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The Crystal Structure of α - $\text{Na}_2\text{Cr}_2\text{O}_7$ and the α - β Phase Transition

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α - $\text{Na}_2\text{Cr}_2\text{O}_7$ is triclinic, $A\bar{1}$, with $a = 7.82$ (3), $b = 10.36$ (3), $c = 9.54$ (3) Å, $\alpha = 89.5$ (3), $\beta = 110.1$ (3), $\gamma = 113.4$ (3)°, $Z = 4$. The structure has been refined ($R_2 = 0.054$) using 714 non-equivalent reflexions measured from precession photographs taken at 260°C. The dichromate geometry is normal with a Cr–O–Cr angle of 135°. The structure is derived from that of β - $\text{Na}_2\text{Cr}_2\text{O}_7$ by a gradual displacement of the atoms in the crystal until the two crystallographically distinct layers become equivalent. The thermal vibrations of the dichromate ion indicate that it is undergoing large antisymmetric torsional vibrations, which are probably associated with the soft mode that causes the transition.

Introduction

In a previous paper (Panagiotopoulos & Brown, 1972a) we reported the crystal structure of the room temperature (β) phase of $\text{Na}_2\text{Cr}_2\text{O}_7$. Above 240°C the crystal undergoes a displacive phase transition to α - $\text{Na}_2\text{Cr}_2\text{O}_7$ in a way which does not destroy the crystal. We now report the crystal structure of the high temperature phase and comment on the nature of the phase transition.

Experimental procedure

The single crystal of β - $\text{Na}_2\text{Cr}_2\text{O}_7$ used for this work was prepared by the method described by Panagiotopoulos & Brown (1972a) and was sealed in a thin-walled quartz capillary tube in a nitrogen atmosphere.

The crystal and tube were heated in a hot air stream and the temperature, which was kept within a 5°C range over the course of each photographic exposure, was monitored by a thermocouple set close above the crystal. The cell constants of the α phase, given in Table 1, were measured from precession photographs

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calibrated with exposures of the same crystal at room temperature.

Table 1. *Crystal data for α and β Na₂Cr₂O₇*

Standard errors in the last figures quoted are given in parentheses

Compound Space group	β -Na ₂ Cr ₂ O ₇ <i>P</i> $\bar{1}$	α -Na ₂ Cr ₂ O ₇ <i>A</i> $\bar{1}$
<i>a</i> (Å)	7.702 (10)	7.82 (3)
<i>b</i> (Å)	10.380 (10)	10.36 (3)
<i>c</i> (Å)	9.402 (10)	9.54 (3)
α (°)	89.41 (10)	89.5 (3)
β (°)	109.57 (10)	110.1 (3)
γ (°)	114.26 (10)	113.4 (3)
<i>V</i> (Å ³)	639	659
<i>Z</i>	4	4
<i>D_m</i>	2.73 (1)	
<i>D_x</i>	2.72	2.64
μ (Mo <i>K</i> α)	3.67 mm ⁻¹	3.56 mm ⁻¹

Intensities were measured from integrated precession photographs taken with Mo *K* α radiation from a single crystal mounted on a precession camera with the [111]* reciprocal axis along the goniometer axis. The crystal had an irregular shape which could be approximated by a sphenoid with a height of 0.29 mm along [111]*, a base of 0.19 mm length parallel to the (011)* reciprocal plane and a length of 0.15 mm normal to it. Integrated intensity photographs were taken of the layers *h*, $-h, l$; *h*, $-(h+1), l$; *h*, $-(h+2), l$; *h, k, -k*; *h, k, -(k+2)*; *h, k, h*; *h, k, h+1* at an estimated temperature of 260 (± 5)°C. The intensities were measured with a Joyce-Loebl microdensitometer and were corrected for Lorentz and polarization effects.

In all, 820 reflexions were measured. After averaging of the common reflexions a set of 512 observed reflexions and 202 unobserved reflexions was obtained. An absorption correction was not considered necessary ($0.5 < \mu R < 1.0$).

The atomic coordinates obtained during the refinement of the structure of the β phase in the superstruc-

ture space group *A* $\bar{1}$ (Panagiotopoulos & Brown, 1972*a*) were used as the initial coordinates for the α phase structure. This model was refined with isotropic temperature factors, individual scale factors for each layer and unit weights. After three cycles the agreement index was $R_1 = 0.09$. Further refinement was attempted with a Cruickshank weighting scheme, anisotropic temperature factors on all atoms and one overall scale factor. With 100 parameters the agreement indices dropped to $R_2 = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2} = 0.054$ and $R_1 = [\sum (|F_o| - |F_c|) / \sum |F_o|] = 0.043$. In the final cycle of the refinement 18 unobserved reflexions, with calculated structure factors larger than the smallest observed amplitude, were included. The reflexions were weighted with the function $w = (3.17 - 0.111|F_o| + 0.0025|F_o|^2)^{-1}$. The scattering factors used for the Na⁺, Cr²⁺, O⁻ (terminal) and O (bridging) were those given in *International Tables for X-ray Crystallography* (1962).

The final atomic positions and temperature factors are given in Table 2. The observed and final calculated structure factors are given by Panagiotopoulos (1972).

The structure is closely related to that of β -Na₂Cr₂O₇ which has been described in detail by Panagiotopoulos & Brown (1972*a*). The two crystallographically distinct (010) layers of β -Na₂Cr₂O₇ become identical in α -Na₂Cr₂O₇ and the conformation of the high temperature layer is almost midway between those of the two low temperature layers giving a structure that is intermediate between the thortveitite-type and the dichromate-type of structure (Brown & Calvo, 1970). Interatomic distances and angles are given in Table 3.

Possible disorder in α -Na₂Cr₂O₇

Although the geometry of the dichromate ion in α -Na₂Cr₂O₇ shows bond lengths and angles close to the ones found in the other dichromates, the temperature factors are considerably larger. Most of this increase can be attributed to the higher temperature at which the measurements were made, but the atoms O(22),

Table 2. *Atomic positional and thermal coordinates for α -Na₂Cr₂O₇*

Standard errors in the last figures quoted as given by the final round of least-squares analysis are shown in parentheses. The temperature factors were calculated using the expression

$$\exp [-2\pi^2(u_{11}h^2a^{*2} + u_{22}k^2b^{*2} + u_{33}l^2c^{*2} + 2u_{12}hka^*b^* + 2u_{13}hla^*c^* + 2u_{23}k|b^*c^*) \times 10^{-4}]$$

Atoms O(11)–O(13) are attached to Cr(1). Atoms O(21)–O(23) are attached to Cr(2). O(B12) bridges Cr(1) and Cr(2).

	<i>x</i>	<i>y</i>	<i>z</i>	<i>u</i> ₁₁	<i>u</i> ₂₂	<i>u</i> ₃₃	<i>u</i> ₁₂	<i>u</i> ₁₃	<i>u</i> ₂₃
Cr(1)	-0.1842 (2)	0.0204 (2)	0.1397 (2)	362 (8)	420 (12)	390 (8)	164 (7)	92 (6)	25 (7)
Cr(2)	0.2391 (2)	0.0680 (2)	0.4086 (2)	427 (9)	438 (13)	336 (7)	206 (8)	74 (7)	21 (8)
O(11)	-0.3569 (12)	0.0439 (9)	0.1782 (8)	649 (45)	747 (72)	693 (45)	366 (44)	340 (43)	106 (47)
O(12)	-0.2347 (11)	-0.1456 (9)	0.1159 (9)	619 (46)	589 (61)	759 (49)	208 (41)	250 (41)	-39 (43)
O(13)	-0.1536 (12)	0.0891 (10)	-0.0088 (8)	784 (52)	867 (73)	565 (42)	310 (48)	385 (44)	269 (46)
O(21)	0.3338 (11)	0.1541 (9)	0.5757 (7)	691 (47)	783 (69)	466 (37)	336 (45)	134 (38)	-3 (43)
O(22)	0.4107 (14)	0.1128 (15)	0.3367 (10)	633 (55)	1820 (137)	852 (60)	98 (69)	425 (56)	-310 (76)
O(23)	0.1523 (18)	-0.0981 (12)	0.4107 (11)	1693 (99)	641 (74)	826 (59)	769 (72)	-22 (65)	22 (65)
O(B12)	0.0504 (11)	0.1137 (9)	0.2914 (9)	667 (46)	450 (52)	896 (55)	238 (39)	-210 (41)	33 (45)
Na(1)	0.3377 (8)	0.3429 (6)	0.7265 (5)	1021 (36)	798 (44)	586 (25)	540 (31)	275 (27)	168 (28)
Na(2)	-0.2351 (7)	0.2382 (6)	0.3934 (5)	690 (27)	697 (37)	608 (25)	93 (24)	302 (24)	95 (23)

Table 3. *Interatomic distances and angles for α - $\text{Na}_2\text{Cr}_2\text{O}_7$*

	Distance (Å)	Angles (°)		
		O(12)	O(13)	O(B12)
Cr(1)-O(11)	1.620	111	112	109
Cr(1)-O(12)	1.604		110	110
Cr(1)-O(13)	1.627			105
Cr(1)-O(B12)	1.785			
		O(22)	O(23)	O(B12)
Cr(2)-O(21)	1.604	109	111	110
Cr(2)-O(22)	1.618		109	107
Cr(2)-O(23)	1.583			110
Cr(2)-O(B12)	1.750			

Cr(1)-O(B12)-Cr(2) 135.1°

The angles are those subtended at Cr by the oxygen atoms at the head of the column and on the left of the row.

	Distance (Å)		Distance (Å)
Na(1)-O(13)	2.367	Na(2)-O(22)	2.405
Na(1)-O(21)	2.423	Na(2)-O(23)	2.425
Na(1)-O(12)	2.561	Na(2)-O(12)	2.448
Na(1)-O(21)	2.601	Na(2)-O(11)	2.526
Na(1)-O(11)	2.654	Na(2)-O(13)	2.660
Na(1)-O(22)	2.762	Na(2)-O(B12)	2.736
Na(1)-O(23)	2.835	Na(2)-O(22)	2.939

Other Na-O distances are all greater than 3.3 Å.

Standard errors derived from least-squares refinement:

Cr-O = 0.010 Å, Na-O = 0.012 Å,

O-Cr-O = 1°, Cr-O-Cr = 0.6°.

O(23), O(B12) and Na(1) have temperature factors that are large even relative to the other atoms in the structure. There are two alternative interpretations of these temperature factors, either they represent real thermal effects or the structure above the phase transition is disordered. To test this latter possibility, a disordered model was obtained by a superposition of the β - $\text{Na}_2\text{Cr}_2\text{O}_7$ structure with itself translated by the A centring operation.

Refinement of this model for α - $\text{Na}_2\text{Cr}_2\text{O}_7$ using isotropic temperature factors (89 variables) gave final

agreement indices of $R_1=0.051$ and $R_2=0.063$. In the final model it was not possible to identify two chemically reasonable dichromate ions; bond lengths for the terminal oxygen atoms, for example, deviated up to 0.1 Å from the normal values of 1.60 to 1.63 Å. Hamilton's (1965) statistical test applied to the α ordered model ($R_1=0.043$, $R_2=0.054$, 100 variables) and the α disordered model ($R_1=0.051$, $R_2=0.063$, 89 variables) showed that the α disordered structure must be rejected at the 0.005 significance level. To test further the correctness of the ordered model, we observed the intensities of the reflexions of a single crystal of $\text{Na}_2\text{Cr}_2\text{O}_7$ as a function of temperature using a series of precession photographs. Reflexions with $k+l$ =odd became weaker as the temperature was raised, becoming zero at the transition temperature, while reflexions with $k+l$ even changed only slightly. No diffuse scattering was observed around the positions of the $k+l$ odd reflexions either below or above 240°C and their shape remained sharp up to the transition, further indicating that the phase transition does not involve disorder.

The statistical test, the molecular geometry, and the absence of any diffuse reflexions in the X-ray photographs all indicate that α - $\text{Na}_2\text{Cr}_2\text{O}_7$ is fully ordered and that the phase transition is a displacive transition rather than an order-disorder transition. It follows that the atoms O(22), O(23), O(B12) and Na(1) have large real thermal motions, an effect that will be discussed below.

Thermal motion and the phase transition

The dichromate ions in all structures reported so far consist of two CrO_4 tetrahedra sharing one oxygen atom (the bridging oxygen). Each of the CrO_4 tetrahedra has almost exact C_{3v} symmetry around the Cr-O (bridging) bond and is essentially invariant between

Table 4. *Conformations and thermal motion in various Cr_2O_7 groups*

Compound	Structure type* (phase)	Cr_2O_7 group	Cr-O(B)-Cr (°)	$\alpha_1 - \alpha_2$ (°)	$\alpha_1 + \alpha_2$ (°)	Δ_B^\dagger (°)	Δ_T^\ddagger (°)	$\langle \text{Cr-O}_B \rangle$ (Å)	$\langle \text{Cr-O}_T \rangle$ (Å)
1 $\text{Ag}_2\text{Cr}_2\text{O}_7$	I		121	16	6			1.78	1.62 (a)
2 $\text{Rb}_2\text{Cr}_2\text{O}_7$	VII		123	7	1			1.80	1.60 (b)
3 $\text{Rb}_2\text{Cr}_2\text{O}_7$	X		123	4	0			1.77	1.62 (c)
4 $\text{Rb}_2\text{Cr}_2\text{O}_7$	VIII	1-2	123	1	1	8	7	1.78	1.63 (d)
5 $\text{K}_2\text{Cr}_2\text{O}_7$	V	1-2	124	7	3	9	7	1.78	1.63 (e)
6 $\text{K}_2\text{Cr}_2\text{O}_7$	V	3-4	128	8	6	12	7	1.78	1.60 (e)
7 $\text{Na}_2\text{Cr}_2\text{O}_7$	β	1-2	131	37	7	13	6	1.79	1.63 (f)
8 $\text{Na}_2\text{Cr}_2\text{O}_7$	β	3-4	131	94	8	14	7	1.80	1.63 (f)
9 $\text{Na}_2\text{Cr}_2\text{O}_7$	α		135	95	17	27	8	1.77	1.61 (g)
10 $\text{NaRbCr}_2\text{O}_7$		1-2	136	53	13	21	6	1.76	1.61 (h)
11 $\text{Rb}_2\text{Cr}_2\text{O}_7$	VIII	3-4	138	7	3	14	5	1.78	1.62 (d)
12 $\text{NaRbCr}_2\text{O}_7$		3-4	141	80	2	25	6	1.76	1.61 (h)

* See Brown & Calvo (1970)

† Δ_B is the apparent r.m.s. libration of the bridging oxygen atom about the Cr-Cr axis.‡ Δ_T is the apparent r.m.s. libration of the terminal oxygen atoms about the Cr-Cr axis.

(a) Hazell (1969); (b) Löfgren (1971); (c) Löfgren & Waltersson (1971); (d) Panagiotopoulos & Brown (1970); (e) Brandon & Brown (1968); (f) Panagiotopoulos & Brown (1972a); (g) present work; (h) Panagiotopoulos & Brown (1972b).

one structure and another. On the other hand, the relative orientations of the CrO_4 groups vary considerably and the various possible conformations can be described by three angles: the angle subtended at the bridging oxygen atom, $\text{Cr-O}(B)\text{-Cr}$, and the torsion angles α_1 and α_2 which describe the orientation of the two tetrahedra around the Cr-O (bridging) bond. The torsion angle is taken to be zero when a terminal oxygen atom lies in the plane of the bridging oxygen and the two chromium atoms in a *trans* arrangement. It is defined as positive when the terminal oxygen atoms are rotated clockwise from this position as viewed from the bridging oxygen atom. The α_1 and α_2 angles can be used to plot the configuration diagram given in Fig. 1.

These conformation coordinates are related to the normal modes of the thermal vibrations of the Cr_2O_7

group as listed by Luu & Lafont (1970) whose energy assignments lead one to expect that the only modes that will be active at room temperature are the three translational, the three librational and the two torsional modes, the normal coordinates for the latter being α_1 and α_2 . In an isolated ion of C_{2v} symmetry the two torsional modes are degenerate and mix to give an A_2 symmetric torsional mode ($\alpha_1 + \alpha_2$) and a B_2 antisymmetric torsional mode ($\alpha_1 - \alpha_2$). It is not possible to assign the amplitudes of thermal motion uniquely on the basis of elastic X-ray diffraction measurements, but it is clear from the thermal parameters given in Table 4 that the apparent librational motion of the bridging oxygen atom around the Cr-Cr axis is larger than the apparent libration motion of the terminal oxygen atoms in all dichromate ions so far studied. Such an effect can be produced by a coupling

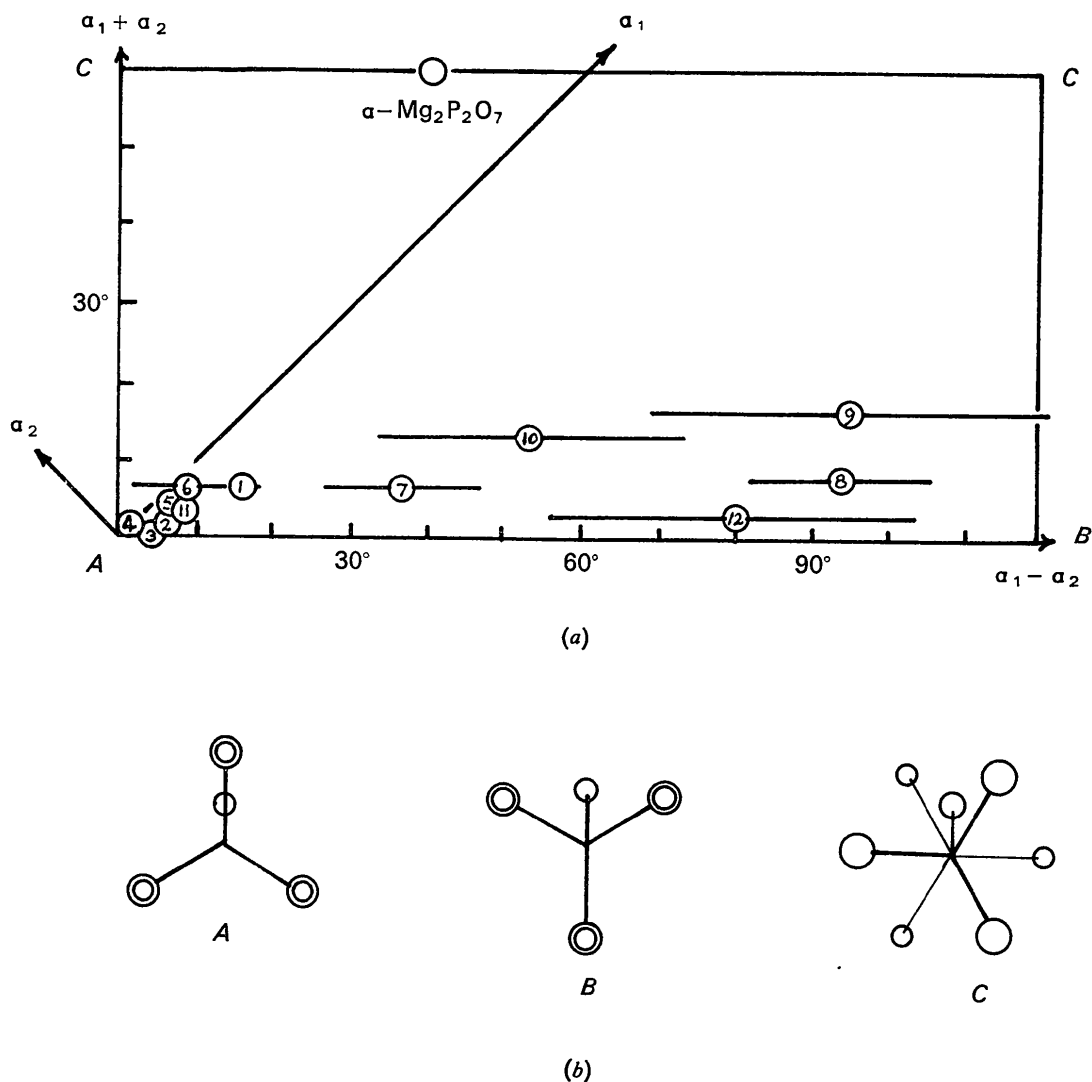


Fig. 1. (a) Conformation diagram for $\text{Cr}_2\text{O}_7^{2-}$ ions. See text for explanation. The numbers refer to Table 4. The horizontal lines indicate the apparent r.m.s. libration of the bridging oxygen atom (Δ_B). (b) The three high symmetry conformations of X_2O_7 groups.

of the antisymmetric torsional mode and the librational mode around the Cr–Cr axis in a manner which allows the bridging oxygen atom to librate with a much larger amplitude than the terminal atoms. Such a motion affects the internal conformation of the $\text{Cr}_2\text{O}_7^{2-}$ ion to a much larger extent than it changes the external shape and orientation of the ion. Consequently the packing of the ions is relatively unaffected by this motion.

Table 4 lists some of the angles and distances, as well as the amplitudes of thermal motion, observed in a number of $\text{Cr}_2\text{O}_7^{2-}$ ions. The conformations of the ions and amplitudes of the asymmetric torsional mode are shown in Fig. 1. Most of the ions lie close to the *A* conformation which has C_{2v} symmetry and the remainder lie close to the line joining *A* and *B*, *B* representing the other eclipsed conformation that has C_{2v} symmetry. There is a correlation between the Cr–O(*B*)–Cr angle, the antisymmetric twist ($\alpha_1 - \alpha_2$) and the amplitude of the antisymmetric torsion mode, each of these tending to increase together. The large amplitudes of vibration observed close to the *B* conformation and the large number of structures containing ions in the *A* conformation suggest that *A* is more stable than *B*. It is this instability that leads to the α - β phase transformation in $\text{Na}_2\text{Cr}_2\text{O}_7$, one of the two $\text{Na}_2\text{Cr}_2\text{O}_7$ groups in the low temperature β phase having a conformation much closer to *A*. Accordingly the antisymmetric torsion–libration mode is expected to be soft immediately above the transition.

This same instability is involved in the V \rightarrow VII phase transition in $\text{K}_2\text{Cr}_2\text{O}_7$. As with the α - β transition in $\text{Na}_2\text{Cr}_2\text{O}_7$, this transition appears to be displacive

in character although in this case there is no doubt that it is first order. Brown & Calvo (1970) have proposed that in this transition the bridging oxygen atoms in one half of the dichromate groups move from one *A* configuration to another passing through an intermediate *B* configuration. The relative instability of *B* compared to *A* results in the transition being very rapid

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Kristall- und Molekularstruktur des chiralen Hexa-*o*-phenylens

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The crystal structure of the chiral isomer of hexa-*o*-phenylene $\text{C}_{36}\text{H}_{24}$ has been determined from counter data. 3291 intensities were measured by diffractometry using Mo $K\alpha$ radiation. Crystals of the orthorhombic form with the space group *Pbcn* were investigated. The lattice dimensions are $a = 14.561$, $b = 10.853$, $c = 15.307$ Å. The structure has been solved by the symbolic addition procedure and refined to $R = 0.055$. In the crystal the molecule has symmetry 2 (C_2) and it has only small deviations from symmetry 222 (D_2). The twelve-membered ring has a loop-like conformation. The benzene rings are slightly distorted in a twist form. Within the twelve-membered ring all the angles are enlarged to a maximum value of 129.3° because of intramolecular repulsion; also because of this effect the bond between the benzene rings of the lateral biphenyl group is lengthened to 1.505 Å compared with 1.482 Å in the central biphenyl group, though the dihedral angles between their ring planes are 46 and 64° respectively.

Einleitung

Im Rahmen unserer Röntgenstrukturuntersuchungen über cyclisch verknüpfte Polyphenyl-Verbindungen

waren die Strukturanalysen der beiden von Wittig & Rümpler (1971) synthetisierten Isomeren des Hexa-*o*-phenylens (I) und (II) von Interesse (siehe auch Staab & Wünsche, 1968). Über die Strukturaufklärung von