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The Crystal Structure of Two Modifications of Chromium(III) Tris(diethyldithiophosphate), $Cr[S_2P(OC_2H_5)_2]_3$

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Chromium(III) tris(diethyldithiophosphate), $Cr[S_2P(OC_2H_5)_2]_3$ crystallises in two different forms, one of which is isomorphous to the corresponding vanadium complex. The structure of the other crystal modification has been determined from three-dimensional X-ray data collected on an automatic diffractometer and refined by the method of least squares to give an R-value of 8.1 % for 1368 reflections. The crystal is monoclinic with $a = 14.28$ Å, $b = 13.60$ Å, $c = 14.35$ Å, $\beta = 89.5°$; space group $C2/c$. The relatively small number of observed reflections is due to large thermal vibration and limits the accuracy of the structure determination. The molecule nearly has the symmetry 32 although only a twofold axis is required by the space group. The only difference between the two modifications is in the packing.

Jørgensen has prepared several diethyldithiophosphate (dtp) complexes [1] and kindly sent us samples of some. Those of chromium, cobalt, ruthenium, rhodium, and iridium were found to be isomorphous as indicated by their powder diagrams and by the fact that all of these complexes can co-crystallise. Lacroix has measured the paramagnetic resonance of $Crdtp_3$ as a 1 % impurity in $Codtp_3$.[2] $Crdtp_3$ was chosen for the structure determination for this reason and because plenty of good crystals were available.

After this structure had been solved the structures of $Vdtp_3$ [3] and $Indtp_3$ [4] have been published. They are mutually isomorphous but different from the structure found in the present investigation. Careful investigation of our sample of $Crdtp_3$ under the polarising microscope showed that about 10 % of the crystals extinguished parallel to their prominent direction. These crystals were found to have cell dimensions and space group similar to those reported for $Vdtp_3$. The quality of the photographs was low, but we consider it proved that this modification of $Crdtp_3$ is isomorphous to $Vdtp_3$, and we intend to do no further work on these crystals.

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EXPERIMENTAL

The unit cell and space group were determined from precession photographs taken with Mo$K\alpha$ radiation ($\lambda = 0.7107$ Å). Preliminary intensities for two projections were obtained from integrated precession films. The three-dimensional intensities were measured on a linear diffractometer of the Arndt-Phillips type [5] with Mo radiation. The crystal was approximately $0.3 \times 0.3 \times 0.2$ mm^3. Balanced filters and pulse height discrimination were applied. The absorption coefficient is $\mu = 10.7$ cm^{-1} for Mo$K\alpha$ radiation, so it was considered justified not to correct for absorption.

STRUCTURE DETERMINATION

The positions of the heavier atoms were found from the *bc* and *ac* projections by aid of the characteristic intensity distributions. For some of the light atoms only approximate coordinates could be obtained because of overlap and lack of data. When three-dimensional data became available the trial structure gave $R = 31$ % and a Fourier map showed that one carbon atom had to be moved 1 Å. Least squares refinement lowered R to 14 % and further refinement including anisotropic temperature factor parameters lowered R to 9 %. At this point a difference Fourier map was calculated which showed no extra atoms heavier than hydrogen. The hydrogen atoms of the CH_2 groups were found in approximately the positions expected. Those of the CH_3 groups could not be found with certainty; one would expect a staggered configuration around the C – C bond but this does not correspond very well to the peaks in the difference map although there are peaks at approximately the right distances from the carbon atoms. One of the three independent ethyl groups shows large temperature factors and an abnormally short C – C bond (1.34 Å). The difference Fourier map gave no indication of the reason for this, and an attempt to move the atoms to the commonly accepted distances failed because least squares refinement immediately brought them back. A riding motion correction to the bond lengths of this OCH_2CH_3 group indicated that the O – C bond appears shortened by 0.04 Å but since both carbon atoms move nearly equally much, little correction was indicated for the C – C bond. The assumption that the carbon atoms moved independently led to a correction of 0.33 Å, so it seems likely that a model with partly correlated motion could lead to a corrected distance of the length of a normal single bond. The reason for the unusually large thermal motion of this ethyl group seems to be the lack of normal van der Waals contacts.

CRYSTAL DATA

$Cr[S_2P(OC_2H_5)_2]_3$
Crystal system: monoclinic

$a = 14.28 \pm 0.03$ Å, $b = 13.60 \pm 0.03$ Å, $c = 14.35 \pm 0.03$ Å, $\beta = 89.5 \pm 0.02°$.
Systematic absences: hkl: $h + k \neq 2n$
$h0l$: $l \neq 2n$

$d_{meas} = 1.44$ g/cm^3, $d_{calc} = 1.45$ g/cm^3 for four molecules per cell.

Possible space groups: $C2/c$ (No. 15) and Cc (No. 9). No piezoelectric effect could be detected so the centrosymmetric space group $C2/c$ was preferred, and the structure analysis confirmed this.

The final atomic coordinates are given in Table 1, temperature factor parameters in Table 2, interatomic distances and angles in Table 3, and Table 4 is a list of observed and calculated structure factors. The scattering factors used were those of International Tables for X-Ray Crystallography, Vol. 3, pp. 202 – 205.

Table 1. Atomic coordinates in fractions of the unit cell. Standard deviations $\times 10^{-4}$ in parentheses.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Cr	0.0000	(0)	0.3185	(2)	0.2500	(0)
S_1	0.0820	(2)	0.3348	(2)	0.1023	(2)
S_2	0.1162	(2)	0.4376	(2)	0.2996	(2)
S_3	0.0907	(2)	0.1845	(2)	0.3139	(2)
P_1	0.0000	(0)	0.0963	(3)	0.2500	(0)
P_2	0.1657	(2)	0.4264	(2)	0.1692	(2)
O_1	0.0477	(6)	0.0184	(5)	0.1839	(5)
O_2	0.1789	(5)	0.5277	(6)	0.1185	(6)
O_3	0.2281	(5)	0.8962	(7)	0.3386	(7)
C_1	0.1152	(12)	0.0482	(9)	0.1155	(11)
C_2	0.1384	(11)	0.0307	(12)	0.0550	(11)
C_3	0.1035	(9)	0.5900	(9)	0.0958	(10)
C_4	0.1411	(10)	0.6807	(9)	0.0593	(9)
C_5	0.1961	(12)	0.8046	(14)	0.3105	(15)
C_6	0.1128	(15)	0.7783	(13)	0.3450	(13)

Table 2. Mean square vibration amplitudes, u_{ij}, in $Å^2 \times 10^{-4}$ with standard deviations in parentheses.

	u_{11}	σu_{11}	u_{22}	σu_{22}	u_{33}	σu_{33}	u_{12}	σu_{12}	u_{13}	σu_{13}	u_{23}	σu_{23}
Cr	603	(13)	551	(13)	769	(12)	0	(0)	19	(11)	0	(0)
S_1	866	(20)	816	(20)	802	(14)	9	(16)	138	(16)	29	(16)
S_2	789	(19)	796	(20)	986	(18)	−188	(16)	−56	(17)	38	(17)
S_3	840	(20)	677	(18)	971	(17)	83	(15)	−31	(16)	84	(16)
P_1	878	(30)	601	(25)	989	(26)	0	(0)	280	(25)	0	(0)
P_2	678	(18)	769	(20)	1193	(22)	21	(16)	102	(18)	159	(19)
O_1	1103	(55)	700	(47)	1210	(49)	−33	(43)	426	(48)	−130	(45)
O_2	858	(51)	835	(53)	1520	(59)	−78	(43)	108	(49)	334	(50)
O_3	671	(49)	1200	(69)	2066	(77)	−89	(49)	−91	(56)	−358	(64)
C_1	1664	(123)	847	(87)	1679	(113)	−5	(87)	1038	(105)	−186	(86)
C_2	1531	(126)	1309	(121)	1649	(106)	307	(105)	712	(104)	11	(102)
C_3	942	(86)	768	(78)	1727	(105)	109	(68)	−5	(85)	378	(80)
C_4	1138	(99)	784	(84)	1483	(99)	−38	(74)	−160	(87)	360	(81)
C_5	1011	(103)	1939	(158)	2184	(146)	−638	(107)	178	(111)	−671	(131)
C_6	2007	(165)	1615	(142)	2114	(152)	−660	(134)	964	(139)	−88	(129)

Table 3. Interatomic distances and angles with standard deviations in parentheses.

Distance	Å	10^{-3} Å	Distance	Å	10^{-3} Å
$Cr-S_1$	2.423	(3)	P_2-O_3	1.574	(9)
$Cr-S_2$	2.430	(3)	O_1-C_1	1.429	(17)
$Cr-S_3$	2.421	(3)	O_2-C_3	1.412	(15)
S_1-P_2	1.980	(4)	O_3-C_5	1.388	(22)
S_2-P_3	2.000	(5)	C_1-C_2	1.418	(21)
S_3-P_1	1.995	(4)	C_3-C_4	1.441	(18)
P_1-O_1	1.572	(8)	C_5-C_6	1.334	(27)
P_2-O_2	1.569	(9)			
Angle	**Degrees**		**Angle**	**Degrees**	
S_1-Cr-S_2	82.5	(0.1)	$S_1-P_2-O_2$	113.5	(0.4)
S_1-Cr-S_3	98.3	(0.1)	$S_1-P_2-O_3$	112.8	(0.4)
S_2-Cr-S_3	91.3	(0.1)	$S_2-P_3-O_2$	114.1	(0.4)
S_3-Cr-S_3	82.4	(0.1)	$S_2-P_3-O_3$	114.7	(0.4)
$Cr-S_1-P_2$	85.5	(0.1)	$O_2-P_2-O_3$	94.8	(0.5)
$Cr-S_2-P_3$	84.9	(0.1)	$P_1-O_1-C_1$	120.6	(0.7)
$Cr-S_3-P_1'$	85.9	(0.1)	$P_2-O_2-C_3$	123.1	(0.8)
$S_3-P_1-S_3$	106.2	(0.2)	$P_2-O_3-C_5$	122.2	(1.0)
$S_3-P_1-O_1'$	113.8	(0.3)	$O_1-C_1-C_2$	111.0	(1.1)
$O_1-P_1-O_1$	95.3	(0.4)	$O_2-C_3-C_4$	108.4	(1.1)
$S_1-P_2-S_2$	107.0	(0.2)	$O_3-C_5-C_6$	115.4	(1.6)

DESCRIPTION ON THE STRUCTURE

The molecule could be expected to have 32 symmetry and indeed this is nearly the case although only one twofold axis is imposed by the space group.

The chromium atoms are surrounded by six sulphur atoms in a slightly flattened octahedron, the angles at chromium in the Cr – S – P – S ring being only 82.3°. The Cr – S distances are in good agreement with those found in other compounds, *e.g.* 2.42 – 2.46 in the chromium sulphides.[6] The average S – P distance is 1.99 Å which is in between the S – P distance in $SP(C_2H_5)_3$ (1.86 Å) (double bond),[7] and the calculated length of a single bond (2.10 Å).[8] It is close to the value (2.01 Å) found in bis(dimethyl dithiophosphinato)-nickel [9] and to the average of those in $Vdtp_3$ [3] (1.98 Å), in $Indtp_3$ [4] (1.99 Å), and in $Bidtp_3$ [10] (1.99 Å). No indication is found for two different S – P bond lengths as reported for $Indtp_3$.[4] The P – O distances are close to the length of the P – O single bond as found in the ferro-electric form of KH_2PO_4 [11] so the double bond character of the thiophosphate group seems distributed between the two P – S bonds.

The S – P – S angles are fairly close to the tetrahedral value; they have to be large so that the ligand can span the Cr-ion. The Cr – S – P angles are slightly smaller than the value of 95 – 100°, usually found for angles of similar sulphur atoms. The positions of oxygen and carbon atoms are of low accuracy, but the distances and angles between them do not differ significantly from the

Table 4. Observed and calculated structure factors, 10 × absolute values.

h	k	l	Fobs	Fcal
-1	17	0	130	-85
0	14	0	201	-236
0	12	0	422	486
-1	13	0	204	-181
-2	14	0	257	-256
-4	16	0	119	-67
-1	11	0	123	-47
-2	12	0	189	212
-3	13	0	292	266
-4	14	0	170	-163
-7	17	0	163	-79
0	8	0	1861	-1830
-1	9	0	888	870
-2	10	0	202	199
-5	13	0	250	246
0	6	0	501	465
-1	7	0	296	286
-2	8	0	483	-486
-4	10	0	427	430
-5	11	0	178	-206
0	4	0	429	-434
-1	5	0	273	256
-2	6	0	1079	1056
-3	7	0	503	-504
-4	8	0	738	724
-5	9	0	158	158
-6	10	0	184	188
-7	11	0	267	-261
0	2	0	758	761
-1	3	0	1033	959
-2	4	0	270	-263
-3	5	0	1187	-1166
-4	6	0	223	262
-6	8	0	98	-55
-7	9	0	213	223
-8	10	0	121	111
-1	1	0	738	-633
-2	2	0	820	-838
-3	3	0	1403	1365
-4	4	0	271	-337
-5	5	0	1261	-1238
-6	6	0	524	-549
-7	7	0	410	443
-8	8	0	250	-287
-9	9	0	183	210
-10	10	0	117	93
-12	12	0	178	131
-2	0	0	1471	1380
-3	1	0	1497	1524
-4	2	0	597	-618
-5	3	0	204	164
-6	4	0	189	167
-7	5	0	386	-365
-8	6	0	352	-344
-9	7	0	205	-169
-10	8	0	391	-383
-12	10	0	138	77
-4	0	0	2275	-2094
-5	1	0	346	375
-6	2	0	748	752
-7	3	0	424	446
-8	4	0	262	-258
-9	5	0	403	-416
-10	6	0	270	287
-11	7	0	102	-109
-12	8	0	248	-259
-14	10	0	144	71
-7	1	0	569	-585
-8	2	0	94	-108
-10	4	0	166	-155
-11	5	0	102	-102
-12	6	0	237	252
-8	0	0	448	470
-9	1	0	190	188
-10	2	0	418	-428
-12	4	0	248	-253
-13	5	0	242	-256
-14	6	0	135	163
-10	0	0	806	787
-11	1	0	319	-348
-12	2	0	245	-250
-13	3	0	224	251
-14	4	0	145	-115
-15	5	0	189	-178
-12	0	0	645	647
-14	2	0	241	-251
-15	3	0	211	205
-17	5	0	129	-71
-18	2	0	139	72
-18	0	0	179	176
11	13	1	149	-108
8	14	1	162	-88
16	4	1	184	-171
15	5	1	137	101
11	9	1	167	181
10	10	1	191	176
6	14	1	145	-172
4	16	1	192	-128
14	4	1	280	-295
13	5	1	263	238
11	7	1	166	-207
10	8	1	226	270
9	9	1	152	150
8	10	1	133	-43
5	13	1	194	-222
14	2	1	155	169
12	4	1	205	-253
11	5	1	290	302
10	6	1	140	178
9	7	1	265	-320
7	9	1	611	626
6	10	1	571	-596
5	11	1	182	-159
4	12	1	507	525
3	13	1	395	-410
13	1	1	303	-299
12	2	1	143	83
11	3	1	141	171
10	4	1	243	-214
8	6	1	338	337
7	7	1	282	286
4	10	1	122	-149
3	11	1	287	-307
1	13	1	244	-231
-1	15	1	199	192
11	1	1	417	-479
10	2	1	441	-454
8	4	1	535	573
7	5	1	566	-546
6	6	1	407	540
5	7	1	276	259
4	8	1	542	569
3	9	1	362	-367
2	10	1	392	-404
1	11	1	370	384
0	12	1	483	-461
-1	13	1	247	-282
9	1	1	161	-191
8	2	1	201	203
7	3	1	847	-881
6	4	1	614	-634
5	5	1	295	309
3	7	1	596	-593
2	8	1	742	778
1	9	1	716	718
0	10	1	654	-640
-3	13	1	330	-322
-4	14	1	135	-140
-5	15	1	143	-54
7	1	1	529	-528
6	2	1	475	535
4	4	1	2712	-2736
3	5	1	1123	1127
1	7	1	831	-811
-1	9	1	608	625
-2	10	1	135	130
-3	11	1	480	-519
-4	12	1	266	288
4	2	1	547	468
3	3	1	1184	1157
2	4	1	1959	-1882
1	5	1	353	317
0	6	1	252	262
-1	7	1	1485	-1472
-2	8	1	395	435
-3	9	1	230	230
-6	12	1	244	238
3	1	1	975	-947
2	2	1	784	-695
1	3	1	1250	-1316
0	4	1	158	157
-1	5	1	90	-107
-2	6	1	562	523
-4	8	1	153	-133
-5	9	1	346	373
-6	10	1	309	-319
-8	12	1	136	132
1	1	1	2641	-2568
0	2	1	1585	1535
-1	3	1	1249	-1210
-2	4	1	551	-514
-3	5	1	401	355
-4	6	1	123	60
-5	7	1	836	868
-6	8	1	162	36
-7	9	1	186	154
-9	11	1	137	83
-1	1	1	1967	-2039
-2	2	1	689	-770
-3	3	1	443	423
-4	4	1	724	-695
-6	6	1	478	498
-7	7	1	266	241
-8	8	1	520	528
-9	9	1	165	139
-10	10	1	200	-183
-11	11	1	150	109
-3	1	1	1184	-1091
-4	2	1	540	-503
-5	3	1	239	217
-6	4	1	616	-626
-8	6	1	394	440
-9	7	1	508	-529
-10	8	1	229	263
-11	9	1	334	330
-5	1	1	826	788
-6	2	1	306	313
-7	3	1	407	-464
-8	4	1	729	-733
-9	5	1	300	332
-10	6	1	240	229
-11	7	1	196	-239
-7	1	1	342	-331
-8	2	1	264	242
-10	4	1	363	-360
-11	5	1	213	235
-12	6	1	125	73
-9	1	1	545	-554
-10	2	1	392	429
-11	3	1	237	-231
-13	5	1	218	214
-11	1	1	750	-780
-12	2	1	136	111
-14	4	1	226	-233
-13	1	1	263	-272
13	11	2	168	115
15	5	2	162	137
10	10	2	150	-188
15	3	2	236	-232
14	4	2	134	100
13	5	2	162	181
10	8	2	214	205
9	9	2	126	-46
8	10	2	170	-173
6	12	2	178	-172
4	14	2	230	222
3	15	2	156	-112
13	3	2	173	-152
12	4	2	146	161
11	5	2	188	221
10	6	2	399	-406
8	8	2	512	518
7	9	2	524	-524
5	11	2	307	326
4	12	2	182	-184
3	13	2	153	-200
2	14	2	177	193
0	16	2	145	-139
14	0	2	198	-156
13	1	2	143	141
8	6	2	267	272
7	7	2	448	-468
6	8	2	295	303
5	9	2	363	-426
4	10	2	220	-247
3	11	2	510	519
2	12	2	261	-304
-1	15	2	175	152
12	0	2	194	-223
11	1	2	362	-340
10	2	2	521	579
9	3	2	306	273
7	5	2	165	195
5	7	2	298	318
4	8	2	516	-509
3	9	2	405	-428
2	10	2	139	-155
1	11	2	164	146
0	12	2	154	-183
-2	14	2	212	235
10	0	2	151	49
9	1	2	309	-332
8	2	2	205	-232
7	3	2	265	239
5	5	2	995	955
4	6	2	816	-826
3	7	2	492	489
2	8	2	245	262
1	9	2	506	-514
0	10	2	293	-302
-1	11	2	481	-464
-3	13	2	208	-215
-4	14	2	175	185
8	0	2	1431	-1400
7	1	2	978	972
6	2	2	187	-157
5	3	2	1852	-1789
4	4	2	649	645
3	5	2	1846	1818
2	6	2	757	-808
1	7	2	519	-554
0	8	2	938	947
-1	9	2	342	-361
-2	10	2	426	-467
6	0	2	730	-761
5	1	2	286	-303
4	2	2	898	925
3	3	2	3283	-3413
2	4	2	173	198
0	6	2	226	239
-1	7	2	189	-174
-2	8	2	316	313
-3	9	2	135	124
-4	10	2	408	-400
-5	11	2	315	328
4	0	2	544	-452
3	1	2	1214	-1262
2	2	2	1246	-1220
1	3	2	888	-869
0	4	2	364	362
-1	5	2	378	-372
-2	6	2	470	-463
-3	7	2	167	180
-4	8	2	214	229
-5	9	2	209	-176
-6	10	2	170	-159
-7	11	2	342	365
-8	12	2	134	-126
2	0	2	2772	-2812
1	1	2	1451	1398
0	2	2	59	16
-1	3	2	2110	1975
-2	4	2	282	-270
-3	5	2	460	450
-4	6	2	132	-119
-6	8	2	160	143
-7	9	2	194	-195
-10	12	2	151	-106
-1	1	2	1244	-1310
-2	2	2	1717	1669
-3	3	2	552	519
-4	4	2	123	-80
-5	5	2	1080	1041
-6	6	2	283	287
-7	7	2	190	210
-9	9	2	425	-466
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5	9	9	305	331
4	10	9	240	-172
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8	4	9	379	-418
7	5	9	229	253
5	7	9	358	-406
4	8	9	293	282
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5	5	9	115	99
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3	7	9	377	-396
2	8	9	266	310
7	1	9	249	-222
6	2	9	133	143
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3	3	9	132	-109
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-1	3	9	242	-243
-2	4	9	137	161
-3	5	9	268	-293
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8	6	10	263	-235

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9	3	10	265	-242
7	5	10	310	290
6	6	10	152	-172
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4	8	10	432	441
3	9	10	143	-74
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7	3	10	330	-305
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6	2	10	146	175
4	4	10	241	262
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4	2	10	153	166
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-7	3	10	335	-336
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7	5	11	223	-218
6	6	11	142	-133
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3	9	11	208	-214
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4	6	11	207	-143
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6	2	11	171	-205
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4	4	11	173	-172
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1	3	11	106	-117
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-4	2	11	237	-249
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-11	9	11	171	-58
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-6	2	11	184	-209
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7	7	12	134	-22
9	3	12	226	229
7	5	12	213	-176
4	8	12	135	-154
2	10	12	177	170
8	2	12	142	-113
7	3	12	198	220
5	5	12	112	-59
2	8	12	210	-178
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-7	3	12	117	-78
-6	0	12	403	388
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3	9	13	155	103
6	4	13	121	-100
3	7	13	153	-101
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0	10	13	175	-98
1	7	13	206	-182
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7	3	14	111	-73
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5	3	14	114	-66
2	6	14	123	-93
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-1	9	14	139	-63
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4	0	14	177	-119
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1	3	14	92	-99
0	4	14	113	80
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-6	6	14	166	115
-2	0	14	85	-54
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-4	2	14	96	-58
-6	2	14	145	-101
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9	1	15	161	28
6	4	15	182	135
3	7	15	123	106
2	8	15	128	-48
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4	2	15	87	-83
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2	2	15	157	-138
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-7	3	15	122	21
-8	4	15	157	109
-9	1	15	196	124
5	5	16	165	-106
5	3	16	198	130
3	3	16	104	94
-1	7	16	133	-10
1	3	16	75	-10
2	0	16	187	189
-1	1	16	112	99
1	1	17	54	-33

normally accepted values. The thiophosphate groups have approximate *mm* symmetry with the ethyl groups as far from one another as possible. The O – P – O angles are small, probably because the carbon atoms attached to oxygen are being repelled by the sulphur atoms.

The structure is only held together by weak van der Waals forces which explains that the temperature movements are large, especially so for the ethyl group $C_5 - C_6$ which has no van der Waals contacts.

The large temperature movements agree well with the rapid fall off in intensity and Fig. 1 shows that the principal directions of the movements are reasonable. If the less symmetric space group *Cc* was chosen as has been

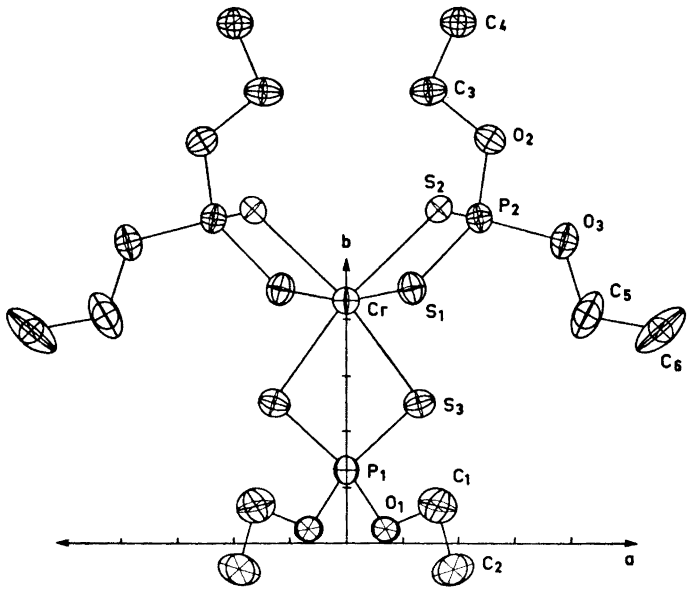

Fig. 1. A molecule of $Crdtp_3$ projected along the *c*-axis showing the ellipsoids of vibration.

suggested for $Indtp_3$ [4] some temperature factor parameters could decrease during the refinement (possibly giving less reasonable directions of largest movement) but correlation between atoms which are in space group $C2/c$ related by the twofold axis would be so large that it would be difficult to test whether the deviation from this symmetry was significant. For this reason, refinement in the acentric space group has not been attempted.

The photographs of the other crystalline form were of such poor quality that we could not hope to get reasonably accurate dimensions of the complex in this modification. The dimensions and geometry of the vanadium complex as described in Ref. 3 are so similar to the results found for the chromium complex in this investigation that the only difference has to be found in the packing of the molecules.

REFERENCES

1. Jørgensen, C. K. *J. Inorg. Nucl. Chem.* **24** (1962) 1571.
2. Gregorio, S., Weber, J. and Lacroix, R. *Helv. Phys. Acta* **38** (1965) 172.
3. Furlani, C., Porta, P., Sgamellotti, A. and Tomlinson, A. A. G. *Chem. Commun.* **1969** 1046.
4. Coggon, P., Lebedda, J. D., McPhail, A. T. and Palmer, R. A. *Chem. Commun.* **1970** 78.
5. Arndt, U. W. and Phillips, D. C. *Acta Cryst.* **14** (1961) 807.
6. Wells, A. F. *Structural Inorganic Chemistry*, Oxford University Press, London 1962, p. 518.
7. van Meersche, M. and Léonard, A. *Acta Cryst.* **12** (1959) 1053.

8. Pauling, L. *The Nature of the Chemical Bond*, Cornell University Press, Ithaca 1960, p. 229.
9. Jones, P. E., Ansell, G. B. and Katz, L. *Chem. Commun.* **1968** 78.
10. Schousboe-Jensen, H. V. F. and Hazell, R. G. *Acta Chem. Scand. In press.*
11. Bacon, G. E. and Pease, R. S. *Proc. Roy. Soc.* **A 230** (1955) 359.

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