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The Crystal Structure of Bis-(2-aminoethylsalicylideneiminato)chromium(III) Iodide

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The crystal structure and molecular configuration of bis-(2-aminoethylsalicylideneiminato)chromium(III) iodide, $C_{18}H_{22}N_4O_2CrI$, has been determined by X-ray diffraction techniques. The compound is triclinic with space group $P\bar{1}$ and cell dimensions $a=9{\cdot}92$, $b=9{\cdot}93$, $c=10{\cdot}45$ Å and $\alpha=93{\cdot}8$, $\beta=92{\cdot}7$, $\gamma=93{\cdot}8°$. There are two molecules in the unit cell and within a molecule there is octahedral coordination around the chromium atom by two tridentate ligands. The structure was solved using the Patterson function and was refined by the full-matrix least-squares method. The R index for the 1481 observed reflexions is 0·114.

Introduction

The structure of this compound was determined as a result of a suggestion that it might be ethylenediamine-*N*,*N*′-ethylenebis(salicylideneiminato)chromium(III) iodide, $C_{18}H_{22}N_4O_2CrI$, (O'Connor, 1966). The postulated structure was that of a planar quadridentate ligand surrounding chromium, with the additional ethylenediamine molecule acting as a bridging group between the chromium atoms, infrared spectral measurements having suggested the presence of a coordinated ethylenediamine molecule (O'Connor & West, 1968). Subsequently, it was realized that the complex could consist of two tridentate ligands about the central chromium atom (Fig. 1.) and this was confirmed by the structure analysis. As far as we are aware, a three-dimensional study of a chromium compound of similar type has not been published (Lingafelter & Braun, 1966).

Experimental

Single crystals used in this determination were provided by Dr M. J. O'Connor and were prepared as described by O'Connor & West (1968).

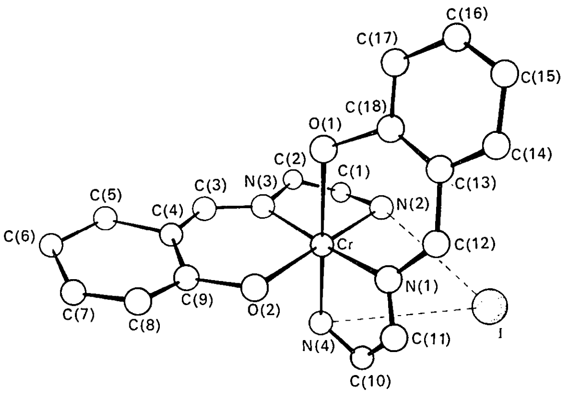

Fig. 1. Numbering scheme used for bis-(2-aminoethylsalicylideneiminato)chromium(III) iodide.

Stoichiometric analysis gave the following values:

	Found	Calculated*
Carbon	43%	42·8%
Hydrogen	4·5	4·4
Iodine	24·9	25·1
Nitrogen	10·7	11·1

* Assuming an empirical formula of $C_{18}H_{22}N_4O_2CrI$.

Crystal data

The crystals of bis-(2-aminoethylsalicylideneiminato)-chromium(III) iodide are dark red triclinic prisms. F.W. 505·3; Space group, $P\bar{1}$; $Z=2$.

Cell parameters:

$a=9{\cdot}92\pm0{\cdot}02$ Å	$\alpha=93°48'\pm30'$
$b=9{\cdot}93\pm0{\cdot}02$	$\beta=92°40'\pm30'$
$c=10{\cdot}45\pm0{\cdot}02$	$\gamma=93°48'\pm30'$

Volume of unit cell = 1024 Å^3
$D_m=1{\cdot}69\pm0{\cdot}01$ g.cm^{-3} (by flotation)
$D_c=1{\cdot}65$ g.cm^{-3}.

The intensities of 1481 independent non-zero reflexions were measured using the integrating multiple-film Weissenberg technique with Cu $K\alpha$ radiation. Six layers were obtained about the k axis, and zero-layer data were obtained about the h and l axes. Precession photographs were used to confirm the cell angles. Since $Z=2$, space group $P\bar{1}$ was assumed and was justified by the subsequent smooth refinement. The usual Lorentz and polarization factors were applied to the observed intensities. The atomic scattering factors based on the Thomas–Fermi–Dirac statistical model were calculated for Cr^{3+}, N, O, C and I (Freeman, 1959).

Calculations were carried out on a CDC 3200 computer.

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Structure determination

The three-dimensional Patterson function contained three very strong peaks arising from the heavy-atom vectors. Assuming that one of these peaks was an (iodine–iodine) vector a Fourier synthesis was calculated, based on the possible iodine position in each case, in order to locate the chromium atoms. Only one pair of chromium and iodine positions satisfied the Patterson function, and gave an initial conventional R value of 0·397. A new Fourier analysis with known chromium and iodine coordinates enabled the nitrogen, oxygen and carbon atom positions to be located. No attempt was made to find the positions of the hydrogen atoms.

The atomic parameters and the individual isotropic temperature factors were refined using a modified version of the Busing & Levy full-matrix least-squares program *ORFLS*, after which the scale factors for $k=0$–5 were adjusted by comparing the observed and calculated structure amplitudes. A three-dimensional difference Fourier synthesis indicated that the chromium and iodine atoms were vibrating somewhat anisotropically. For the final stages of the calculation, anisotropic temperature factors were refined for the chromium and iodine atoms only. A weighting scheme based on that described by Cruickshank (1952) was used.

The final value of the discrepancy factor

$$R=\frac{(\sum||F_o|-|F_c||)}{\sum|F_o|}$$

was 0·114, using the 1481 observed reflexions.

Table 1 shows the final atomic coordinates as fractions of unit-cell parameters, and standard deviations as calculated from the inverse matrix. At this stage the parameter shifts were all less than $\frac{1}{3}$ of the standard deviations.

A final three-dimensional difference Fourier synthesis showed no significant peaks.

Table 2 lists the observed and calculated structure

Table 1. *Atomic positional and thermal parameters with standard deviations for the former in parentheses*

Terms are × 10^4.

	x/a	y/c	z/b	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I	2599 (2)	3337 (2)	290 (2)	321	485	549	74	5	−3
Cr	7929 (4)	2963 (5)	2547 (3)	312	371	406	89	−3	−62
O(1)	9420 (18)	2288 (23)	3526 (15)	342					
O(2)	6489 (18)	2263 (22)	3493 (16)	418					
N(1)	7834 (22)	1243 (27)	1353 (18)	317					
N(2)	9356 (22)	4121 (27)	1561 (19)	747					
N(3)	8083 (25)	4781 (29)	3666 (22)	380					
N(4)	6391 (21)	3534 (25)	1215 (18)	304					
C(1)	114 (26)	5161 (33)	2477 (22)	443	$U_{11}=U_{22}=U_{33}$				
C(2)	9040 (24)	5854 (31)	3253 (20)	355	$U_{12}=U_{13}=U_{23}=0$				
C(3)	7465 (27)	4966 (33)	4777 (23)	380					
C(4)	3480 (25)	6008 (32)	4744 (22)	329					
C(5)	4046 (27)	5550 (34)	3557 (22)	456					
C(6)	5015 (26)	6451 (34)	3079 (22)	418					
C(7)	5556 (26)	7629 (34)	3674 (23)	557					
C(8)	4958 (26)	1883 (34)	5132 (22)	545					
C(9)	5989 (27)	2767 (33)	4615 (23)	304					
C(10)	6408 (29)	2546 (36)	30 (24)	456					
C(11)	6665 (26)	1101 (34)	425 (22)	393					
C(12)	8649 (31)	296 (38)	1353 (26)	1342					
C(13)	9816 (26)	254 (34)	2291 (22)	355					
C(14)	583 (26)	9037 (32)	2132 (22)	545					
C(15)	1638 (26)	8823 (33)	3039 (22)	494					
C(16)	1960 (27)	9858 (33)	4123 (23)	481					
C(17)	1123 (26)	961 (33)	4247 (22)	456					
C(18)	64 (27)	1155 (35)	3328 (22)	329					

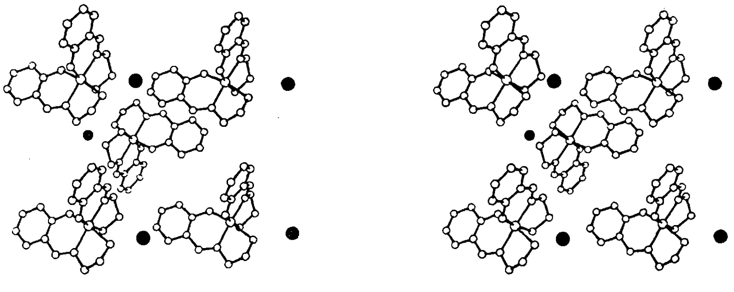

Fig. 2. Stereo view of the packing in the crystal. Black circles indicate iodide ions.

amplitudes. Fig. 2 shows a stereo view of the packing in the crystal.

Bond lengths and angles

Table 3 gives the values for important internuclear distances and bond angles. These, together with the appropriate standard deviations were calculated using program *ORFFE* (Busing, Martin & Levy, 1964). The corrections to covalent bond lengths to allow for thermal motion were calculated assuming random correlation of motion. Corrections were approximately 0·02 Å, but were not applied to the bond lengths.

A survey of some related structures (Baker, Hall & Waters, 1966; Frasson, Panattoni & Sacconi, 1964; Wei, Stogsdill & Lingafelter, 1964; Gerloch & Mabbs, 1967; Braun & Lingafelter, 1966) revealed that the distances C(8)–C(9) & C(17)–C(18) and C(3)–C(4) & C(12)–C(13) (Fig. 1) are normally the largest in the salicylideneiminato group; while in individual structures this is not statistically significant, the fact that the feature is common to most structures is probably significant. Throughout the structure, bond lengths and angles are entirely compatible with the corresponding dimensions in related structures.

The salicylideneiminato group is planar within the limits of our experimental error, and the two such groups in one cation are almost perpendicular (87°).

In the ethylenediamine group, N(2) lies in the plane of the salicylideneiminato group while C(1) and C(2) are spaced above and below the planes, their distances out of the plane being −0·50 and +0·23 Å respectively. The equation for the mean plane through the atoms O(2), N(2), C(3), C(4), C(5), C(6), C(7), C(8), and C(9) is

$$-0\cdot7127x+0\cdot4898y-0\cdot5021z+1\cdot837=0\ ,$$

and for the mean plane through atoms O(1), N(1), N(4), C(12), C(13), C(14), C(16), C(17), and C(18) the equation is

$$0\cdot6452x+0\cdot5223y-0\cdot5575z-2\cdot5983=0\ .$$

Table 2

H	K	L	F(OBS)	F(CAL)
0	0	1	4.0	3.0
0	0	2	5.0	3.6
0	0	3	9.3	8.4
0	0	4	6.4	5.9
0	0	5	2.1	2.9
0	0	6	2.9	2.6
0	0	8	3.4	3.1
0	0	10	2.0	-2.2
1	0	0	2.2	2.9
1	0	1	6.1	5.9
1	0	2	9.3	-8.3
1	0	3	6.3	-5.9
1	0	4	4.4	-3.5
1	0	5	3.9	-3.3
1	0	6	6.3	-6.2
1	0	7	5.6	-5.3
1	0	9	3.1	-3.4
1	0	10	2.6	-3.0
1	0	11	2.2	-2.7
1	0	-1	3.5	-4.7
1	0	-2	5.8	-5.5
1	0	-3	3.9	3.9
1	0	-4	2.6	2.5
1	0	-5	6.0	5.3
1	0	-6	3.3	3.3
1	0	-7	5.3	5.8
1	0	-8	3.8	3.5
1	0	-11	1.9	2.3
1	0	-12	1.8	1.5
-1	0	13	1.4	2.0
2	0	1	6.5	-7.7
2	0	2	7.8	-6.7
2	0	3	7.6	-6.9
2	0	4	5.6	-5.4
2	0	5	3.0	-2.8
2	0	7	3.0	-2.9
2	0	8	1.9	-1.3
2	0	9	2.5	2.3
2	0	-2	7.7	-9.3
2	0	-3	3.2	-3.3
2	0	-4	6.5	-7.3
2	0	-5	3.3	-2.9
2	0	-7	2.1	-2.3
2	0	-8	2.5	-2.3
2	0	-10	2.0	1.3
-2	0	-11	1.4	.9
3	0	0	1.0	.4
3	0	1	3.7	4.1
3	0	2	5.4	5.3
3	0	3	7.5	6.7
3	0	4	4.4	4.6
3	0	5	3.0	2.5
3	0	6	6.1	6.0
3	0	7	3.6	3.8
3	0	8	1.8	2.2
3	0	9	2.0	2.6
3	0	10	1.9	2.1
3	0	11	1.2	1.6
3	0	-1	2.3	3.1
3	0	-2	.8	1.4
3	0	-3	3.9	-5.0
3	0	-4	3.8	-4.0
3	0	-6	2.0	-2.0
3	0	-7	5.4	-6.0
3	0	-8	2.7	-3.5
3	0	-10	2.0	-2.4
3	0	-11	1.8	-2.0
4	0	0	5.7	7.1
4	0	1	2.8	3.8
4	0	3	5.1	6.0
4	0	4	5.8	6.1
4	0	5	1.7	-1.2
4	0	7	1.9	1.5
4	0	9	2.4	-2.2
4	0	-2	3.2	4.2
4	0	-3	1.8	2.6
4	0	-4	5.4	6.1
4	0	-5	4.8	6.1
4	0	-6	1.2	1.2
4	0	-8	2.4	1.8
-4	0	1	6.6	7.1
5	0	0	2.0	1.8
5	0	1	2.3	-2.3
5	0	2	6.4	-7.3
5	0	3	3.2	-3.9
5	0	4	3.1	-3.1
5	0	5	3.0	-3.0
5	0	6	5.4	-6.3
5	0	7	2.5	-2.7
5	0	10	3.0	-2.9
5	0	-3	1.9	2.1
5	0	-4	3.4	4.1
5	0	-5	2.1	2.3
5	0	-6	1.3	1.4
5	0	-7	2.4	2.8
5	0	-8	4.0	4.3
5	0	-9	2.4	2.1
5	0	-10	1.7	1.4
-5	0	1	2.0	-1.6
-5	0	2	1.4	-1.7
-5	0	11	1.9	2.0
-5	0	12	1.7	1.9
-5	0	-9	1.9	-1.8
6	0	0	3.0	-3.4
6	0	1	1.1	-1.2
6	0	2	3.5	-4.0
6	0	3	2.5	-3.1
6	0	9	1.7	2.0
-6	0	0	3.4	-3.4
-6	0	1	7.7	-7.9
-6	0	2	5.4	-5.2
-6	0	3	2.7	-2.6
-6	0	4	3.3	-3.6
-6	0	5	4.6	-4.6
-6	0	6	2.4	-2.5
-6	0	9	1.9	-1.9
7	0	2	3.9	4.0
7	0	3	2.8	3.1
7	0	4	2.8	2.3
7	0	5	2.9	3.7
7	0	6	3.7	4.0
7	0	9	1.6	2.0
-7	0	0	2.1	-1.4
-7	0	2	1.7	1.5
-7	0	4	2.3	-2.4
-7	0	5	1.4	-1.4
-7	0	6	3.0	-2.3
-8	0	6	3.3	2.8
-8	0	9	1.9	1.8
-8	0	-2	3.7	3.6
-8	0	-3	2.3	2.3
-9	0	2	2.0	-1.6
-9	0	3	2.0	-0.6
-9	0	4	1.4	1.0
-9	0	5	1.9	1.9
-9	0	8	2.0	2.2
-9	0	9	1.5	1.9
-9	0	-1	2.6	-2.8
-9	0	-2	3.3	-2.9
-9	0	-3	1.4	-1.0
-9	0	-4	2.2	-2.3
-9	0	-5	3.2	-3.0
-9	0	-6	2.9	-2.7
-10	0	0	1.9	-1.4
-10	0	1	3.8	-3.2
-10	0	2	4.5	-4.0
-10	0	3	2.3	-1.9
-10	0	4	1.8	-1.4
-10	0	5	3.5	-2.9
-10	0	6	2.8	-2.8
-10	0	8	1.2	-0.9
-10	0	-1	2.3	-1.8
-10	0	-2	2.3	-2.1
-10	0	-3	1.3	-1.4
-11	0	0	2.1	1.6
-11	0	3	2.6	1.9
-11	0	5	1.4	-1.1
-11	0	-1	2.9	2.4
-11	0	-2	2.0	1.9
-11	0	-3	1.1	1.1
-11	0	-4	1.7	1.7
-12	0	0	.9	.9
-12	0	1	1.3	1.2
-12	0	2	2.5	2.3
-12	0	3	2.3	2.2
-12	0	-2	1.6	1.5
-12	0	-1	1.3	.9
0	1	0	5.9	-5.4
0	1	1	9.1	-9.1
0	1	2	7.4	-6.4
0	1	3	7.8	-6.4
0	1	4	8.2	-7.1
0	1	5	5.5	-5.3
0	1	6	5.5	-4.9
0	1	7	1.6	-1.7
0	1	8	3.9	-4.3
0	1	9	3.6	-3.9
0	1	-2	1.6	.5
0	1	-5	4.8	4.4
0	1	-6	1.8	1.8
0	1	-7	1.5	1.4
0	1	-8	1.6	2.1
0	1	-9	4.5	4.9
0	1	-10	1.7	2.8
1	1	0	3.5	4.7
1	1	1	12.0	-11.6
1	1	2	3.3	-2.5
1	1	3	2.7	-2.1
1	1	5	1.5	-1.4
1	1	7	2.9	2.4
1	1	8	3.5	3.0
1	1	9	1.9	1.9
1	1	11	1.8	2.1
1	1	12	1.0	1.4
1	1	-1	2.5	-3.0
1	1	-2	13.9	-15.7
1	1	-3	7.9	-7.3
1	1	-4	6.2	-5.4
1	1	-5	6.2	-5.9
1	1	-6	5.5	-5.2
1	1	-7	3.6	-3.7
1	1	-10	2.3	-2.0
1	1	-11	.9	-0.7
-1	1	1	4.1	4.7
-1	1	2	7.3	6.9
-1	1	3	3.4	2.9
-1	1	4	1.5	-1.3
-1	1	5	3.4	3.1
-1	1	7	1.6	-1.4
-1	1	8	1.5	-3.3
-1	1	9	1.0	-1.5
-1	1	11	1.3	-1.7
-1	1	12	1.1	-2.0
-1	1	-1	6.8	6.3
-1	1	-2	12.2	10.9
-1	1	-3	7.9	6.8
-1	1	-4	5.5	4.3
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-1	1	-6	5.5	5.1
-1	1	-7	2.5	2.4
-1	1	-9	3.7	2.5
-1	1	-10	1.9	1.0
2	1	0	3.2	4.9
2	1	1	4.3	5.1
2	1	2	6.4	6.1
2	1	3	3.3	2.7
2	1	4	8.6	8.4
2	1	5	8.1	6.8
2	1	6	2.0	1.7
2	1	8	4.1	4.3
2	1	9	2.4	2.6
2	1	-1	.9	1.1
2	1	-2	5.5	-5.9
2	1	-3	2.5	2.2
2	1	-4	1.8	1.5
2	1	-5	2.9	-2.7
2	1	-6	2.5	-2.4
2	1	-7	1.2	-1.4
2	1	-8	1.6	-1.4
2	1	-9	3.8	-3.9
2	1	-10	2.1	-2.4
2	1	-11	1.3	-1.3
-2	1	0	3.4	4.1
-2	1	2	5.7	6.6
-2	1	3	3.8	3.2
-2	1	4	7.5	7.4
-2	1	5	8.0	8.0
-2	1	6	6.1	6.2
-2	1	7	1.2	1.9
-2	1	8	3.1	3.5
-2	1	9	4.1	4.4
-2	1	-1	6.7	-6.7
-2	1	-2	9.1	8.1
-2	1	-3	.8	.5
-2	1	-4	2.2	-1.4
-2	1	-5	5.4	-4.9
-2	1	-6	2.8	-2.4
-2	1	-7	3.3	-3.0
-2	1	-8	3.2	-3.4
-2	1	-9	2.5	-2.7
3	1	0	3.8	4.8
3	1	1	4.1	4.3
3	1	2	3.7	3.8
3	1	3	1.2	1.5
3	1	4	1.3	-1.4
3	1	5	4.2	4.1
3	1	6	.9	.8
3	1	7	5.3	-5.2
3	1	8	2.4	-2.1
3	1	10	1.8	-2.2
3	1	11	1.2	-1.8
3	1	12	.9	-1.4
3	1	-1	5.9	7.0
3	1	-2	5.4	6.7
3	1	-3	9.5	11.3
3	1	-4	5.6	6.2
3	1	-5	2.8	2.8
3	1	-6	5.2	6.0
3	1	-7	3.5	3.8
3	1	-10	1.8	1.8
-3	1	0	6.1	-7.0
-3	1	1	5.5	-7.0
-3	1	2	4.6	-5.8
-3	1	3	2.6	-3.3
-3	1	4	2.5	-2.6
-3	1	6	3.3	-3.2
-3	1	7	1.7	-1.7
-3	1	8	2.0	2.4
-3	1	-1	4.9	-6.5
-3	1	-2	12.6	-14.4
-3	1	-3	1.2	-0.9
-3	1	-5	5.7	-5.6
-3	1	-6	4.5	-4.2
-3	1	-7	1.3	-1.5
-3	1	-8	1.7	-1.5
4	1	0	4.7	-5.5
4	1	1	4.4	-5.3
4	1	2	4.7	-4.8
4	1	3	3.0	-3.6
4	1	4	8.1	-8.0
4	1	5	2.9	-2.7
4	1	7	4.1	-4.1
4	1	8	2.5	-2.8
4	1	-2	1.7	1.7
4	1	-3	2.0	-2.5
4	1	-4	3.0	-3.5
4	1	-6	3.6	3.7
4	1	-9	2.1	2.6
4	1	-10	2.3	2.5
-4	1	0	1.5	-1.5
-4	1	1	4.2	-4.0
-4	1	2	3.5	-4.6
-4	1	3	2.2	-2.9
-4	1	4	3.6	-3.6
-4	1	5	7.1	-7.9
-4	1	6	5.8	-5.9
-4	1	7	1.9	-1.9
-4	1	8	2.4	-2.9
-4	1	9	2.4	-2.6
-4	1	10	1.9	-1.9
-4	1	-2	3.6	-2.6
-4	1	-3	2.0	-1.7
-4	1	-4	4.8	4.7
-4	1	-5	3.6	3.9
-4	1	-6	2.3	1.9
-4	1	-7	1.6	2.1
-4	1	-8	3.1	3.7
-4	1	-9	2.0	2.9
5	1	1	5.1	-5.8
5	1	2	3.4	-3.7
5	1	5	1.6	-1.3
5	1	6	2.4	2.5
5	1	7	1.7	2.0
5	1	8	1.0	1.1
5	1	-3	6.9	-7.6
5	1	-4	4.2	-5.5
5	1	-5	2.3	-2.4
5	1	-6	4.2	-4.6
5	1	-7	2.7	-2.9
5	1	-8	3.1	-3.3
-5	1	0	5.7	6.0
-5	1	1	2.9	2.3
-5	1	2	6.0	7.1
-5	1	3	6.3	7.1
-5	1	4	.8	.9
-5	1	6	3.0	3.3
-5	1	7	2.2	2.1
-5	1	8	1.0	-1.0
-5	1	9	1.0	-1.1
-5	1	-1	8.1	9.2
-5	1	-2	4.8	5.6
-5	1	-3	3.1	3.8
-5	1	-4	3.5	3.9
-5	1	-5	3.7	3.7
-5	1	-6	2.4	3.1
-5	1	-9	1.4	1.5
6	1	1	2.8	2.9
6	1	2	2.6	2.8
6	1	3	4.9	5.1
6	1	4	3.9	4.4
6	1	5	3.2	3.5
6	1	6	1.4	1.6
6	1	7	2.5	3.0
6	1	8	2.3	2.5
6	1	-7	2.0	-1.9
-6	1	0	3.3	-3.0
-6	1	1	3.0	2.7
-6	1	2	5.2	4.9
-6	1	3	3.2	3.3
-6	1	4	1.8	1.7
-6	1	5	3.8	3.6
-6	1	6	3.9	4.3
-6	1	7	2.4	2.5
-6	1	8	1.0	1.3
-6	1	9	1.9	2.3
-6	1	10	1.9	2.1
-6	1	-3	1.5	-1.6
-6	1	-4	2.4	-2.9
-6	1	-5	2.4	-2.9
-6	1	-7	2.2	-2.6
-6	1	-8	3.6	-4.1
-6	1	-9	1.8	-2.1
7	1	6	2.0	-1.5
7	1	7	1.9	-2.1
-7	1	0	4.5	-4.9
-7	1	1	2.1	-1.7
-7	1	2	4.3	-3.5
-7	1	3	4.9	-5.1
-7	1	4	2.8	-2.4
-7	1	5	2.9	-2.6
-7	1	7	2.0	-1.9
-7	1	9	1.8	1.4
-7	1	-1	6.9	-6.5
-7	1	-2	3.5	-3.9
-7	1	-3	2.9	-3.1
-7	1	-4	3.9	-3.7
-7	1	-5	2.0	-2.7
-7	1	-9	1.1	-1.9
8	1	4	4.3	-3.9
8	1	5	1.4	-1.4
8	1	7	2.2	-2.5
-8	1	1	1.7	1.6
-8	1	2	3.2	-2.6
-8	1	3	2.6	-2.1
-8	1	4	2.0	-1.7
-8	1	5	2.1	-1.9
-8	1	6	3.7	-3.4
-8	1	9	1.7	-1.5
-8	1	10	2.3	-2.7
-8	1	-1	1.4	1.0
-8	1	-2	1.4	-1.3
-8	1	-3	2.3	2.3
-8	1	-4	2.3	2.3
-8	1	-5	1.9	1.7
-8	1	-6	1.4	1.7
-8	1	-7	1.7	1.9
-8	1	-8	2.4	2.7
-9	1	0	4.2	4.0
-9	1	1	1.7	1.6
-9	1	2	2.9	2.6
-9	1	3	4.3	3.7
-9	1	4	2.4	2.5
-9	1	5	1.0	.6
-9	1	6	.9	.7
-9	1	7	1.9	1.8
-9	1	-1	5.3	4.9
-9	1	-2	2.3	2.3
-9	1	-3	1.4	.9
-9	1	-4	2.2	2.4
-9	1	-5	2.3	2.5
-10	1	1	1.4	-1.5
-10	1	2	2.1	1.7
-10	1	3	1.3	1.5
-10	1	4	1.3	.8
-10	1	5	.9	1.0
-10	1	6	2.9	2.4
-10	1	7	2.6	2.3
-10	1	-3	3.2	-2.6
-10	1	-4	2.8	-2.6
-10	1	-6	1.2	-1.1
-10	1	-7	1.6	-2.1
-11	1	0	3.4	-3.1
-11	1	1	2.6	-2.2
-11	1	2	2.4	-2.1
-11	1	3	1.6	-1.6
-11	1	4	2.9	-2.4
-12	1	1	1.3	.9
-12	1	-2	1.1	.9
0	2	0	4.9	-5.2
0	2	1	2.5	-2.1
0	2	2	3.1	2.3
0	2	3	6.5	-5.0
0	2	4	2.3	-2.0
0	2	5	5.0	4.3
0	2	6	3.3	2.6
0	2	7	.4	1.1
0	2	8	1.8	1.7
0	2	9	2.5	3.0
0	2	10	1.9	2.3
0	2	-1	7.3	-5.5
0	2	-2	8.9	-7.3
0	2	-3	8.3	-6.1
0	2	-4	8.3	-6.1
0	2	-5	8.1	-6.1
0	2	-6	4.9	-4.1
0	2	-7	2.4	-2.1
0	2	-8	4.9	-4.7
0	2	-9	2.2	-2.5
0	2	-12	.8	-1.1
1	2	1	6.7	5.8
1	2	2	13.0	10.6
1	2	3	9.3	7.2
1	2	4	6.0	4.4
1	2	5	5.0	3.7
1	2	6	6.4	5.7
1	2	7	4.7	4.2
1	2	12	.7	-1.2
1	2	-1	13.0	10.6
1	2	-2	3.0	2.8
1	2	-3	3.9	3.8
1	2	-4	1.4	1.4
1	2	-5	2.8	2.6
1	2	-6	1.9	1.4
1	2	-7	2.5	-2.1
1	2	-8	2.4	-2.3
1	2	-11	2.6	-3.0
-1	2	0	1.1	-1.1
-1	2	1	4.7	-5.0
-1	2	2	10.2	-9.1
-1	2	3	13.9	-12.5
-1	2	4	4.9	-4.1
-1	2	5	2.8	-2.2
-1	2	6	5.7	-4.8
-1	2	7	4.2	-4.1
-1	2	-1	11.8	-12.3
-1	2	-2	4.2	-3.2
-1	2	-3	2.2	-1.9
-1	2	-5	3.0	-2.2
-1	2	-6	1.1	-1.2
-1	2	-7	3.0	2.5
-1	2	-8	2.3	2.0
-1	2	-10	1.7	1.8
-1	2	-11	2.0	2.3
2	2	0	4.6	5.1
2	2	1	2.5	2.0
2	2	2	2.7	-2.1
2	2	3	3.3	2.6
2	2	4	1.7	-1.8
2	2	5	7.9	-6.2
2	2	6	4.7	-3.6
2	2	8	2.3	-2.4
2	2	9	1.2	-1.5
2	2	10	1.8	-2.5
2	2	12	.9	-1.3
2	2	-1	6.0	8.1
2	2	-2	3.2	-2.8
2	2	-3	1.0	.1
2	2	-4	9.0	8.7
2	2	-5	6.5	6.6
2	2	-6	3.5	3.4
2	2	-7	2.7	2.4
2	2	-8	4.1	4.4
2	2	-9	2.5	2.9
2	2	-11	.9	.1
-2	2	0	3.1	4.2
-2	2	1	3.7	3.9
-2	2	2	1.0	.8
-2	2	3	1.0	1.1
-2	2	4	1.6	1.3
-2	2	5	1.8	1.6
-2	2	6	6.0	-5.0
-2	2	7	2.1	-1.9
-2	2	9	2.9	-2.9
-2	2	10	2.8	-3.4
-2	2	11	1.2	-1.3
-2	2	-1	11.9	12.3
-2	2	-2	3.7	-2.5
-2	2	-3	8.3	6.7
-2	2	-4	13.8	11.6
-2	2	-5	6.9	5.2
-2	2	-7	5.4	4.5
-2	2	-8	2.5	2.0
-2	2	-9	1.9	1.8
-2	2	-12	1.3	1.5
3	2	0	5.4	-6.3
3	2	1	6.9	-7.1
3	2	2	7.2	-6.5
3	2	3	8.0	-7.1
3	2	5	5.3	-4.6
3	2	6	7.2	-6.3
3	2	7	4.2	-3.7
3	2	9	1.7	-1.6
3	2	-2	12.3	-14.1
3	2	-3	2.2	-2.9
3	2	-4	3.8	3.8
3	2	-5	2.3	-2.2
3	2	-6	1.5	-1.5
3	2	-9	1.7	1.5
3	2	-11	.9	1.3
3	2	-12	1.6	1.7
-3	2	0	6.1	6.2
-3	2	1	4.7	5.3
-3	2	2	5.1	5.4
-3	2	3	6.9	6.8
-3	2	4	7.1	6.6
-3	2	6	3.1	3.1
-3	2	7	5.1	4.9
-3	2	8	2.2	2.0
-3	2	-1	5.8	6.5
-3	2	-2	3.2	3.0
-3	2	-3	5.9	-5.3
-3	2	-4	4.7	3.6
-3	2	-5	2.0	2.1
-3	2	-6	2.0	-2.0
-3	2	-7	2.3	-2.0
-3	2	-10	1.9	-2.1
4	2	3	1.8	1.5
4	2	4	3.5	2.6
4	2	5	7.2	6.7
4	2	6	1.7	2.2
4	2	8	3.5	3.3
4	2	9	3.0	3.4
4	2	-2	2.9	-3.1
4	2	-4	1.4	-1.5
4	2	-5	5.8	-5.8
4	2	-6	3.9	-4.0
4	2	-7	3.5	-3.8
4	2	-8	2.8	-2.9
4	2	-9	2.5	-2.5
4	2	-10	.4	-1.6
-4	2	0	5.2	-5.6
-4	2	1	4.7	-5.4
-4	2	2	1.4	-1.0
-4	2	3	1.2	-1.1
-4	2	4	1.7	-2.0
-4	2	6	4.3	3.8
-4	2	7	3.9	3.2
-4	2	9	2.2	2.0
-4	2	10	1.8	2.4
-4	2	11	1.4	1.7
-4	2	-1	2.7	-2.5
-4	2	-2	1.6	-2.3
-4	2	-3	9.4	-8.3
-4	2	-4	6.1	-6.2
-4	2	-5	3.0	-3.7
-4	2	-6	3.6	-3.3
-4	2	-7	3.3	-3.2
-4	2	-8	2.0	-2.4
5	2	1	5.3	5.0
5	2	2	5.9	5.8
5	2	3	3.6	3.0
5	2	4	1.7	2.2
5	2	5	5.1	4.7
5	2	6	2.1	1.8
5	2	-3	3.4	3.3
5	2	-6	2.3	2.3
5	2	-7	1.0	-0.7
-5	2	0	5.3	-5.1
-5	2	1	3.6	-2.5
-5	2	2	5.8	-5.8
-5	2	3	5.3	-4.8
-5	2	4	5.8	-5.2
-5	2	5	2.4	-2.7
-5	2	6	2.4	-2.3
-5	2	7	5.3	-4.7
-5	2	8	2.2	-2.6
-5	2	9	1.4	-1.5
-5	2	-1	5.5	-5.9
-5	2	-3	2.0	1.5
-5	2	-4	2.5	-2.6
-5	2	-6	2.4	2.1
-5	2	-7	1.8	2.1
-5	2	-10	1.5	2.0
-5	2	-11	.7	1.5
6	2	1	3.1	-2.6
6	2	4	3.3	-3.1
6	2	5	2.9	-2.7
6	2	6	2.4	-2.2
6	2	7	2.3	-2.0
6	2	8	2.3	-2.5
6	2	9	2.4	-2.4
6	2	-5	5.4	5.7
6	2	-6	2.0	2.4
6	2	-7	2.3	2.1
6	2	-8	1.4	1.9
6	2	-9	2.8	3.0
-6	2	0	5.5	5.1
-6	2	1	6.2	5.6
-6	2	3	.4	.3
-6	2	4	2.1	1.8
-6	2	6	2.6	-2.8
-6	2	7	2.4	-2.2
-6	2	10	1.6	-2.0
-6	2	11	1.5	-1.7
-6	2	-1	2.3	2.6
-6	2	-2	3.4	3.7
-6	2	-3	5.1	5.6
-6	2	-4	3.8	4.6
-6	2	-5	2.7	2.2
-6	2	-6	2.0	2.7
-6	2	-7	3.1	3.8
-6	2	-8	1.9	2.2
7	2	2	5.0	-4.6
7	2	3	1.4	-1.5
7	2	4	1.0	-1.2
7	2	5	2.4	-2.6
7	2	6	1.8	-2.0
-7	2	0	6.0	5.1
-7	2	1	3.3	2.8
-7	2	3	5.6	4.9
-7	2	4	7.3	5.8
-7	2	5	2.6	2.5
-7	2	6	2.5	2.4
-7	2	7	3.7	3.0
-7	2	8	3.8	3.6
-7	2	9	1.2	1.4
-7	2	-1	2.6	2.1
-7	2	-5	1.0	-1.1
-7	2	-6	1.4	-1.6
-7	2	-7	1.4	-2.2
-7	2	-10	1.4	-2.1
8	2	4	1.8	3.1
8	2	5	1.8	2.0
-8	2	0	3.6	-3.2
-8	2	1	6.7	-5.2
-8	2	2	2.8	-2.2
-8	2	4	1.7	-1.0
-8	2	5	1.7	-1.5
-8	2	7	1.5	1.4
-8	2	-2	4.6	-4.4
-8	2	-3	4.8	-4.8
-8	2	-4	3.2	-3.2
-8	2	-5	1.7	-1.8
-8	2	-6	1.6	-2.2
-8	2	-7	2.4	-2.6
-9	2	0	2.8	-2.2
-9	2	1	4.1	-3.5
-9	2	2	1.4	-1.1
-9	2	3	3.8	-3.0
-9	2	4	3.3	-3.3
-9	2	5	4.2	-3.4
-9	2	7	2.5	-2.3
-9	2	8	2.6	-2.4
-9	2	9	1.1	-1.2
-9	2	-2	1.7	1.1
-9	2	-5	1.6	1.3
-9	2	-6	1.7	1.9
-9	2	-7	1.1	1.1
-10	2	0	2.6	2.2
-10	2	1	2.3	2.3
-10	2	2	2.1	1.7
-10	2	3	2.5	1.9
-10	2	5	2.8	2.1
-10	2	7	1.5	-1.1
-10	2	-1	2.3	1.8
-10	2	-2	3.3	3.0
-10	2	-3	4.4	4.0
-10	2	-4	1.4	1.4
-10	2	-5	1.1	1.2
-10	2	-6	1.3	1.6
-11	2	0	2.0	1.7
-11	2	1	1.2	1.3
-11	2	2	1.5	1.4
-11	2	3	2.0	1.5
-11	2	4	3.0	2.7
-11	2	5	2.6	2.6
-11	2	-1	.8	-0.6
-11	2	-2	.8	-0.9
-11	2	-5	1.5	-1.8
-12	2	0	.9	-0.8
-12	2	1	2.4	-2.1
-12	2	2	1.7	-2.2
-12	2	-1	1.5	-1.5
-12	2	-2	2.6	-2.6
-12	2	-3	1.6	-2.0
0	3	0	6.9	7.4
0	3	1	10.8	11.9
0	3	2	.9	-0.1
0	3	3	.9	.7
0	3	4	7.6	7.2
0	3	5	4.5	4.3
0	3	6	.8	-0.0
0	3	7	2.2	2.2
0	3	10	1.2	-1.3
0	3	11	.8	-1.2
0	3	-1	6.0	6.4
0	3	-2	9.8	7.6
0	3	-3	8.2	7.2
0	3	-4	6.2	6.6
0	3	-5	1.8	1.4
0	3	-6	1.1	-0.5
0	3	-7	1.1	2.7
0	3	-8	2.3	1.9
1	3	0	1.2	-1.1
1	3	1	2.0	1.5
1	3	2	.7	.3
1	3	3	[illegible]	[illegible]
1	3	4	4.2	-3.5
1	3	5	1.7	-1.7
1	3	6	3.5	-3.1
1	3	7	5.7	-5.7
1	3	8	2.8	-2.8
1	3	9	2.4	-2.2
1	3	10	1.3	-1.4
1	3	11	[illegible]	[illegible]
1	3	-2	2.7	2.8
1	3	-3	[illegible]	4.7
1	3	-4	1.2	.5
1	3	-5	1.8	1.9
1	3	-6	4.7	4.4
1	3	-7	4.8	4.7
1	3	-8	2.6	2.8
1	3	-9	1.4	1.4
1	3	-10	2.1	2.5
1	3	-11	1.5	1.5
-1	3	0	.7	.1
-1	3	1	1.8	-1.8
-1	3	2	1.0	-0.5
-1	3	3	6.7	5.7
-1	3	4	7.2	6.7
-1	3	5	4.3	3.5
-1	3	6	3.8	4.0
-1	3	7	3.9	3.9
-1	3	8	3.3	3.9
-1	3	9	2.0	1.9
-1	3	11	1.9	2.7
-1	3	-1	1.5	-1.2
-1	3	-2	2.7	-2.3
-1	3	-3	5.7	-4.4
-1	3	-4	2.8	-1.9
-1	3	-5	2.5	-2.1
-1	3	-6	8.2	-6.8
-1	3	-7	4.4	-4.6
-1	3	-8	1.3	-1.7
-1	3	-9	1.8	-2.1
-1	3	-10	2.3	-2.6
-1	3	-11	1.2	-1.6
2	3	1	4.8	-4.8
2	3	2	3.9	-3.2
2	3	3	6.6	-5.8

Table 2 (*cont.*)

H	K	L	F(OBS)	F(CAL)
2	3	4	5.1	-4.5
2	3	5	1.9	-2.0
2	3	6	2.1	1.4
2	3	10	1.8	2.1
2	3	-1	3.7	-4.2
2	3	-2	4.3	-4.9
2	3	-3	5.4	-6.1
2	3	-4	6.6	-6.8
2	3	-5	3.0	-3.1
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1	5	-3	2.9	3.0
1	5	-5	4.3	3.3
1	5	-6	.8	1.1
1	5	-7	2.7	-2.0
1	5	-9	1.3	.7
1	5	-10	2.0	-1.8
-1	5	1	1.4	-2.0
-1	5	2	4.4	-4.6
-1	5	3	7.4	-7.4
-1	5	4	6.3	-5.8
-1	5	5	1.1	-1.0
-1	5	6	3.6	-3.4
-1	5	7	2.4	-2.3
-1	5	8	2.5	-2.0
-1	5	11	.9	-1.0
-1	5	-1	2.2	-2.6
-1	5	-2	2.6	-2.3
-1	5	-3	2.1	-1.4
-1	5	-4	2.5	-2.1
-1	5	-5	4.6	-3.9
-1	5	-6	2.1	1.6
-1	5	-7	2.3	2.0
-1	5	-10	1.7	1.7
2	5	0	4.0	4.9
2	5	1	1.5	1.3
2	5	2	2.4	-2.1
2	5	3	1.1	-0.8
2	5	4	.7	-0.9
2	5	5	3.2	-2.7
2	5	6	3.4	-2.8
2	5	9	2.5	-2.6
2	5	10	2.0	-2.1
2	5	-1	3.1	3.0
2	5	-2	2.1	2.1
2	5	-3	1.8	2.0
2	5	-4	4.5	3.9
2	5	-5	3.6	3.0
2	5	-6	2.3	2.3
2	5	-7	1.9	1.5
2	5	-8	4.5	4.0
-2	5	0	4.4	5.9
-2	5	1	1.4	1.8
-2	5	2	[illegible]	[illegible]
-2	5	3	.6	-0.4
-2	5	4	2.6	2.3
-2	5	5	1.3	-1.6
-2	5	6	1.5	-0.8
-2	5	7	3.4	-3.4
-2	5	8	.9	-0.9
-2	5	9	1.5	-1.4
-2	5	10	2.8	-2.7
-2	5	11	1.3	-1.6
-2	5	-1	3.3	3.0
-2	5	-2	4.7	4.5
-2	5	-3	6.6	6.0
-2	5	-4	6.1	5.5
-2	5	-5	3.3	3.1
-2	5	-6	2.5	2.5
-2	5	-7	4.5	4.5
-2	5	-8	2.9	2.8
-2	5	-11	1.3	1.4
-2	5	-12	.8	.2
3	5	0	2.0	-1.8
3	5	1	1.4	-1.1
3	5	2	5.5	-5.8
3	5	3	4.5	-4.4
3	5	4	3.3	-2.6
3	5	5	2.3	-2.2
3	5	6	3.2	-2.8
3	5	7	2.5	-1.9
3	5	-2	4.3	-4.9
3	5	-3	2.0	-1.6
3	5	-4	2.2	-2.0
3	5	-5	1.7	-1.7
3	5	-7	1.3	.7
3	5	-8	1.3	.9
3	5	-11	1.5	1.4
-3	5	0	3.8	4.7
-3	5	2	3.4	3.5
-3	5	3	5.0	5.4
-3	5	4	5.6	5.7
-3	5	5	3.2	3.2
-3	5	6	1.2	1.0
-3	5	7	3.5	3.8
-3	5	8	2.9	2.6
-3	5	11	1.1	1.4
-3	5	-1	4.9	5.8
-3	5	-3	1.5	1.0
-3	5	-4	1.0	.8
-3	5	-5	.8	-0.4
-3	5	-6	2.0	-1.3
-3	5	-7	2.0	-1.5
-3	5	-8	2.3	-2.1
4	5	1	2.9	2.4
4	5	2	.8	.8
4	5	5	2.7	2.7
4	5	6	2.5	2.4
4	5	7	.9	.8
4	5	8	.9	1.2
4	5	9	2.5	2.3
4	5	-3	2.4	-2.0
4	5	-4	3.1	-2.6
4	5	-5	2.9	-2.2
4	5	-6	2.5	-2.5
4	5	-7	1.8	-1.4
4	5	-8	2.3	-2.5
4	5	-9	2.5	-2.3
-4	5	0	4.9	-5.1
-4	5	1	5.3	-5.9
-4	5	7	3.0	2.9
-4	5	10	2.0	1.9
-4	5	-2	2.8	-2.5
-4	5	-3	5.9	-6.1
-4	5	-4	4.2	-4.3
-4	5	-5	2.5	-1.8
-4	5	-6	3.3	-3.3
-4	5	-7	3.6	-3.3
-4	5	-8	3.1	-2.5
5	5	1	3.8	4.2
5	5	2	5.3	4.8
5	5	3	2.9	3.2
5	5	4	1.5	1.3
5	5	5	2.1	1.9
5	5	6	2.1	1.9
5	5	-4	1.2	.9
5	5	-5	2.7	2.6
5	5	-8	1.3	-1.1
-5	5	0	5.7	-6.1
-5	5	1	2.8	-2.9
-5	5	2	1.4	-1.3
-5	5	3	3.4	-3.9
-5	5	4	4.7	-4.7
-5	5	5	2.8	-2.6
-5	5	6	3.4	-3.2
-5	5	7	2.6	-2.5
-5	5	8	2.6	-2.7
-5	5	9	1.1	-1.2
-5	5	-1	2.5	-2.3
-5	5	-2	1.1	1.1
-5	5	-3	.8	-1.2
-5	5	-4	2.4	-1.9
-5	5	-5	2.9	2.3
-5	5	-6	2.8	2.5
-5	5	-7	1.6	1.5
-5	5	-9	1.6	1.4
6	5	2	.9	.9
6	5	4	.9	-0.9
6	5	5	2.2	-1.9
6	5	6	1.3	-1.4
6	5	7	1.2	-1.1
6	5	8	1.5	-1.5
6	5	9	1.4	-1.7
-6	5	0	2.2	2.4
-6	5	1	4.3	3.7
-6	5	2	3.5	3.0
-6	5	3	2.0	-1.3
-6	5	6	2.0	-1.3
-6	5	7	2.0	-1.9
-6	5	-1	2.7	2.4
-6	5	-2	4.8	4.8
-6	5	-3	1.5	2.1
-6	5	-4	3.8	3.5
-6	5	-6	2.7	2.9
-6	5	-7	3.3	3.3
-6	5	-9	1.1	.3
7	5	3	2.7	-2.2
7	5	4	1.3	-1.8
7	5	5	1.2	-1.5
7	5	6	1.9	-1.6
-7	5	0	3.1	2.3
-7	5	1	4.3	4.5
-7	5	3	3.2	2.7
-7	5	4	5.3	5.2
-7	5	5	3.4	3.1
-7	5	6	1.6	1.7
-7	5	7	2.3	2.3
-7	5	8	2.9	2.6
-7	5	9	1.2	1.2

Table 3. *Interatomic distances and angles and their standard deviations*

Distances

Cr—O(1)	1·92 (0·02) Å	C(5)—C(6)	1·40 Å	C(16)-C(17)	1·39 Å
Cr—O(2)	1·93	C(6)—C(7)	1·36	C(17)-C(18)	1·44
Cr—N(1)	2·02	C(7)—C(8)	1·44	N(1)—C(11)	1·58
Cr—N(2)	2·10	C(8)—C(9)	1·45	N(1)—C(12)	1·32
Cr—N(3)	2·06	C(10)-C(11)	1·56	N(2)—C(1)	1·52
Cr—N(4)	2·15	C(12)-C(13)	1·48	N(3)—C(2)	1·53
C(1)-C(2)	1·52 (0·04)	C(13)-C(18)	1·37	N(3)—C(3)	1·33
C(3)-C(4)	1·42	C(13)-C(14)	1·46	N(4)—C(10)	1·46
C(4)-C(5)	1·43	C(14)-C(15)	1·42	O(1)—C(18)	1·35 (0·03)
C(4)-C(9)	1·47	C(15)-C(16)	1·50	O(2)—C(9)	1·35 (0·03)
I——N(2)	3·90 (0·02)	I——N(4)	3·70 (0·02)	I——Cr	4·90 (0·02)

Angles

O(1)-Cr—O(2)	98·4 (2·0)°	Cr—N(2)—C(1)	115·8 (2·0)°	C(6)—C(7)—C(8)	119·2 (2·5)°
O(1)-Cr—N(1)	90·8	Cr—N(3)—C(2)	105·4	C(7)—C(8)—C(9)	122·9
O(1)-Cr—N(2)	87·8	Cr—N(3)—C(3)	126·9	C(12)-C(13)-C(18)	127·2
O(1)-Cr—N(3)	90·1	Cr—N(4)—C(10)	116·8	C(13)-C(14)-C(15)	117·9
O(2)-Cr—N(1)	92·7	O(1)-C(18)-C(13)	121·0	C(13)-C(18)-C(17)	124·5
O(2)-Cr—N(3)	91·1	O(2)-C(9)—C(4)	125·0	C(14)-C(15)-C(16)	126·1
O(2)-Cr—N(4)	86·2	N(1)-C(11)-C(10)	104·1	C(15)-C(16)-C(17)	121·0
N(1)-Cr—N(2)	95·8	N(1)-C(12)-C(13)	122·3	C(16)-C(17)-C(18)	114·2

Table 3 (*cont.*)

N(1)-Cr—N(4)	82·7	N(2)-C(1)—C(2)	106·7	C(14)-C(13)-C(18)	119·1
N(2)-Cr—N(3)	80·3	N(3)-C(2)—C(1)	110·1	N(2)—I——N(4)	46·08
N(2)-Cr—N(4)	88·6	N(3)-C(3)—C(4)	123·8	C(1)—N(2)—I	95·8
N(3)-Cr—N(4)	96·0	N(4)-C(10)-C(11)	107·9	N(1)—C(11)-I	89·6
Cr—O(1)-C(18)	130·4	C(3)-C(4)—C(9)	123·8 (2·5)	Cr—I——N(4)	24·0
Cr—O(2)-C(9)	129·0	C(4)-C(5)—C(6)	121·2	Cr—I——N(2)	24·3
Cr—N(1)-C(11)	108·8	C(4)-C(9)—C(8)	118·7		
Cr—N(1)-C(12)	127·0	C(5)-C(6)—C(7)	116·8		
		C(5)-C(4)—C(9)	120·1		

Bond distances and angles involving the chromium and the iodine atoms are given in Table 3. The close approaches of N(2) and N(4) to the iodide anion could be taken to indicate hydrogen bonding and these may contribute to the apparent lengthening of the Cr–N(2) and Cr–N(4) bonds compared with the Cr–N(1) and Cr–N(3) bonds.

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Nuclear Magnetic Resonance Investigation of Fluoride Ions in Hydroxyapatite

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The position of fluoride minorities in hydroxyapatite [Ca$_5$OH(PO$_4$)$_3$] has been investigated by nuclear magnetic resonance methods. There is experimental evidence for the existence of OH–F–vacancy and OH–F–HO configurations parallel to the *c* axis. The internuclear distances permit the formation of weak OH···F hydrogen bonds. A discussion of the energy-level system for a linear array of two protons and a fluorine nucleus, coupled by dipolar interaction, is included.

Introduction

Fluorapatite [Ca$_5$F(PO$_4$)$_3$] and hydroxyapatite [Ca$_5$OH(PO$_4$)$_3$] are nearly isomorphous. Detailed descriptions of the structural aspects of apatites can be found in review articles by Young & Elliott (1966) and by Elliott (1969). Of the two compounds, fluorapatite is the more stable and, consequently, most mineral samples of hydroxyapatite contain some fluoride.

The space group attributed to fluorapatite is $P6_3/m$ (Hentschel, 1923; Náray-Szabó, 1930; Mehmel, 1930). According to Young & Elliott (1966) the lattice constants a and c are 9·364 and 6·879 Å respectively; slightly different values are mentioned by de Boer (1957), and in *Structure Reports* (1952, 1953, 1956). According to Náray-Szabó (1930) a fluoride ion in fluorapatite is situated at the intersection of a 6_3-axis and a mirror plane, exactly at the centre of a triangle of Ca^{2+} ions. The fluoride ions form linear chains along the 6_3-axis, the F–F distance being 3·44 Å.

The unit-cell dimensions of hydroxyapatite differ only slightly from those of fluorapatite. Kay, Young & Posner (1964) found $a = 9{\cdot}422$ and $c = 6{\cdot}883$ Å, and Bhatnagar (1969) $a = 9{\cdot}4192$ and $c = 6{\cdot}8867$ Å; see also *Structure Reports* (1952, 1953). For a hydroxyl ion, the