1	0.0 *	9.5	-9	3	5	71.2	-59.7
-6	2	3	0.0 *	-0.8	8	2	6	0.0 *	9.6	13	3	1	40.3	-42.1	-8	3	5	57.2	56.9
-5	2	3	49.7	-48.0	9	2	6	12.2	-17.4	14	3	1	25.5	24.9	-7	3	5	60.7	62.6
-4	2	3	36.2	-33.8	10	2	6	16.8	16.1	15	3	1	43.1	37.5	-6	3	5	59.2	-55.5
-3	2	3	101.8	-120.8	11	2	6	9.8	12.4	16	3	1	13.8	-13.3	-5	3	5	67.5	67.8
-2	2	3	113.1	133.9	12	2	6	14.7	-16.9	-15	3	2	13.8	10.5	-4	3	5	110.3	-121.9
-1	2	3	68.3	66.9	-12	2	7	27.8	27.9	-14	3	2	18.2	18.6	-3	3	5	111.4	-114.9
0	2	3	54.1	55.6	-11	2	7	23.9	-18.0	-13	3	2	0.0 *	2.0	-2	3	5	39.6	41.9
1	2	3	69.8	75.2	-10	2	7	0.0 *	14.7	-12	3	2	0.0 *	-2.8	-1	3	5	33.8	-34.7
2	2	3	172.0 *	-214.6	-9	2	7	27.4	-21.3	-11	3	2	0.0 *	-0.7	0	3	5	78.5	89.3
3	2	3	74.5	-76.9	-8	2	7	49.9	-45.4	-10	3	2	19.3	-20.4	1	3	5	97.1	101.6
4	2	3	52.9	-47.1	-7	2	7	0.0 *	11.3	-9	3	2	61.2	53.4	2	3	5	0.0 *	-12.5
5	2	3	44.4	-38.6	-6	2	7	0.0 *	2.3	-8	3	2	36.6	31.4	3	3	5	0.0 *	2.1
6	2	3	69.9	71.4	-5	2	7	14.3	15.0	-7	3	2	21.4	-22.0	4	3	5	0.0 *	-6.6
7	2	3	59.9	60.7	-4	2	7	17.1	20.4	-6	3	2	11.4	8.5	5	3	5	48.6	-46.0
8	2	3	48.9	-49.5	-3	2	7	13.7	10.0	-5	3	2	119.1	-107.5	6	3	5	0.0 *	6.7
9	2	3	21.8	22.3	-2	2	7	27.1	-22.8	-4	3	2	49.0	-45.1	7	3	5	0.0 *	-7.5
10	2	3	12.6	15.9	-1	2	7	16.5	14.5	-3	3	2	24.3	24.6	8	3	5	19.3	-22.5
11	2	3	37.7	-38.0	0	2	7	37.0	34.0	-2	3	2	54.3	53.3	9	3	5	18.8	19.5
12	2	3	37.8	41.8	1	2	7	0.0 *	-5.4	-1	3	2	46.6	40.7	10	3	5	0.0 *	-0.7
13	2	3	0.0 *	-12.1	2	2	7	37.8	37.3	0	3	2	70.4	81.9	11	3	5	24.4	26.8
14	2	3	31.5	-37.0	3	2	7	31.6	-30.8	1	3	2	11.3	-6.0	12	3	5	0.0 *	5.4
15	2	3	9.8	9.4	4	2	7	39.8	-40.0	2	3	2	106.7	-114.5	13	3	5	0.0 *	-0.6
16	2	3	0.0 *	-16.4	5	2	7	26.1	-21.8	3	3	2	104.1	115.2	-13	3	6	15.6	-17.4
-15	2	4	31.7	-30.1	6	2	7	27.0	-25.7	4	3	2	13.7	-12.8	-12	3	6	36.1	-31.0
-14	2	4	40.9	-38.5	7	2	7	0.0 *	14.8	5	3	2	60.0	59.6	-11	3	6	24.8	24.1

Table 5. Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-10	3	6	16.9	12.6	3	4	0	20.6	-16.5	13	4	2	0.0 *	8.4	-1	4	5	42.3	-44.7
-9	3	6	0.0 *	12.2	4	4	0	49.9	-49.7	14	4	2	43.6	45.6	0	4	5	42.3	36.7
-8	3	6	20.4	16.1	5	4	0	83.8	83.6	15	4	2	0.0 *	-7.7	1	4	5	74.4	90.7
-7	3	6	0.0 *	9.7	6	4	0	43.3	-43.0	-15	4	3	0.0 *	0.7	2	4	5	18.0	-17.3
-6	3	6	0.0 *	9.4	7	4	0	39.7	39.9	-14	4	3	27.0	24.9	3	4	5	0.0 *	2.8
-5	3	6	17.5	16.6	8	4	0	91.1	99.5	-13	4	3	0.0 *	-1.7	4	4	5	19.8	-13.1
-4	3	6	11.9	-9.8	9	4	0	64.3	-65.1	-12	4	3	10.9	9.5	5	4	5	65.7	-70.4
-3	3	6	28.9	-27.8	10	4	0	25.5	25.7	-11	4	3	0.0 *	8.9	6	4	5	0.0 *	0.7
-2	3	6	38.1	-37.3	11	4	0	41.1	-40.1	-10	4	3	13.2	-12.4	7	4	5	32.9	30.7
-1	3	6	15.9	-15.5	12	4	0	83.1	-87.2	-9	4	3	30.5	21.7	8	4	5	0.0 *	7.3
0	3	6	50.5	46.5	13	4	0	19.7	19.0	-8	4	3	0.0 *	12.0	9	4	5	0.0 *	16.1
1	3	6	16.1	15.3	14	4	0	17.3	-13.9	-7	4	3	42.1	-45.8	10	4	5	24.6	22.3
2	3	6	66.5	74.5	15	4	0	28.1	29.5	-6	4	3	36.6	31.5	11	4	5	36.6	-37.7
3	3	6	30.1	-26.9	16	4	0	32.7	41.3	-5	4	3	27.1	25.1	12	4	5	16.0	18.0
4	3	6	44.3	-44.9	-16	4	1	15.0	-15.7	-4	4	3	32.5	-35.1	-12	4	6	48.6	-52.9
5	3	6	0.0 *	13.3	-15	4	1	0.0 *	-2.5	-3	4	3	117.0	120.9	-11	4	6	28.8	24.3
6	3	6	54.1	-52.6	-14	4	1	0.0 *	-11.4	-2	4	3	98.2	-105.6	-10	4	6	0.0 *	7.0
7	3	6	63.4	63.6	-13	4	1	0.0 *	5.3	-1	4	3	93.7	-104.4	-9	4	6	43.3	35.7
8	3	6	58.5	61.7	-12	4	1	13.6	15.6	0	4	3	7.9	-7.2	-8	4	6	69.5	63.3
9	3	6	0.0 *	-16.5	-11	4	1	22.7	-20.3	1	4	3	115.4	-130.1	-7	4	6	0.0 *	-1.3
10	3	6	27.5	29.6	-10	4	1	0.0 *	-7.0	2	4	3	82.2	86.4	-6	4	6	31.5	-24.8
11	3	6	50.0	-53.8	-9	4	1	27.0	24.9	3	4	3	105.1	110.4	-5	4	6	35.2	-32.4
12	3	6	38.0	-47.8	-8	4	1	52.0	-44.4	4	4	3	45.2	40.9	-4	4	6	24.1	-23.2
-11	3	7	16.4	-13.9	-7	4	1	25.4	20.9	5	4	3	60.7	53.7	-3	4	6	14.9	-14.3
-10	3	7	17.5	15.4	-6	4	1	0.0 *	8.7	6	4	3	30.5	-27.0	-2	4	6	35.3	35.8
-9	3	7	65.4	56.0	-5	4	1	116.4	-114.4	7	4	3	60.2	-66.1	-1	4	6	23.0	16.8
-8	3	7	33.5	-31.8	-4	4	1	81.2	80.7	8	4	3	33.1	-30.8	0	4	6	29.7	-23.9
-7	3	7	22.7	21.5	-3	4	1	9.4	9.5	9	4	3	23.4	21.0	1	4	6	0.0 *	9.8
-6	3	7	42.7	-40.7	-2	4	1	14.8	14.7	10	4	3	0.0 *	-6.5	2	4	6	30.5	-30.2
-5	3	7	71.8	-72.9	-1	4	1	137.5	164.6	11	4	3	28.3	29.3	3	4	6	0.0 *	13.7
-4	3	7	49.1	54.8	0	4	1	61.5	-70.6	12	4	3	0.0 *	-6.6	4	4	6	43.7	41.8
-3	3	7	0.0 *	0.6	1	4	1	55.2	-52.4	13	4	3	40.9	-40.4	5	4	6	13.6	13.3
-2	3	7	47.8	44.3	2	4	1	59.7	-61.0	14	4	3	0.0 *	7.9	6	4	6	17.5	13.4
-1	3	7	85.0	81.2	3	4	1	153.3	-164.1	-14	4	4	51.2	49.2	7	4	6	21.0	-21.2
0	3	7	41.5	-47.8	4	4	1	64.0	61.0	-13	4	4	29.2	-23.8	8	4	6	23.4	-24.3
1	3	7	22.9	-23.1	5	4	1	49.4	42.4	-12	4	4	30.4	23.0	9	4	6	15.7	-14.3
2	3	7	35.5	-31.9	6	4	1	104.4	112.5	-11	4	4	39.5	-35.5	10	4	6	0.0 *	5.1
3	3	7	53.0	-54.9	7	4	1	76.8	75.3	-10	4	4	100.4	-86.5	11	4	6	11.6	11.7
4	3	7	0.0 *	9.6	8	4	1	0.0 *	5.7	-9	4	4	0.0 *	11.1	-10	4	7	16.1	8.5
5	3	7	15.9	14.6	9	4	1	33.0	-33.4	-8	4	4	0.0 *	-10.9	-9	4	7	23.6	-17.4
6	3	7	0.0 *	5.2	10	4	1	29.9	-27.9	-7	4	4	55.1	57.2	-8	4	7	0.0 *	2.7
7	3	7	0.0 *	11.2	11	4	1	0.0 *	11.8	-6	4	4	74.3	77.4	-7	4	7	0.0 *	9.8
8	3	7	17.3	14.1	12	4	1	0.0 *	3.2	-5	4	4	42.8	33.0	-6	4	7	15.6	19.2
9	3	7	0.0 *	-0.4	13	4	1	24.9	24.7	-4	4	4	0.0 *	3.4	-5	4	7	0.0 *	6.3
10	3	7	0.0 *	12.4	14	4	1	0.0 *	-6.3	-3	4	4	55.7	-57.6	-4	4	7	21.1	15.1
-9	3	8	15.5	-14.3	15	4	1	13.2	-14.8	-2	4	4	11.9	-13.0	-3	4	7	0.0 *	-7.3
-8	3	8	24.4	-18.7	-15	4	2	16.2	12.9	-1	4	4	58.4	-54.7	-2	4	7	0.0 *	-17.5
-7	3	8	0.0 *	-5.5	-14	4	2	62.9	-60.1	0	4	4	48.8	25.0	-1	4	7	23.0	26.8
-6	3	8	19.9	-18.7	-13	4	2	47.7	49.8	1	4	4	0.0 *	-2.5	0	4	7	20.6	-15.2
-5	3	8	18.0	-13.5	-12	4	2	68.2	62.6	2	4	4	33.9	-27.2	1	4	7	0.0 *	-2.7
-4	3	8	0.0 *	-3.5	-11	4	2	18.9	15.7	3	4	4	18.5	-20.1	2	4	7	0.0 *	2.8
-3	3	8	0.0 *	-1.4	-10	4	2	72.9	71.0	4	4	4	23.1	-24.7	3	4	7	37.5	-39.0
-2	3	8	19.2	22.7	-9	4	2	46.6	-45.4	5	4	4	0.0 *	-5.1	4	4	7	0.0 *	3.7
-1	3	8	19.2	20.0	-8	4	2	84.5	-84.9	6	4	4	22.3	16.0	5	4	7	22.4	21.9
0	3	8	21.5	19.1	-7	4	2	42.1	-33.1	7	4	4	30.3	28.7	6	4	7	0.0 *	4.6
1	3	8	0.0 *	-2.8	-6	4	2	49.3	-50.4	8	4	4	22.0	-22.9	7	4	7	0.0 *	14.8
2	3	8	35.5	-40.3	-5	4	2	62.1	70.5	9	4	4	16.6	16.7	8	4	7	15.9	16.2
3	3	8	0.0 *	5.6	-4	4	2	67.6	73.5	10	4	4	0.0 *	3.7	9	4	7	16.1	-24.1
4	3	8	16.2	-18.3	-3	4	2	60.5	59.3	11	4	4	0.0 *	-10.7	-8	4	8	30.7	-30.0
5	3	8	22.1	21.9	-2	4	2	0.0 *	9.9	12	4	4	27.1	29.9	-7	4	8	0.0 *	-6.4
6	3	8	36.7	39.7	-1	4	2	27.7	-33.2	13	4	4	0.0 *	-9.9	-6	4	8	0.0 *	-12.9
7	3	8	24.1	-26.2	0	4	2	6.3	-7.4	-13	4	5	0.0 *	0.4	-5	4	8	0.0 *	6.5
-4	3	9	0.0 *	9.0	1	4	2	14.0	-9.2	-12	4	5	33.2	-26.2	-4	4	8	40.8	39.7
-3	3	9	41.1	40.5	2	4	2	28.1	22.4	-11	4	5	0.0 *	5.0	-3	4	8	0.0 *	6.3
-2	3	9	47.5	-48.0	3	4	2	29.3	26.7	-10	4	5	0.0 *	-1.3	-2	4	8	27.2	-22.1
-1	3	9	26.2	-24.8	4	4	2	18.9	-18.0	-9	4	5	0.0 *	-12.8	-1	4	8	0.0 *	10.0
0	3	9	26.2	-20.3	5	4	2	18.4	-15.8	-8	4	5	0.0 *	3.1	0	4	8	17.2	-16.5
1	3	9	35.6	-37.0	6	4	2	46.0	42.8	-7	4	5	0.0 *	-0.8	1	4	8	0.0 *	-2.4
2	3	9	28.2	28.2	7	4	2	54.7	-56.9	-6	4	5	29.8	-30.5	2	4	8	57.9	53.5
3	3	9	18.6	21.0	8	4	2	13.3	11.3	-5	4	5	27.9	28.5	3	4	8	27.6	-28.1
4	3	9	18.1	15.9	9	4	2	0.0 *	-6.4	-4	4	5	24.4	-17.2	4	4	8	0.0 *	-1.6
0	4	0	0.0 *	-7.6	10	4	2	64.7	-70.0	-3	4	5	53.9	-56.7	5	4	8	22.9	-26.5
1	4	0	37.2	-32.4	11	4	2	29.0	32.6	-2	4	5	40.7	38.6	6	4	8	41.0	-48.3
2	4	0	26.8	22.4	12	4	2	0.0 *	7.7										

O(4) to the terminal oxygens range from 2.744 to 2.790 $\pm$ 22 Å, and the distances between opposite feet are 3.286 and 3.478 $\pm$ 18 Å (see Figs. 2a – b).

The Rb – O distances are given in Table 7 together with the estimated standard deviations. The coordination polyhedron around the Rb atoms is irregular as indicated in Fig. 1. Both Rb(1) and Rb(2) are coordinated to eight oxygen atoms. The Rb(1) – O distances range from 2.897 to 3.173 $\pm$ 19 Å including oxygens of five different Cr_2O_7 complexes. Rb(2) has six Cr_2O_7 neighbours, and the contact distances range from 2.857 to 3.084 $\pm$ 20 Å. In Ref. 5 the distance range for 7-fold coordination is given as 2.90 – 3.20 Å.

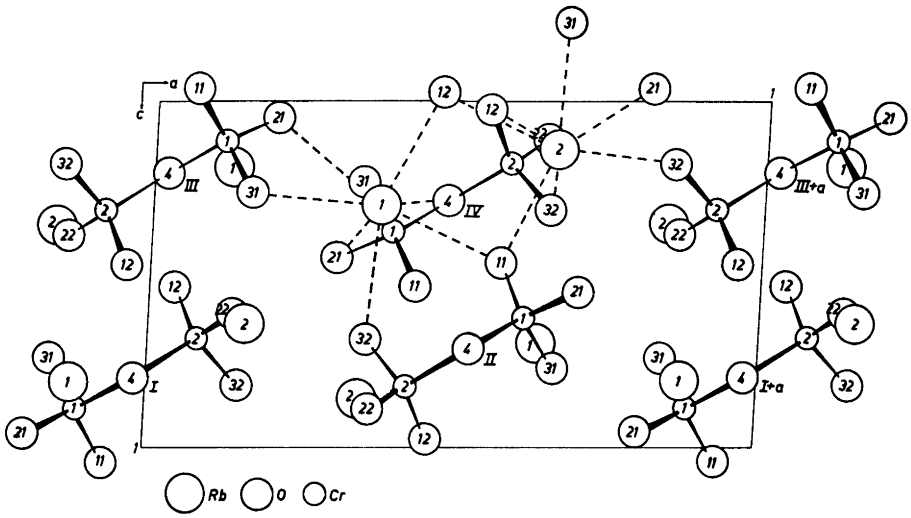

Fig. 1. Projection of the structure of $Rb_2Cr_2O_7$ ($P2_1/n$) along [010]. Rb – O bonds are broken.

Structure determinations of dichromates carried out with reasonable accuracy are relatively scarce. Some refined data are given in Table 8. It is seen that the Cr – O (terminal) distances are all in the range 1.58 – 1.64 Å, whereas the Cr – O (bridging) distances range from 1.76 to 1.80 Å. These values are comparable with those given by Stephens and Cruickshank [8] for the refined chromium trioxide structure, which consists of infinite chains of

Table 6. Distances (Å) and angles (°) within the dichromate ion with estimated standard deviations in $Rb_2Cr_2O_7$ ($P2_1/n$). The numbering of the atoms refers to Fig. 1. The standard errors are those indicated by the least squares refinement.

Distances:

Cr(1) – Cr(2)	3.155 ± 4	O(11) – O(21)	2.610 ± 23	O(12) – O(32)	2.604 ± 17
Cr(1) – O(11)	1.594 ± 15	O(11) – O(31)	2.614 ± 19	O(12) – O(4)	2.781 ± 19
Cr(1) – O(21)	1.598 ± 16	O(11) – O(4)	2.788 ± 21	O(22) – O(32)	2.620 ± 24
Cr(1) – O(31)	1.595 ± 16	O(11) – O(32)	3.478 ± 18	O(22) – O(4)	2.744 ± 20
Cr(1) – O(4)	1.796 ± 14	O(21) – O(31)	2.601 ± 23	O(32) – O(4)	2.790 ± 19
Cr(2) – O(12)	1.596 ± 14	O(21) – O(4)	2.762 ± 18		
Cr(2) – O(22)	1.580 ± 18	O(31) – O(4)	2.755 ± 22		
Cr(2) – O(32)	1.617 ± 14	O(31) – O(12)	3.286 ± 18		
Cr(2) – O(4)	1.795 ± 13	O(12) – O(22)	2.604 ± 24		

Angles:

Cr(1) – O(4) – Cr(2)	122.9 ± 0.9	O(12) – Cr(2) – O(22)	110.2 ± 0.8
O(11) – Cr(1) – O(21)	109.7 ± 0.7	O(12) – Cr(2) – O(32)	108.3 ± 0.8
O(11) – Cr(1) – O(31)	110.1 ± 0.9	O(12) – Cr(2) – O(4)	110.1 ± 0.6
O(11) – Cr(1) – O(4)	110.5 ± 0.7	O(22) – Cr(2) – O(32)	110.1 ± 0.8
O(21) – Cr(1) – O(31)	109.1 ± 0.8	O(22) – Cr(2) – O(4)	108.6 ± 0.8
O(21) – Cr(1) – O(4)	108.8 ± 0.8	O(32) – Cr(2) – O(4)	109.6 ± 0.6
O(31) – Cr(1) – O(4)	108.6 ± 0.7		

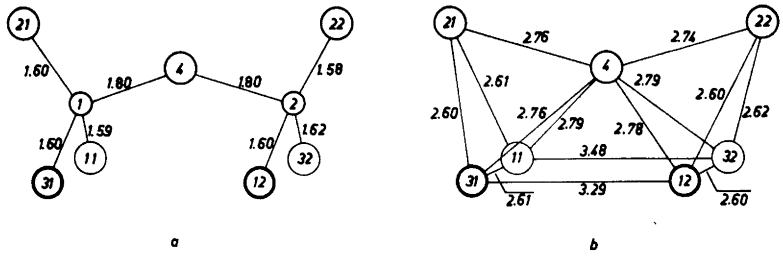

Fig. 2a. Chromium-oxygen distances within the dichromate ion.
Fig. 2b. Oxygen-oxygen distances within the dichromate ion.

corner-sharing CrO_4 tetrahedra with an average terminal Cr – O distance of 1.599 Å and a Cr – O(bridging) distance of 1.748 Å. The Cr – O(bridging) – Cr angles generally have values between 122° and 128°. Quite large, significantly deviating values are reported for $Rb_2Cr_2O_7$ ($P\bar{1}$) (138° in one of the crystallographically nonequivalent anions) [3] and in CrO_3 (143°).[8]

Table 7. Interatomic distances (Å) Rb – O < 3.7 Å with estimated standard deviations in $Rb_2Cr_2O_7$ ($P2_1/n$). The numbering of the atoms refers to Fig. 1. The notation $+a$, $+b$, $-c$ indicates an atom translated $+a$, $+b$, and $-c$, respectively. Corresponding distances in $Rb_2Cr_2O_7$ ($C2/c$) within parentheses.

$Rb(1)-O(12)_{II(-c)}$	2.897 ± 12	(2.887)	$Rb(2)-O(11)_{II}$	2.857 ± 13	(2.990)
$Rb(1)-O(11)_{II}$	2.899 ± 14	(2.990)	$Rb(2)-O(32)_{III(+a)}$	2.914 ± 14	(2.900)
$Rb(1)-O(32)_{II}$	2.926 ± 12	(2.889)	$Rb(2)-O(31)_{II(-c)}$	2.919 ± 13	(2.889)
$Rb(1)-O(31)_{IV}$	3.002 ± 19	(2.926)	$Rb(2)-O(32)_{IV}$	2.931 ± 17	(2.926)
$Rb(1)-O(31)_{III}$	3.016 ± 14	(2.900)	$Rb(2)-O(22)_{IV(+b)}$	2.946 ± 20	(2.952)
$Rb(1)-O(4)_{IV(+b)}$	3.058 ± 16	(3.695)	$Rb(2)-O(21)_{I(+a-c)}$	3.031 ± 16	(3.143)
$Rb(1)-O(21)_{III}$	3.172 ± 14	(3.449)	$Rb(2)-O(12)_{II(-c)}$	3.043 ± 12	(2.887)
$Rb(1)-O(21)_{IV(+b)}$	3.173 ± 18	(2.952)	$Rb(2)-O(12)_{IV}$	3.084 ± 15	(3.120)
$Rb(1)-O(11)_{IV}$	3.584 ± 17	(3.120)	$Rb(2)-O(22)_{III(+a)}$	3.394 ± 16	(> 4)

The Cr – O(terminal) bond angles, not given in Table 8, are all very close to the value of the regular tetrahedron; which is 109.5°.

The values of the oxygen-oxygen distances within the dichromate ion observed in $P2_1/n$ (see Fig. 2b) also correspond very closely to those reported for $Rb_2Cr_2O_7$ ($C2/c$)[4] and $K_2Cr_2O_7$ ($P1$).[9]

The magnitude of the deviation from the ideal eclipsed conformation is expressed in terms of the twist angle (see Table 8). This angle is defined according to Ref. 7 as the average of the three smallest angles subtended by the projections of opposite Cr – O(terminal) bond distances onto the plane perpendicular to the Cr – Cr line.

In Table 7, the Rb – O distances of $Rb_2Cr_2O_7$ ($P2_1/n$) are given for comparison with those observed in $Rb_2Cr_2O_7$ ($C2/c$). It can be seen that the coordination number of the rubidium atoms is eight in both compounds. The eight oxygen atoms of the coordination polyhedra, RbO_8 in the $C2/c$ type

and $Rb(2)O_8$ in the $P2_1/n$ type, belong to six different Cr_2O_7 groups. The $Rb(1)O_8$ polyhedron of the latter structure is formed by oxygen atoms from five different Cr_2O_7 groups. This difference is due to the different roles of the bridging oxygen atoms O(4), in the two structures. The O(4) atom is in contact with the Rb(1) atom in the $P2_1/n$ structure but not in the $C2/c$ structure. The coordination numbers of the rubidium atoms around a dichromate ion in the $P2_1/n$ and $C2/c$ structure are eleven and twelve, respectively.

Recently, Brown and Calvo [7] have proposed a scheme for classifying the different structures with $A_2M_2O_7$ stoichiometry in which the A atom is a large cation (>0.97 Å), and M is a small atom (<0.60 Å).[7] The structures can be described in terms of the possible ways of stacking sheets composed of two centrosymmetrically related $A_2M_2O_7$ groups forming the so-called 'basic unit'. Two translation vectors B and C generate a sheet from the basic unit. The C vector lies in the plane of the feet and makes an angle of 60° with the backbone of the M_2O_7 group, while the B vector which usually is perpendicular to C is almost normal to the plane of the feet. Different orientations of the sheets give rise to several structure types which have been summarized by Brown and Calvo.[7]

It is possible to describe the structure of $Rb_2Cr_2O_7$ ($C2/c$) and $Rb_2Cr_2O_7$ ($P2_1/n$) in terms of two alternating sheets parallel with the bc-plane. One of

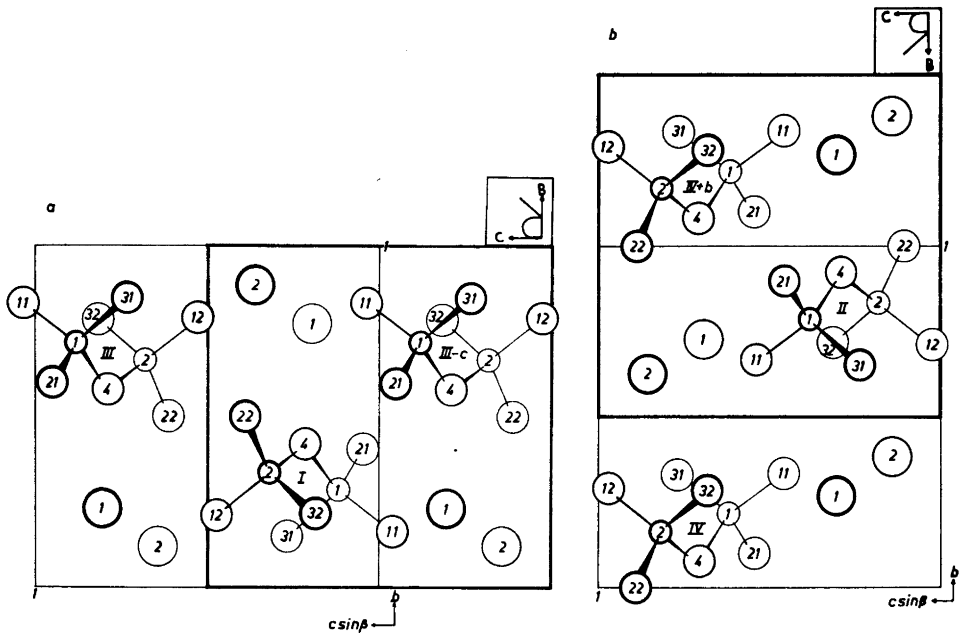

Fig. 3a–b. Projections along [100] of the two first sheets of $Rb_2Cr_2O_7$ ($P2_1/n$). To allow a comparison with $Rb_2Cr_2O_7$ ($C2/c$) the parameters x, y, and z given in Table 4 are transformed to $x'=x$, $y'=y$, and $z'=1-z$. The basic units are indicated by heavy lines. The symbol R represents the translation vectors B and C according to Ref. 7 (see text).

those is composed of $Rb_2Cr_2O_7$ (I) + $Rb_2Cr_2O_7$ (III) (sheet 1), the other of $Rb_2Cr_2O_7$ (II) + $Rb_2Cr_2O_7$ (IV) (sheet 2) (Fig. 1).

In Figs. 3a and 3b the basic units of the first and second sheet of $Rb_2Cr_2O_7$ ($P2_1/n$) are illustrated. This structure belongs to the group VII-type of the

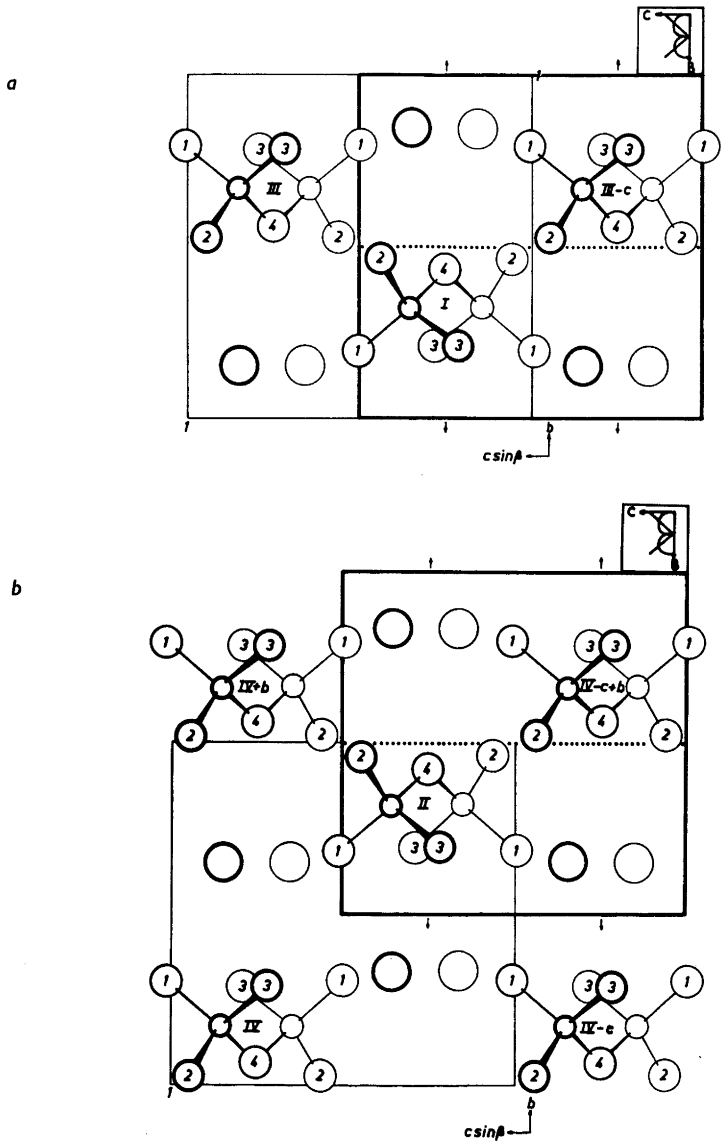

Fig. 4a–b. Projections along [100] of the two first sheets of $Rb_2Cr_2O_7$ ($C2/c$). The basic units are indicated by heavy lines. The symbol R represents the translation vectors B and C according to Ref. 7 (see text).

Brown-Calvo scheme, where adjacent sheets are related by two-fold screw axes lying between them and glide planes perpendicular to them.

The corresponding illustrations for the $C2/c$ structure are given in Figs. 4a and 4b. In this case the sheets, in addition to a center of symmetry, have a two-fold axis along B, running through the bridging oxygen atoms, and a glide plane along C. Adjacent sheets are related by a translation $\left(\frac{a}{2}+\frac{b}{2}\right)$, characteristic for group X.

Table 8. Distances and angles with estimated standard deviations within the dichromate ion for some dichromates.

Compound	Structure type according to Ref. 7	Cr—O (terminal) distances (Å)	Cr—O (bridging) distances (Å)	Cr—O—Cr angle (°)	Twist angle (°) (see text)	Ref.
$Rb_2Cr_2O_7$ ($P2_1/n$)	VII	1.580—1.614±18	1.795, 1.796±14	122.9±0.9	2	This paper
$Rb_2Cr_2O_7$ ($C2/c$)	X	1.609—1.627±14	1.774±10	122.9±1.2	4	4
$K_2Cr_2O_7$ ($P\bar{1}$)	V	1.583—1.633±4 [a] 1.602—1.643 [b]	1.775—1.784±3 [a] 1.781—1.790 [b]	124.0,127.6±0.2	5, 10	9
$Ag_2Cr_2O_7$ ($P\bar{1}$)	I	1.624±8 (mean)	1.782, 1.786	121.2	—	10 [c]
$Rb_2Cr_2O_7$ ($P\bar{1}$)	VIII	1.593—1.630±10 [a] 1.607—1.636 [b]	1.759—1.789±10 [a] 1.768—1.799 [b]	123.0, 137.5±1.0	1, 9	3

[a] and [b] Data corresponding to structure obtained after refinement with isotropic and anisotropic thermal parameters, respectively.
[c] Preliminary report.

The comparison of the structures of $Rb_2Cr_2O_7$ ($P2_1/n$) and $Rb_2Cr_2O_7$ ($C2/c$) has revealed a very close relationship between the two structures with regard to the dimensions of the $Cr_2O_7^{2-}$ anions and also to the Rb—O bonding scheme between the sheets. The widths of the sheets are, however, different. This difference is mainly reflected in the increase of the a parameter from 13.332 Å ($C2/c$) to 13.714 Å ($P2_1/n$) and a corresponding increase of the unit cell volume from 778.1 Å^3 to 801.5 Å^3. The difference is due to a more symmetrical and denser packing in the $C2/c$ compound.

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