ACTA CHEMICA SCANDINAVICA 26 (1972) 1023–1044

Structural Studies on the Rare Earth Carboxylates

12. The Crystal and Molecular Structure of Triclinic Trisodium Tris-(pyridine-2,6-dicarboxylato)neodymate(III) 15-Hydrate

JÖRGEN ALBERTSSON

Inorganic Chemistry 1, Chemical Center, University of Lund, P.O. Box 740, S-220 07 Lund 7, Sweden

The crystal and molecular structure of $Na_3[Nd(C_7H_3NO_4)_3].15H_2O$ has been determined from three-dimensional X-ray intensity data collected with the Weissenberg multi-film technique. The crystals are triclinic, space group $P\bar{1}$, with two formula units in a cell with the dimensions $a = 10.535(15)$ Å, $b = 11.019(12)$ Å, $c = 17.948(23)$ Å, $\alpha = 107.61(5)°$, $\beta = 90.76(7)°$, and $\gamma = 110.45(11)°$. The elements Ce – Dy form isomorphous compounds. In the mononuclear tris(pyridine-2,6-dicarboxylato) complexes the lanthanoid ions are surrounded by six carboxylate oxygen atoms and three nitrogen atoms which form a distorted tri-capped trigonal prism. The Nd – O and Nd – N bond distances are in the ranges 2.37 – 2.61 Å and 2.57 – 2.59 Å, respectively. The structure is held together by a three-dimensional network of sodium-carboxylate oxygen bonds. Five of the fifteen water molecules have not been located but are assumed to be occluded in the fairly large cavities in the structure. The structure is compared with the previously investigated structures of sodium salts of the mononuclear tris(pyridine-2,6-dicarboxylato)- and tris(oxydiacetato)-lanthanoidate complexes.

The tridentate ligand pyridine-2,6-dicarboxylate (or dipicolinate) forms mononuclear complexes of the composition $[ML_3]^{3-}$ with the trivalent lanthanoid ions. The sodium salts of these complexes with the elements Ce – Dy have almost identical triclinic structures, but hexagonal, monoclinic, and orthorhombic phases have been prepared for the elements Ho – Lu. The crystal structures of the three ytterbium compounds have been published earlier.[1–3]

This paper is a report of the crystal and molecular structure of the triclinic neodymium compound trisodium tris(dipicolinato)neodymate(III) 15-hydrate, $Na_3[Nd(C_7H_3NO_4)_3].15H_2O$, here referred to as NDP. The variation of the unit cell dimensions in the isomorphous series of Ce – Dy compounds is also studied.

The tridentate ligand oxydiacetate is very similar to the dipicolinate ion. Like the latter ion, oxydiacetate forms mononuclear tris-complexes with the trivalent lanthanoid ions. The present series of investigations includes two reports dealing with the isostructural compounds $Na_3[M(C_4H_4O_5)_3].2NaClO_4.6H_2O$, M = Ce – Lu.[4,5] In this paper the different structures containing the tris(dipicolinato)- and tris(oxydiacetato)lanthanoidate complexes are compared.

EXPERIMENTAL

The triclinic lanthanoid dipicolinates $Na_3[M(C_7H_3NO_4)_3].15H_2O$, M = Ce – Dy, were prepared by mixing water solutions of the lanthanoid nitrates or perchlorates and disodium dipicolinate in the molar ratio 1 : 3. The pH of the resulting solutions had values near 7. Slow evaporation at room temperature gave prismatic crystals, which were stored in the mother liquor to prevent efflorescing. NDP was analysed for Nd, Na, N, C, H, and H_2O as described before.[1] The relative amounts found are compared with those calculated for $Na_3[Nd(C_7H_3NO_4)_3].15H_2O$, F.W. 978.8.

	Nd	Na	N	C	H	H_2O
Found	14.7	7.3	4.4	26.9	4.1	28.8 (%)
Calc.	14.7	7.3	4.3	25.8	4.0	27.6 (%)

In the structure determination only 10 H_2O per Nd were located.

The compounds prepared for the lanthanoids Ce – Dy gave the same powder pattern as NDP. The preparation method described for the orthorhombic ytterbium dipicolinate, denoted ORTYBDIPIC, using lanthanoid hydroxide, dipicolinic acid, and sodium hydroxide resulted in the NDP phase if M = Ce – Dy.

In the batch prepared using dysprosium perchlorate a crystal was found by chance to be an intergrowth in the *ac* plane of the triclinic NDP phase and the hexagonal HEXYBDIPIC phase.[3] This latter phase is the stable one in the region Ho – Lu if perchlorate ions are present in the mother liquor. Lines from the hexagonal phase could not be detected in the powder photographs of the dysprosium compound.

Powder photographs were taken as described for ORTYBDIPIC in a Guinier-Hägg focusing camera using Cu$K\alpha$-radiation (a = 1.54178 Å) for the cerium (CDP), neodymium (NDP), samarium (SDP), gadolinium (GDP), and dysprosium (DDP) compounds. These films were used for determination of unit cell dimensions.

A freshly prepared single crystal of NDP was mounted along the a axis in a capillary together with mother liquor. It had the approximate dimensions $0.25 \times 0.15 \times 0.10$ mm^3 and was elongated in the a direction. The intensity data of $0kl - 5kl$ could be recorded before the crystal disintegrated. Another single crystal of the approximate dimensions $0.10 \times 0.10 \times 0.15$ mm^3 elongated in the c direction could be used to record the intensity data of layers $hk0 - hk6$ before it disintegrated. The nonintegrated Weissenberg multifilm technique was used with Ni-filtered Cu-radiation. The intensities were measured visually by comparison with a calibrated scale. Since most reflexions with $l \neq 2n$ were absent or very weak it was not possible to index and thus to use the few reflexions in the layers $kh1$, $hk3$, and $hk5$. 2366 intensities were used in the refinement of the structure.

The intensity data were corrected for Lorentz, polarization, and spot shape effects. The linear absorption coefficient, μ, is 116 cm^{-1}. No absorption corrections were applied.

UNIT CELL AND SPACE GROUP

NDP, and thus the isomorphous Ce – Dy dipicolinates, crystallize in the Laue class $\bar{1}$. The possible space groups are $P1$ (No. 1) and $P\bar{1}$ (No. 2).[6]

The unit cell dimensions of CDP, NDP, SDP, GDP, and DDP were obtained as described before.[2] The observed powder patterns are given in Table 1.

Table 1. X-Ray powder data: observed and calculated values of $10^5 \times \sin^2 \theta$ for the compounds $Na_3[M(C_7H_3NO_4)_3].15H_2O$, M = Ce, Nd, Sm, Gd, and Dy. The observed powder intensities of the neodynium compound are also given.

h k l	CDP		NDP		SDP		GDP		DDP		I_{obs}
	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc	NDP
0 -1 1	615	591	614	596	623	597	627	602	631	603	vvs
0 1 0		618		625		625		630		630	
0 0 2	828	822	833	827	841	828	852	834	852	839	m
1 -1 1	865	861	868	866	876	871	885	872	887	876	s
1 0 1	928	940	934	947	942	951	945	952	945	954	m
-1 0 2	1263	1270	1269	1276	1284	1280	1288	1287	1301	1293	w
1 -1 2	1349	1340	1363	1348	1373	1394	1379	1358	1384	1366	m
1 0 2	1628	1652	1659	1665	1669	1670	1676	1674	1683	1680	m
1 1 0	1700	1722	1704	1738	1718	1742	1733	1750	1742	1749	w
0 -1 3	1730	1767	1742	1779	1752	1783	1762	1797	1767	1808	m
1 -2 1	2035	2018	2058	2034	2068	2040	2083	2052	2104	2060	m
-2 1 0	2249	2246	2262	2257	2281	2271	2285	2269	2288	2278	s
1 1 1		2256		2277		2282		2292		2292	
1 -2 2	2288	2264	2297	2281	2332	2288	2343	2302	2341	2312	m
0 2 0	2453	2474	2490	2499	2504	2501	2516	2520	2513	2521	m
-2 1 1	2482	2494	-	2505	2520	2519	2542	2521	2549	2531	
2 0 0	-	-	-	-	-	2586		2586		2590	
1 -2 3	2897	2920	2917	2940	2939	2950	2941	2968	2952	2984	m
0 -2 3		2923		2946		2952		2978		2989	
-2 0 2	3005	2996	3023	3010	3046	3025	3082	3034	3068	3042	w
1 1 2	3211	3201	3219	3231	3243	3236	3263	3250	3282	3254	vvw
0 0 4	3280	3286	3326	3309	-	-	3363	3336	3378	3355	vw
2 -2 2	3471	3444	3487	3464	3511	3482	3500	3488	3540	3506	vs
1 -1 4	3529	3529	3535	3553	-	3563	3579	3580	3584	3602	m
-1 0 4		3543		3563	-	3570		3596		3616	
-2 2 1	3665	3653	3676	3675	3670	3689	3705	3700	3728	3716	vvw
-1 -2 1		3685	-	3720	3706	3727	-	3753		3751	
-1 -1 4	-	3693	-	3715		3724	-	3754	-	3771	
-1 2 2	3776	3747		3777		3782	-	3806		3818	
2 0 2		3761	3794	3788	3828	3805	-	3807	3855	3815	s
-2 0 3	-	3832	-	3850		3865	3852	3883		3897	
-2 -1 1	-	3885	3939	3913	3925	3929	3937	3942	3945	3942	vw
0 -2 4	3890	3894		3922		3930		3965		3984	
2 -1 3	3963	3969	3998	3994	3981	4013	4010	4017	4021	4035	vvw
1 -2 4		3987		4014		4026		4051		4076	
1 -3 1	4417	4413	4443	4452	4461	4461	4488	4493	4504	4503	w
1 -3 2	4464	4425		4463		4473		4506		4519	
-2 2 2	-	-	4513	4572	4552	4586	-	4605	4604	4625	w
2 1 1	4754	4734	4769	4774	4791	4790	4798	4802	4793	4802	w
0 1 4	4886	4838	-	4876	-	-	-	-	-	-	
1 -3 3		4848	4930	4887	-	4899	4989	4936	5004	4955	w
-3 1 0	4992	4978		5002	5049	5034		5029		5044	
0 -3 2		4988	5025	5035		5042	5093	5086	5095	5092	vvw
0 -3 1	-	5071		5122		5127		5170		5173	
-2 0 4	5089	5078	5113	5103	5146	5120	5157	5149	5176	5171	vw
-1 -2 4		5080		5116		5128		5172		5186	
-1 -1 5	5214	5213	5233	5243	5288	5255		5297	5370	5325	vs
3 -1 1		5237		5264		5298		5291		5307	
1 -1 5		5240		5275		5289		5317		5330	
0 -2 5	5270	5277	5297	5312	5331	5323	5318	5369		5398	s
-1 0 5		5296		5327		5337		5376	-	5406	
-3 2 0	5428	5439	5449	5466	5476	5498	5499	5498	5520	5521	w
0 3 0	5538	5566	5659	5622	5622	5627	5659	5671	5665	5673	vw
-1 3 1	5627	5621		5672	5689	5679		5718	-	5730	
2 -3 3		5659	-	5697		5721	5720	5745	-	5774	
-3 0 1	-	5671	-	5702		5734		5738	-	5746	
1 -3 4		5682	-	5725	-	5740		5783		5810	
-2 1 4		5701	-	5725	-	5747	5810	5777	5806	5806	
0 2 3		5721	5763	5774	5786	5778		5815		5828	m
3 0 0	5758	5753		5787		5819		5819		5827	
2 1 2		5775	5819	5824	5836	5842	5871	5857		5861	m
3 -2 2	5912	5902	5945	5933	5969	5970	5992	5967	5903	5994	s
3 -1 2		5906		5941		5976		5970		5989	
-3 0 2		6001	6032	6031	6070	6063		6073	6013	6085	w
1 2 2	6028	5987		6046		6053	6108	6087		6089	
-2 3 1		6049	-	6094	-	6110		6140	6136	6162	
3 0 1	6212	6245	6263	6285	-	-	6292	6318	6316	6326	vvw
1 0 5		6252		6299	-	-		6343	-	6373	
-2 -2 2	6426	6395	6474	6446	6479	6465	6519	6503	6516	6501	vvw
0 3 1	-	6471	-	6536	-	6540		6589	-	6592	
2 -3 4	6582	6588	6632	6632	6662	6659		6688		6725	m
2 0 4		6608		6658		6680	6717	6696	6751	6718	
0 -1 6	-	6614	-	6655	-	6668		6718		6760	
-3 0 3		6741	-	6773	6804	6806		6826		6844	
3 -2 3	6763	6749	-	6788		6827	6845	6827	6851	6859	
-2 0 5		6736	-	6770	6804	6789		6832		6865	
-2 -2 3		6764	6817	6815		6834		6879		6883	vw
-1 -3 2	-	6829	-	-	6903	6905	6931	6960	6951	6959	
-1 3 2	-	6841	-	-		6909		6956		6972	
-1 2 4	6961	6953	6998	7007	7024	7014	7055	7060	7079	7088	w
-1 -3 1	-	7008	-	-	-	7087		7141	-	7137	
-1 -3 3	-	7061	-	-	7147	7136	7167	7197		7201	
0 -2 6	7061	7070	-	-		7130		7190		7231	
-3 3 0	-	7138		7179	7224	7213		7228	7218	7259	
3 0 2	-	7148	7205	7197		7233	7241	7234		7246	w
2 -1 5	7187	7171		7220	-	7246		7267		7255	
-2 3 2		7174	-	7227	-	7243		7281		-	
0 0 6	7416	7394	7442	7445	7474	7456	7499	7506		-	m

Table 1. Continued.

h k l	CDP obs	CDP calc	NDP obs	NDP calc	SDP obs	SDP calc	GDP obs	GDP calc	DDP obs	DDP calc	I_{obs} NDP
1 2 3	7588	7576	7637	7649		7657		7699		7708	vvw
-2 1 5		7591		7633	7673	7651	7724	7697	7748	7736	
1 3 0	-	7598	-	7675		7684		7738		7733	
0 2 4		7626	-	7693		7698		7748		7769	
0 3 2		7786		7864		7868		7923	7846	7931	
3 1 0		7765		7822	7899	7855		-		7870	
1 -4 2	7817	7823	7869	7894		7908	7942	7970	7958	7987	m
-3 2 3		7827		7866		7898		7923		7958	
2 -4 1	8189	8199	8243	8266	8276	8287	8335	8335	8347	8362	m
2 -4 3		-	-	-	8450	8450	8498	8499		8534	
-1 3 3		8472	-	8546		8553		8611		8633	
3 0 3	8471	8462	8503	8523	8541	8561	8567	8566	8588	8584	w
3 -1 4		8478		8534		8575		8580		8613	
3 1 1		8490	-	8556		8591		8605		8607	
-2 3 3	-	-	-	8774	-	8790	8852	8840	8886	8872	
-2 4 0	-	8735		8807	-	8827		8879		8906	
0 -3 6	8789	8762	8838	8824	8856	8843	-	-		-	m
-3 3 2		8785		8837		8870		8900		8938	
0 -4 2	-	8850	8952	8937	8985	8948		9024		9031	m
2 -2 6	8896	6916		8974		9006	9023	9042	9077	9097	
-4 1 0	-	-	9053	9033		9090		9081	-	9104	w
-1 1 6	-	-		9073		9085	-	9147		-	
2 2 2	-	-		9110	9136	9129	-	9168	-	-	
2 -4 4	-	9055	-	9122		9153	9214	9206	-	-	
2 1 4	9118	9088	9179	9166		9188		9219	-	-	m
4 -1 1	-	-	9415	9393		-		-	-	-	vw
0 -4 4	-	-	9558	9533		-		9635	-	-	vvw
-4 1 2	-	-		9554		9609		9614	-	9643	
1 2 4	-	9576		9665	9672	9676		9728	-	-	
4 -2 2	9618	-	9672	9689		9750	9740	9739	-	-	w
3 -4 2		9603		9667		9708		9741	-	-	
-2 2 5		9683		9746		9764	9822	9822	-	9868	
-2 4 1		-	9758	9762		9781		9840	-	9868	w
3 -2 5	9700	-		9737	9798	9783		-	-	-	
2 -3 6		-		9743		9778		-	9898	9886	
-1 4 1		9718		9811		9820	9907	9891		9907	
-3 1 5	9812	9845	9859	9892		-	-	9973		-	w

The unit cell dimensions with estimated standard deviations are given in Table 2. For NDP the density 1.8 g/cm³ was estimated by flotation. With two formula units in the cell the calculated density is 1.79 g/cm³.

DETERMINATION AND REFINEMENT OF THE STRUCTURE

Assuming the space group to be $P\bar{1}$ the neodymium atom were found in the position 2(*i*) with coordinates (0,0,0.25) from the vector map obtained in a three-dimensional Patterson synthesis. This location of the heavy atom is in accordance with the special condition *hkl*: $l = 2n$, limiting the strong reflexions. The preliminary neodymium parameters and the inter-layer scale factors were improved by least-squares calculations. The non-hydrogen atoms of the ligands, the sodium ions and ten water oxygen atoms could then be located in the electron density maps obtained in a series of three-dimensional

Table 2. The unit cell parameters and volumes with estimated standard deviations of the triclinic compounds $Na_3[M(C_7H_3NO_4)_3].15H_2O$, M = Ce, Nd, Sm, Gd, and Dy.

Compound	*a*/Å	*b*/Å	*c*/Å	*α*/°	*β*/°	*γ*/°	*V*/Å³
CDP	10.377(14)	11.067(14)	18.003(22)	107.60(5)	90.72(7)	110.38(12)	1831
NDP	10.353(15)	11.019(12)	17.948(23)	107.61(5)	90.76(7)	110.45(11)	1812
SDP	10.319(16)	11.007(12)	17.930(43)	107.57(7)	90.79(7)	110.39(10)	1804
GDP	10.324(13)	10.967(13)	17.860(25)	107.53(7)	90.70(7)	110.49(11)	1791
DDP	10.308(20)	10.953(15)	17.803(38)	107.49(9)	90.74(9)	110.35(15)	1782

difference syntheses. To locate the tris(dipicolinato) complex a model of the corresponding ytterbium complex found in ORTYBDIPIC was fitted to the peaks of the maps.

The coordinates and isotropic temperature factors of the 51 atoms found were improved together with the scale factors in a series of least-squares refinements. In the quantity minimized, $\sum w(|F_o| - |F_c|)^2$, the weights w were calculated according to the expression $w = 1/(a + |F_o| + c|F_o|^2 + d|F_o|^3)$. An analysis of the weighting scheme suggested suitable values for a, c, and d. In the last cycle of refinement the values $a = 50$, $c = 0.01$, and $d = 0.0005$ were used. Reflexions not obeying the condition $0.80 \leq |F_o|/|F_c| \leq 1.25$ were given zero weight. After a number of cycles the discrepancy indices $R = \sum||F_o| - |F_c||/\sum|F_o|$ and $wR = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{\frac{1}{2}}$ had converged to 0.126 and 0.097, respectively. 353 of the 2366 reflexions were given zero weight in the last cycle. The shifts in all parameters were less than 1/3 of their estimated standard deviations in this cycle. As the refinement of the structure was successful using space group $P\bar{1}$ the other possible choice, $P1$, was not considered, due to the very large number of parameters then necessary.

Table 3. Positional parameters and isotropic temperature factors in NDP with estimated standard deviations. The space group is $P\bar{1}$ (No. 2).[6]

Atom	x	y	z	B/Å^2
Nd	−0.0085(02)	0.0020(01)	0.2496(01)	0.8(0.1)
N(1)	−0.2614(27)	−0.0067(16)	0.2452(12)	1.5(0.4)
C(1)	−0.1509(27)	0.2104(19)	0.2260(12)	1.2(0.4)
C(2)	−0.2902(26)	0.0874(17)	0.2266(10)	1.4(0.3)
C(3)	−0.4326(36)	0.0805(28)	0.2249(16)	3.8(0.5)
C(4)	−0.5269(28)	−0.0121(27)	0.2437(15)	2.2(0.5)
C(5)	−0.4886(36)	−0.1165(27)	0.2522(16)	3.8(0.6)
C(6)	−0.3582(26)	−0.1138(18)	0.2502(10)	1.4(0.5)
C(7)	−0.2990(23)	−0.2233(15)	0.2613(09)	0.1(0.3)
O(1)	−0.0611(18)	0.1759(13)	0.2162(07)	1.6(0.2)
O(2)	−0.2041(19)	0.2931(16)	0.2086(10)	2.5(0.3)
O(3)	−0.1717(18)	−0.1920(13)	0.2723(08)	1.8(0.2)
O(4)	−0.3878(18)	−0.3313(15)	0.2622(09)	2.2(0.3)
N(2)	0.1079(23)	−0.0039(16)	0.3751(09)	2.1(0.3)
C(8)	0.1767(24)	−0.1877(18)	0.2922(11)	0.9(0.3)
C(9)	0.1736(22)	−0.0851(15)	0.3742(09)	0.6(0.2)
C(10)	0.2461(34)	−0.0892(27)	0.4367(15)	3.8(0.5)
C(11)	0.2566(31)	0.0162(26)	0.5091(15)	3.0(0.5)
C(12)	0.2032(30)	0.1185(23)	0.5221(13)	2.9(0.4)
C(13)	0.1211(25)	0.1011(18)	0.4498(10)	1.7(0.3)
C(14)	0.0466(23)	0.1963(17)	0.4465(10)	0.8(0.3)
O(5)	0.1115(15)	−0.1697(11)	0.2316(06)	1.1(0.2)
O(6)	0.1989(18)	−0.2962(15)	0.2741(09)	2.2(0.3)
O(7)	−0.0191(17)	0.1727(13)	0.3847(07)	1.7(0.2)
O(8)	0.0536(19)	0.2999(16)	0.5084(09)	2.4(0.3)
N(3)	0.1379(20)	0.0148(14)	0.1345(08)	1.5(0.2)
C(5)	0.3007(37)	0.2166(30)	0.2260(17)	3.8(0.7)
C(16)	0.2502(20)	0.1196(14)	0.1464(08)	0.5(0.2)

Table 3. Continued.

C(17)	0.3372(30)	0.1301(23)	0.0901(13)	2.7(0.4)
C(18)	0.3003(35)	0.0192(28)	0.0124(16)	3.4(0.5)
C(19)	0.1609(32)	−0.0929(26)	−0.0022(14)	3.2(0.4)
C(20)	0.0983(28)	−0.0835(21)	0.0644(12)	2.2(0.3)
C(21)	−0.0279(30)	−0.2054(26)	0.0569(15)	2.4(0.5)
O(9)	0.2151(18)	0.1986(14)	0.2837(08)	2.0(0.2)
O(10)	0.4096(21)	0.3266(17)	0.2537(10)	3.2(0.3)
O(11)	−0.1004(19)	−0.1709(15)	0.1141(08)	2.4(0.3)
O(12)	−0.0812(20)	−0.3119(18)	−0.0060(11)	3.4(0.4)
Na(1)	0	1/2	0	1.9(0.2)
Na(2)	0.0051(13)	0.4962(11)	0.2449(06)	2.5(0.1)
Na(3)	0	1/2	1/2	4.0(0.4)
Na(4)	−0.3526(12)	0.4609(09)	0.2591(05)	3.1(0.2)
O(13)	0.2115(22)	0.6562(19)	0.0919(11)	4.3(0.4)
O(14)	−0.0913(24)	0.5361(21)	0.1293(12)	4.1(0.5)
O(15)	0.1258(22)	0.3611(19)	0.1636(11)	3.8(0.4)
O(16)	−0.1187(17)	0.6241(14)	0.3210(08)	1.8(0.3)
O(17)	0.0688(19)	0.4484(16)	0.3647(09)	2.0(0.3)
O(18)	−0.2130(25)	0.3197(21)	0.4261(12)	4.9(0.4)
O(19)	−0.3222(33)	0.4380(31)	0.1358(17)	8.5(0.7)
O(20)	−0.3679(25)	0.4813(22)	0.3909(12)	5.1(0.4)
O(21)	−0.3546(35)	0.6329(33)	0.0512(18)	8.5(0.7)
O(22)	0.3806(23)	0.3391(19)	0.4364(11)	3.8(0.4)

Table 3 gives the final parameters with estimated standard deviations. A three-dimensional difference synthesis based upon these parameters showed only small spurious peaks above a slowly varying background. The highest peak, about 2 e/Å^3, is situated at the neodymium position.

The atomic scattering factors used in the calculations were taken from the *International Tables* [7] (Na^+, O, N, and C) and from Cromer *et al.*[8] (Nd). Observed and calculated structure factors are compared in Table 4.

Selected interatomic distances and angles in the structure are given in Table 5. The standard deviations are calculated from the estimated standard deviations of the atomic coordinates and the unit cell dimensions.

The computations were performed on the computers CDC 3600 in Uppsala and UNIVAC 1108 in Lund using the programs PIRUM,[9] CELSIUS, DRF, LALS, DISTAN, PLANE, and ORTEP.[10]

DESCRIPTION OF THE STRUCTURE

The superscipts (i) – (vii) are used to indicate the following symmetry-related sites in the structure

(i) $1+x,y,z$	(ii) $x,1+y,z$	(iii) $x-1,y,z$
(iv) $x,y-1,z$	(v) $\bar{x},\bar{y},\bar{z}$	(vi) $\bar{x},\bar{y},1-z$
(vii) $\bar{x},1-y,1-z$		

where x,y,z are the coordinates of the "crystal-chemical" unit given in Table 3.

The mononuclear tris(dipicolinato)neodymate complex in NDP is located in layers around the plane $y=0$ as illustrated in Fig. 1. The ligand

Table 4. Observed and calculated structure factors of NDP. The 353 reflexions not obeying the condition $0.80 \leq |F_o|/|F_c| \leq 1.25$ are denoted by asterisks.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
0	2	-22	47	45	0	4	-10	21	17	0	8	-2	33	42	1	-9	-10	44	36
0	3	-22	45	47	0	5	-10	101	71 *	0	9	-2	36	60	1	-8	-10	39	59
0	4	-22	44	36	0	6	-10	60	55	0	10	-2	82	59	1	-6	-10	67	76
0	5	-22	36	39	0	7	-10	61	58	0	-7	-1	39	21 *	1	-5	-10	43	54
0	6	-22	43	50	0	8	-10	74	83	0	5	-1	34	43 *	1	-2	-10	91	88
0	7	-22	49	40	0	9	-10	46	114 *	0	-12	0	45	40	1	-1	-10	97	92
0	8	-22	37	46	0	10	-10	54	55	0	-11	0	67	66	1	0	-10	75	66
0	-2	-20	33	54 *	0	11	-10	39	46	0	-10	0	100	90	1	1	-10	56	56
0	0	-20	40	40	0	12	-10	53	40	0	-9	0	61	77	1	2	-10	99	87
0	1	-20	40	35 *	0	13	-10	44	37	0	-8	0	53	51	1	3	-10	118	87 *
0	2	-20	40	45	0	-2	-9	32	24 *	0	-7	0	42	45	1	4	-10	81	82
0	3	-20	39	40 *	0	-1	-9	30	30 *	0	-6	0	104	104	1	5	-10	91	82
0	4	-20	49	47	0	0	-9	19	10 *	0	-5	0	61	50	1	6	-10	44	45
0	5	-20	33	55	0	1	-9	46	39	0	-4	0	126	99 *	1	7	-10	82	57
0	6	-20	34	37	0	2	-9	57	51	0	2	0	140	119	1	8	-10	56	67
0	7	-20	34	42	0	3	-9	70	64	0	3	0	34	64	1	9	-10	71	56 *
0	8	-20	56	53	0	4	-9	77	90	1	2	-22	44	47	1	10	-10	41	33
0	9	-20	42	47	0	5	-9	56	56	1	3	-22	43	43	1	11	-10	43	42
0	10	-20	40	36	0	6	-9	104	116	1	5	-22	40	40	1	12	-10	34	33
0	-3	-18	37	52	0	-2	-8	173	157	1	6	-22	45	47	1	-1	-9	54	47
0	-2	-18	66	61	0	-1	-8	69	92	1	7	-22	34	42 *	1	6	-9	41	40
0	-1	-18	66	47 *	0	0	-8	230	219	1	-1	-20	41	37	1	-10	-8	67	62
0	0	-18	49	34 *	0	1	-8	31	73 *	1	0	-20	31	27	1	-8	-8	59	54
0	1	-18	39	42	0	2	-8	140	144	1	1	-20	30	34	1	-7	-8	79	76
0	2	-18	76	76	0	4	-8	244	159 *	1	2	-20	30	45	1	-6	-8	71	76
0	3	-18	70	67	0	5	-8	39	34	1	3	-20	39	43	1	-4	-8	89	89
0	4	-18	37	41	0	6	-8	111	124	1	4	-20	42	40	1	-3	-8	84	96 *
0	5	-18	37	36	0	7	-8	104	97	1	5	-20	40	39	1	-2	-8	103	102
0	6	-18	33	35	0	8	-8	104	81 *	1	6	-20	36	39	1	-1	-8	98	94
0	7	-18	77	79	0	9	-8	106	93	1	7	-20	33	40	1	0	-8	134	120
0	8	-18	79	77	0	10	-8	79	70	1	8	-20	36	36	1	1	-8	137	87 *
0	9	-18	64	66	0	11	-8	33	44 *	1	9	-20	38	33	1	2	-8	112	80 *
0	-5	-16	43	41	0	12	-8	59	56	1	-3	-18	33	63	1	3	-8	133	104 *
0	-4	-16	59	59	0	5	-7	59	53 *	1	-2	-18	53	76	1	4	-8	133	120 *
0	-3	-16	54	51	0	-10	-6	41	48	1	-1	-18	43	44	1	5	-8	94	89 *
0	-2	-16	66	61	0	-9	-6	56	54	1	1	-18	39	45	1	6	-8	108	110 *
0	-1	-16	36	46	0	-8	-6	54	48	1	2	-18	71	74	1	7	-8	56	59
0	0	-16	92	87	0	-7	-6	59	60	1	3	-18	60	50	1	8	-8	58	64
0	1	-16	67	58	0	-6	-6	72	73	1	4	-18	53	54	1	9	-8	57	50
0	2	-16	122	111	0	-5	-6	60	70	1	7	-18	60	63	1	10	-8	60	55
0	3	-16	76	70	0	-4	-6	56	63	1	8	-18	61	64	1	11	-8	49	37 *
0	4	-16	65	68	0	-3	-6	40	36 *	1	9	-18	40	45	1	12	-8	44	44
0	5	-16	51	56	0	-2	-6	89	80 *	1	-5	-16	37	39	1	-6	-7	48	48 *
0	6	-16	57	56	0	-1	-6	155	154	1	-4	-16	59	54	1	-1	-7	46	40
0	7	-16	65	63	0	0	-6	227	204 *	1	-3	-16	57	56	1	0	-7	58	57 *
0	8	-16	77	80	0	1	-6	102	75 *	1	-2	-16	59	49	1	-10	-6	60	50
0	9	-16	61	56	0	4	-6	90	55 *	1	-1	-16	54	59	1	-9	-6	59	54
0	10	-16	48	51	0	5	-6	102	77 *	1	0	-16	74	80	1	-8	-6	49	46
0	11	-16	43	51	0	6	-6	140	109	1	1	-16	74	75	1	-7	-6	53	62
0	12	-16	64	61	0	7	-6	84	77	1	2	-16	114	112	1	-6	-6	64	64
0	-6	-14	54	48	0	8	-6	134	122	1	3	-16	80	80	1	-5	-6	79	74
0	-5	-14	59	49	0	9	-6	69	69	1	4	-16	63	61	1	-4	-6	43	43
0	-4	-14	60	44 *	0	10	-6	43	36	1	5	-16	74	56 *	1	-3	-6	35	36
0	-3	-14	57	52	0	11	-6	33	34	1	7	-16	53	49	1	-2	-6	43	40
0	-2	-14	60	76	0	12	-6	54	71 *	1	8	-16	54	54	1	-1	-6	210	148 *
0	-1	-14	71	81	0	-5	-5	60	27	1	9	-16	49	31	1	0	-6	98	98
0	0	-14	48	48	0	-2	-5	48	33 *	1	10	-16	55	55	1	1	-6	134	127
0	2	-14	65	76	0	-1	-5	48	37 *	1	11	-16	33	34	1	2	-6	71	49
0	3	-14	59	67	0	0	-5	48	38 *	1	2	-15	23	27	1	3	-6	142	86 *
0	4	-14	65	59	0	1	-5	48	37 *	1	-6	-14	34	37	1	4	-6	220	144 *
0	5	-14	85	75	0	-11	-4	41	40	1	-5	-14	50	50	1	5	-6	134	119 *
0	6	-14	57	62	0	-10	-4	69	64	1	-4	-14	46	47	1	6	-6	72	72
0	7	-14	69	56	0	-9	-4	43	42	1	-3	-14	59	61	1	7	-6	73	49 *
0	8	-14	106	94	0	-8	-4	46	40	1	-2	-14	58	63	1	8	-6	73	45
0	9	-14	73	60	0	-7	-4	46	51	1	-1	-14	76	83	1	9	-6	46	39
0	11	-14	47	30	0	-6	-4	114	114	1	1	-14	57	46	1	10	-6	66	63
0	12	-14	39	43	0	-5	-4	41	22	1	2	-14	69	64	1	11	-6	66	56
0	-1	-13	37	44	0	-4	-4	143	130	1	3	-14	69	73	1	-2	-5	119	90 *
0	-7	-12	41	36	0	-3	-4	76	82	1	4	-14	69	64	1	-1	-5	54	61
0	-6	-12	66	60	0	-2	-4	29	31	1	5	-14	69	74	1	0	-5	33	37 *
0	-5	-12	26	31	0	-1	-4	260	242	1	6	-14	59	46	1	1	-5	38	23 *
0	-4	-12	60	59	0	0	-4	244	254	1	8	-14	53	53	1	2	-5	37	33
0	-3	-12	68	70	0	2	-4	81	90	1	7	-14	59	66 *	1	4	-5	46	20
0	-2	-12	99	97	0	3	-4	135	98 *	1	9	-14	53	46 *	1	-11	-4	46	39
0	-1	-12	46	46	0	4	-4	169	119 *	1	10	-14	39	49	1	-10	-4	46	74
0	0	-12	94	89	0	5	-4	94	76	1	11	-14	28	33	1	-8	-4	57	41
0	1	-12	39	37	0	6	-4	71	85	1	12	-14	44	41	1	-7	-4	57	66
0	2	-12	73	70	0	7	-4	33	36	1	6	-13	35	38	1	-6	-4	103	110
0	3	-12	131	112 *	0	8	-4	74	76	1	-3	-12	78	57 *	1	-4	-4	146	130
0	4	-12	74	70	0	9	-4	86	75	1	-7	-12	34	37	1	-3	-4	51	29 *
0	5	-12	24	27	0	10	-4	149	95 *	1	-6	-12	53	46	1	-2	-4	134	73 *
0	6	-12	75	80	0	-5	-3	47	42	1	-4	-12	64	69	1	-1	-4	134	120
0	7	-12	94	96	0	-3	-3	17	14	1	-3	-12	65	76	1	0	-4	134	97
0	8	-12	93	92	0	-1	-3	29	31	1	-2	-12	67	80	1	3	-4	74	67
0	9	-12	78	70	0	0	-3	63	25	1	-1	-12	50	40 *	1	4	-4	166	141
0	10	-12	77	49 *	0	6	-3	18	17	1	0	-12	38	66	1	5	-4	51	44
0	11	-12	43	26	0	-11	-2	60	60	1	1	-12	51	27	1	6	-4	66	55
0	12	-12	55	47	0	-10	-2	67	59	1	2	-12	91	89	1	7	-4	63	60
0	13	-12	40	38	0	-9	-2	67	59	1	3	-12	111	106	1	8	-4	63	64
0	-2	-11	28	29	0	-8	-2	65	56	1	4	-12	61	62	1	9	-4	34	37
0	-8	-10	43	44	0	-7	-2	57	34	1	5	-12	51	45	1	10	-4	74	66
0	-7	-10	55	43 *	0	-6	-2	103	95	1	6	-12	56	54	1	-2	-3	42	28 *
0	-6	-10	55	52	0	-5	-2	104	115	1	7	-12	33	36	1	-1	-3	87	64
0	-5	-10	59	63	0	-4	-2	132	140	1	8	-12	61	57	1	0	-3	89	69 *
0	-4	-10	76	90	0	-3	-2	77	61 *	1	10	-12	56	54	1	1	-3	63	59 *
0	-3	-10	55	54	0	-2	-2	103	92	1	11	-12	29	32	1	2	-3	101	72 *
0	-2	-10	92	103	0	-1	-2	110	103	1	12	-12	39	41	1	3	-3	25	41 *
0	-1	-10	90	107	0	3	-2	31	30	1	-4	-11	43	27 *	1	6	-3	27	36 *
0	0	-10	125	109	0	4	-2	72	76	1	-9	-10	44	36 *	1	-12	-2	43	42
0	1	-10	62	44 *	0	5	-2	142	110	1	-8	-10	43	36	1	-11	-2	36	58
0	2	-10	131	90 *	0	6	-2	104	91	1	-7	-10	45	39	1	-10	-2	72	59
0	3	-10	122	101	0	7	-2	128	100 *						1	-9	-2	56	40 *

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$	
1	-9	-2	36	34		1	-9	7	36	34		2	3	-22	43	44		2	5	-10	68	59	
1	-7	-2	46	44		1	-5	7	26	27		2	4	-22	41	37		2	6	-10	101	73	*
1	-6	-2	110	111		1	-4	7	45	39		2	5	-22	44	43		2	7	-10	50	47	
1	-5	-2	141	111	*	1	-3	7	40	34	*	2	6	-22	47	45		2	8	-10	53	51	
1	-4	-2	145	134		1	-1	7	47	43		2	-1	-20	35	31		2	9	-10	64	56	
1	-3	-2	42	37		1	0	7	49	46		2	0	-20	56	53		2	10	-10	65	58	
1	-2	-2	50	50		1	1	7	41	27	*	2	1	-20	25	32	*	2	11	-10	43	49	
1	-1	-2	117	62	*	1	2	7	25	20		2	2	-20	39	44		2	12	-10	30	26	
1	1	-2	100	124	*	1	-13	8	53	47		2	3	-20	41	49		2	0	-9	26	48	*
1	2	-2	104	136		1	-12	8	66	60		2	4	-20	45	50		2	3	-9	43	45	
1	3	-2	140	85	*	1	-11	8	42	38		2	6	-20	36	38		2	4	-9	27	20	*
1	4	-2	77	69		1	-10	8	78	63		2	8	-20	36	37		2	7	-9	41	19	*
1	5	-2	156	154	*	1	-9	8	43	44		2	9	-20	49	51		2	-10	-8	46	44	
1	6	-2	26	36		1	-8	8	69	79		2	-4	-18	63	51		2	-9	-8	41	44	
1	7	-2	97	93		1	-7	8	66	66		2	-3	-18	51	51		2	-8	-8	40	53	
1	8	-2	65	64		1	-6	8	131	115		2	-2	-18	36	49		2	-7	-8	67	74	
1	-2	-1	49	42	*	1	-5	8	46	30		2	-1	-18	20	31		2	-6	-8	84	76	
1	0	-1	21	22		1	-4	8	165	160		2	0	-18	36	39		2	-5	-8	51	53	
1	-12	0	60	52		1	-3	8	28	29		2	1	-18	50	53		2	-4	-8	86	83	
1	-11	0	79	73		1	-2	8	169	130	*	2	2	-18	74	76		2	-3	-8	90	89	
1	-10	0	111	102		1	-1	8	67	56		2	3	-18	60	61		2	-2	-8	31	55	
1	-9	0	49	49		1	0	8	83	94		2	4	-18	75	66		2	0	-8	150	150	
1	-8	0	51	51		1	2	8	69	74		2	5	-18	59	41		2	1	-8	21	23	
1	-7	0	57	51		1	-9	9	51	34	*	2	6	-18	48	40		2	2	-8	30	106	*
1	-6	0	131	106		1	-1	9	18	37	*	2	7	-18	37	41		2	3	-8	102	90	*
1	-5	0	87	89		1	-13	10	38	34		2	8	-18	37	39		2	4	-8	150	114	*
1	-4	0	93	106		1	-12	10	47	45		2	9	-18	43	40		2	5	-8	23	20	
1	-2	0	104	90		1	-11	10	55	34		2	10	-18	35	34		2	6	-8	108	120	*
1	-1	0	140	140		1	-10	10	41	47		2	-5	-16	46	36	*	2	7	-8	36	34	
1	0	0	110	70	*	1	-9	10	72	69		2	-4	-16	36	50		2	8	-8	70	67	
1	1	0	64	35	*	1	-8	10	73	64		2	-3	-16	41	45		2	9	-8	28	25	
1	2	0	133	134		1	-7	10	54	54		2	-2	-16	36	46		2	10	-8	84	53	
1	3	0	120	119		1	-6	10	87	87		2	-1	-16	34	35		2	11	-8	34	36	
1	4	0	91	82		1	-5	10	89	74		2	0	-16	38	37		2	12	-8	32	36	
1	7	0	60	59		1	-4	10	110	101		2	1	-16	51	51		2	-1	-7	34	55	*
1	8	0	52	50		1	-3	10	59	60		2	2	-16	62	63		2	0	-7	60	60	
1	-2	1	43	49	*	1	-2	10	61	46		2	3	-16	37	38		2	1	-7	42	27	*
1	-13	2	34	32	*	1	-1	10	114	117		2	4	-16	84	87		2	2	-7	30	30	
1	-12	2	53	38	*	1	0	11	27	27		2	5	-16	28	27		2	3	-7	32	31	
1	-11	2	32	35		1	1	10	49	59		2	6	-16	93	82		2	4	-7	17	15	
1	-10	2	47	47		1	-5	11	44	43		2	7	-16	38	37		2	7	-7	29	21	*
1	-9	2	36	31		1	-4	11	55	57		2	8	-16	54	36	*	2	-11	-6	33	32	
1	-8	2	74	69		1	-3	11	49	28	*	2	9	-16	38	39	*	2	-10	-6	40	39	
1	-7	2	58	65		1	-13	12	30	33		2	10	-16	52	56		2	-9	-6	59	56	
1	-6	2	141	124		1	-12	12	42	43		2	7	-15	31	20	*	2	-8	-6	63	63	
1	-5	2	139	134		1	-11	12	33	34		2	-7	-14	36	38		2	-7	-6	34	70	
1	-4	2	90	27	*	1	-10	12	53	50		2	-6	-14	47	49		2	-6	-6	45	57	*
1	-3	2	91	98		1	-8	12	68	71		2	-5	-14	50	45		2	-5	-6	133	123	
1	-2	2	136	136		1	-6	12	66	63		2	-4	-14	43	46		2	-4	-6	33	32	
1	-1	2	143	93	*	1	-5	12	50	50		2	-3	-14	64	66		2	-3	-6	90	91	
1	0	2	76	48	*	1	-4	12	109	106		2	-2	-14	51	51		2	-2	-6	108	122	
1	1	2	82	68		1	-3	12	61	65		2	-1	-14	71	74		2	-1	-6	36	66	*
1	2	2	45	47		1	-2	12	73	72		2	0	-14	63	64		2	0	-6	49	54	
1	3	2	85	89		1	-1	12	64	57		2	1	-14	53	55		2	1	-6	113	222	*
1	4	2	100	106	*	1	0	12	30	34		2	2	-14	57	60		2	2	-6	50	42	
1	5	2	117	115		1	-10	13	43	34	*	2	3	-14	62	65		2	3	-6	44	46	
1	6	2	75	78		1	-9	13	48	36	*	2	4	-14	72	75		2	4	-6	136	147	
1	7	2	59	65		1	-4	13	58	50		2	5	-14	60	64		2	5	-6	69	63	*
1	-11	3	47	29		1	0	13	58	52		2	6	-14	50	55		2	6	-6	90	86	
1	-10	3	36	26		1	-13	14	41	41		2	7	-14	47	45		2	7	-6	74	67	
1	-9	3	41	46		1	-12	14	37	38		2	8	-14	60	60		2	8	-6	53	60	
1	-8	3	64	54	*	1	-11	14	36	37		2	9	-14	65	61		2	9	-6	56	56	
1	-7	3	91	99		1	-10	14	38	38		2	10	-14	60	60		2	10	-6	71	69	
1	-1	3	73	68		1	-9	14	47	45		2	11	-14	26	34	*	2	11	-6	41	49	
1	0	3	82	75		1	-8	14	79	66		2	-1	-13	43	39		2	-8	-5	21	21	
1	1	3	41	45		1	-6	14	70	70		2	3	-13	24	26		2	-7	-5	25	24	
1	-13	4	36	33		1	-5	14	64	61		2	6	-13	30	26		2	-3	-5	67	73	
1	-12	4	42	37		1	-4	14	68	74		2	-8	-12	61	57		2	-2	-5	40	40	
1	-11	4	50	27	*	1	-3	14	50	61		2	-7	-12	43	42		2	-1	-5	53	46	
1	-10	4	114	96		1	-2	14	58	62		2	-6	-12	44	52		2	0	-5	53	35	*
1	-9	4	33	32		1	-1	14	58	65		2	-4	-12	69	69		2	3	-5	64	67	
1	-8	4	79	76		1	0	14	68	72		2	-3	-12	54	53		2	4	-5	58	58	
1	-7	4	53	33		1	-12	16	39	45		2	-2	-12	53	47		2	-11	-4	22	28	*
1	-6	4	107	94		1	-11	16	39	43		2	0	-12	79	82		2	-10	-4	48	43	
1	-5	4	84	71		1	-10	16	42	51		2	1	-12	52	51		2	-9	-4	40	39	
1	-4	4	134	140		1	-8	16	58	59		2	2	-12	94	79		2	-8	-4	59	56	
1	-3	4	40	33	*	1	-7	16	48	48		2	3	-12	87	69	*	2	-7	-4	22	30	*
1	-2	4	79	92	*	1	-6	16	81	63		2	4	-12	97	89		2	-6	-4	101	99	
1	-1	4	32	28		1	-5	16	40	37		2	5	-12	37	41		2	-5	-4	21	24	
1	0	4	42	61	*	1	-4	16	82	84		2	6	-12	54	51		2	-4	-4	70	83	
1	1	4	37	66	*	1	-2	16	66	71		2	7	-12	53	40	*	2	-3	-4	76	112	*
1	2	4	145	116		1	-1	16	65	53		2	8	-12	43	40		2	-2	-4	103	153	
1	-10	5	35	24	*	1	0	16	64	68		2	9	-12	38	41		2	-1	-4	28	27	
1	-9	5	44	44		1	-11	18	30	31		2	10	-12	80	77		2	0	-4	131	116	
1	-8	5	44	28	*	1	-10	18	33	36		2	11	-12	24	37	*	2	1	-4	89	83	
1	-2	5	23	24		1	-9	18	33	42	*	2	12	-12	36	36		2	2	-4	31	34	
1	-1	5	36	15	*	1	-8	18	44	49		2	0	-11	32	28		2	3	-4	175	98	*
1	0	5	74	49	*	1	-7	18	48	48		2	3	-11	61	56		2	4	-4	97	94	
1	1	5	52	77	*	1	-6	18	36	36		2	4	-11	37	37		2	6	-4	89	86	
1	2	5	36	46	*	1	-5	18	38	57		2	-9	-10	52	50		2	7	-4	72	65	
1	-12	6	66	53		1	-4	18	38	64		2	-8	-10	45	42		2	8	-4	70	66	
1	-11	6	33	52	*	1	2	18	55	35	*	2	-7	-10	33	37		2	9	-4	61	55	
1	-10	6	70	61		1	3	18	45	53		2	-6	-10	63	63		2	10	-4	67	60	
1	-9	6	50	33	*	1	-10	20	54	52		2	-5	-10	72	78		2	-9	-3	27	24	
1	-8	6	52	42		1	-9	20	31	31		2	-4	-10	52	52		2	-8	-3	37	35	
1	-7	6	45	46		1	-8	20	51	34		2	-3	-10	93	88		2	-7	-3	28	24	
1	-6	6	90	97		1	-6	20	36	35		2	-2	-10	79	85		2	-5	-3	20	20	
1	-5	6	192	147	*	1	-5	20	31	36		2	-1	-10	23	22		2	2	-3	98	73	*
1	-4	6	166	121	*	1	-4	20	60	63		2	0	-10	156	131		2	3	-3	109	85	*
1	-2	6	105	165		1	-8	22	31	31		2	1	-10	90	87		2	4	-3	30	30	
1	-1	6	55	70	*	1	-7	22	30	33		2	2	-10	73	63		2	-12	-2	49	47	
1	1	6	166	148		1	-6	22	45	45		2	3	-10	76	79		2	-11	-2	49	40	
1	2	6	49	52		2	2	-22	49	47		2	4	-10	143	104	*	2	-10	-2	59	33	

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
2	-9	-2	76	80	2	-4	7	44	49	3	6	-20	32	34	3	0	-8	127	104
2	-8	-2	62	63	2	-3	7	46	44	3	7	-20	27	27	3	2	-8	124	104
2	-7	-2	65	71	2	-2	7	66	65	3	-4	-18	43	43	3	3	-8	56	57
2	-6	-2	107	104	2	0	7	20	22	3	-3	-18	30	33	3	4	-8	84	76
2	-5	-2	44	72 *	2	1	7	34	39	3	-2	-18	36	40	3	5	-8	93	64 *
2	-4	-2	98	109	2	-13	8	33	33	3	-1	-18	37	40	3	6	-8	100	92
2	-3	-2	84	87	2	-12	8	59	57	3	0	-18	42	45	3	7	-8	32	41 *
2	-2	-2	124	113	2	-11	8	65	43 *	3	1	-18	51	47	3	8	-8	66	68
2	-1	-2	51	53	2	-10	8	42	41	3	2	-18	51	51	3	9	-8	36	37
2	1	-2	232	230	2	-9	8	42	32 *	3	3	-18	47	55	3	10	-8	32	37
2	2	-2	273	239	2	-8	8	75	73	3	4	-18	48	62 *	3	11	-8	31	43 *
2	4	-2	66	70	2	-7	8	102	92	3	5	-18	34	49 *	3	-2	-7	29	31
2	5	-2	48	51	2	-6	8	64	63	3	6	-18	32	36	3	-1	-7	39	41
2	6	-2	64	53	2	-5	8	64	57	3	7	-18	27	35 *	3	1	-7	76	70
2	7	-2	85	90	2	-4	8	90	76	3	8	-18	28	39 *	3	7	-7	30	31
2	8	-2	74	72	2	-3	8	46	49	3	-5	-16	47	41	3	-11	-6	29	29
2	9	-2	31	37	2	-2	8	57	81 *	3	-4	-16	58	61	3	-10	-6	53	51
2	-3	-1	27	32	2	-1	8	60	61	3	-3	-16	35	36	3	-9	-6	65	60
2	2	-1	70	70	2	0	8	66	71	3	-2	-16	44	43	3	-8	-6	72	62
2	5	-1	26	27	2	1	8	48	49	3	-1	-16	49	51	3	-7	-6	21	31 *
2	-12	0	66	57	2	-5	9	29	34	3	0	-16	71	66	3	-6	-6	55	54
2	-11	0	62	53	2	-4	9	50	58	3	1	-16	27	26	3	-5	-6	62	64
2	-10	0	45	36	2	0	9	19	21	3	2	-16	24	25	3	-4	-6	91	93
2	-9	0	44	42	2	-12	10	59	58	3	3	-16	32	38	3	-3	-6	66	79
2	-8	0	66	63	2	-11	10	57	53	3	4	-16	76	74	3	-2	-6	106	120
2	-7	0	65	66	2	-10	10	63	53	3	5	-16	66	60	3	-1	-6	97	68 *
2	-6	0	105	100	2	-9	10	56	56	3	6	-16	69	65	3	0	-6	75	83
2	-5	0	153	120 *	2	-8	10	58	59	3	7	-16	24	33 *	3	1	-6	114	100
2	-4	0	36	54 *	2	-7	10	102	81	3	8	-16	29	42 *	3	2	-6	84	62
2	-3	0	63	76	2	-6	10	96	57 *	3	9	-16	36	44 *	3	3	-6	99	49 *
2	-2	0	93	104	2	-5	10	115	103	3	10	-16	51	46	3	4	-6	38	28 *
2	1	0	97	78	2	-4	10	36	50 *	3	-7	-14	36	36	3	5	-6	90	89
2	2	0	163	151	2	-3	10	81	87	3	-6	-14	60	52	3	7	-6	41	45
2	4	0	207	146 *	2	-2	10	97	83	3	-5	-14	32	34	3	8	-6	68	66
2	5	0	25	26	2	-1	10	118	113	3	-4	-14	51	54	3	8	-6	68	75
2	6	0	122	105	2	0	10	35	36	3	-3	-14	54	50	3	9	-6	50	55
2	7	0	47	48	2	1	10	64	79	3	-2	-14	68	73	3	10	-6	29	34
2	8	0	78	59 *	2	-5	11	49	31 *	3	-1	-14	76	86	3	11	-6	46	45
2	-8	1	21	20	2	-4	11	56	70	3	0	-14	51	66	3	-9	-5	28	33
2	-7	1	32	34	2	-3	11	32	30	3	1	-14	50	54	3	-8	-5	27	25
2	-6	1	28	33	2	-12	12	66	57	3	2	-14	47	35 *	3	-3	-5	31	45 *
2	-3	1	43	26 *	2	-11	12	59	46	3	3	-14	54	49	3	-2	-5	39	37
2	-2	1	74	81	2	-10	12	63	53	3	4	-14	67	70	3	0	-5	46	30 *
2	-13	2	35	35	2	-8	12	68	63	3	5	-14	63	74	3	3	-5	27	33
2	-12	2	69	65	2	-7	12	49	45	3	7	-14	53	47	3	4	-5	34	33
2	-11	2	50	46	2	-6	12	76	85	3	8	-14	52	49	3	6	-5	21	24
2	-9	2	72	69	2	-5	12	42	43	3	9	-14	47	51	3	-12	-4	53	43
2	-8	2	73	84	2	-4	12	90	80	3	10	-14	41	48	3	-11	-4	27	32
2	-7	2	61	54	2	-3	12	50	48	3	11	-14	46	37 *	3	-10	-4	63	57
2	-6	2	96	88	2	-2	12	61	67	3	-3	-13	29	32	3	-9	-4	56	41
2	-5	2	123	120	2	-1	12	72	77	3	-2	-13	30	22 *	3	-8	-4	71	73
2	-4	2	81	29 *	2	0	12	68	71	3	-8	-12	49	45	3	-7	-4	56	34
2	-3	2	209	300 *	2	-10	13	43	49	3	-7	-12	30	32	3	-6	-4	50	53
2	-2	2	214	185	2	-4	13	53	58	3	-6	-12	34	36	3	-5	-4	43	23
2	-1	2	116	96	2	-12	14	45	44	3	-4	-12	77	87	3	-4	-4	166	167
2	0	2	47	54	2	-11	14	69	65	3	-3	-12	43	41	3	-3	-4	47	47
2	1	2	158	114 *	2	-10	14	62	58	3	-2	-12	52	66 *	3	-2	-4	128	155
2	2	2	175	113 *	2	-9	14	61	53	3	1	-12	47	35 *	3	-1	-4	62	60
2	3	2	144	93 *	2	-8	14	101	49 *	3	0	-12	74	100 *	3	0	-4	74	77
2	4	2	50	53	2	-7	14	44	45	3	1	-12	38	35	3	1	-4	152	89 *
2	5	2	62	46 *	2	-6	14	70	75	3	2	-12	93	86	3	2	-4	178	177
2	6	2	74	68	2	-5	14	63	58	3	3	-12	44	44	3	3	-4	101	95
2	-5	3	65	58	2	-4	14	38	44	3	4	-12	84	76	3	4	-4	93	83
2	-4	3	69	73	2	-3	14	49	52	3	5	-12	72	68	3	5	-4	63	56
2	-3	3	61	55	2	-2	14	56	60	3	6	-12	61	62	3	6	-4	103	70 *
2	-4	5	83	73	2	-1	14	72	78	3	7	-12	58	54	3	7	-4	63	57
2	0	3	11	11	2	0	14	71	83	3	8	-12	60	61	3	8	-4	81	78
2	-12	4	46	46	2	-12	16	31	30	3	9	-12	60	56	3	9	-4	74	63
2	-11	4	36	38	2	-11	16	48	48	3	10	-12	59	59	3	10	-4	31	38
2	-10	4	74	62	2	-10	16	42	46	3	11	-12	54	41 *	3	-9	-3	31	30
2	-9	4	34	32	2	-8	16	60	60	3	-9	-10	64	53	3	-8	-3	29	25
2	-8	4	113	105	2	-7	16	61	62	3	-8	-10	44	39	3	-7	-3	27	22
2	-7	4	33	36	2	-6	16	80	77	3	-7	-10	39	38	3	-3	-3	44	40
2	-6	4	63	62	2	-5	16	59	52	3	-6	-10	66	60	3	0	-3	29	24
2	-5	4	58	62	2	-4	16	29	30	3	-5	-10	67	65	3	1	-3	23	14 *
2	-4	4	175	125 *	2	-2	16	50	47	3	-4	-10	52	53	3	2	-3	20	21
2	-3	4	19	51 *	2	-1	16	56	56	3	-3	-10	57	56	3	-12	-2	40	40
2	-2	4	108	121	2	-11	18	37	37	3	-2	-10	75	68	3	-11	-2	53	55
2	-1	4	29	29	2	-10	18	34	45 *	3	-1	-10	89	59	3	-10	-2	51	46
2	0	4	88	83	2	-9	18	34	38	3	0	-10	90	96	3	-9	-2	94	97
2	1	4	49	91 *	2	-8	18	37	40	3	1	-10	76	67	3	-8	-2	61	70
2	-9	5	31	30	2	-7	18	52	52	3	2	-10	105	96	3	-7	-2	48	47
2	-6	5	49	43	2	-6	18	60	56	3	3	-10	63	46 *	3	-6	-2	37	34
2	-5	5	73	67	2	-5	18	64	61	3	4	-10	68	73	3	-5	-2	86	52 *
2	-4	5	72	69	2	-4	18	33	32	3	5	-10	107	87	3	-4	-2	76	116 *
2	-3	5	44	30 *	2	-3	18	55	54	3	6	-10	25	25	3	-3	-2	85	84
2	-2	5	38	20 *	2	-2	18	46	48	3	7	-10	52	44	3	-2	-2	120	113
2	-13	6	47	39	2	-10	20	60	59	3	8	-10	66	64	3	-1	-2	40	19 *
2	-12	6	66	61	2	-8	20	34	36	3	9	-10	53	55	3	0	-2	138	91 *
2	-11	6	82	70	2	-7	20	32	35	3	10	-10	36	41	3	1	-2	164	141
2	-10	6	38	36	2	-6	20	50	52	3	11	-10	57	51	3	2	-2	164	155
2	-9	6	38	44	2	-5	20	53	55	3	-1	-9	21	21	3	3	-2	61	42 *
2	-8	6	111	84 *	2	-4	20	53	50	3	5	-9	40	37	3	4	-2	54	39 *
2	-7	6	61	58	3	2	-22	33	31	3	-10	-8	47	41	3	5	-2	53	48
2	-6	6	105	88	3	3	-22	35	40	3	-9	-8	37	36	3	6	-2	68	74
2	-5	6	144	140	3	4	-22	33	34	3	-8	-8	55	53	3	7	-2	88	82
2	-4	6	36	36	3	5	-22	46	49	3	-7	-8	41	36	3	8	-2	59	58
2	-3	6	59	67	3	-1	-20	39	46	3	-6	-8	59	64	3	9	-2	72	62
2	-2	6	204	204	3	0	-20	50	60	3	-5	-8	39	36	3	-2	-1	16	21 *
2	-1	6	40	46	3	2	-20	23	36 *	3	-4	-8	91	95	3	1	-1	59	54
2	0	6	65	90 *	3	3	-20	25	46 *	3	-3	-8	65	70	3	2	-1	101	90 *
2	1	6	153	157	3	4	-20	41	56 *	3	-2	-8	84	77	3	3	-1	69	62
2	-5	7	40	40	3	5	-20	42	43	3	-1	-8	19	17	3	-13	0	35	33

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
3	-12	0	47	45	3	-13	10	42	39	4	8	-10	50	49 *	4	0	-6	59	40 *
3	-11	0	35	27 *	3	-12	10	54	51	4	9	-10	35	35	4	1	-6	131	109 *
3	-10	0	50	45	3	-11	10	50	46	4	-7	-14	54	40 *	4	2	-6	89	64 *
3	-9	0	38	43	3	-10	10	64	56	4	-6	-14	50	54	4	3	-6	88	82
3	-8	0	71	63	3	-9	10	55	56	4	-5	-14	55	52	4	4	-6	49	35 *
3	-7	0	74	68	3	-7	10	69	67	4	-4	-14	20	30	4	5	-6	110	86 *
3	-6	0	64	70	3	-6	10	71	69	4	-3	-14	51	59	4	6	-6	51	54
3	-5	0	17	26 *	3	-5	10	61	62	4	-2	-14	77	84	4	7	-6	71	59
3	-4	0	210	203	3	-4	10	84	72	4	-1	-14	60	65	4	8	-6	67	60
3	-3	0	34	34	3	-3	10	97	97	4	0	-14	37	33	4	9	-6	36	36
3	-2	0	40	39	3	-2	10	45	50	4	1	-14	81	86	4	10	-6	55	43 *
3	0	0	139	116	3	-1	10	53	56	4	2	-14	39	40	4	-8	-5	34	37
3	1	0	117	52 *	3	0	10	76	79	4	3	-14	65	69	4	-7	-5	34	33
3	2	0	340	227 *	3	1	10	45	39	4	4	-14	65	69	4	-6	-5	29	31
3	3	0	46	25 *	3	-9	11	27	30	4	4	-14	76	74	4	-4	-5	25	26
3	4	0	60	65	3	-8	11	29	26	4	5	-14	55	56	4	-3	-5	27	27
3	5	0	93	67 *	3	-13	12	34	34	4	6	-14	39	37	4	3	-5	64	54
3	6	0	116	97	3	-12	12	56	56	4	7	-14	45	50	4	4	-5	39	36
3	7	0	50	41	3	-11	12	51	50	4	8	-14	34	38	4	5	-5	32	32
3	8	0	80	72	3	-10	12	78	73	4	9	-14	33	36	4	-12	-4	55	45
3	-8	1	32	27	3	-9	12	41	39	4	10	-14	39	43	4	-11	-4	51	55
3	-7	1	50	40	3	-8	12	61	60	4	-8	-12	58	52	4	-10	-4	74	71
3	-4	1	73	84	3	-7	12	80	75	4	-7	-12	41	41	4	-9	-4	39	37
3	0	1	46	32 *	3	-6	12	90	86	4	-6	-12	65	60	4	-8	-4	111	118
3	1	1	27	11 *	3	-4	12	113	116	4	-5	-12	43	46	4	-7	-4	70	67
3	3	1	50	43	3	-3	12	39	39	4	-4	-12	88	90	4	-6	-4	78	72
3	-13	2	53	51	3	-2	12	70	72	4	-3	-12	57	68	4	-5	-4	105	95
3	-12	2	67	59	3	-1	12	83	89	4	-2	-12	99	106	4	-4	-4	147	153
3	-11	2	50	46	3	0	12	77	87	4	-1	-12	84	79	4	-2	-4	176	198
3	-10	2	37	36	3	-13	14	36	35	4	0	-12	78	84	4	-1	-4	77	83
3	-9	2	75	71	3	-12	14	39	43	4	1	-12	50	56	4	0	-4	134	101 *
3	-8	2	50	63	3	-11	14	50	56	4	2	-12	104	97	4	1	-4	91	105
3	-7	2	50	46	3	-10	14	46	56	4	3	-12	52	47	4	2	-4	190	172
3	-6	2	37	37	3	-9	14	63	55	4	4	-12	80	82	4	3	-4	155	116 *
3	-5	2	39	39	3	-7	14	44	45	4	5	-12	70	77	4	4	-4	127	102
3	-4	2	116	113	3	-6	14	75	80	4	6	-12	90	83	4	5	-4	53	51
3	-3	2	77	121 *	3	-5	14	49	45	4	7	-12	76	69	4	6	-4	86	83
3	-2	2	84	109 *	3	-4	14	58	59	4	8	-12	68	59	4	7	-4	68	61
3	-1	2	82	74	3	-3	14	56	56	4	9	-12	33	39	4	8	-4	74	64
3	0	2	98	27 *	3	-2	14	53	61	4	10	-12	53	54	4	9	-4	43	44
3	1	2	141	97 *	3	-1	14	39	46	4	-4	-11	35	35	4	-6	-3	31	32
3	2	2	167	146 *	3	0	14	83	83	4	-3	-11	36	39	4	-4	-3	38	44
3	3	2	95	64 *	3	-12	16	35	36	4	-9	-10	59	50	4	-3	-3	24	36 *
3	4	2	82	42 *	3	-11	16	38	45	4	-8	-10	61	57	4	-1	-3	58	46 *
3	5	2	45	40	3	-10	16	34	39	4	-7	-10	61	53	4	-12	-2	45	36
3	6	2	42	36	3	-9	16	40	37	4	-6	-10	61	54	4	-11	-2	51	48
3	-9	3	24	22	3	-8	16	42	45	4	-5	-10	71	72	4	-10	-2	51	56
3	-7	3	26	26	3	-7	16	63	60	4	-4	-10	40	30 *	4	-9	-2	79	72
3	-13	4	42	37	3	-6	16	63	76	4	-3	-10	44	46	4	-8	-2	62	64
3	-12	4	54	45	3	-5	16	42	39	4	-2	-10	89	95	4	-7	-2	51	46
3	-11	4	60	54	3	-4	16	41	49	4	-1	-10	86	82	4	-6	-2	39	36
3	-10	4	70	69	3	-3	16	38	40	4	0	-10	56	43 *	4	-5	-2	118	116
3	-9	4	42	43	3	-2	16	55	56	4	1	-10	111	94	4	-4	-2	97	99
3	-8	4	119	100	3	-11	18	35	37	4	2	-10	103	94	4	-3	-2	116	105
3	-7	4	29	19 *	3	-10	18	32	37	4	3	-10	40	38	4	-2	-2	173	140
3	-6	4	90	85	3	-9	18	39	44	4	4	-10	107	92	4	-1	-2	47	47
3	-5	4	51	42 *	3	-8	18	38	37	4	5	-10	79	71	4	0	-2	130	89 *
3	-4	4	183	211	3	-7	18	49	49	4	6	-10	50	50	4	1	-2	158	122 *
3	-3	4	177	134 *	3	-6	18	44	41	4	7	-10	75	71	4	2	-2	82	88
3	-2	4	63	75	3	-5	18	36	39	4	8	-10	65	66	4	3	-2	91	79
3	-1	4	51	43	3	-4	18	35	25 *	4	9	-10	38	37	4	4	-2	84	75
3	0	4	117	102 *	3	-3	18	54	54	4	10	-10	50	46	4	5	-2	67	47 *
3	-11	5	33	36	3	-10	20	43	44	4	11	-10	57	39 *	4	6	-2	80	79
3	-10	5	30	21 *	3	-9	20	23	27	4	0	-9	27	32	4	7	-2	84	86
3	-9	5	17	19	3	-8	20	44	46	4	1	-9	39	45	4	8	-2	66	61
3	-5	5	79	75	3	-7	20	40	43	4	7	-9	27	27	4	-6	-1	45	40
3	-4	5	33	56 *	3	-6	20	56	63	4	-10	-8	45	37	4	-2	-1	35	37
3	-14	6	20	15 *	3	-5	20	57	53	4	-9	-8	33	33	4	-1	-1	58	52
3	-13	6	43	41	3	-4	20	50	49	4	-8	-8	83	76	4	0	-1	86	82
3	-12	6	44	45	4	-1	-20	49	49	4	-7	-8	40	40	4	1	-1	21	19
3	-11	6	71	64	4	0	-20	56	59	4	-6	-8	64	65	4	-12	0	41	40
3	-10	6	70	63	4	1	-20	30	35	4	-5	-8	70	77	4	-11	0	54	44
3	-9	6	69	64	4	2	-20	40	43	4	-4	-8	91	70	4	-10	0	95	82
3	-8	6	76	63	4	3	-20	45	54	4	-3	-8	93	94	4	-9	0	36	35
3	-7	6	68	69	4	4	-20	51	72 *	4	-2	-8	100	91	4	-8	0	66	75
3	-6	6	50	59	4	5	-20	37	52 *	4	-1	-8	94	68 *	4	-7	0	67	70
3	-5	6	43	29 *	4	6	-20	49	51	4	0	-8	53	58	4	-6	0	67	64
3	-4	6	95	96	4	7	-20	44	38	4	1	-8	89	78	4	-5	0	75	80
3	-3	6	73	65	4	-3	-18	53	47	4	2	-8	123	107	4	-4	0	174	182
3	-2	6	72	85	4	-2	-18	51	53	4	3	-8	111	80 *	4	-3	0	152	41 *
3	-1	6	87	80	4	-1	-18	44	46	4	4	-8	71	52 *	4	0	0	57	53
3	0	6	142	129	4	0	-18	39	45	4	5	-8	45	40	4	1	0	99	61 *
3	1	6	40	43	4	1	-18	52	52	4	6	-8	80	81	4	2	0	116	107
3	-11	7	43	29 *	4	2	-18	40	44	4	7	-8	58	48	4	3	0	115	66 *
3	-5	7	27	22	4	3	-18	40	45	4	8	-8	76	74	4	4	0	111	82 *
3	-4	7	46	31 *	4	4	-18	49	57	4	10	-8	51	53	4	5	0	57	44 *
3	0	7	53	47 *	4	5	-18	38	46	4	11	-8	50	42	4	6	0	70	68
3	-14	8	44	33 *	4	6	-18	35	50 *	4	-2	-7	56	58	4	7	0	65	53
3	-13	8	37	34	4	7	-18	36	42	4	-1	-7	48	46	4	-6	1	29	45 *
3	-12	8	41	35	4	8	-18	49	49	4	0	-7	22	26	4	-2	1	80	75
3	-11	8	43	39	4	-5	-16	51	54	4	1	-7	28	25	4	0	1	38	35
3	-10	8	56	57	4	-4	-16	51	48	4	2	-7	40	31 *	4	1	1	51	31 *
3	-8	8	70	81	4	-3	-16	44	44	4	-11	-6	47	45	4	5	1	35	36
3	-7	8	105	92	4	-2	-16	65	63	4	-10	-6	59	57	4	-13	2	37	34
3	-6	8	55	41 *	4	-1	-16	66	62	4	-9	-6	53	48	4	-12	2	51	46
3	-5	8	51	56	4	0	-16	52	51	4	-8	-6	68	69	4	-11	2	55	48
3	-4	8	105	120	4	1	-16	56	53	4	-7	-6	27	27	4	-10	2	83	78
3	-2	8	109	101	4	2	-16	43	38	4	-6	-6	58	60	4	-9	2	53	59
3	-1	8	23	21	4	3	-16	44	41	4	-5	-6	86	85	4	-8	2	104	94
3	0	8	107	123	4	4	-16	69	77	4	-4	-6	78	71	4	-7	2	42	40
3	1	8	29	40 *	4	5	-16	48	51	4	-3	-6	103	101	4	-6	2	75	73
3	-6	9	27	19 *	4	6	-16	62	65	4	-2	-6	76	88	4	-5	2	47	58
3	-1	9	26	27	4	7	-16	29	37 *	4	-1	-6	106	113	4	-4	2	95	89

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$		h	k	l	$\|F_o\|$	$\|F_c\|$	
4	-3	2	131	81	*	4	-4	12	89	96		5	1	-12	49	43		5	-3	-4	54	46	
4	-2	2	95	74	*	4	-3	12	34	34		5	2	-12	77	89		5	-2	-4	65	70	
4	-1	2	110	116		4	-2	12	83	92		5	3	-12	46	41		5	-1	-4	44	45	
4	0	2	47	46		4	-1	12	58	58		5	4	-12	64	63		5	0	-4	157	144	
4	1	2	191	146	*	4	0	12	65	69		5	5	-12	50	46		5	2	-4	71	120	*
4	2	2	116	80	*	4	-6	13	46	44		5	6	-12	88	80		5	3	-4	107	103	
4	3	2	95	96		4	-13	14	32	46		5	7	-12	33	33		5	4	-4	61	65	
4	4	2	77	65		4	-12	14	36	41		5	8	-12	26	26		5	5	-4	66	49	*
4	5	2	57	40	*	4	-10	14	42	47		5	9	-12	37	39		5	6	-4	89	82	
4	-10	3	34	30		4	-9	14	56	61		5	10	-12	49	47		5	7	-4	25	23	
4	-9	3	30	32		4	-8	14	44	48		5	-5	-11	40	35		5	8	-4	36	41	
4	-7	3	44	40		4	-7	14	45	50		5	-3	-11	26	42	*	5	9	-4	32	37	
4	-5	3	28	24		4	-9	15	31	32		5	0	-11	20	16		5	-12	-3	34	36	
4	-2	3	41	44		4	-12	16	53	47		5	9	-10	28	39	*	5	-6	-3	23	22	
4	-1	3	56	45		4	-11	16	37	35		5	-8	-10	50	38	*	5	-5	-3	49	54	*
4	0	3	32	50	*	4	-10	16	42	48		5	-7	-10	64	64		5	-4	-3	16	19	
4	-13	4	40	36		4	-8	16	54	56		5	-6	-10	59	55		5	-1	-3	43	31	*
4	-12	4	48	44		4	-7	16	44	42		5	-5	-10	44	43		5	0	-3	20	17	
4	-11	4	58	61		4	-6	16	69	67		5	-4	-10	41	44		5	1	-3	26	26	
4	-10	4	101	93		4	-5	16	54	50		5	-3	-10	71	72		5	3	-3	26	25	
4	-9	4	49	47		4	-4	16	70	65		5	-2	-10	60	53		5	-12	-2	32	30	
4	-8	4	93	104		4	-2	16	61	64		5	-1	-10	73	70		5	-11	-2	44	42	
4	-7	4	35	34		4	-9	17	30	30		5	0	-10	53	59		5	-10	-2	55	50	
4	-6	4	132	139		4	-11	18	35	34		5	1	-10	36	33		5	-9	-2	51	51	
4	-5	4	94	95		4	-10	18	33	40		5	2	-10	66	76		5	-8	-2	31	30	
4	-4	4	182	209		4	-9	18	39	46		5	3	-10	68	63		5	-7	-2	64	69	
4	-3	4	85	87		4	-8	18	46	51		5	4	-10	84	81		5	-6	-2	66	51	*
4	-2	4	112	102		4	-7	18	34	31		5	5	-10	61	67		5	-5	-2	85	88	
4	-1	4	87	93		4	-6	18	45	46		5	6	-10	99	79	*	5	-4	-2	85	81	
4	0	4	149	146		4	-5	18	41	37		5	7	-10	43	44		5	-3	-2	86	105	
4	-7	5	36	33		4	-4	18	41	40		5	8	-10	35	38		5	-2	-2	75	94	*
4	-6	5	44	44		4	-3	18	52	54		5	9	-10	27	39	*	5	-1	-2	45	39	
4	-5	5	44	42		4	-9	19	31	28		5	10	-10	44	38	*	5	0	-2	89	101	
4	-4	5	52	42		4	-10	20	40	43		5	-5	-9	32	45	*	5	1	-2	85	84	
4	-3	5	16	25	*	4	-9	20	30	33		5	-1	-9	38	50	*	5	2	-2	65	61	
4	0	5	22	22		4	-8	20	52	58		5	0	-9	41	36		5	3	-2	88	69	
4	-13	6	46	36	*	4	-7	20	33	35		5	1	-9	44	45		5	4	-2	110	97	
4	-12	6	41	30	*	4	-6	20	47	56		5	5	-9	32	26		5	5	-2	27	38	*
4	-11	6	73	64		4	-5	20	40	47		5	-10	-8	38	38		5	6	-2	84	93	
4	-10	6	74	75		5	-1	-20	39	37		5	-9	-8	35	32		5	7	-2	57	57	
4	-8	6	61	57		5	0	-20	52	53		5	-8	-8	67	62		5	-11	-1	37	33	
4	-8	6	56	57		5	1	-20	29	27		5	-7	-8	50	46		5	-9	-1	19	20	
4	-7	6	77	64		5	2	-20	49	54		5	-6	-8	41	38		5	-8	-1	25	25	
4	-6	6	72	83		5	3	-20	35	41		5	-5	-8	27	21	*	5	-7	-1	48	55	
4	-5	6	90	92		5	4	-20	45	54		5	-4	-8	74	68		5	-6	-1	58	52	
4	-4	6	70	75		5	5	-20	40	47		5	-3	-8	55	52		5	-5	-1	28	28	
4	-3	6	99	95		5	6	-20	43	48		5	-2	-8	55	55		5	0	-1	47	46	
4	-2	6	92	88		5	-3	-18	31	52		5	-1	-8	79	78		5	1	-1	50	49	
4	-1	6	49	55		5	-2	-18	44	49		5	0	-8	112	89	*	5	3	-1	29	29	
4	0	6	77	99	*	5	-1	-18	34	40		5	1	-8	36	32		5	-12	0	40	41	
4	-7	7	31	29		5	0	-18	45	47		5	2	-8	132	116		5	-11	0	48	41	
4	-6	7	53	48		5	1	-18	41	46		5	3	-8	23	22		5	-10	0	73	68	
4	-4	7	25	23		5	2	-18	39	47		5	4	-8	50	33	*	5	-9	0	35	34	
4	-2	7	54	44		5	3	-18	31	32		5	6	-8	69	72		5	-8	0	40	37	
4	-1	7	22	22		5	4	-18	30	36		5	7	-8	29	30		5	-7	0	51	53	
4	0	7	51	60		5	5	-18	29	42	*	5	8	-8	51	50		5	-6	0	70	77	
4	-13	8	43	32	*	5	6	-18	33	49	*	5	9	-8	29	31		5	-5	0	54	61	
4	-12	8	37	35		5	7	-18	29	36		5	10	-8	63	53		5	-4	0	58	58	
4	-11	8	43	42		5	8	-18	37	42		5	-3	-7	24	29		5	-3	0	55	64	
4	-10	8	81	85		5	-5	-16	32	29		5	-2	-7	40	39		5	0	0	130	98	*
4	-9	8	48	39		5	-4	-16	46	43		5	-1	-7	76	69		5	1	0	47	30	*
4	-7	8	66	89		5	-3	-16	35	36		5	1	-7	26	40	*	5	2	0	83	55	*
4	-7	8	53	38		5	-2	-16	54	49		5	2	-7	39	35		5	3	0	80	67	
4	-6	8	53	57		5	-1	-16	41	42		5	4	-7	33	30		5	4	0	120	90	*
4	-5	8	90	74		5	0	-16	56	60		5	5	-7	25	36	*	5	6	0	113	81	*
4	-4	8	110	105		5	1	-16	35	34		5	6	-7	22	21		5	-13	1	22	26	
4	-3	8	56	60		5	2	-16	52	52		5	-11	-6	52	46		5	-8	1	36	38	
4	-2	8	83	105	*	5	3	-16	38	37		5	-10	-6	39	37		5	-7	1	32	31	
4	-1	8	73	64		5	4	-16	37	42		5	-9	-6	54	48		5	-5	1	29	35	
4	0	8	30	93	*	5	5	-16	30	36		5	-8	-6	42	44		5	-3	1	53	56	
4	-13	9	33	29		5	6	-16	44	58	*	5	-7	-6	53	52		5	3	1	37	36	
4	-9	9	31	26		5	7	-16	18	16		5	-6	-6	76	84		5	2	1	17	6	*
4	-7	9	26	24		5	8	-16	34	35		5	-5	-6	70	66		5	-13	2	34	28	
4	-5	9	34	32		5	9	-16	37	37		5	-4	-6	36	41		5	-12	2	42	41	
4	-4	9	31	34		5	-1	-15	35	31		5	-3	-6	84	83		5	-11	2	43	38	
4	-3	9	53	53		5	-7	-14	39	53		5	-1	-6	42	36		5	-10	2	76	73	
4	-2	9	53	66	*	5	-6	-14	45	44		5	0	-6	144	119		5	-9	2	71	77	
4	-13	10	46	44		5	-5	-14	32	32		5	1	-6	77	67		5	-8	2	25	23	
4	-12	10	49	42		5	-4	-14	31	34		5	2	-6	87	57		5	-7	2	84	82	
4	-10	10	61	80		5	-3	-14	43	48		5	3	-6	111	93		5	-6	2	62	89	
4	-9	10	42	41		5	-2	-14	63	61		5	4	-6	69	61		5	-4	2	59	62	
4	-8	10	51	52		5	-1	-14	30	41		5	5	-6	51	57		5	-3	2	112	124	
4	-7	10	51	46		5	0	-14	47	54		5	6	-6	74	70		5	-1	2	85	80	
4	-6	10	92	67		5	1	-14	64	66		5	7	-6	51	38	*	5	0	2	107	68	*
4	-5	10	72	74		5	2	-14	73	72		5	8	-6	28	27		5	1	2	86	70	
4	-4	10	66	66		5	3	-14	62	58		5	9	-6	34	37		5	2	2	80	74	
4	-3	10	58	57		5	4	-14	46	47		5	-7	-5	47	48		5	3	2	101	85	
4	-2	10	66	71		5	5	-14	42	44		5	-6	-5	25	24		5	4	2	81	70	
4	-1	10	59	62		5	6	-14	40	46		5	-5	-5	32	35		5	5	2	60	40	*
4	0	10	57	61		5	7	-14	31	33		5	-3	-5	33	31		5	-12	3	37	33	
4	-9	11	56	55		5	8	-14	28	30		5	-1	-5	52	50		5	-9	3	34	29	
4	-8	11	36	37		5	9	-14	39	36		5	0	-5	12	11		5	-8	3	40	46	
4	-7	11	27	25		5	-3	-13	36	36		5	1	-5	33	28		5	-4	3	16	15	
4	-3	11	27	33		5	3	-13	28	26		5	3	-5	33	29		5	-3	3	57	62	
4	-13	12	51	43		5	-8	-12	56	48		5	-12	-4	48	39		5	-2	3	53	33	
4	-12	12	57	54		5	-7	-12	58	53		5	-11	-4	52	49		5	-13	4	23	22	
4	-11	12	42	43		5	-6	-12	63	56		5	-10	-4	58	53		5	-12	4	44	33	*
4	-10	12	80	82		5	-5	-12	48	45		5	-9	-4	45	41		5	-11	4	48	43	
4	-9	12	70	52	*	5	-4	-12	84	79		5	-8	-4	73	73		5	-10	4	72	66	
4	-8	12	96	96		5	-3	-12	46	48		5	-7	-4	81	90		5	-9	4	64	59	
4	-7	12	56	59		5	-2	-12	60	75		5	-6	-4	61	92		5	-8	4	71	67	
4	-6	12	91	88		5	-1	-12	49	47		5	-5	-4	37	38		5	-7	4	54	49	
4	-5	12	47	48		5	0	-12	76	85		5	-4	-4	114	97		5	-6	4	101	103	

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
5	-5	4	48	49	5	-10	14	37	37	7	3	3	66	64	-6	-6	4	39	46
5	-4	4	95	106	5	-9	14	61	57	7	4	3	75	77	-6	-5	4	53	60
5	-3	4	43	38	5	-8	14	40	34	7	5	3	45	41	-6	-4	4	69	71
5	-2	4	103	112	5	-7	14	56	56	7	6	3	36	46 *	-6	-3	4	56	71
5	-1	4	38	44 *	5	-6	14	54	51	8	0	3	56	73	-6	0	4	131	125
5	0	4	82	89	5	-5	14	65	63	8	2	3	47	101 *	-6	1	4	93	89 *
5	-15	5	23	22	5	-4	14	65	68	8	3	3	76	59 *	-6	2	4	99	93
5	-7	5	37	34	5	-3	14	64	62	8	4	3	47	46	-6	4	4	66	62
5	-4	5	22	22	5	-2	14	61	56	8	6	3	40	47	-6	6	4	76	69
5	-3	5	27	27	5	-1	14	45	42	9	0	3	60	58	-6	7	4	59	63
5	-13	6	36	30	5	0	14	42	36	9	2	3	48	55	-6	8	4	38	46
5	-12	6	39	28 *	5	-12	15	50	47	9	5	3	38	36	0	-3	4	38	35 *
5	-11	6	43	34 *	5	-11	15	29	28	10	0	3	56	56	0	-2	4	134	127
5	-10	6	48	36	5	-10	15	25	26	10	3	3	39	35	0	-1	4	72	65 *
5	-9	6	69	85	5	-8	15	41	42	10	4	3	33	39	0	0	4	73	66
5	-8	6	41	40	5	-7	15	35	34	11	0	3	62	48 *	0	2	4	63	56
5	-7	6	51	71 *	5	-6	15	43	48	11	2	3	39	47	0	4	4	56	56
5	-6	6	126	131	5	-5	15	46	49	-9	3	2	60	73	7	-3	4	36	27 *
5	-5	6	73	73	5	-4	15	39	42	-8	3	2	76	81	7	-2	4	114	99
5	-4	6	36	33	5	-3	15	42	40	-7	-4	2	58	50	7	-1	4	53	56
5	-3	6	118	138	5	-4	17	37	35	-7	-3	2	60	62	7	0	4	51	50
5	-2	6	42	54	5	-10	18	27	30 *	-7	-2	2	49	49	7	1	4	37	35
5	0	6	83	82	5	-9	18	36	39	-7	-1	2	58	60	7	2	4	75	67
5	-13	7	33	33	5	-8	18	39	39	-7	1	2	53	55	8	-2	4	53	54
5	-8	7	37	48	5	-7	18	37	36	-7	2	2	51	45	8	-1	4	38	36
5	-7	7	42	54 *	5	-6	18	38	41	-7	3	2	76	99 *	8	0	4	56	64
5	-5	7	47	35 *	5	-5	18	35	37	-7	5	2	48	67 *	8	2	4	64	59
5	-3	7	36	31	5	-4	18	37	36	-7	6	2	57	56	9	-2	4	53	54 *
5	-2	7	37	42	-13	4	0	34	41	-6	-4	2	66	69	9	0	4	70	59
5	-1	7	41	43	-12	2	0	51	51	-6	-1	2	76	71	-9	-2	5	63	56
5	-13	8	30	29	-12	6	0	35	40	-6	0	2	116	95	-9	-1	5	77	80
5	-12	8	47	43	-12	8	0	36	37	-6	1	2	59	61	-9	0	5	72	67
5	-11	8	33	35	-11	2	0	56	56	-6	2	2	64	70	-9	1	5	61	61
5	-10	8	46	57	-11	4	0	48	50	-6	3	2	114	100	-9	2	5	57	55
5	-8	8	46	41	-11	6	0	42	27 *	-6	4	2	58	76	-9	4	5	74	73
5	-6	8	33	36	-11	10	0	32	36	-6	5	2	76	80	-8	-1	6	58	64
5	-5	8	56	57	-10	2	0	49	49	0	-3	2	56	55	-8	0	6	74	76
5	-4	8	66	70	-10	3	0	54	51	0	-2	2	105	101	-8	1	6	57	61
5	-3	8	109	99	-10	6	0	76	69	0	-1	2	44	46	-8	2	6	55	54
5	-2	8	67	72	-9	2	0	62	73	0	0	2	79	76	-8	4	6	58	61
5	-1	8	46	46	-9	3	0	57	47 *	0	1	2	91	87	-8	5	6	51	51
5	0	8	49	47	-9	4	0	57	79	0	2	2	47	54	-8	6	6	66	69
5	-9	9	37	26 *	-9	6	0	66	61	7	-3	2	59	65	-7	-4	6	79	81
5	-8	9	51	55	-9	8	0	59	73	7	-2	2	66	63	-7	-1	6	74	61
5	-6	9	31	29	-8	1	0	56	55	7	-1	2	54	45	-7	0	6	61	61
5	-3	9	47	52	-8	2	0	51	73	7	0	2	51	64	-7	1	6	70	79
5	-2	9	45	47	-8	4	0	71	71	7	1	2	77	84	-7	2	6	60	59
5	-13	10	34	32	-8	5	0	59	41 *	8	-3	2	57	57	-7	3	6	54	55
5	-12	10	38	35	-8	6	0	61	80	8	-2	2	63	74	-7	5	6	67	76
5	-10	10	66	57	-8	7	0	56	63	8	-1	2	54	63	-7	6	6	69	93 *
5	-9	10	56	54	-8	8	0	96	94	8	0	2	60	56	-6	-5	6	69	91 *
5	-8	10	57	59	-8	10	0	56	53	9	-3	2	56	54	-6	-4	6	68	70
5	-7	10	50	53	-8	12	0	41	45	9	-2	2	54	80	-6	-1	6	54	51
5	-6	10	72	66	-7	1	0	92	63 *	9	-1	2	56	47	-6	0	6	124	116
5	-5	10	87	79	-7	2	0	127	114	9	0	2	60	61	-6	1	6	73	69
5	-3	10	73	79	-7	3	0	76	38	-10	4	4	59	53	-6	2	6	62	54
5	-2	10	63	69	-7	4	0	60	66	-10	6	4	47	42	-6	4	6	69	77
5	-1	10	41	43	-7	6	0	54	67	-9	2	4	63	63	-6	5	6	54	56
5	0	10	47	44	-7	8	0	60	83	-9	4	4	75	84	-6	6	6	81	110 *
5	-8	11	36	35	-7	12	0	35	49	-9	8	4	59	64	0	-4	6	69	72
5	-4	11	33	49 *	-6	1	0	66	46	-8	-2	4	60	103	0	-3	6	101	97
5	-3	11	33	34	-6	2	0	191	113 *	-8	1	4	51	47	0	-2	6	79	81
5	-2	11	26	30	-6	4	0	104	94	-8	2	4	79	84	0	-1	6	60	55
5	-15	12	27	32	-6	5	0	46	56	-8	3	4	51	51	0	0	6	46	47
5	-12	12	39	44	-6	6	0	84	66	-8	4	4	59	56	0	1	6	60	56
5	-10	12	47	49	-6	8	0	61	65	-8	6	4	56	46	0	2	6	68	80
5	-9	12	57	54	-6	10	0	59	71	-8	7	4	50	55	7	-4	6	63	59
5	-8	12	80	82	-6	12	0	43	46	-8	8	4	64	62	7	-3	6	69	77
5	-7	12	47	44	0	0	0	102	84	-7	-4	4	74	77	7	-2	6	70	68
5	-6	12	56	63	0	1	0	59	57	-7	-2	4	51	49	7	-1	6	58	66
5	-5	12	34	35	0	3	0	44	26 *	-7	0	4	105	94	7	1	6	51	52
5	-4	12	58	61	0	4	0	68	73	-7	1	4	49	57	8	-4	6	67	60
5	-3	12	51	46	0	5	0	56	53	-7	2	4	117	120	8	-3	6	53	63
5	-2	12	66	80	0	8	0	56	52	-7	3	4	55	60	8	-2	6	48	45
5	-13	14	41	39	7	0	0	37	52 *	-7	5	4	48	46	8	-1	6	52	74
5	-12	14	34	32	7	1	0	129	92 *	-7	6	4	54	76 *	9	-3	6	57	54
5	-11	14	24	25	7	2	0	56	63	-7	8	4	54	56					

atoms are designated in Fig. 2. The sodium coordination polyhedra, which are composed of carboxylate and water oxygen atoms, form infinite chains around the lines $x=0$, $y=1/2$ (Fig. 3). The six carboxylate oxygen atoms of each tris(dipicolinato) complex not coordinated to neodymium are coordinated to seven sodium ions. The complex around the neodymium ion at $(-0.01,0.00,0.25)$ thus takes part in the bonds $Na(1^{iv})-O(12)$, $Na(2)-O(2)$, $Na(2^{iv})-O(6)$, $Na(3)-O(8)$, $Na(4)-O(2)$, $Na(4^{i})-O(10)$, and $Na(4^{iv})-O(4)$.

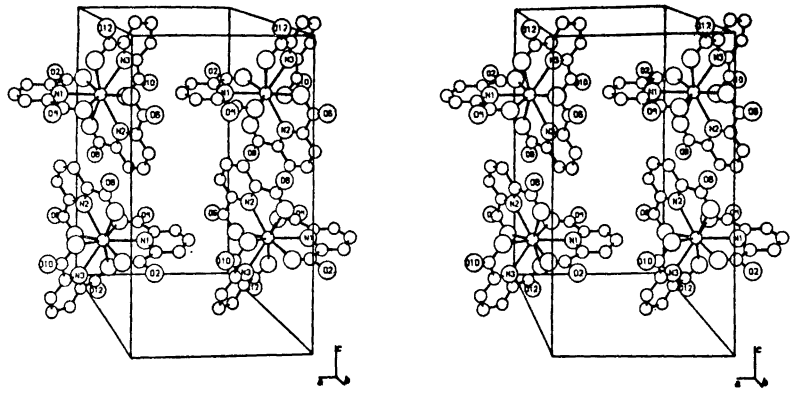

Fig. 1. A stereoscopic pair of drawing showing the layer around $y = 0$ in NDP containing the tris(dipicolinato) complexes (the box is drawn between $y = -1/2$ and $y = 1/2$). Figs. 1, 3, and 4 were drawn with the program ORTEP, written by C. K. Johnson, Oak Ridge.

In this way NDP is held together by a three-dimensional network of sodium-carboxylate oxygen bonds.

The neodymium coordination polyhedron. The structure of the tris-(dipicolinato)neodymiumate ion in NDP is, as expected, a distorted version of the structure of the corresponding ytterbium complex in HEXYBDIPIC.[3] The tri-capped trigonal prism has the carboxylate oxygen atoms O(1), O(3), O(5), O(7), O(9), and O(11) at the corners of the prism and the nitrogen atoms N(1), N(2), and N(3) in the equatorial plane. Selected distances in the coordination polyhedron are given in Table 5 A. The metal – oxygen bond distances are in the range 2.37 – 2.61 Å, with an average of 2.49 Å. The metal – nitrogen bond distances are almost equal, their average being 2.58 Å.

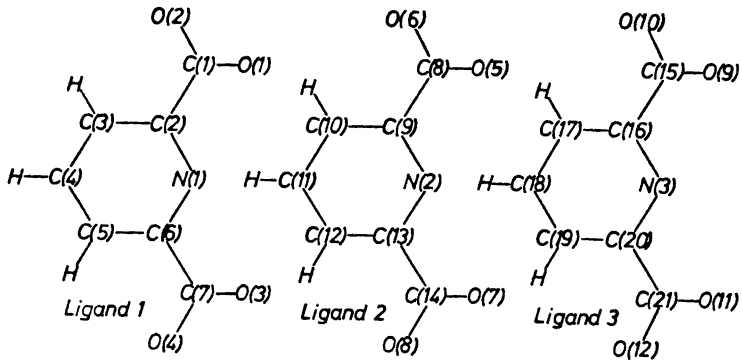

Fig. 2. Designation of the atoms in the three different dipicolinate ligands in NDP.

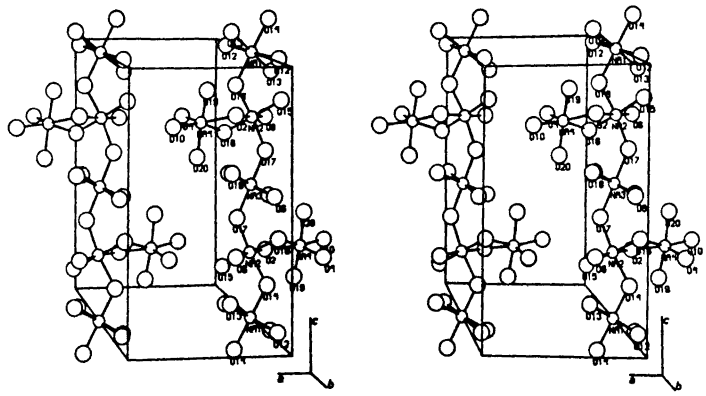

Fig. 3. A stereoscopic pair of drawings of the layer around $y = 1/2$ in NDP showing the sodium coordination (the box is drawn between $y = 0$ and $y = 1$).

The neodymium ion is located only 0.01 Å from the plane formed by the equatorial nitrogen atoms. The two triangular faces of the prism are slightly tilted; the perpendicular distances from O(3), O(5), and O(11) to the plane through O(1), O(7), and O(9) are 3.55, 3.60, and 3.47 Å, respectively. Within the limits of error the triangles O(1)O(7)O(9) and O(3)O(5)O(11) are equilateral

Table 5. Selected interatomic distances (Å) and angles (°) with estimated standard deviations in NDP.

A. The ytterbium coordination polyhedron

Distance		Distance	
Nd – O(1)	2.40(1)	O(3) – N(1)	2.68(2)
Nd – O(3)	2.37(2)	O(3) – N(2)	3.07(3)
Nd – O(5)	2.55(1)	O(5) – O(9)	3.62(2)
Nd – O(7)	2.61(1)	O(5) – O(11)	3.02(2)
Nd – O(9)	2.47(2)	O(5) – N(2)	2.68(2)
Nd – O(11)	2.51(2)	O(5) – N(3)	3.00(2)
Nd – N(1)	2.59(3)	O(7) – O(9)	3.03(2)
Nd – N(2)	2.57(2)	O(7) – N(1)	3.14(3)
Nd – N(3)	2.58(2)	O(7) – N(2)	2.67(2)
O(1) – O(7)	3.06(2)	O(9) – N(2)	3.07(2)
O(1) – O(9)	2.99(2)	O(9) – N(3)	2.73(2)
O(1) – O(11)	3.58(2)	O(11) – N(1)	3.35(3)
O(1) – N(1)	2.52(3)	O(11) – N(3)	2.53(2)
O(1) – N(3)	3.26(2)	N(1) – N(2)	4.03(2)
O(3) – O(5)	2.98(2)	N(1) – N(3)	4.10(2)
O(3) – O(7)	3.65(2)	M(2) – N(3)	4.39(2)
O(3) – O(11)	3.00(2)		

Table 5. Continued.

B. Ligand No. 1

Distance		Angle	
N(1)–C(2)	1.30(3)	C(2)–N(1)–C(6)	121(3)
C(2)–C(3)	1.45(4)	N(1)–C(2)–C(3)	118(2)
C(3)–C(4)	1.28(4)	C(2)–C(3)–C(4)	121(3)
C(4)–C(5)	1.39(4)	C(3)–C(4)–C(5)	115(3)
C(5)–C(6)	1.34(4)	C(4)–C(5)–C(6)	123(3)
C(6)–N(1)	1.28(3)	C(5)–C(6)–N(1)	119(2)
C(1)–C(2)	1.60(3)	N(1)–C(2)–C(1)	111(2)
C(6)–C(7)	1.59(3)	C(3)–C(2)–C(1)	130(2)
C(1)–O(1)	1.12(3)	C(2)–C(1)–O(1)	111(2)
C(1)–O(2)	1.32(3)	C(2)–C(1)–O(2)	100(2)
C(7)–O(3)	1.24(3)	O(1)–C(1)–O(2)	141(2)
C(7)–O(4)	1.23(2)	N(1)–C(6)–C(7)	112(2)
N(1)–C(4)	2.73(4)	C(5)–C(6)–C(7)	129(2)
O(1)–O(2)	2.31(2)	C(6)–C(7)–O(3)	119(2)
O(3)–O(4)	2.19(2)	C(6)–C(7)–O(4)	115(2)
		O(3)–C(7)–O(4)	126(2)

C. Ligand No. 2

Distance		Angle	
N(2)–C(9)	1.30(3)	C(9)–N(2)–C(13)	117(2)
C(9)–C(10)	1.36(3)	N(2)–C(9)–C(10)	127(2)
C(10)–C(11)	1.43(4)	C(9)–C(10)–C(11)	114(2)
C(11)–C(12)	1.38(4)	C(10)–C(11)–C(12)	128(2)
C(12)–C(13)	1.46(3)	C(11)–C(12)–C(13)	140(2)
C(13)–N(2)	1.45(2)	C(12)–C(13)–N(2)	124(2)
C(8)–C(9)	1.57(2)	N(2)–C(9)–C(8)	118(2)
C(13)–C(14)	1.52(3)	C(10)–C(9)–C(8)	115(2)
C(8)–O(5)	1.37(2)	C(9)–C(8)–O(5)	111(2)
C(8)–O(6)	1.24(2)	C(9)–C(8)–O(6)	132(2)
C(14)–O(7)	1.20(2)	O(5)–C(8)–O(6)	115(2)
C(14)–O(8)	1.31(2)	N(2)–C(13)–C(14)	114(2)
N(2)–C(11)	2.75(3)	C(12)–C(13)–C(14)	122(2)
O(5)–O(6)	2.20(2)	C(13)–C(14)–O(7)	117(2)
O(7)–O(8)	2.19(2)	C(13)–C(14)–O(8)	121(2)
		O(7)–C(14)–O(8)	121(2)

Table 5. Continued.

D. Ligand No. 3

Distance		Angle	
N(3) – C(16)	1.28(2)	C(16) – N(3) – C(20)	120(2)
C(16) – C(17)	1.37(3)	N(3) – C(16) – C(17)	122(2)
C(17) – C(18)	1.49(3)	C(16) – C(17) – C(18)	120(2)
C(18) – C(19)	1.49(4)	C(17) – C(18) – C(19)	117(2)
C(19) – C(20)	1.36(3)	C(18) – C(19) – C(20)	111(2)
C(20) – N(3)	1.33(2)	C(19) – C(20) – N(3)	129(2)
C(15) – C(16)	1.46(3)	N(3) – C(16) – C(15)	119(2)
C(20) – C(21)	1.48(4)	C(17) – C(16) – C(15)	118(2)
C(15) – O(9)	1.39(4)	C(16) – C(15) – O(9)	117(3)
C(15) – O(10)	1.29(4)	C(16) – C(15) – O(10)	131(3)
C(21) – O(11)	1.32(3)	O(9) – C(15) – O(10)	111(2)
C(21) – O(12)	1.30(3)	N(3) – C(20) – C(21)	117(2)
N(3) – C(18)	2.78(3)	C(19) – C(20) – C(21)	113(2)
O(9) – O(10)	2.20(3)	C(20) – C(21) – O(11)	108(2)
O(11) – O(12)	2.30(2)	C(20) – C(21) – O(12)	126(2)
		O(11) – C(21) – O(12)	123(2)

E. The sodium coordination

Distance		Distance	
Na(1) – O(12^{v})	2.52(2)	Na(3) – O(18)	2.42(2)
Na(1) – O(13)	2.46(2)	Na(4) – O(2)	2.76(2)
Na(1) – O(14)	2.49(2)	Na(4) – O(4^{i})	2.43(2)
Na(2) – O(2)	2.41(2)	Na(4) – O(10^{iii})	2.37(2)
Na(2) – O(6^{ii})	2.36(2)	Na(4) – O(16)	2.45(2)
Na(2) – O(14)	2.51(2)	Na(4) – O(19)	2.18(3)
Na(2) – O(15)	2.44(2)	Na(4) – O(20)	2.32(2)
Na(2) – O(16)	2.37(2)	Na(1) – Na(2)	4.40(2)
Na(2) – O(17)	2.49(2)	Na(2) – Na(3)	4.56(2)
Na(3) – O(8)	2.50(2)	Na(2) – Na(4)	3.61(2)
Na(3) – O(17)	2.49(2)		

F. Possible hydrogen bond distances

Distance		Distance	
O(13) – O(5^{ii})	3.09(2)	O(17) – O(7)	2.99(2)
O(13) – O(6^{ii})	3.16(2)	O(18) – O(7)	2.97(3)
O(14) – O(5^{ii})	3.13(2)	O(18) – O(20)	2.97(3)
O(14) – O(19)	2.29(4)	O(18) – O(21)	3.08(4)
O(15) – O(1)	2.69(2)	O(20) – O(22^{iii})	2.81(3)
O(15) – O(12^{v})	2.71(3)	O(20) – O(22^{vii})	3.16(3)
O(16) – O(3^{ii})	2.65(2)	O(21) – O(11^{ii})	2.71(4)
O(16) – O(8^{vii})	2.92(2)	O(21) – O(12)	2.95(4)
O(17) – O(1)	3.19(2)	O(22) – O(9)	2.88(2)

Table 6. The deviations (in Å) from the least-squares planes through the seven carbon atoms and the nitrogen atom of each ligand.

Atom	Distance	Atom	Distance	Atom	Distance
N(1)	−0.05	N(2)	−0.06	N(3)	0.05
C(1)	−0.07	C(8)	0.09	C(15)	−0.08
C(2)	0.09	C(9)	−0.04	C(16)	0.08
C(3)	0.10	C(10)	0.06	C(17)	0.01
C(4)	−0.08	C(11)	0.00	C(18)	−0.08
C(5)	−0.04	C(12)	0.07	C(19)	0.05
C(6)	0.02	C(13)	−0.01	C(20)	0.04
C(7)	0.03	C(14)	0.01	C(21)	−0.08
O(1)	0.16	O(5)	0.21	O(9)	−0.10
O(2)	0.07	O(6)	−0.19	O(10)	−0.11
O(3)	−0.17	O(7)	−0.07	O(11)	0.29
O(4)	0.17	O(8)	0.11	O(12)	−0.13
Nd	−0.08	Nd	0.16	Nd	0.05

but the triangle N(1)N(2)N(3) is not. The twelve independent distances in the coordination polyhedron between adjacent atoms not belonging to the same ligand lie in the interval 2.98 – 3.35 Å, with an average of 3.08 Å.

The ligands. Each dipicolinate ion in NDP acts as a tridentate ligand forming two five-membered rings with the neodymium ion. The bond angles Nd–O–C and Nd–N–C lie in the intervals 120 – 132° and 118 – 123°, respectively. The bond distances and angles in the ligand are given in Table 5, B – D. The least-squares planes through the seven carbon atoms and the nitrogen atom of each ligand have been calculated. As shown in Table 6 these atoms are coplanar within 0.10, 0.09, and 0.08 Å for ligands 1, 2, and 3. The carboxylate groups appear to be more or less twisted out of the ligand planes. The neodymium ion is loacted rather near the ligand planes.

The packing of the complex ions. The large mononuclear tris(dipicolinato) complexes are well separated in NDP, all carbon – carbon packing distances except C(4) – C(8^{iii}) and C(5) – C(8^{iii}) being longer than 3.50 Å. C(4) – C(8^{iii}) is 3.30 ± 0.04 Å and C(5) – C(8^{iii}) is 3.40 ± 0.04 Å. The separation distance along the *b* axis between the layers of complex ions is about 3.8 Å. All neodymium – neodymium distances are very long. The shortest distances are Nd – Nd^{v} and Nd – Nd^{vi} along the *c* axis. They are 8.94 Å and 8.99 Å, respectively.

The coordination around the sodium ions. The sodium ions Na(1), Na(2), and Na(3) are located on or very near the line $x = 0$, $y = 1/2$. The octahedra of oxygen atoms around them form an infinite chain by sharing corners (Fig. 3). Na(1) and Na(2) are bridged by O(14), Na(2) and Na(3) by O(17). The sodium ions Na(4) are connected to this infinite chain by sharing the edges O(2)O(16) with Na(2). The sodium – oxygen bond distances and the sodium – sodium distances are given in Table 5 E. The bond distances except Na(4) – O(2) and Na(4) – O(19) are in the range 2.32 – 2.52 Å. Na(4) – O(2) is 2.76 Å and Na(4) – O(19) is 2.18 Å. The average sodium – oxygen bond distance in NDP is 2.45 Å.

The 36 different oxygen – oxygen "contact" distances along the edges of the octahedra are in the interval 2.89 – 4.21 Å. Three of these distances are less than 3.20 Å, *viz.*, O(2) – O(19) (2.89 ± 0.04 Å), O(13) – O(14) (3.12 ± 0.03 Å), and O(17) – O(18) (3.14 ± 0.03 Å). The O – Na – O bond angles with adjacent oxygen atoms have values between 71 and 110°.

Possible hydrogen bonds. According to the chemical analyses the asymmetric unit of NDP contains fifteen water molecules. As five out of these are not located in the structure determination only some of the possibilities of hydrogen bonding in NDP could be outlined. In Table 5 F the possible hydrogen bond distances less than 3.20 Å are given, and the most probable bond scheme among the known oxygen atoms is shown in Fig. 4. This choice is based upon considerations similar to those made in Ref. 2. Donor angles O···O(H_2O)···O between 84 and 115° with an average of 102° are accepted in this structure. The acceptor angles C – O···O(H_2O) are in the range 99 – 137°. The hydrogen bond distance O(14) – O(19) is extremely short, 2.29 Å. As the bond length Na(4) – O(19) and contact distance O(2) – O(19) also are short it seems reasonable to conclude that the coordinates obtained for the oxygen atom O(19) in the refinement of the structure most probably are erroneous. All the carboxylate oxygen atoms coordinated to the neodymium ion are also hydrogen bonded to a water molecule. The carboxylate oxygen O(7) which is coordinated to Nd is perhaps even bonded to two water oxygen atoms, *viz.*, O(17) and O(18). The angle O(17) – O(7) – O(18) is 64 ± 1°. In this connection one may observe that Nd – O(7) is the longest neodymiumoxygen bond distance in NDP (*cf.* Table 5 A).

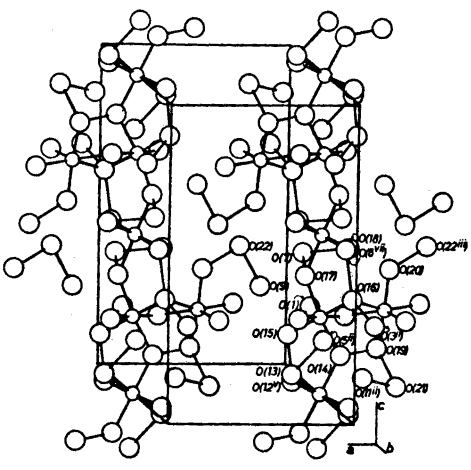

Fig. 4. A projection of NDP along *b** showing the layer around $y = 1/2$. The sodium – oxygen bonds are filled, hydrogen bonds are open. Only the most probable hydrogen bonds among those listed in Table 5 F are included.

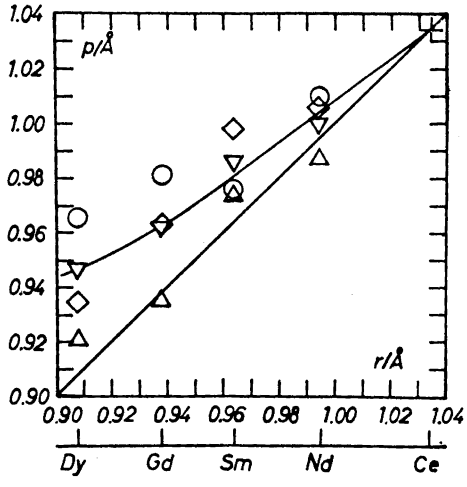

Fig. 5. Values of p of eqn. (1) with $q = a$ (○), b (△), $c/2$ (◇), and $(V/2)^{1/3}$ (▽) plotted *versus* the crystal radius r of the trivalent lanthanoid ions. The curves $p = (V/2)^{1/3}$ and $p = r$ are drawn.

The structure of NDP contains fairly large cavities around the lines $x = 1/2$, $z = 0$, and $x = 1/2$, $z = 1/2$ (Figs. 1, 3, and 4). In these cavities at least some of the ten missing water molecules per unit cell might be occluded. As the 2366 measured intensities should suffice to determine even their positions if they were ordered, they are assumed to be disordered.[11,2,3] These disordered water molecules most probably interact *via* hydrogen bonds with the rest of the structure.

The variation of the unit cell dimensions. In each unit cell of the triclinic Ce–Dy compounds there are two tris(dipicolinato) complexes stacked along the c axis. Hence, the decrease in the cell edges a and b between cerium and dysprosium may be compared with the decrease in $c/2$. This is done in Fig. 5 where the quantities

$$p(\mathrm{M}) = r(\mathrm{Ce}) + q(\mathrm{M}) - q(\mathrm{Ce}) \quad (1)$$

for the different triclinic lanthanoid dipicolinates represented by M are plotted *versus* the crystal radii for the lanthanoid ions.[12] In eqn. (1) q represents a, b, $c/2$, or $(V/Z)^{1/3}$. Due to the approximately parallel decrease in a, b, and $c/2$ it should be possible to use $(V/Z)^{1/3}$ as a "mean parameter" when the shrinking dimensions of the unit cell are compared with the lanthanoid contraction. Z is the number of lanthanoid ions per unit cell.

COMPARISON WITH PREVIOUS WORK

In this section the result of the present investigation of NDP are compared with those previously reported for the ytterbium dipicolinate compounds $Na_3[Yb(C_7H_2NO_4)_3].14H_2O$, ORTYBDIPIC,[1] $Na_3[Yb(C_7H_3NO_4)_3].13H_2O$, MONYBDIPIC,[2] and $Na_3[Yb(C_7H_3NO_4)_3].NaClO_4.10H_2O$, HEXYBDIPIC.[3] Due to the above mentioned similarity between the dipicolinate and oxydiacetate (or diglycolate) ions, it is convenient to include the lanthanoid oxydiacetate compounds $Na_3[M(C_4H_4O_5)_3].2NaClO_4.6H_2O$, M = Nd (NDG) and Yb (YDG), in the comparison.[5] The structures of the latter two compounds have been investigated using a single crystal diffractometer while the structures of the four dipicolinates are based on photographic intensity data.

All the six compounds contain mononuclear tris-complexes. These complexes are located in parallel layers alternating with layers containing the sodium ions and water molecules. The lanthanoid coordination polyhedra are distorted versions of the tri-capped trigonal prism, *i.e.*, the idealised ground geometry for nine-coordination.[13] The lanthanoid ions in HEXYBDIPIC, NDG, and YDG have the point symmetry **32** ($D3$). The distortion from the idealised geometry of the complexes is, in these cases, a rotation of the triangular faces of the prism relative each other. The point symmetry of the lanthanoid ions in NDP, ORTYBDIPIC, and MONYBDIPIC is approximately **32**. Besides the rotation, the triangular faces of the prism are slightly tilted in these latter compounds. The lanthanoid ion is in or very near the equatorial plane of all the six coordination polyhedra. Both ligands are approximately planar. The lanthanoid ion is also in or very near these planes except in MONYBDIPIC in which case the ytterbium ion deviates up to 0.6 Å from them.

Table 7 gives a survey of the coordination and "contact" distances in the six lanthanoid coordination polyhedra. The average decrease in both the M–O and M–N bond lengths between the neodymium and ytterbium dipicolinates is 0.14 Å. The corresponding decrease in the crystal radius of the lanthanoid ions is 0.137 Å. On the other hand the decrease in the M–O bond distances between NDG and YDG is 0.090 Å. The structures of the oxydiacetate compounds are more accurately determined than those of the dipicolinates. There is also a large spread in the M–O and M–N bond distances in the latter compounds. In view of these facts one should not yet rule out the possibility that the contraction of the lanthanoid coordination polyhedra in the dipicolinates is similar to the corresponding contraction in the oxydiacetates.

The coordination polyhedra in the ytterbium dipicolinate complexes appear to be not much affected by the different surroundings of the complex in the three structures. This corroborates further the assumption that the mononuclear tris(dipicolinato)- as well as the tris(oxydiacetato)lanthanoidate complexes are exposed only to weak interactions in their solid sodium salts and have almost the same structure there as in solution. The O–O and N–O distances in the ytterbium coordination polyhedra indicate van der Waals

Table 7. Coordination and "contact" distances (Å) in the lanthanoid coordination polyhedra in some sodium salts formed with the tris(dipicolinato)- and tris(oxydiacetato)lanthanoidate complexes.

Compound	M–O		M–N		O–O		O–N	
	range	average	range	average	range	average	range	average
NDP	2.37–2.61	2.49	2.57–2.59	2.58	2.99–3.06	3.01	3.00–3.35	3.15
ORTYBDIPIC [1]	2.33–2.34	2.33	2.33–2.41	2.38	2.87–2.92	2.89	2.76–2.84	2.80
MONYBDIPIC [2]	2.34–2.43	2.36	2.50–2.53	2.51	2.88–3.01	2.94	2.72–3.14	2.90
HEXYBDIPIC [3]	2.38	2.38	2.43	2.43	2.96	2.96	2.88	2.88
NDG [5]	2.428–2.523	2.476		–	3.026	3.026		–
YDG [5]	2.339–2.431	2.385		–	2.851–2.924	2.888		–

Table 8. A comparison of the bond distances (Å) in the dipicolinate ion obtained from some of its metal complexes.

Compound	C–N		C–C (pyridine)		C–C (carboxylate)		C–O	
	range	average	range	average	range	average	range	average
$Ca(C_7H_3NO_4).3H_2O$ [16]	1.33–1.34	1.33	1.37–1.40	1.39	1.50–1.51	1.50	1.24–1.26	1.25
$VO(C_7H_3NO_4).4H_2O$ [17]	1.29	1.29	1.39–1.42	1.40	1.53	1.53	1.24–1.26	1.25
$Ag(C_7H_4NO_4)_2.H_2O$ [18]	1.32–1.36	1.34	1.37–1.39	1.38	1.49–1.53	1.51	1.18–1.32	1.26
ORTYBDIPIC [1]	1.35–1.42	1.39	1.30–1.47	1.36	1.51–1.63	1.58	1.20–1.28	1.25
MONYBDIPIC [2]	1.23–1.41	1.32	1.32–1.52	1.41	1.44–1.57	1.53	1.23–1.32	1.28
HEXYBDIPIC [3]	1.34	1.34	1.37–1.40	1.39	1.49	1.49	1.23–1.25	1.24
NDP	1.28–1.45	1.32	1.28–1.49	1.40	1.48–1.60	1.54	1.12–1.39	1.27

contacts between these atoms.[14] As discussed before [1,5] such contacts should impose a steric hindrance for the formation of the third dipicolinate and oxydiacetate complexes with the heaviest lanthanoid ions. In solution such an increasing difficulty has been observed for the elements dysprosium through lutetium.[15]

In view of the composition of the dipicolinate ion, it would be expected to behave as a rigid body in its various compounds. In Table 8 the interatomic distances obtained for the dipicolinate ion in the investigated lanthanoid compounds are compared with those reported in the literature. Even if the variations between the individual "lanthanoid" distances are fairly large, the average values are in good agreement with the literature data.

It is the angles including the carboxylate atoms in the dipicolinate ion that may be changed. Small differences of the ligand conformation are actually found between the investigated compounds. The carboxylate groups are sometimes slightly twisted out of the plane of the pyridine ring and in some cases the C – COO groups are bent. Strahs and Dickerson [16] and Bersted *et al.*[17] report that the angles N – C – C(carboxylate) are significantly less than 120° due to attraction of the carboxylate group to the central ions of the complexes,[16] but in the lanthanoid compounds, with the possible exception of ORTYB-DIPIC, no clear-cut evidence for this is obtained. The carboxylate C – O distances are reported as equal within the limits of error with a value near 1.25 Å, except in $Ag(C_7H_4NO_4)_2.H_2O$ where the carboxylic acid hydrogens seem to cause a disparity of the C – O bond lengths.[18]

The N – O distances between the coordinated atoms in each dipicolinate ion (*i.e.*, the ligand bites) are not appreciably different in the four lanthanoid compounds. The average value is 2.60 Å. Due to the small changes of the ligand conformation the distances between the two coordinated carboxylate oxygens in each dipicolinate ion, on the other hand, seem to decrease somewhat between the neodymium and ytterbium compounds. The average distance is 4.41 Å in NDP and 4.28 Å in the ytterbium dipicolinates. A similar behaviour is found for the ligand in the oxydiacetate compounds. The O – O bites decrease only from 2.582 to 2.546 Å between NDG and YDG, while the distance between the two carboxylate oxygen atoms decreases from 4.320 to 4.222 Å.

The variation of the unit cell dimensions of the triclinic lanthanoid dipicolinates is illustrated in Fig. 5. The contraction between the cerium and dysprosium compounds described by the parameter $(V/Z)^{1/3}$ may be compared with the corresponding quantity for the lanthanoid oxydiacetates (*cf.* Ref. 5). Both series of compounds show the same decrease in $(V/Z)^{1/3}$, slightly less than the decrease in crystal radius. The oxydiacetates are isostructural for all lanthanoid ions. For the heaviest ions the decrease in $(V/Z)^{1/3}$ is appreciably smaller than the decrease in crystal radius. In Ref. 5 this trend in $(V/Z)^{1/3}$ through the lanthanoid series is given the following interpretation. The sodium coordination and hydrogen bonding in the oxydiacetates obstruct the contraction imposed on the structure by the shrinking complex ion. These counteracting forces are increased for the heaviest central ions by van der Waals repulsions between the oxygen atoms coordinated to the lanthanoid ions.

In the case of the lanthanoid dipicolinates there is a phase change at dysprosium. If sodium perchlorate is present in the mother liquor the hexagonal

HEXYBDIPIC phase is formed for the lanthanoids Ho – Lu. In absence of sodium perchlorate the monoclinic MONYBDIPIC phase is formed for the elements Ho – Yb and the orthorhombic ORTYBDIPIC phase for Yb and Lu. Within the limits of error, no contraction of the monoclinic unit cell is found between Ho and Tm, while the hexagonal unit cell dimensions decrease from Ho to Lu. Most probably there are van der Waals repulsions between the atoms coordinated to the lanthanoid ions in the tris(dipicolinato) complexes formed with the heaviest lanthanoids (*cf.* Refs. 1 – 3). In spite of this the actual contractions of the unit cells seem essentially to depend upon the sodium coordination and hydrogen bonding (including bonds with disordered water molecules) in the structures.

I am indebted to Drs. Karin Aurivillius, Ingmar Grenthe, and Eva Hansson for useful discussions and valuable suggestions. I also wish to thank Professor Sture Fronæus for his kind interest and for the facilities he has put at my disposal. This work is part of a research project supported by the *Swedish Natural Science Research Council.*

REFERENCES

1. Albertsson, J. *Acta Chem. Scand.* **24** (1970) 1213.
2. Albertsson, J. *Acta Chem. Scand.* **26** (1972) 985.
3. Albertsson, J. *Acta Chem. Scand.* **26** (1972) 1005.
4. Albertsson, J. *Acta Chem. Scand.* **22** (1968) 1563.
5. Albertsson, J. *Acta Chem. Scand.* **24** (1970) 3527.
6. *International Tables for X-Ray Crystallography*, 3rd Ed., Kynoch Press, Birmingham 1969, Vol. I.
7. *International Tables for X-Ray Crystallography*, 2nd Ed., Kynoch Press, Birmingham 1968, Vol. III.
8. Cromer, D. T., Larsson, A. C. and Waber, J. T. *Acta Cryst.* **17** (1964) 1044.
9. Werner, P.-E. *Arkiv Kemi* **31** (1970) 513.
10. Liminga, R. *Acta Chem. Scand.* **21** (1967) 1206.
11. Hansson, E. *Acta Chem. Scand.* **24** (1970) 2969.
12. Templeton, D. H. and Dauben, C. H. *J. Am. Chem. Soc.* **76** (1954) 5237.
13. Muetterties, E. L. and Wright, C. H. *Quart. Rev.* **21** (1967) 109.
14. Pauling, L. *The Nature of the Chemical Bond*, 3rd Ed., Cornell University Press, Ithaka 1960, p. 260.
15. Grenthe, I. and Hansson, E. *Acta Chem. Scand.* **23** (1969) 611.
16. Strahs, G. and Dickerson, R. E. *Acta Cryst.* **B 24** (1968) 571.
17. Bersted, B. H., Belford, R. L. and Paul, I. C. *Inorg. Chem.* **7** (1968) 1557.
18. Drew, M. G. B., Fowles, G. W. A., Matthews, R. W. and Walton, R. A. *J. Am. Chem. Soc.* **91** (1969) 7769.

Received June 25, 1971.