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The Crystal Structure of Trisacetylacetonatochromium(III)*

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The crystal structure and molecular configuration of trisacetylacetonatochromium(III), $Cr(CH_3COCHCOCH_3)_3$, has been determined by X-ray diffraction techniques. The structure consists of four discrete molecules linked together by van der Waals forces in a unit cell of $P2_1/c$ symmetry and of dimensions $a_0 = 14{\cdot}031$, $b_0 = 7{\cdot}551$, $c_0 = 16{\cdot}379$ Å, $\beta = 99{\cdot}06°$. Unusually large anisotropic thermal parameters for one of the chelate rings appear to be associated with a lattice wave of large amplitude rather than a slight disordering in the molecular packing along the *b* direction.

Introduction

Early crystal studies (Astbury, 1926) indicated that the trivalent metal acetylacetonates may be grouped into three isomorphous series. From cell dimensions and space group considerations it appeared that the complexes of aluminum(III), manganese(III), cobalt(III), and chromium(III) and one allotrope of the gallium(III) complex formed a monoclinic isomorphic set. However, the structure determination of the cobalt(III) complex (Padmanabhan, 1958) and preliminary results for the aluminum(III) and chromium(III) complexes (Shkolnikova & Shugam, 1956) differed with respect to the molecular packing. Later Shkolnikova & Shugam (1960) reported the crystal structure determination of the chromium(III) complex with, however, a relatively high discrepancy index ($R = 28{\cdot}8$). Together with Roof's (1956) results on the iron(III) complex, Forman & Orgel (1959) found their infrared data for the chromium, manganese and iron complexes consistent with a Jahn–Teller distortion for the manganese complex. In order to gain an insight to the nature or extent of the distortion, the crystal structure of the manganese complex was determined (Morosin & Brathovde, 1964). During the refinement of the manganese complex it became apparent that an error in the structure determination of the chromium complex had been made by Shkolnikova and Shugam.

Experimental

Standard methods (Bryant & Fernelius, 1957) were used to prepare the chromium complex. The compound was purified by recrystallization and, subsequently, vacuum sublimation. Crystals were grown by slow evaporation at room temperature from an acetone solution.

Equi-inclination Weissenberg photographs were taken with Cu $K\alpha$ radiation to verify the symmetry of the lattice. The following cell dimensions were obtained from a modified Picker diffractometer (λMo $K\alpha_1 = 0{\cdot}70926$ Å) equipped with a G. E. single-crystal orienter and are compared with values determined by Astbury (1926).

		Astbury
a_0	$14{\cdot}031 \pm 0{\cdot}009$	14·2
b_0	$7{\cdot}551 \pm 0{\cdot}005$	7·62
c_0	$16{\cdot}379 \pm 0{\cdot}011$	16·5
β	$99{\cdot}06 \pm 0{\cdot}020$	99·13°

Systematic absences of $0k0$ for $k = 2n+1$ and of $h0l$ for $l = 2n+1$ are consistent with the space group $P2_1/c$. With four molecules per unit cell, the calculated and observed densities are 1·362 and 1·374 g.cm^{-3}, respectively.

Intensity data were collected with a Picker diffractometer equipped with a G.E. single-crystal orienter, a scintillation counter, and a molybdenum X-ray tube employing balanced zirconium and yttrium filters. All independent reflections permitted by the space group in the sphere with $\sin\theta/\lambda$ less than 0·5 (2θ less than 45°) were measured by a fixed count (10^n counts with $n \geq 3$, time > 10 seconds) technique. There were 1490 unique reflections of which 224 were considered to be unobserved. No absorption corrections were applied to the intensity data for the spherically ground ($r = 0{\cdot}03$ cm; $\mu = 8{\cdot}5$ cm^{-1}) crystal specimen.

Lorentz and polarization factors were applied to the observed data. Structure factors were calculated with Cr (Thomas & Umeda, 1957), C, O, and H (Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal, 1955) scattering factors and Cr dispersion corrections (Dauben & Templeton, 1955). Dispersion corrections were included by the method of Templeton (1955). Four reflections, 002, 200, 020, and 110 were found to be considerably affected by secondary extinction and were omitted from all difference and differential syntheses and in the calculation of *R*. Calculations were carried out on a CDC 1604 computer.

Determination of structure

A comparison of the observed and calculated structure factors listed by Shkolnikova & Shugam for their chrom-

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ium complex with the corresponding ones for the manganese complex was made. This comparison, together with their reported discrepancy index of 0·288, suggested that the crystal structure of the chromium complex is probably more similar to that of the manganese complex than to the molecular packing proposed by Shkolnikova & Shugam. Refinement to about $R=0·30$ for the manganese complex (Morosin & Brathovde) with a trial structure similar to Shkolnikova & Shugam's had led to an incorrect determination for that crystal structure.

A three-dimensional Patterson synthesis was calculated. The general features of the synthesis were similar to that of the manganese complex. With the slightly different positional parameter coordinates for the chromium and oxygen, a three-dimensional Fourier synthesis clearly showed the parameter positions of all the carbon atoms although for one chelate ring the electron density distribution at several atomic sites was elliptical rather than spherical. These parameter positions together with individual isotropic temperature factors were refined by several differential syntheses (Booth, 1946), corrected for series termination, to a discrepancy index, R, of 0·11. For the numbering scheme illustrated in Fig. 1, the isotropic thermal parameters for the three carbon atoms [C(12), C(X1) and C(X2)] which appeared with elliptical electron density distributions were large (9·0, 10·8, and 9·4 Å^2, respectively) compared with the average value of the other carbon atoms (6·4 Å^2). A three-dimensional difference synthesis was calculated. Anisotropic motion for several other carbon atoms was evident; therefore, refinement was continued with anisotropic therma parameters. Inclusion of hydrogen atoms as fixed atom contributions ($B=8·0$) did not significantly change the value of R nor positions of the methyl carbons. Since there is uncertainty as to the proper coordinates for these hydrogen atoms because of the large anisotropic motion of several methyl groups, the final refined structure (shifts $<0·1\sigma$) does not include any contribution from hydrogen atoms. The final discrepancy index, omitting the four reflections strongly affected by secondary extinction and unobserved reflections, is 0·066 (with no omitted reflections, $R=0·074$).

The final values of the positional and thermal parameters are given in Tables 1 and 2 respectively. Observed and calculated structure factors are listed in Table 3. The molecular packing arrangement is illustrated in Fig. 2. The average estimated standard deviations (e.s.d.) of the positional parameters calculated by the method of Cruickshank (1949) are 0·0014, 0·0065 and

Table 1. *Final atomic coordinates*

Cr	x	y	z
Cr	0·23771	0·26578	0·47071
O(1)	0·11258	0·33858	0·41152
O(2)	0·18055	0·18920	0·56564
O(3)	0·36548	0·19218	0·52550
O(4)	0·29698	0·34347	0·37596
O(5)	0·21717	0·02765	0·42352
O(6)	0·25741	0·50286	0·51967
C(4)	0·30395	0·25276	0·31081
C(5)	0·23428	−0·02536	0·35347
C(45)	0·27410	0·08116	0·29743
C(X4)	0·34770	0·35068	0·24573
C(X5)	0·21023	−0·21721	0·33426
C(6)	0·33308	0·56527	0·56060
C(3)	0·43110	0·29259	0·56316
C(36)	0·41881	0·47041	0·58000
C(X3)	0·52800	0·20577	0·59184
C(X6)	0·32767	0·75494	0·59008
C(1)	0·03285	0·31731	0·43459
C(2)	0·09222	0·18624	0·57122
C(12)	0·01789	0·23933	0·50880
C(X1)	−0·05339	0·37613	0·37093
C(X2)	0·06938	0·11372	0·65401

Table 2. *Anisotropic temperature factors*

Anisotropic thermal parameters are of the form $\exp\left(-\frac{1}{4}\sum_{i=1}^{3}\sum_{j=1}^{3}B_{ij}h_ih_ja^*_ia^*_j\right)$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr	4·794	4·627	3·485	0·748	0·669	−0·138
O(1)	6·246	7·364	5·254	2·132	−0·838	−0·538
O(2)	5·489	7·315	4·509	−0·050	1·249	−0·056
O(3)	5·112	5·123	5·337	0·760	0·681	0·295
O(4)	7·745	5·030	4·577	−0·192	1·543	0·063
O(5)	6·401	4·477	5·157	−0·176	0·646	−0·387
O(6)	6·550	5·001	4·713	1·299	0·573	−0·564
C(4)	5·373	6·081	4·061	0·792	0·357	−0·483
C(5)	4·878	5·262	4·256	1·246	−0·483	−0·800
C(45)	6·342	5·899	5·140	0·353	0·633	−0·474
C(X4)	8·354	8·497	5·255	−1·188	2·248	0·150
C(X5)	7·175	5·133	6·936	−0·319	−0·550	−1·337
C(6)	7·363	5·653	3·895	−0·371	1·399	−0·227
C(3)	5·294	7·309	4·398	0·495	1·606	1·208
C(36)	7·110	6·238	6·306	−0·433	1·562	0·025
C(X3)	4·923	11·476	7·961	1·401	0·515	0·750
C(X6)	11·839	5·213	7·084	−0·225	1·582	−0·347
C(1)	6·240	14·890	6·471	3·017	−0·685	−2·320
C(2)	6·511	13·964	4·819	−1·298	1·934	−0·356
C(12)	5·679	30·443	6·772	2·218	1·368	−1·854
C(X1)	8·150	25·291	9·983	7·838	−3·212	−3·833
C(X2)	10·097	25·953	7·334	−6·253	4·570	0·176

Table 3. *Observed and calculated structure factors*

H	10FO	10FC
K= 0 L= 0		
1	231	236
2	1151	-1624
3	550	542
4	868	973
5	119	142
6	642	-616
7	410	-415
8	214	218
9	357	363
10	162	-168
11	136	-129
12	70	73
13	68	74
K= 1 L= 0		
1	1399	-1880
2	163	-165
3	242	192
4	237	-203
5	408	-445
6	227	-199
7	416	411
8	< 21	15
9	159	-171
10	175	-182
11	71	75
12	22	-30
K= 2 L= 0		
0	1375	-1370
1	162	-103
2	224	151
3	116	-75
4	155	-119
5	82	-46
6	71	-41
7	188	198
8	82	-87
9	172	-174
10	55	47
11	44	35
12	< 21	-17
K= 3 L= 0		
1	406	473
2	32	15
3	286	-317
4	37	-30
5	288	285
6	257	246
7	178	-202
8	93	-93
9	207	181
10	167	162
11	101	-117
K= 4 L= 0		
0	823	765
1	128	-111
2	641	-631
3	154	-156
4	551	564
5	333	334
6	357	-363
7	172	-178
8	180	183
9	190	200
10	105	-103
K= 5 L= 0		
1	98	-99
2	64	-76
3	275	270
4	230	234
5	327	-333
6	127	-132
7	194	185
8	32	32
9	33	-54
K= 6 L= 0		
0	< 19	-30
1	< 19	19
2	130	129
3	< 19	-10
4	139	-146
5	< 22	-34
6	37	48
K= 7 L= 0		
1	< 21	35
2	< 21	32
K= 1 L= 1		
0	292	317
1	448	-477
3	724	777
4	256	259
5	748	-587
6	249	-254
7	346	373
8	24	19
9	69	-66
10	122	-130
11	26	-26
12	73	79
K= 2 L= 1		
0	830	760
1	257	239
2	129	4
3	244	225
4	47	-64
5	432	-422
6	375	370
7	50	-29
8	90	-87
9	< 21	-18
10	141	-144
11	45	-54
12	< 21	25
K= 3 L= 1		
0	< 32	-19
1	797	-772
2	179	137
3	131	127
4	378	-369
5	74	106
6	191	183
7	< 17	18
8	< 18	8
9	155	-164
10	44	-45
11	146	144
K= 4 L= 1		
0	361	-382
1	225	227
2	169	171
3	201	-206
4	55	45
5	302	296
6	109	-103
7	34	-30
8	54	-57
9	75	71
10	76	84
K= 5 L= 1		
0	54	55
1	< 17	19
2	95	-86
3	39	36
4	293	305
5	116	-110
6	268	-271
7	124	123
8	114	120
9	< 21	26
K= 6 L= 1		
0	< 19	30
1	< 19	-19
2	32	-44
3	40	40
4	104	98
5	155	-156
6	< 21	6
K= 7 L= 1		
0	< 21	-23
1	< 21	30
K= 0 L= -1		
5	24	0
K= 1 L= -1		
1	59	66
2	43	-21
3	55	55
4	290	-282
5	117	66
6	91	-60
7	285	255
8	33	37
9	< 22	7
10	26	-38
11	66	-69
12	< 20	29
13	69	-69
K= 2 L= -1		
1	231	-220
2	< 32	12
3	< 22	-65
4	253	229
5	132	-150
6	209	193
7	289	292
8	< 17	-7
9	93	-93
10	150	-142
11	< 20	-12
12	56	57
K= 3 L= -1		
1	282	277
2	87	95
3	53	-36
4	89	-101
5	74	76
6	221	229
7	119	-101
8	39	-50
9	146	-134
10	53	49
11	139	146
K= 4 L= -1		
1	294	277
2	161	-142
3	240	-248
4	134	133
5	238	247
6	142	-145
7	63	-65
8	56	-45
9	53	39
10	159	155
K= 5 L= -1		
1	< 17	-10
2	64	-67
3	29	24
4	95	96
5	40	-17
6	221	-211
7	117	119
8	37	41
9	< 21	12
K= 6 L= -1		
1	61	-66
2	21	40
3	97	101
4	41	21
5	194	-209
6	< 21	12
7	141	127
K= 7 L= -1		
1	< 21	-31
2	50	63
K= 0 L= 2		
0	1353	2187
1	624	850
2	124	-128
3	1274	-1404
4	284	364
5	935	972
6	576	-607
7	459	-482
8	185	202
9	431	441
10	32	-35
11	120	115
12	< 21	-8
K= 1 L= 2		
0	329	366
1	659	-722
2	1126	-1136
3	884	912
4	585	702
5	902	-965
6	590	-614
7	472	506
8	275	285
9	181	-179
10	202	-209
11	58	-60
12	< 21	10
K= 2 L= 2		
0	822	-783
1	358	-312
2	139	183
3	153	131
4	350	-385
5	454	-449
6	372	380
7	206	217
8	276	-277
9	202	-207
10	83	86
11	< 20	-1
12	< 21	-23
K= 3 L= 2		
0	57	54
1	672	663
2	160	143
3	509	-498
4	165	-165
5	369	357
6	352	348
7	223	-227
8	195	-203
9	135	128
10	113	110
11	31	-42
K= 4 L= 2		
0	591	632
1	58	69
2	246	-283
3	287	-276
4	225	229
5	352	375
6	102	-88
7	179	-172
8	52	47
9	162	160
10	81	-89
K= 5 L= 2		
0	63	62
1	222	-221
2	199	-205
3	260	261
4	272	275
5	186	-185
6	133	-142
7	67	70
8	134	129
K= 6 L= 2		
0	< 19	-32
1	< 19	-6
2	67	64
3	70	76
4	< 20	-6
5	104	-128
6	< 21	8
K= 7 L= 2		
0	51	-72
1	49	58
K= 0 L= -2		
1	384	-464
2	614	-684
3	1056	1134
4	867	736
5	134	113
6	252	-281
7	< 23	5
8	591	604
9	84	89
10	415	-391
11	304	-296
12	314	294
13	74	83
K= 1 L= -2		
1	1055	-1157
2	59	55
3	505	-524
4	371	321
5	89	-122
6	465	-474
7	266	283
8	< 23	43
9	353	-348
10	193	-185
11	247	255
12	32	37
13	100	-116
K= 2 L= -2		
1	129	-113
2	77	-43
3	50	60
4	231	-144
5	37	-50
6	42	58
7	120	139
8	260	-264
9	56	-73
10	227	224
11	83	83
12	147	-136
K= 3 L= -2		
1	686	682
2	82	-94
3	228	-255
4	< 14	-4
5	286	301
6	99	101
7	279	-287
8	50	-51
9	275	272
10	71	65
11	199	-201
K= 4 L= -2		
1	188	-182
2	596	-616
3	251	239
4	541	544
5	42	-18
6	495	-525
7	85	-83
8	355	360
9	65	51
10	199	-199
K= 5 L= -2		
1	178	-180
2	87	85
3	186	197
4	122	116
5	293	-295
6	209	-216
7	260	262
8	97	98
9	123	-127
K= 6 L= -2		
1	< 19	8
2	104	97
3	< 20	-24
4	170	-177
5	< 20	-9
6	100	101
7	45	51
K= 7 L= -2		
1	< 21	-9
K= 1 L= 3		
0	508	511
1	234	-243
2	59	-37
3	42	48
4	643	637
5	184	-186
6	457	-499
7	55	-43
8	257	248
9	< 18	27
10	182	-187
11	41	46
12	114	120
K= 2 L= 3		
0	764	801
1	531	550
2	265	-297
3	94	-44
4	60	-46
5	584	-572
6	35	-10
7	47	62
8	36	-22
9	93	-98
10	< 19	11
11	105	94
K= 3 L= 3		
0	326	320
1	759	-789
2	482	-467
3	351	337
4	58	-73
5	184	-179
6	73	-47
7	159	149
8	53	56
9	52	-40
10	55	-62
11	< 21	17
K= 4 L= 3		
0	358	-393
1	213	-202
2	118	137
3	75	69
4	88	-85
5	123	125
6	63	65
7	< 19	-9
8	69	-72
9	50	50
10	27	40
K= 5 L= 3		
0	138	142
1	128	109
2	87	-102
3	121	-100
4	320	328
5	< 29	-31
6	110	-115
7	< 20	-8
8	52	55
K= 6 L= 3		
0	40	47
1	< 19	3
2	77	-66
3	71	88
4	< 20	22
5	121	-105
6	41	60
K= 1 L= -3		
1	272	253
2	844	-916
3	73	-123
4	77	107
5	98	92
6	103	-108
7	41	24
8	34	-15
9	50	20
10	41	26
11	84	-87
12	22	24
13	< 21	-5
K= 2 L= -3		
1	304	-248
2	159	-103
3	69	-81
4	145	-89
5	< 18	-16
6	36	-26
7	81	71
8	102	-98
9	< 26	17
10	50	-47
11	42	43
12	30	46
K= 3 L= -3		
1	597	613
2	72	86
3	43	25
4	72	-76
5	< 28	-24
6	119	111
7	106	-107
8	< 29	30
9	97	-93
10	34	64
11	105	113
K= 4 L= -3		
1	139	118
2	36	-9
3	310	-303
4	37	-5
5	220	229
6	70	-68
7	157	-156
8	40	13
9	66	60
10	142	141
K= 5 L= -3		
1	31	-18
2	233	-242
3	76	-90
4	167	167
5	151	141
6	231	-241
7	42	-10
8	106	113
9	21	32
K= 6 L= -3		
1	< 19	-30
2	< 19	40
3	39	43
4	49	-33
5	91	-83
6	< 21	28
K= 0 L= 4		
0	83	14
1	686	-746
2	172	229
3	194	101
4	279	257
5	825	848
6	72	48
7	308	-356
8	214	212
9	366	367
10	58	-54
11	59	56
12	< 21	-26
K= 1 L= 4		
0	281	173
1	288	-222
2	221	-208
3	344	382
4	553	557
5	203	-195
6	647	-712
7	95	-112
8	479	494
9	73	-65
10	204	-202
11	102	-94
K= 2 L= 4		
0	349	-371
1	183	-140
2	218	262
3	247	271
4	303	-279
5	837	-852
6	217	248
7	526	522
8	200	-189
9	431	-436
10	21	29
11	219	228
K= 3 L= 4		
0	115	-83
1	426	411
2	279	265
3	191	-208
4	422	-441
5	90	94
6	359	358
7	99	-88
8	302	-313
9	54	58
10	167	162
K= 4 L= 4		
0	312	316
1	179	169
2	140	-130
3	270	-278
4	124	112
5	139	145
6	69	-74
7	100	-98
8	< 25	22
9	112	108
K= 5 L= 4		
0	208	207
1	268	-269
2	316	-327
4	279	177
5	37	-29
6	29	-47
7	61	-76
8	118	123
K= 6 L= 4		
0	118	-134
1	79	-87
2	87	74
3	140	134
4	< 21	-3
5	96	-99
K= 0 L= -4		
1	252	314
2	1334	-1399
3	520	541
4	374	449
5	60	-71
6	406	-389
7	168	182
8	802	808
9	271	-262
10	501	-468
11	39	-19
12	324	331
13	< 21	12
K= 1 L= -4		
1	1196	-1178
2	826	776
3	231	291
4	214	-260
5	383	-406
6	197	-193
7	458	453
8	74	-42
9	653	-641
10	38	33
11	350	360
12	37	42
13	133	-123
K= 2 L= -4		
1	54	1
2	155	204
3	246	-286
4	193	-198
5	< 25	1
6	180	182
7	112	-118
8	304	-298
9	138	143
10	255	249
11	< 20	10
12	155	-151
K= 3 L= -4		
1	554	600
2	335	-322
3	297	-313
4	261	267
5	363	367
6	147	-158
7	208	-219
8	77	77
9	183	185
10	< 20	2
11	109	-118
K= 4 L= -4		
1	68	58
2	402	-392
3	277	266
4	404	420
5	185	-197
6	361	-390
7	128	125
8	286	280
9	101	-102
10	112	-111
K= 5 L= -4		
1	313	-309
2	212	210
3	287	294
4	163	-165
5	314	-316
6	63	43
7	289	297
8	33	-14
9	164	-159
K= 6 L= -4		
1	< 20	5
2	156	161
3	< 20	-22
4	224	-245
5	94	95
6	205	192
K= 1 L= 5		
0	200	-160
1	< 29	-5
2	66	-38
3	170	-192
4	269	264
5	84	80
6	107	91
7	150	-147
8	42	29
9	126	122
10	29	-25
11	< 21	-4
K= 2 L= 5		
0	186	214
1	142	172
2	263	-259
3	169	190
4	53	-22
5	44	-23
6	67	-73
7	124	-121
8	183	171
9	72	66
10	62	-63
11	119	116
K= 3 L= 5		
0	354	332
1	343	-345
2	290	-271
3	154	143
4	< 21	22
5	31	-33
6	241	-240
7	51	48
8	113	125
9	38	-31
10	< 21	27
K= 4 L= 5		
0	306	-304
1	102	-93
2	181	188
3	79	-75
4	126	-118
5	< 20	-10
6	94	93
7	45	55
8	71	-64
9	< 21	-23
K= 5 L= 5		
0	137	141
1	205	206
2	162	-130
3	136	-134
4	190	190
5	< 20	-29
6	< 23	30
7	38	-45
K= 6 L= 5		
0	60	60
1	104	-87
2	145	-131
3	163	171
4	36	40
K= 1 L= -5		
1	167	173
2	452	-486
3	368	-365
4	349	342
5	306	257
6	229	-216
7	175	-164
8	109	110
9	100	-83
10	27	-24
11	31	-14
12	< 20	18
K= 2 L= -5		
1	209	202
2	140	-134
3	97	86
4	371	396
5	83	-64
6	423	-433
7	92	94
8	70	-67
9	74	-16
10	47	41
11	< 20	7
12	< 21	-6
K= 3 L= -5		
1	545	544
2	102	-78
3	135	126
4	64	-65
5	121	-116
6	190	197
7	90	-85
8	62	-55
9	120	119
10	< 20	4
11	< 21	-18
K= 4 L= -5		
1	139	127
2	36	-26
3	271	-271
4	39	-25
5	261	271
6	89	-79
7	214	-209
8	209	209
9	< 20	-3
10	55	-66
K= 5 L= -5		
1	143	-143
2	239	-247
3	51	27
4	201	195
5	40	-23
6	167	-173
7	62	47
8	123	117
K= 6 L= -5		
1	96	-112
2	< 20	-20
3	72	82
4	< 20	-9
5	< 21	0
6	< 21	-24
K= 0 L= 6		
0	316	-301
1	364	251
2	74	-130
3	151	-211
4	644	630
5	635	657
6	< 16	-43
7	235	-240
8	175	184
9	80	72
10	< 20	20
11	114	-114
K= 1 L= 6		
0	135	181
1	< 26	40
2	62	-64
3	196	197
4	433	433
5	60	-69
6	296	-308
7	85	-85
8	249	254
9	< 22	-21
10	111	-111
11	102	-110
K= 2 L= 6		
0	116	-87
1	402	-400
2	202	210
3	517	531
4	197	-182
5	636	-660
6	63	-44
7	350	360
8	81	86
9	248	-245
10	178	-174
K= 3 L= 6		
0	191	-205
1	190	190
2	146	155
3	91	-87
4	343	-330
5	72	58
6	220	211
7	26	2
8	170	-176
9	< 20	7
K= 4 L= 6		
0	222	228
1	381	363
2	228	-228
3	257	-259
4	147	142
5	82	66
6	65	-64
7	103	-102
8	< 22	35
K= 5 L= 6		
0	393	396
1	150	-141
2	325	-322
3	52	40
4	89	85
5	< 20	24
6	50	-51
K= 6 L= 6		
0	114	-108
1	312	-304
2	78	58
3	266	255
K= 0 L= -6		
1	220	146
2	811	-866
3	685	764
4	675	683
5	487	-461
6	551	-546
7	212	210
8	437	422
9	284	-275
10	345	-317
11	< 19	26
12	194	198
K= 1 L= -6		
1	106	-129
2	903	952
3	487	496
4	909	-952
5	300	-279
6	339	349
7	192	192
8	237	-235
9	409	-407
10	146	145
11	247	245
12	62	67
K= 2 L= -6		
1	466	504
2	343	350
3	681	-693
4	192	-176
5	123	107
6	196	200
7	171	-168
8	232	-236
9	105	97
10	208	205
11	< 20	6
12	134	-144
K= 3 L= -6		
1	392	393
2	397	-398
3	322	-313
4	380	375
5	222	219
6	150	-177
7	172	-182
8	94	91
9	251	247
10	< 20	16
11	151	-152
K= 4 L= -6		
1	198	-203
2	252	-265
3	279	279
4	145	157
5	130	-130
6	146	-148
7	57	44
8	277	274
9	< 20	1
10	206	-185
K= 5 L= -6		
1	147	-140
2	349	357
3	31	9
4	327	-333
5	48	40
6	155	159
7	79	78
8	28	-25
K= 6 L= -6		
1	219	211
2	< 31	25
3	118	-117
4	< 21	-19
5	95	88
K= 1 L= 7		
0	100	91
1	112	-113
2	245	-235
3	65	73
4	127	111
5	< 22	-6
6	113	120
7	49	33
8	< 25	-26
9	78	-86
10	< 21	13
K= 2 L= 7		
0	< 37	-24
1	240	-237
2	46	-41
3	343	335
4	93	89
5	263	243
6	29	-14
7	94	-103
8	71	62
9	65	71
10	< 21	-13
K= 3 L= 7		
0	< 16	-16
1	< 16	-22
2	105	93
3	152	145
4	29	28
5	79	-86
6	137	-137
7	56	62
8	58	68
9	87	-82
K= 4 L= 7		
0	95	-93
1	78	-70
2	219	218
3	27	-31
4	149	-134
5	144	-140
6	45	46
7	72	77
8	< 21	-22
K= 5 L= 7		
0	38	37
1	136	138
2	140	-143
3	97	-85
4	43	40
5	< 21	6
6	67	64
K= 6 L= 7		
0	180	187
1	118	-79
2	222	-222
K= 0 L= -7		
10	33	0
K= 1 L= -7		
1	272	306
2	445	-426
3	284	-293
4	449	462
5	59	-60
6	163	-172
7	< 18	-29
8	42	48
9	84	-78
10	44	40
11	69	63
12	< 21	-4
K= 2 L= -7		
1	270	-261
2	102	90
3	458	455
4	46	-44
5	154	-153
6	86	70
7	98	87
8	< 19	-26
9	61	59
10	103	93
11	< 20	0
K= 3 L= -7		
1	165	166
2	72	47
3	130	-120
4	< 17	-20
5	207	217
6	55	51
7	132	-124
8	55	71
9	228	222
10	104	-91
11	97	-91
K= 4 L= -7		
1	< 27	-26
2	59	-70
3	42	-47
4	66	67
5	116	112
6	92	-89
7	71	-61
8	186	184
9	51	-73
K= 5 L= -7		
1	54	-52
2	47	-57
3	< 19	-24
4	55	63
5	31	-27
6	< 20	-17
7	< 21	-8
8	31	31
K= 6 L= -7		
1	62	-71
2	53	57
3	86	85
4	< 21	-24
K= 0 L= 8		
0	299	-317
1	249	270
2	< 23	19
3	257	-273
4	145	-145
5	203	212
6	135	171
7	213	-199
8	19	27
9	< 20	-17
10	85	88
K= 1 L= 8		
0	114	136
1	< 26	-6
2	77	-63
3	76	-63
4	370	390
5	203	201
6	294	-290
7	< 28	-33
8	194	186
9	< 20	-14
K= 2 L= 8		
0	100	75
1	474	-477
2	131	-129
3	486	508
4	92	83
5	375	-361
6	149	-155
7	232	237
8	46	50
9	108	-105
K= 3 L= 8		
0	349	-336
1	110	-103
2	417	414
3	96	83
4	435	-422
5	52	48
6	321	321
7	< 20	-1
8	158	-160
K= 4 L= 8		
0	109	-116
1	360	368
2	70	81
3	239	-246
4	< 25	-7
5	166	163
6	< 20	28
7	146	-140
K= 5 L= 8		
0	160	154
1	115	117
2	169	-165
3	101	-97
4	117	104
5	60	46
K= 6 L= 8		
0	71	58
K= 0 L= -8		
1	290	-303
2	53	-2
3	849	898
4	464	479
5	386	-403
6	208	-216
7	169	184
8	424	414
9	110	-99
10	390	-371
11	172	177
12	126	135
K= 1 L= -8		
1	151	121
2	626	614
3	472	488
4	678	-723
5	< 22	-27
6	280	281
7	130	132
8	110	-103
9	283	-276
10	124	126
11	131	126
12	85	-84
K= 2 L= -8		
1	627	664
2	191	195
3	777	-821
4	104	-82
5	323	351
6	146	138
7	167	-166
8	264	-256
9	72	75
10	176	164
11	94	-95
K= 3 L= -8		
1	60	78
2	257	-271
3	303	-300
4	316	325
5	214	210
6	229	-219
7	259	-249
8	208	196
9	216	201
10	115	-124
K= 4 L= -8		
1	232	-243
2	40	-39
3	131	127
4	96	103
5	151	-154
6	153	-154
7	129	130
8	226	222
9	99	-98
K= 5 L= -8		
1	55	67
2	< 19	14
3	< 20	-12
4	76	-69
5	< 20	-5
6	90	95
7	< 22	35
K= 6 L= -8		
1	131	126
2	< 21	-35
K= 1 L= 9		
0	170	129
1	236	229
2	333	-338
3	106	-112
4	36	23
5	< 31	-25
6	38	-44
7	< 19	13
8	< 20	-7
9	< 21	-46
K= 2 L= 9		
0	255	238
1	248	-253
2	220	-237
3	192	192
4	85	81
5	169	150
6	< 21	-4
7	56	-56
8	< 28	41
K= 3 L= 9		
0	52	-40
1	229	-222
2	272	264
3	228	224
4	48	39
5	59	-39
6	90	-76
7	52	59
K= 4 L= 9		
0	< 19	-7
1	97	108
2	131	126
3	144	-148
4	< 20	0
5	< 31	-32
6	52	-51
K= 5 L= 9		
0	65	75
1	87	92
2	163	-165
3	< 21	-32
4	< 21	-31
K= 1 L= -9		
1	< 16	20
2	115	115
3	132	-134
4	122	121
5	< 23	18
6	154	-146
7	90	-86
8	36	32
9	< 19	19
10	< 20	20
11	< 21	14
K= 2 L= -9		
1	78	57
2	167	161
3	217	-220
4	224	-203
5	46	39
6	148	144
7	< 18	-27
8	< 19	-3
9	94	83
10	116	115
11	< 21	-15
K= 3 L= -9		
1	107	108
2	202	-211
3	218	-203
4	187	181
5	174	174
6	77	-69
7	87	-86
8	84	77
9	214	217
10	103	-106
K= 4 L= -9		
1	123	-103
2	< 19	-14
3	105	113
4	< 19	11
5	< 19	4
6	64	-63
7	< 20	-32
8	136	130
K= 5 L= -9		
1	< 20	-26
2	27	-50
3	< 20	-11
4	86	97
5	30	-44
6	37	-54
K= 0 L= 10		
0	157	-140
1	267	257
2	62	82
3	210	-212
4	179	-174
5	39	-31
6	< 19	10
7	24	29
8	< 21	-36
K= 1 L= 10		
0	404	421
1	113	126
2	416	-417
3	239	-250
4	250	260
5	168	171
6	180	-194
7	59	-65
8	< 21	-3
K= 2 L= 10		
0	70	54
1	414	-395
2	227	-226
3	332	338
4	203	200
5	171	-171
6	162	-164
7	158	168
K= 3 L= 10		
0	291	-290
1	205	-213
2	325	319
3	235	229
4	196	-194
5	202	-197
6	153	155
K= 4 L= 10		
0	42	-45
1	38	45
2	90	95
3	32	45
4	135	-125
5	< 29	40
K= 5 L= 10		
0	79	83
1	< 21	3
2	< 21	-22
K= 0 L= -10		
1	341	-364
2	118	125
3	312	332
4	135	146
5	458	-460
6	127	125
7	357	363
8	117	103
9	< 19	-10
10	< 20	-3
11	244	222
K= 1 L= -10		
1	56	52
2	365	372
3	337	336
4	514	-539
5	30	-36
6	469	462
7	89	89
8	117	-117
9	32	-34
10	62	63
11	< 21	-11
K= 2 L= -10		
1	437	449
2	91	84
3	621	-619
4	107	-97
5	518	527
6	51	-46
7	233	-225
8	102	-96
9	68	73
10	22	48
K= 3 L= -10		
1	115	-111
2	274	-276
3	135	-139
4	317	323
5	73	87
6	214	-212
7	139	-134
8	161	160
9	104	105
K= 4 L= -10		
1	149	-143
2	< 19	16
3	150	147
4	< 20	26
5	171	-179
6	< 20	3
7	115	131
8	< 21	23
K= 5 L= -10		
1	30	-42
2	88	95
3	84	90
4	118	-114
5	59	-65
K= 1 L= 11		
0	< 17	-8
1	64	55
2	55	-63
3	76	-64
4	134	-133
5	< 20	-15
6	< 20	7
7	< 21	24
K= 2 L= 11		
0	48	-31
1	76	-91
2	80	-66
3	120	-113
4	41	27
5	70	77
6	50	-42
K= 3 L= 11		
0	65	68
1	110	-109
2	69	72
3	118	117
4	93	86
5	< 21	-36
K= 4 L= 11		
0	92	-95
1	91	106
2	138	137
3	85	-79
K= 1 L= -11		
1	< 32	-33
2	167	159
3	< 19	-16
4	44	34
5	55	47
6	111	-111
7	< 19	8
8	111	98
9	< 20	21
10	129	-125
K= 2 L= -11		
1	127	121
2	45	-12
3	170	-175
4	77	-75
5	52	40
6	59	74
7	91	-93
8	< 20	-23
9	< 21	-29
K= 3 L= -11		
1	< 30	-14
2	185	-192
3	108	-97
4	152	165
5	83	90
6	149	-150
7	79	-70
8	< 21	41
K= 4 L= -11		
1	106	-100
2	< 20	-31
3	133	134
4	< 20	23
5	< 21	-79
6	38	-46
K= 0 L= 12		
0	< 18	24
1	321	332
2	269	269
3	150	-161
4	218	-228
5	214	103
6	24	22
K= 1 L= 12		
0	355	360
1	254	244
2	408	-406
3	390	-403
4	102	98
5	126	131
6	148	-142
K= 2 L= 12		
0	137	133
1	381	-380
2	277	-268
3	250	253
4	250	250
5	149	-154
K= 3 L= 12		
0	242	-238
1	169	-165
2	199	197
3	99	105
4	38	-47
K= 4 L= 12		
0	< 21	-30
1	< 21	37
2	< 21	22
K= 0 L= -12		
1	221	-234
2	172	192
3	< 18	-5
4	181	-161
5	87	-86
6	198	181
7	< 20	-18
8	< 20	4
9	115	115
K= 1 L= -12		
1	274	183
2	207	219
3	155	-168
4	250	-233
5	149	135
6	241	234
7	48	-45
8	33	-35
9	< 21	-25
K= 2 L= -12		
1	328	328
2	96	-86
3	355	-361
4	32	-11
5	406	410
6	165	-151
7	228	-221
8	< 21	-14
K= 3 L= -12		
1	197	-200
2	229	-224
3	62	73
4	189	209
5	51	-48
6	196	-191
7	< 21	0
K= 4 L= -12		
1	104	-101
2	29	41
3	106	106
4	< 21	18
5	122	-114
K= 1 L= 13		
0	87	-87
1	37	-11
2	73	72
3	88	76
4	31	-40
5	48	-57
K= 2 L= 13		
0	49	44
1	< 28	27
2	34	9
3	88	-85
4	< 21	-7
K= 3 L= 13		
0	43	49
1	52	-64
2	< 21	11
K= 1 L= -13		
1	92	-93
2	147	161
3	< 19	0
4	117	-127
5	42	50
6	< 20	-4
7	91	89
8	< 21	64
K= 2 L= -13		
1	104	113
2	30	21
3	66	-72
4	47	48
5	< 20	2
6	69	64
7	33	24
K= 3 L= -13		
1	< 22	35
2	96	-93
3	< 28	42
4	< 21	29
5	< 21	-13
6	< 21	1
K= 0 L= 14		
0	< 20	-28
1	144	127
2	354	325
3	47	-55
K= 1 L= 14		
0	< 23	23
1	220	205
2	159	-145
3	315	-314
K= 2 L= 14		
0	228	232
1	149	-141
2	326	-310
K= 0 L= -14		
1	< 34	28
2	< 31	43
3	< 20	2
4	89	91
5	51	-63
6	< 21	-6
7	145	159
K= 1 L= -14		
1	125	126
2	< 20	2
3	73	-77
4	< 20	-33
5	< 21	-3
6	201	202
7	125	-112
K= 2 L= -14		
1	56	57
2	173	-178
3	92	-89
4	31	34
5	256	244
K= 1 L= 15		
0	< 21	-22
1	< 26	38
2	< 21	39
3	< 21	-16
4	104	-104

0·015 Å for Mn, O, and C, respectively. (The e.s.d. value along the *b* axis for C(12), C(*X*1) and C(*X*2) is 0·017 Å).

Anisotropic thermal motion

Because of the large values* obtained for the anisotropic thermal parameters (Table 2) of several atoms, in particular C(12), C(*X*1) and C(*X*2), it was considered desirable to obtain some experimental evidence, if possible, to distinguish between thermal motion and a disordered packing of molecules. Patterns involving streaks between reciprocal lattice points or large isolated diffuse spots or areas on Laue photographs which vary strongly in intensity with temperature are known to indicate the existence of large-amplitude high-frequency thermal waves in the solid (Lonsdale, 1959). Therefore, photographs, employing the Laue technique

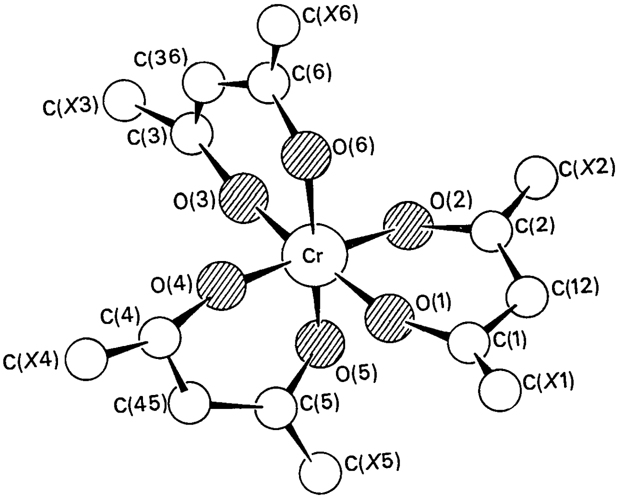

Fig. 1. Labeling scheme for trisacetylacetonatochromium(III).

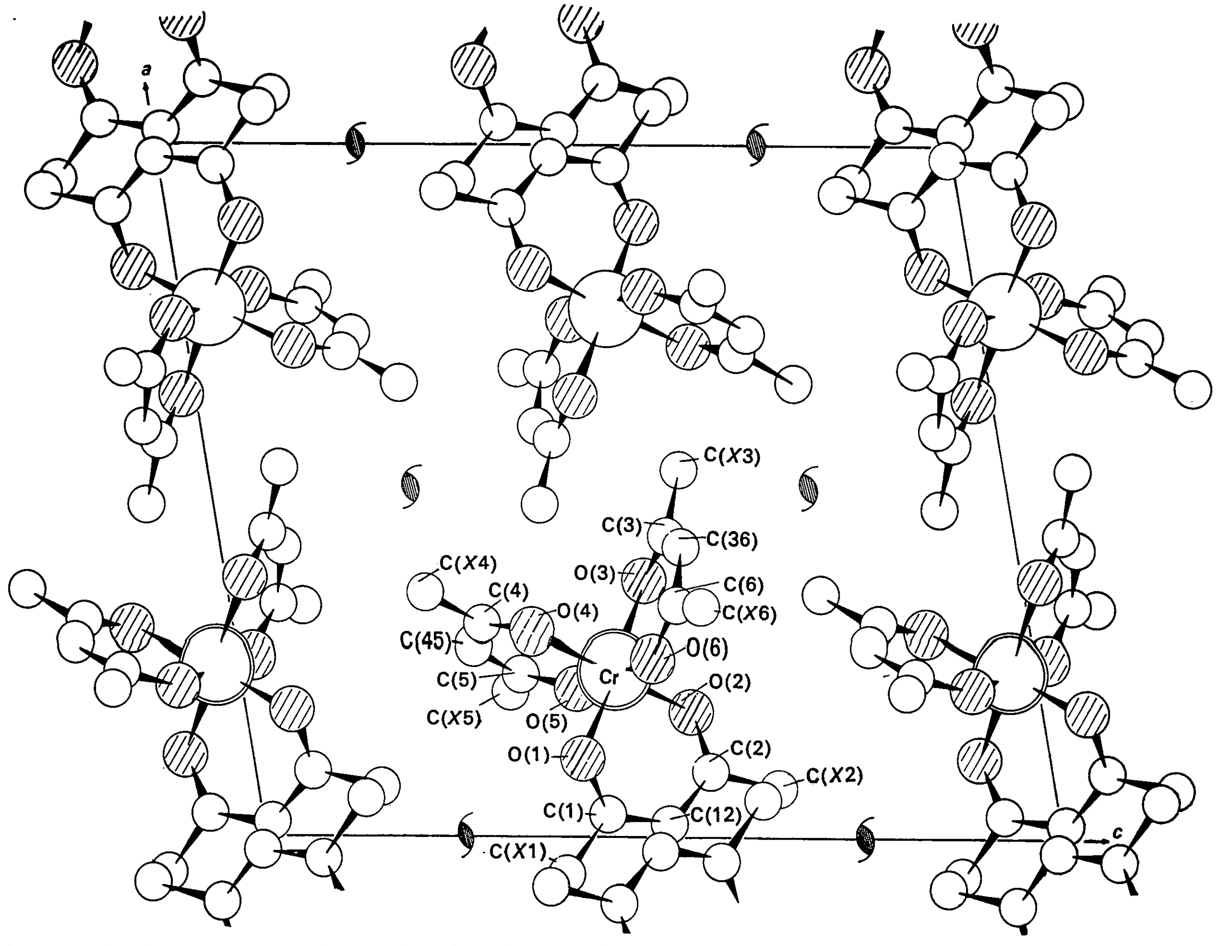

Fig. 2. Molecular arrangement viewed along b_0 axis. Chromium atoms near $y=\frac{1}{4}$ are denoted by double circles; those near $y=\frac{3}{4}$ by a single open circle.

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* The thermal parameters, B_{ij}, have the units Å^2, hence the corresponding root mean square (r.m.s.) amplitude of displacement might be better for comparative purposes. B_{ij}'s of 3·5, 7·0, and 30·0 would correspond to r.m.s. amplitudes of 0·21, 0·30, and 0·61 Å, respectively. Simple crystal structures, such as the hexagonal-packed metals of Cd and Zn, show r.m.s. displacements along *a* and *c* which approach ratios of 2:1 (*International Tables for X-ray Crystallography*, 1962).

both with unfiltered Mo and filtered Mo $K\alpha$ radiation, were taken at room temperature and in the range −35 to −65 °C along the a^* and c^* axes. The well defined patterns of diffuse spots in the room temperature Mo $K\alpha$ photographs disappeared at the lower temperature; however, the patterns were reproduced when the crystal was returned to room temperature. This indicates that the thermal parameters, though large, are probably real and that the 'smearing' of the Fourier syntheses at the atom sites of C(12), C(X1) and C(X2) is due to thermal motion rather than to a disordered packing of the molecules in the lattice.

Discussion

Soon after the structure determination of the manganese(III) complex appeared (to which was appended a note added in proof concerning the chromium(III) complex), the redetermination of the cobalt (III) complex (Pfluger, 1964) and the determination of the rhodium(III) complex (Morrow, 1964) became known to us and established the same molecular packing for all these compounds. These results appear to corroborate the earlier crystal studies of Astbury which indicated these compounds formed a monoclinic isomorphic series.

Tables 4 and 5 list the intramolecular bond lengths and bond angles, respectively, for the molecule (e.s.d. by the method of Cruickshank). There do not appear to be any values which are significantly different from the averages. In Fig. 3, the average values of these intramolecular bond lengths and angles are compared with those for the manganese complex. The average metal–oxygen distances of the two compounds are significantly different (difference ~10σ), and, although the difference between the average values for the C–O bond distances cannot be considered significant, the C–O bonds of the chromium compound appear to be shorter than those for the manganese compound. The carbon–carbon distances of the two complexes are in agreement with each other. The 1·39 Å ring C–C bond distance lies between the single-bond (1·54 Å) and the double-bond (1·33 Å) values. This partial double bond character is consistent with the expected delocalization of π electrons in the chelate rings.

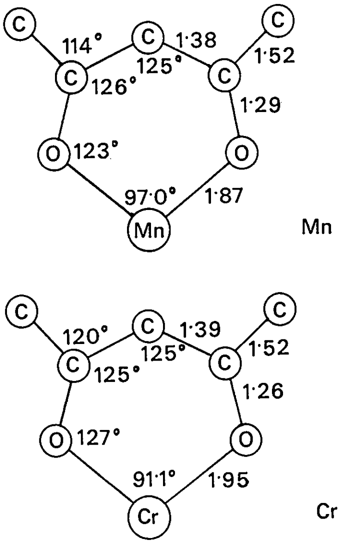

Fig. 3. Average intramolecular bond lengths for the chromium and manganese complexes.

Table 4. *Intramolecular bond lengths*

		Average
Cr–O(1)	1·943 ± 0·007 Å	
Cr–O(2)	1·942	
Cr–O(3)	1·951	
Cr–O(4)	1·958	
Cr–O(5)	1·956	
Cr–O(6)	1·959	1·951 Å
O(1)–C(1)	1·244 ± 0·016	
O(2)–C(2)	1·255	
O(3)–C(3)	1·272	
O(4)–C(4)	1·282	
O(5)–C(5)	1·271	
O(6)–C(6)	1·252	1·263
C(1)–C(12)	1·393 ± 0·021	
C(2)–C(12)	1·396	
C(3)–C(36)	1·384	
C(6)–C(36)	1·390	
C(4)–C(45)	1·366	
C(5)–C(45)	1·398	1·388
C(1)–C(X1)	1·530 ± 0·021	
C(2)–C(X2)	1·539	
C(3)–C(X3)	1·513	
C(4)–C(X4)	1·502	
C(5)–C(X5)	1·506	
C(6)–C(X6)	1·514	1·517

The oxygen–oxygen separations in the octahedron surrounding the chromium ion are listed in Table 6. This octahedron is very much more regular (the 2·786 Å average intrachelate O–O separation mightbe considered possibly significantly different from the average interchelate O–O separation of 2·751 Å) than that found for the manganese(III) complex (the corresponding distances are 2·805 and 2·596 Å, respectively). Preliminary results on the redetermination of the iron(III) complex (Iball & Morgan, 1964) indicate that this octahedron is also regular. The regular environments about the chromium and iron, but not manganese, complexes are in agreement with the infrared data for the respective compounds (Forman & Orgel).

Fig. 4* best illustrates the large anisotropic motion of one of the chelate rings, in particular carbon atoms C(12), C(X1) and C(X2). The ellipsoids, whose principal axes are proportional to thc root mean square thermal displacement along the axes, represent atoms and are drawn to scale with respect to the interatomic

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* This figure was drawn at Oak Ridge National Laboratory on a Cal Comp $x-y$ plotter with computer code OR TEP written by Dr C. K. Johnson.

Table 5. *Intramolecular bond angles*

O(1)–Cr–O(2)	91·66 ± 0·6°	C(1)–C(12)–C(2)	124·06 ± 1·0°
O(3)–Cr–O(6)	90·67	C(3)–C(36)–C(6)	125·61
O(4)–Cr–O(5)	90·95	C(4)–C(45)–C(5)	125·60
Cr–O(1)–C(1)	126·51 ± 0·9	O(1)–C(1)–C(X1)	113·98 ± 1·0
Cr–O(2)–C(2)	126·75	O(2)–C(2)–CX(2)	114·46
Cr–O(3)–C(3)	126·41	O(3)–C(3)–C(X3)	115·92
Cr–O(4)–C(4)	126·49	O(4)–C(4)–C(X4)	114·79
Cr–O(5)–C(5)	127·50	O(5)–C(5)–C(X5)	114·95
Cr–O(6)–C(6)	127·84	O(6)–C(6)–C(X6)	116·36
O(1)–C(1)–C(12)	125·70 ± 1·0	C(12)–C(1)–C(X1)	120·23 ± 1·0
O(2)–C(2)–C(12)	124·87	C(12)–C(2)–C(X2)	120·66
O(3)–C(3)–C(36)	124·69	C(36)–C(3)–C(X3)	119·38
O(4)–C(4)–C(45)	125·19	C(45)–C(4)–C(X4)	120·01
O(5)–C(5)–C(45)	124·14	C(45)–C(5)–C(X5)	120·90
O(6)–C(6)–C(36)	123·81	C(36)–C(6)–C(X6)	119·83

Table 6. *Oxygen–oxygen distances found in the octahedron about the metal ion*

O(1)–O(2)	2·786 ± 0·013 Å	O(1)–O(5)	2·754 Å
O(3)–O(6)	2·781	O(3)–O(4)	2·731
O(4)–O(5)	2·791	O(2)–O(6)	2·750
O(1)–O(4)	2·735	O(2)–O(3)	2·770
O(4)–O(6)	2·771	O(3)–O(5)	2·746
O(6)–O(1)	2·764	O(5)–O(2)	2·742

separation. Preliminary results on the cobalt(III) and rhodium(III) complexes also indicate anisotropic motion of this particular ring. The packing arrangement of the molecules requires this particular chelate ring to be stacked directly above itself along the *b* axis. Hence, the shorter intermolecular (nearest neighbours) separations (Table 7) for C(*X*1), C(12) and C(*X*2) involve the C(*X*2′), C(12′) and C(*X*1′), respectively, which are translated $\frac{1}{2}$ of a unit cell *b* by the 2_1 axis. This type of molecular packing allows this chelate ring sufficient freedom to oscillate (in unison with its symmetry related members) with a large amplitude. The

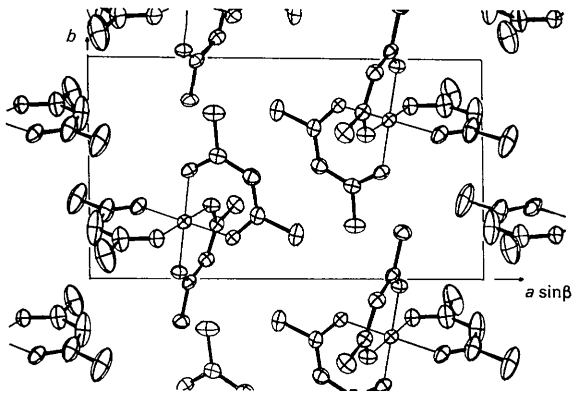

Fig. 4. Perspective view for trisacetylacetonatochromium(III) along c_0 axis. Atoms are represented as ellipsoids with principal axes proportional to the root mean square thermal displacements along the axes. Only two molecules per cell (those contained between $z \simeq -0·25$ and 0·25) are included. The second set (z between ~ 0·25 and ~0·75) are related to the illustrated set by the *c* glide and their inclusion would only serve to obscure the drawing.

Table 7. *Intermolecular* (*nearest neighbour*) *distances and angles*

Intermolecular distances (Å) for each methyl carbon are followed in parentheses by the C(*i*)–C(*Xi*)–C(intermolecular) angle (°). The C(*i*)–C(*Xi*) intramolecular bond distances are shown after each methyl carbon atom. The numbers preceding the atom involved in the intermolecular contacts refer to the following positions:

1 $x, 1+y, z$
2 $x, -1+y, z$
3 $1-x, 1-y, 1-z$
4 $1-x, -y, 1-z$
5 $-x, 1-y, 1-z$
6 $-x, -y, 1-z$
7 $x, \frac{1}{2}-y, \frac{1}{2}+z$
8 $x, 1\frac{1}{2}-y, \frac{1}{2}+z$
9 $x, -\frac{1}{2}-y, \frac{1}{2}+z$
10 $x, 1\frac{1}{2}-y, -\frac{1}{2}+z$
11 $x, \frac{1}{2}-y, -\frac{1}{2}+z$
12 $x, -\frac{1}{2}-y, -\frac{1}{2}+z$

C(*X*1)	1·530	C(*X*2)	1·539	C(*X*3)	1·513
5 C(12)	3·493 (80)	6 C(*X*1)	3·716 (106)	4 C(*X*5)	3·676 (153)
6 C(*X*2)	3·716 (79)	5 C(*X*1)	3·868 (65)	4 C(45)	3·748 (163)
5 C(*X*2)	3·868 (113)	7 C(*X*4)	3·959 (89)	7 C(*X*4)	3·858 (67)
5 C(*X*6)	4·112 (119)	7 C(45)	4·109 (95)	4 C(*X*6)	3·861 (106)
11 C(*X*2)	4·188 (100)	7 C(*X*1)	4·178 (156)	3 C(36)	3·880 (77)
5 C(*X*1)	4·641 (53)	9 C(*X*5)	4·435 (134)	4 (C*X*3)	4·302 (92)
1 C(*X*5)	4·907 (75)	2 C(*X*6)	4·762 (75)	2 C(*X*6)	4·403 (78)
				3 C(*X*4)	4·449 (96)
C(*X*4)	1·502	C(*X*5)	1·506	C(*X*6)	1·514
11 C(*X*3)	3·858 (142)	2 O(4)	3·555 (143)	3 C(*X*3)	3·861 (77)
11 C(36)	3·877 (112)	4 C(*X*3)	3·676 (74)	8 C(*X*4)	3·893 (158)
10 C(*X*6)	3·893 (147)	2 O(1)	3·900 (147)	5 C(*X*1)	4·112 (85)
11 C(*X*2)	3·951 (77)	2 C(*X*4)	4·157 (136)	1 C(*X*5)	4·251 (76)
1 C(*X*5)	4·157 (83)	2 C(*X*6)	4·251 (79)	7 C(45)	4·389 (74)
3 C(*X*3)	4·449 (96)	12 C(*X*2)	4·435 (148)	1 C(*X*3)	4·403 (132)
		11 C(*X*6)	4·559 (98)	7 C(*X*5)	4·559 (105)
		2 C(*X*1)	4·907 (136)	1 C(*X*2)	4·762 (133)

other two chelate rings are arranged in the crystal in such a manner as to prevent the existence of a similar lattice wave or mode. The reproducibility of the diffuse patterns with temperature cycling together with the packing arrangement suggest the interpretation that the large anisotropic temperature parameters arise from the thermal motion rather than a slight disorder in the molecular packing is correct.

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Diffraction by a One-Dimensionally Disordered Crystal. I. The Intensity Equation

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The intensity equation $I(\varphi)=N\text{spur}\,\mathbf{VF}+\sum_{m=1}^{N-1}(N-m)\,\text{spur}\,\mathbf{VFQ}^m+\text{conj.}$ for X-rays diffracted by a one-dimensionally disordered crystal has been solved by three methods, *viz.* (i) by using the inverse matrix $(\mathbf{1}-\mathbf{Q})^{-1}$, (ii) by diagonalizing $\mathbf{Q}$ by the similarity transformation $\mathbf{OQO}^{-1}$ and by using the solutions of a characteristic equation $\det(y\mathbf{1}-\mathbf{Q})=0$ and a set of simultaneous equations with respect to b_ν's, $\sum_{\nu=1}^{R} b_\nu^\nu y_\nu^m=B_m=\text{spur}\;\mathbf{VFQ}^m$ $(m=0, 1, \cdots, R-1)$, and (iii) by using B_m and the relation between roots and coefficients of the characteristic equation without solving these equations explicitly. These methods should be applied to the problem only after the order of matrices has been reduced to lower order by considering the symmetry character involved in the matrices.

Explicit expressions are given for three cases, namely the case of different thickness, that of equal thickness, and that of displacement stacking faults. In the case of displacement stacking faults, the three-dimensional Patterson function is given with respect to the distribution of origins of layers. The result is compared with that obtained by Allegra.

Introduction

The intensity of X-rays diffracted by a one-dimensionally disordered crystal such as SiC, $CdBr_2$, CdI_2, some alloys of the Laves phase, many other alloys and metals showing the stacking faults between cubic and hexagonal close-packed structures, and some kinds of antiphase domain structures and minerals, has been studied by many researchers, for example, Wilson (1942, 1943), Hendricks & Teller (1942), Zachariasen (1947), Jagodzinski (1949 *a*, *b*, *c*, 1954), Méring (1949), Paterson (1952), Kakinoki & Komura (1952; 1954 *a*, *b*), Gevers