terminal nitrogen atom. In the present investigation the χ^1 values correspond to position II and they are 191·6°, 182·6° and 169·5° (Fig. 6(*b*), (*c*) and (*d*) respectively) and their average is 181·2°. This agrees well with the average of 184° given by Ramachandran and Lakshminarayanan.

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The Crystal Structures of Nitrates of Divalent Hexaquocations. III. Hexaquonickel nitrate

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The crystals of hexaquonickel nitrate, $Ni(OH_2)_6(NO_3)_2$, are triclinic, space group $P\bar{1}$ with the following unit-cell constants: $a = 7{\cdot}694$, $b = 11{\cdot}916$, $c = 5{\cdot}817$ Å; $\alpha = 102{\cdot}3$, $\beta = 102{\cdot}4$, $\gamma = 105{\cdot}9°$, $Z = 2$. The structure consists of hexaquonickel cations and nitrato anions joined to one another by a network of hydrogen bonds. The symmetry of the cation is nearly octahedral deformed to an orthorhombic bipyramid, with pairs of opposite distances Ni–OH_2 = 2·03, 2·07, 2·09 Å. The nitrato anions are not exactly trigonal; they present, in both crystallographically independent anions, two short bonds with average N–O = 1·23 Å and one long bond N–O = 1·28 Å. Not all the water molecules form equal hydrogen bonds, either in direction or in strength. The packing of the cation and anion units in the nickel compound cannot be reduced to the same scheme as the zinc and magnesium nitrates. On the whole there are significant differences compared with the structure of $Zn(OH_2)_6(NO_3)_2$ and $Mg(OH_2)_6(NO_3)_2$ in (i) environment of hexaquocation, (ii) structure of nitrato anions and (iii) packing.

Introduction

The crystals of nitrates of divalent hexaquocations $Zn(OH_2)_6(NO_3)_2$ (Ferrari, Braibanti, Manotti Lanfredi & Tiripicchio, 1967), $Mg(OH_2)_6(NO_3)_2$ (Braibanti, Tiripicchio, Manotti Lanfredi & Bigoli, 1969), $Ni(OH_2)_6(NO_3)_2$ (Jayaraman, 1957; Weigel, Imelik & Laffitte, 1962; Weigel, Imelik & Prettre, 1964) and $Co(OH_2)_6(NO_3)_2$ (Weigel, Imelik & Prettre, 1964) are not isostructural. This is rather surprising because the structures of these cations are generally assumed to be octahedral and of similar size. It is not clear yet if the differences are due either to different types of bonding of water molecules with the metal ion or to packing strains or to other causes such as the thermal energy state of the nitrato groups.

Experimental

Preparation

Crystals of the compound were obtained in the form of needles or elongated plates by evaporation of an aqueous solution at room temperature. The crystals are very often twinned. They are hygroscopic and had to be sealed in Lindemann capillary tubes for the crystal analysis.

Crystal data

Compound: hexaquonickel dinitrate, $Ni(OH_2)_6(NO_3)_2$; F.W. 290·82

Crystal class: triclinic, pinacoidal

Unit cell: (Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å) from rotation and Weissenberg photographs around [001],

$a = 7{\cdot}694$ (10), $b = 11{\cdot}916$ (12), $c = 5{\cdot}817$ (8) Å;

$\alpha = 102{\cdot}3$ (2), $\beta = 102{\cdot}4$ (1), $\gamma = 105{\cdot}9$ (3)°;

$V = 479{\cdot}5$ Å^3, $Z = 2$; $D_x = 2{\cdot}014$, $D_m = 2{\cdot}01$ g.cm^{-3};

μ(Cu $K\alpha$) = 17·56 cm^{-1}.

Space group: $P\bar{1}$ ($C_i(1)$, no. 2).

Intensity data

Integrated reflexions ($hk0$, ..., $hk5$) were measured by a microdensitometer on Weissenberg photographs. The intensities were corrected for absorption by assuming the crystal to be cylindrical ($\mu\bar{R} = 0{\cdot}49$ for l = 0,1,2,3,4 and $\mu\bar{R} = 0{\cdot}39$ for $l = 4,5$. Individual scale factors for each layer have been calculated as independent parameters. The calculations were performed on the Olivetti 6001/S computer of Centro di Calcolo Elettronico of the University of Parma.

Table 1. *Fractional atomic coordinates* ($\times 10^4$)

E.s.d.'s are given in parentheses.

	x	y	z
Ni	0900 (2)	2325 (2)	0606 (3)
$H_2O(1)$	0135 (12)	3655 (7)	2694 (18)
$H_2O(2)$	1112 (11)	1448 (8)	3330 (16)
$H_2O(3)$	1700 (11)	1038 (7)	−1467 (17)
$H_2O(4)$	0773 (11)	3149 (8)	−2240 (16)
$H_2O(5)$	3614 (9)	3496 (7)	2179 (16)
$H_2O(6)$	−1834 (9)	1262 (7)	−0969 (15)
O(1)	4696 (13)	8648 (11)	8845 (18)
O(2)	5323 (12)	8766 (8)	5422 (19)
O(3)	2611 (13)	8757 (10)	5844 (20)
O(4)	5386 (13)	6283 (9)	2804 (19)
O(5)	2587 (14)	6035 (8)	3158 (22)
O(6)	3106 (18)	5904 (14)	−0424 (23)
N(1)	4207 (12)	8730 (9)	6648 (19)
N(2)	3707 (12)	6081 (7)	1900 (17)

The structure has been solved by Patterson and Fourier maps and refined by differential syntheses (final $R = 10{\cdot}3$ %). The results of the structure refinement are quoted in Tables 1 to 6. The hydrogen atoms were tentatively located on a difference map, but could not be confirmed with certainty.

Table 3. *Probable coordinates and isotropic thermal parameters of hydrogen atoms*

	x	y	z	B
H(1)	−0980	3750	1583	2·0
H(2)	1088	4180	2750	2·0
H(3)	−0233	1283	3550	2·5
H(4)	2020	1350	4420	1·8
H(5)	3033	1153	−0950	1·7
H(6)	0700	0400	−2567	2·5
H(7)	−0353	2655	−3517	2·0
H(8)	1356	3861	−2483	2·0
H(9)	4000	3550	3917	1·7
H(10)	4704	3451	1817	1·7
H(11)	−2833	1417	−0167	2·0
H(12)	−2333	1167	−3000	2·3

Discussion of the structure

Hexaquonickel cations and nitrato anions, joined to one another by hydrogen bonds, form the network of the whole structure (Fig. 1). The coordination around the metal (Fig. 2) is only approximately octahedral; Ni–O distances in the hexaquocation range from 2·02 to 2·09 Å. By averaging bonds opposite to each other one obtains $Ni{-}O_{av} = 2{\cdot}03$, 2·07 and 2·09 Å, indicating that the octahedron is deformed, roughly speaking, to an orthorhombic bipyramid. The same pairs averaged in $Zn(OH_2)_6^{2+}$ are 2·06, 2·09, 2·13 Å again indicating an orthorhombic deformation of the octahedron, whereas in $Mg(OH_2)_6^{2+}$ they are 2·05, 2·06, 2·06 Å thus indicating no deformation of the octahedron. The two shortest pairs of distances in $Zn(OH_2)_6^{2+}$ and all of them in $Mg(OH_2)_6^{2+}$ seem to be correlated to a bonding of

Table 2. *Anisotropic thermal parameters** (Å^2)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	1·649	2·336	2·121	0·686	0·486	0·532
$H_2O(1)$	1·677	1·962	1·855	0·833	0·675	0·339
$H_2O(2)$	1·141	2·289	1·566	0·698	0·536	0·786
$H_2O(3)$	1·430	1·852	1·581	0·893	0·429	0·337
$H_2O(4)$	1·434	2·224	1·658	0·620	0·143	0·550
$H_2O(5)$	1·086	2·230	1·894	0·388	0·264	0·287
$H_2O(6)$	1·072	2·290	1·735	0·202	0·047	0·294
O(1)	1·968	4·103	2·206	1·440	0·565	1·053
O(2)	1·587	2·624	1·965	0·757	0·752	0·484
O(3)	1·350	3·652	1·792	0·939	0·278	0·631
O(4)	1·695	3·159	2·829	0·601	0·614	0·275
O(5)	2·373	2·531	2·450	0·617	0·993	0·460
O(6)	2·816	4·507	2·614	1·649	0·630	1·039
N(1)	0·909	1·547	1·439	0·359	0·108	0·306
N(2)	1·618	1·563	1·729	0·500	0·443	0·199

* In the last cycle for all the atoms the average shift, $|\Delta B_{ij}|_{av}$, and the maximum shift, $|\Delta B_{ij}|_{max}$, were:

$|\Delta B_{ij}|_{av} = 0{\cdot}044$

$|\Delta B_{ij}|_{max} = 0{\cdot}265$

the water molecules along the bisector of the lone pairs in the oxygen atom, because the hydrogen bonds radiating from water molecules lie in nearly the same plane as the metal–oxygen bond. This type of bonding corresponds to class 1D in the classification of Chidambaram, Sequeira & Sikka (1964). On the other hand the longest

Table 4. *Observed and calculated structure factors*

$10F_o$ values followed by – are not observed

h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$
1	0	0	152	201	2	6	0	38	13	1	13	0	143	130	8	2	1	53	-65	6	5	1	309	-332	7	8	1	165	-174	4	12	1	156	-144
2	0	0	255	306	2	6	0	175	161	2	13	0	122	115	8	2	1	100	99	6	5	1	63	-62	7	8	1	81	73	4	12	1	41	-40
3	0	0	187	-164	3	6	0	103	-103	3	13	0	31	17	8	2	1	61	50	7	5	1	5	81	8	8	1	64	65	5	12	1	150	-148
4	0	0	137	-148	3	6	0	188	211	4	13	0	129	-127	8	2	1	35	24	7	5	1	88	-102	8	8	1	63	52	5	12	1	96	-94
5	0	0	285	-306	4	6	0	59	49	5	13	0	152	-143	9	2	1	94	-88	7	5	1	75	-71	9	8	1	64	59	6	12	1	84	-81
6	0	0	197	-192	4	6	0	55	94	6	13	0	138	-132	9	2	1	10-	-70	7	5	1	114	-107	9	8	1	12-	100	6	12	1	43	24
7	0	0	126	-113	5	6	0	41	18	7	13	0	76	-77	0	3	1	151	115	8	5	1	64	-60	0	9	1	54	-51	7	12	1	76	84
8	0	0	29-	-9	5	6	0	320	332	0	14	0	15-	-13	0	3	1	175	-199	8	5	1	268	-252	0	9	1	394	406	7	12	1	99	95
9	0	0	24	33	6	6	0	90	78	1	14	0	84	88	1	3	1	101	105	9	5	1	32	-8	1	9	1	96	93	8	12	1	6-	16
0	1	0	101	96	8	6	0	109	108	2	14	0	151	138	1	3	1	291	-285	9	5	1	48	-36	1	9	1	103	112	0	13	1	127	149
1	1	0	118	-92	7	6	0	13-	28	3	14	0	118	106	1	3	1	468	-527	0	6	1	254	-255	1	9	1	422	417	0	13	1	20-	9
1	1	0	195	227	7	6	0	32-	25	4	14	0	71	66	1	3	1	80	89	0	6	1	346	-348	1	9	1	194	192	1	13	1	125	114
2	1	0	539	-631	8	6	0	19-	0	5	14	0	54	57	2	3	1	367	373	1	6	1	494	-489	2	9	1	92	-71	1	13	1	31	22
2	1	0	606	685	9	6	0	47	-36	6	14	0	16-	-18	2	3	1	834	-884	1	6	1	513	-482	2	9	1	28	21	1	13	1	48	46
3	1	0	1011	-1144	0	7	0	49	-36	3	15	0	12-	15	2	3	1	261	298	1	6	1	263	241	2	9	1	120	121	2	13	1	156	153
3	1	0	236	292	1	7	0	253	-239	4	15	0	10-	38	2	3	1	35	15	1	6	1	26	-5	2	9	1	91	98	2	13	1	72	72
4	1	0	310	-303	1	7	0	100	-110	0	0	1	437	496	3	3	1	392	382	2	6	1	154	-142	3	9	1	150	-139	2	13	1	37	26
4	1	0	291	321	2	7	0	89	86	1	0	1	181	195	3	3	1	657	-684	2	6	1	112	101	3	9	1	22	4	3	13	1	69	65
5	1	0	72	-51	2	7	0	436	-450	1	0	1	582	344	3	3	1	45	-71	2	6	1	251	251	3	9	1	30	-32	3	13	1	38	-25
5	1	0	26-	28	3	7	0	59	56	2	0	1	61	-42	3	3	1	435	488	2	6	1	291	-309	3	9	1	34	-14	4	13	1	41	-51
6	1	0	39	33	3	7	0	371	-370	2	0	1	131	105	4	3	1	151	161	3	6	1	115	-111	4	9	1	111	-111	4	13	1	90	-92
6	1	0	102	-95	4	7	0	92	94	3	0	1	79	-82	4	3	1	353	-361	3	6	1	61	52	4	9	1	111	108	5	13	1	57	-55
7	1	0	129	120	4	7	0	137	135	3	0	1	13-	10	4	3	1	49	-62	3	6	1	384	415	4	9	1	64	-52	5	13	1	127	-117
7	1	0	66-	69	5	7	0	57	47	4	0	1	488	-478	4	3	1	207	245	3	6	1	138	-150	4	9	1	167	-181	6	13	1	127	-143
8	1	0	161	144	5	7	0	29-	23	4	0	1	107	-122	5	3	1	66	54	4	6	1	147	162	5	9	1	64	-64	6	13	1	105	-99
8	1	0	270	-261	6	7	0	38	-29	5	0	1	32	-61	5	3	1	136	-132	4	6	1	224	221	5	9	1	136	-130	7	13	1	9-	-62
9	1	0	87	-89	6	7	0	120	137	5	0	1	304	-349	5	3	1	32	34	4	6	1	206	234	5	9	1	103	-97	7	13	1	12-	-2
0	2	0	510	-542	7	7	0	83	86	6	0	1	141	-134	5	3	1	39	63	4	6	1	33	-21	6	9	1	50-	52	0	14	1	15-	-20
1	2	0	520	-518	8	7	0	85	77	8	0	1	77	58	6	3	1	149	-142	5	6	1	129	124	6	9	1	139	-131	1	14	1	92	97
1	2	0	393	-433	9	7	0	33	20	7	0	1	52	-36	6	3	1	152	152	5	6	1	49	46	7	9	1	141	-146	1	14	1	9-	16
2	2	0	468	-518	0	8	0	502	538	7	0	1	273	-297	6	3	1	38	18	5	6	1	416	431	7	9	1	207	-205	2	14	1	97	128
2	2	0	138	-148	1	8	0	238	237	8	0	1	42	-32	6	3	1	36	32	5	6	1	23-	25	8	9	1	180	-184	2	14	1	42	35
3	2	0	116	102	1	8	0	303	-283	8	0	1	153	-155	7	3	1	49	-43	6	6	1	42	35	8	9	1	73	66	3	14	1	90	101
3	2	0	50	-35	2	8	0	128	126	9	0	1	41	33	7	3	1	23-	27	6	6	1	39	-19	9	9	1	102	-101	3	14	1	80	75
4	2	0	59	68	2	8	0	90	81	0	1	1	292	-329	7	3	1	111	116	6	6	1	103	95	9	9	1	54	-7	4	14	1	77	90
4	2	0	408	448	3	8	0	54	40	0	1	1	124	139	7	3	1	63	63	6	6	1	177	174	0	10	1	114	-115	4	14	1	77	64
5	2	0	360	359	3	8	0	317	-313	1	1	1	126	104	8	3	1	306	319	7	6	1	171	186	0	10	1	57	64	5	14	1	12-	1
5	2	0	98	125	4	8	0	61	63	1	1	1	154	114	8	3	1	187	168	7	6	1	60	-65	1	10	1	33	22	5	14	1	16-	16
6	2	0	267	272	4	8	0	107	-123	1	1	1	601	622	8	3	1	45	-33	7	6	1	44	41	1	10	1	76	68	6	14	1	41	-45
6	2	0	179	177	5	8	0	27-	-7	1	1	1	145	-175	9	3	1	166	155	8	6	1	27	-13	1	10	1	22-	4	1	15	1	7-	-63
7	2	0	205	196	5	8	0	87	-85	2	1	1	668	-736	9	3	1	47	53	8	6	1	224	-202	1	10	1	213	-222	2	15	1	12-	-22
7	2	0	83	84	6	8	0	11-	-14	2	1	1	417	428	0	4	1	445	463	9	6	1	31	-19	2	10	1	62	-59	3	15	1	64	74
8	2	0	55	47	6	8	0	69	-65	2	1	1	317	372	0	4	1	185	220	9	6	1	136	-131	2	10	1	68	-60	4	15	1	30	31
8	2	0	30-	-6	7	8	0	31-	19	2	1	1	106	97	1	4	1	372	386	0	7	1	207	-161	2	10	1	228	231	0	0	2	116	-118
9	2	0	88	-81	8	8	0	120	110	3	1	1	506	-511	1	4	1	455	451	0	7	1	207	-217	2	10	1	180	-176	1	0	2	31	-10
0	3	0	196	-200	9	8	0	75	75	3	1	1	49	17	1	4	1	124	97	1	7	1	139	114	3	10	1	17-	-12	1	0	2	224	186
1	3	0	446	458	0	9	0	255	240	3	1	1	97	136	1	4	1	58	-75	1	7	1	173	178	3	10	1	98	102	2	0	2	112	-118
1	3	0	539	-566	1	9	0	73	68	3	1	1	155	-221	2	4	1	310	292	1	7	1	358	-379	3	10	1	217	214	2	0	2	139	-92
2	3	0	216	216	1	9	0	236	226	4	1	1	207	-208	2	4	1	330	287	1	7	1	211	-225	3	10	1	190	-185	3	0	2	271	-275
2	3	0	67	-47	2	9	0	38	28	4	1	1	358	417	2	4	1	85	-109	2	7	1	157	147	4	10	1	120	125	3	0	2	18	42
3	3	0	446	469	2	9	0	131	119	4	1	1	176	173	2	4	1	271	323	2	7	1	52	-59	4	10	1	155	164	4	0	2	104	-106
3	3	0	298	281	3	9	0	175	-165	4	1	1	326	-376	3	4	1	139	-119	2	7	1	595	-622	4	10	1	150	-142	4	0	2	86	88
4	3	0	297	297	3	9	0	29-	16	5	1	1	43	29	3	4	1	159	-160	2	7	1	20-	23	5	10	1	236	247	5	0	2	234	-213
4	3	0	334	-365	4	9	0	177	-156	5	1	1	252	271	3	4	1	93	-147	3	7	1	165	166	5	10	1	37	-26	5	0	2	253	-243
5	3	0	208	199	4	9	0	56	44	5	1	1	67	55	3	4	1	284	298	3	7	1	135	-124	5	10	1	11-	-9	6	0	2	157	-141
5	3	0	26-	-23	5	9	0	64	-58	5	1	1	84	-102	4	4	1	43	-7	3	7	1	33	-36	6	10	1	165	162	6	0	2	26	-22
6	3	0	63	50	5	9	0	121	-123	6	1	1	154	150	4	4	1	284	-284	3	7	1	26	4	6	10	1	22-	-2	7	0	2	59	57
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9	3	0	146	130	1	10	0	106	-94	7	1	1	221	-217	5	4	1	68	48	5	7	1	78	-71	0	11	1	26	-3	0	1	2	359	422
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3	4	0	39	10	4	10	0	250	256	9	1	1	75	97	7	4	1	34	15	7	7	1	216	220	2	11	1	182	-179	2	1	2	427	426
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6	5	0	123	-131	3	12	0	105	-106	6	2	1	305	313	4	5	1	126	-128	4	8	1	117	112	1	12	1	35	-8	8	1	2	63	-62
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8	5	0	48	4	6	12	0	38	35	6	2	1	39	-18	5	5	1	45	40	5	8	1	22-	-33	2	12	1	63	-64	9	1	2	51	59
9	5	0	83	-76	7	12	0	66	76	7	2	1	133	120	5	5	1	265	-273	5	8	1	65	-61	2	12	1	74	61	0	2	2	253	237
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Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$
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1	2	2	350	-450	1	5	2	149	126	2	8	2	283	-317	1	12	2	18	-7	2	2	3	428	443	3	5	3	28	3	6	8	3	246	-273
2	2	2	27	-30	1	5	2	731	774	2	8	2	120	131	2	12	2	47	41	2	2	3	271	-337	3	5	3	30	2	6	8	3	93	81
2	2	2	744	-695	1	5	2	282	312	3	8	2	39	-25	2	12	2	44	-30	3	2	3	207	196	4	5	3	88	84	6	8	3	51	-46
2	2	2	284	304	2	5	2	227	-233	3	8	2	65	-71	2	12	2	58	58	3	2	3	391	-377	4	5	3	334	315	7	8	3	34	-9
2	2	2	179	148	2	5	2	368	350	3	8	2	275	-302	3	12	2	58	-47	3	2	3	339	308	4	5	3	116	-106	7	8	3	103	83
3	2	2	142	-108	2	5	2	83	100	3	8	2	82	90	3	12	2	30	-32	3	2	3	38	-81	4	5	3	103	-113	8	8	3	107	-115
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4	2	2	158	153	3	5	2	37	-8	4	8	2	165	183	5	12	2	53	-50	4	2	3	218	-225	5	5	3	105	-118	1	9	3	65	-64
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7	3	2	8-	-20	7	6	2	167	-143	2	10	2	37	-50	7	0	3	22	9	9	3	3	92	83	2	7	3	63	58	0	11	3	89	-83
7	3	2	123	125	7	6	2	198	218	2	10	2	199	207	8	0	3	168	-167	0	4	3	219	211	2	7	3	152	-145	1	11	3	47	-48
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8	3	2	25	-11	9	6	2	80	-85	3	10	2	234	244	0	1	3	310	374	1	4	3	197	197	3	7	3	138	134	2	11	3	118	-132
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1	4	2	95	-99	2	7	2	100	-107	6	10	2	76	73	2	1	3	46	53	3	4	3	57	-33	5	7	3	190	-197	4	11	3	20	2
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4	4	2	432	-441	5	7	2	176	-171	2	11	2	206	-203	5	1	3	400	-422	6	4	3	60	48	0	8	3	89	83	2	12	3	26	6
4	4	2	56	60	5	7	2	297	289	2	11	2	65	-59	6	1	3	115	98	6	4	3	69	57	0	8	3	44	45	3	12	3	36	-48
5	4	2	98	-97	5	7	2	235	251	2	11	2	49	-54	6	1	3	232	224	7	4	3	269	-288	1	8	3	93	110	3	12	3	135	-138
5	4	2	115	-145	6	7	2	138	135	3	11	2	95	-87	6	1	3	376	-327	7	4	3	85	58	1	8	3	185	196	3	12	3	54	57
5	4	2	77	-83	6	7	2	202	196	3	11	2	74	75	6	1	3	253	-264	7	4	3	220	-241	1	8	3	69	-81	4	12	3	19	-14
5	4	2	116	101	6	7	2	258	271	3	11	2	38	-32	7	1	3	35	25	8	4	3	35	-26	1	8	3	22	-21	4	12	3	43	22
6	4	2	15-	-4	7	7	2	142	151	4	11	2	86	-81	7	1	3	169	-155	8	4	3	113	-122	2	8	3	43	31	5	12	3	22	-26
6	4	2	381	-403	7	7	2	196	176	4	11	2	58	47	7	1	3	74	-67	9	4	3	44	-44	2	8	3	157	162	5	12	3	75	65
6	4	2	25	3	7	7	2	31	33	4	11	2	41	37	8	1	3	37	-34	0	5	3	30	3	2	8	3	149	-154	6	12	3	6-	-39
6	4	2	134	-143	8	7	2	275	289	5	11	2	46	53	8	1	3	78	78	0	5	3	417	445	2	8	3	86	93	0	13	3	102	94
7	4	2	62	-72	8	7	2	15-	-13	5	11	2	22	9	9	1	3	59	-46	1	5	3	197	-185	3	8	3	65	-80	0	13	3	103	95
7	4	2	34	35	9	7	2	88	94	6	11	2	25	-21	9	1	3	63	63	1	5	3	136	-116	3	8	3	76	76	1	13	3	29	11
7	4	2	125	-125	0	8	2	114	117	6	11	2	53	50	0	2	3	169	-173	1	5	3	407	435	3	8	3	35	20	1	13	3	165	180
8	4	2	70	-72	0	8	2	7	-25	7	11	2	39	37	0	2	3	22	-26	1	5	3	316	369	3	8	3	131	146	2	13	3	127	-131
8	4	2	253	233	1	8	2	84	84	7	11	2	15-	-7	1	2	3	25	12	2	5	3	99	-92	4	8	3	159	-172	2	13	3	101	109
8	4	2	71	-77	1	8	2	249	244	8	11	2	9-	-21	1	2	3	219	-247	2	5	3	39	36	4	8	3	43	-27	3	13	3	92	-76
9	4	2	31	24	1	8	2	128	-142	0	12	2	72	83	1	2	3	136	118	2	5	3	99	-92	5	8	3	68	77	4	13	3	153	-152
0	5	2	282	-285	1	8	2	129	142	0	12	2	50	-49	1	2	3	427	-444	2	5	3	91	104	5	8	3	235	-254	5	13	3	67	-65
0	5	2	393	425	2	8	2	147	137	1	12	2	19	-9	2	2	3	34	-10	3	5	3	180	-176	5	8	3	22	-3	6	13	3	10-	32

pair in $Zn(OH_2)_6^{2+}$ seems to indicate a bonding of water through one of the lone pairs of O, which corresponds to class 2*J* according to Chidambaram, Sequeira & Sikka (1964). No correlation can be found in the nickel compound between the nickel–oxygen bonds and the angles formed by the hydrogen bonds radiating from water molecules; therefore the bonding of water molecules to metal cannot be assigned to a definite class. On the other hand the uncertainties in the location of the hydrogen atoms do not allow the use of the angles

Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$
0	14	3	104	109
1	14	3	126	135
1	14	3	12-	51
2	14	3	47	45
3	14	3	49	48
4	14	3	80	-91
5	14	3	10-	-5
1	15	3	7-	68
2	15	3	10-	17
3	15	3	8-	65
0	0	4	29	3
1	0	4	139	-161
1	0	4	201	222
2	0	4	288	-318
2	0	4	590	665
3	0	4	36	37
3	0	4	282	281
4	0	4	151	-143
4	0	4	238	253
5	0	4	92	-83
5	0	4	58	55
6	0	4	26	21
6	0	4	54	-36
7	0	4	263	-230
8	0	4	43	-45
9	0	4	80	-80
0	1	4	408	-456
0	1	4	21	14
1	1	4	351	-422
1	1	4	138	-133
1	1	4	170	226
1	1	4	321	357
2	1	4	122	-133
2	1	4	202	-200
2	1	4	84	97
2	1	4	218	226
3	1	4	63	-51
3	1	4	142	127
3	1	4	85	91
3	1	4	23	-33
4	1	4	148	139
4	1	4	277	283
4	1	4	218	-206
4	1	4	188	-203
5	1	4	108	96
5	1	4	152	168
5	1	4	48	-18
5	1	4	409	-421
6	1	4	150	148
6	1	4	146	-139
6	1	4	228	-205
7	1	4	97	92
7	1	4	171	-142
8	1	4	31	-14
8	1	4	35	-13
9	1	4	33	-34
9	1	4	10-	28
0	2	4	78	-70
0	2	4	28	-4
1	2	4	40	71
1	2	4	197	-197
1	2	4	86	117
1	2	4	84	-154
2	2	4	147	175
2	2	4	371	-367
2	2	4	157	176
2	2	4	116	-112
3	2	4	228	245
3	2	4	250	-248
3	2	4	81	95
3	2	4	231	-289
4	2	4	235	224
4	2	4	155	-155
4	2	4	44	34
4	2	4	194	-214
5	2	4	33	30
5	2	4	49	45
5	2	4	69	60
5	2	4	173	-164
6	2	4	107	106
6	2	4	113	-100
8	2	4	26-	9
7	2	4	156	137
7	2	4	125	110
8	2	4	138	123
8	2	4	168	131
9	2	4	74	65
0	3	4	352	397
0	3	4	38	16
1	3	4	219	239
1	3	4	71	71
1	3	4	38	-23
1	3	4	271	-292
2	3	4	160	165
2	3	4	43	-18
2	3	4	31	3
2	3	4	14-	-10
3	3	4	51	-37
3	3	4	157	-139
3	3	4	108	111
3	3	4	22	8
4	3	4	48	49
4	3	4	124	-120
4	3	4	46	2
4	3	4	36	9
5	3	4	146	-165
5	3	4	40	-42
5	3	4	166	156
5	3	4	234	209
6	3	4	182	-195
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6	3	4	242	212
7	3	4	33	-27
7	3	4	144	125
8	3	4	31	36
8	3	4	80	58
0	4	4	137	135
0	4	4	61	-70
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1	4	4	126	118
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2	4	4	66	-89
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3	4	4	257	254
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6	4	4	59	-61
7	4	4	55	-85
7	4	4	6-	55
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8	4	4	245	-215
8	4	4	159	-146
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1	5	4	80	116
2	5	4	97	-107
2	5	4	87	82
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2	5	4	25	-12
3	5	4	45	-35
3	5	4	49	46
3	5	4	191	-193
3	5	4	37	55
4	5	4	12	-14
4	5	4	154	146
4	5	4	33	-19
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5	5	4	232	230
5	5	4	218	-196
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6	5	4	99	95
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6	5	4	85	-68
7	5	4	35	20
7	5	4	17	-39
7	5	4	56	-57
8	5	4	38	-28
8	5	4	75	-81
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1	6	4	40	19
1	6	4	135	-133
1	6	4	212	208
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2	6	4	98	-103
2	6	4	172	169
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4	6	4	71	62
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5	6	4	56	-35
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7	6	4	84	-118
7	6	4	26	-10
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1	7	4	56	58
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3	7	4	102	100
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6	7	4	45	23
7	7	4	63	-56
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7	8	4	54	-65
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6	9	4	121	-121
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3	10	4	38	-43
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3	10	4	94	-118
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5	10	4	167	-142
5	10	4	102	-129
6	10	4	53	83
6	10	4	192	-181
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2	11	4	205	183
2	11	4	96	-117
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5	11	4	180	181
6	11	4	14-	16
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5	12	4	73	63
0	13	4	45	49
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3	13	4	161	-152
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1	14	4	82	81
2	14	4	20	24
3	14	4	14	-17
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4	0	5	60	-58
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3	1	5	37	-11
3	1	5	37-	-19
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5	1	5	122	102
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7	1	5	46	-8
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8	1	5	45	-9
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1	2	5	97	115
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2	2	5	88	115
2	2	5	164	-123
2	2	5	34-	85
3	2	5	31	17
3	2	5	37-	37
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6	2	5	52	-79
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7	3	5	194	-184
7	3	5	19-	0
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5	4	5	53	64
5	4	5	221	172
6	4	5	68	-60
6	4	5	70	56
7	4	5	166	-154
7	4	5	14-	-9
8	4	5	207	-191
8	4	5	115	-119
9	4	5	112	134
0	5	5	65	-68
0	5	5	175	-183
1	5	5	34	20
1	5	5	62	73
2	5	5	87	-95
2	5	5	69	19
3	5	5	60	-72
3	5	5	195	220
3	5	5	71	51
4	5	5	179	-203
4	5	5	69	59
4	5	5	196	172
5	5	5	224	-224
5	5	5	47	-40
5	5	5	264	244
6	5	5	72	-79
6	5	5	82	-69
6	5	5	212	205
6	5	5	56	-22
7	5	5	51	-44
7	5	5	148	-117
8	5	5	29	12
0	6	5	45	-57
0	6	5	165	203
1	6	5	25	14
1	6	5	103	-107
1	6	5	179	203
1	6	5	115	123
2	6	5	218	-217
2	6	5	212	226
2	6	5	104	-108
3	6	5	256	-249
3	6	5	75	99
3	6	5	50	17
4	6	5	41	37
4	6	5	39	-21
4	6	5	194	-192
5	6	5	43	-16
5	6	5	56	-64
5	6	5	134	-98
6	6	5	71	58
6	6	5	118	-80
7	6	5	29	6
7	6	5	14-	40
0	7	5	95	108
0	7	5	194	-217
1	7	5	64	85
1	7	5	62	72
1	7	5	176	-193
2	7	5	59	-61
2	7	5	101	130
2	7	5	141	-144
3	7	5	17-	10
3	7	5	175	192
3	7	5	122	-106
4	7	5	80	-72
4	7	5	224	259
4	7	5	140	-123
5	7	5	92	-94
5	7	5	55	52
5	7	5	93	73
6	7	5	55	-69
6	7	5	30	11
7	7	5	23	20
0	8	5	227	-269
1	8	5	78	83
1	8	5	184	-224
1	8	5	106	-101
2	8	5	61	81
2	8	5	123	-155
2	8	5	115	-119
3	8	5	71	98
3	8	5	29-	-16
3	8	5	237	236
4	8	5	49	-55
4	8	5	56	50
4	8	5	112	99
5	8	5	49	65
5	8	5	59	83
5	8	5	83	62
6	8	5	54	-69
6	8	5	13-	70
0	9	5	68	17
1	9	5	45	-65
1	9	5	175	185
2	9	5	90	-106
2	9	5	160	153
3	9	5	11-	56
3	9	5	69	-82
3	9	5	198	176
4	9	5	16-	17
4	9	5	53	-36
4	9	5	135	114
5	9	5	64	-84
5	9	5	23-	19
0	10	5	109	122
1	10	5	103	108
1	10	5	53	45
2	10	5	29-	14
2	10	5	97	93
3	10	5	48	15
3	10	5	113	-89
4	10	5	45	-36
4	10	5	91	-59
5	10	5	70	-89
0	11	5	51	40
1	11	5	26-	-7
1	11	5	36-	-22
2	11	5	62	64
2	11	5	114	-107
3	11	5	33-	29
3	11	5	135	-125
4	11	5	27-	1
0	12	5	40	10
1	12	5	33-	-29
2	12	5	60	-61
2	12	5	30-	-20
3	12	5	43	42
3	12	5	27-	7
4	12	5	14-	24
1	13	5	33	-35
2	13	5	22-	17
3	13	5	11-	-14
2	13	5	22-	-53

involving hydrogen atoms with confidence. The distances of oxygen to hydrogen in water molecules range from 0·81 to 1·13 Å and the angles H–O–H from 98·5 to 131·3°. Almost all the hydrogen bonds formed by water molecules are directed toward the nitrato groups (Fig. 3). Four water molecules, namely $H_2O(1)$, $H_2O(2)$, $H_2O(5)$ and $H_2O(6)$, each form two hydrogen bonds; $H_2O(3)$ forms one strong hydrogen bond (2·74 Å) with an NO_3^- anion and one weak hydrogen bond (2·96 Å) with a water molecule of another cation. There are three possible hydrogen bonds (2·95, 2·95 and 2·99 Å) formed by $H_2O(4)$; two of them could be due to a bifurcated hydrogen bond. Different interpretations of them, however, are equally acceptable, for instance if one of the distances is not considered a hydrogen bond or if a statistical distribution of the water molecule is assumed between two possible orientations.

The two nitrato anions (Fig. 4) which are crystallographically independent show very similar features. The groups are planar, as shown by the sums of the angles around N(1) and N(2), but deviate significantly from trigonal symmetry. There are two short N–O bonds, averaging 1·23 Å in both independent anions and one long bond N–O = 1·28 Å in both anions. These results are in contrast with those obtained for hexaquomagnesium nitrate where there is one short bond N–O= 1·196 (12) Å and two long bonds 1·257 (9) Å. In the zinc compound the distances, although statistically less

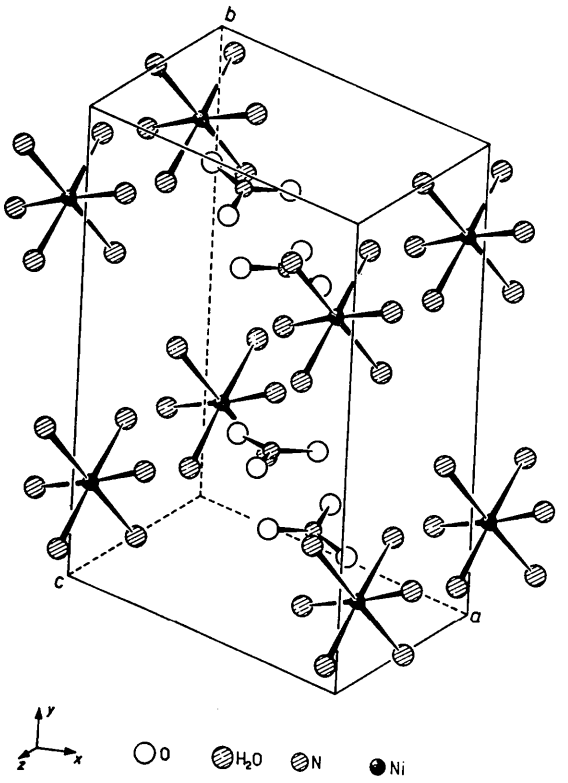

Fig. 1. Clinographic projection of the structure of $Ni(OH_2)_6(NO_3)_2$.

significant, are closer to those in the magnesium than to those in the nickel compound. Even the angles O–N–O in each nitrato group of $Ni(OH_2)_6(NO_3)_2$ are significantly different; they differ also from those of the anions in the other hexaquonitrates. The differences in the anions are not related to the number or strength of the hydrogen bonds of each oxygen (Fig. 5). The packing of cation and anion units in the structure (Fig. 6) cannot be reduced to the same scheme as those of the analogous nitrates previously studied.

Altogether the structure of the nickel compound, we have now determined, is different from those of the compounds of zinc and magnesium for (i) environment of the hexaquocation, (ii) structure of nitrato groups, and (iii) packing. It is worth mentioning, however, that the nickel compound presents (Jaffray & Rodier, 1955)

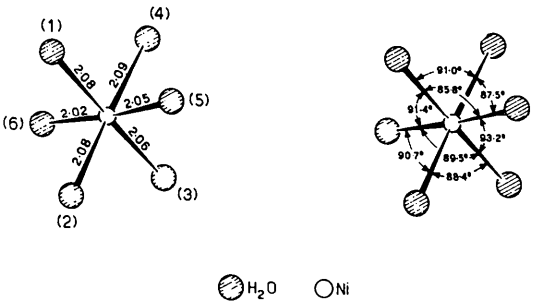

Fig. 2. Hexaquonickel cation, $Ni(OH_2)_6^{2+}$. Bond lengths are in Å.

Table 5. *Atomic peak heights* (e.Å^{-3}), *curvatures* (e.Å^{-5}), *and e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{hk}	A_{hl}	A_{kl}
Ni	obs	72·8	820	752	600	235	127	117
	calc	75·0	822	762	610	236	130	120
$H_2O(1)$	obs	16·2	175	167	125	58	36	20
	calc	16·5	176	169	127	57	35	21
$H_2O(2)$	obs	16·8	176	169	140	57	22	37
	calc	16·9	175	173	139	57	23	35
$H_2O(3)$	obs	16·6	183	171	130	60	29	25
	calc	17·0	182	173	134	59	31	26
$H_2O(4)$	obs	16·3	166	169	137	52	22	35
	calc	16·5	167	173	137	53	24	34
$H_2O(5)$	obs	15·8	179	139	118	37	24	20
	calc	16·1	178	143	120	38	24	21
$H_2O(6)$	obs	15·8	177	129	119	30	23	18
	calc	15·9	175	133	120	32	24	19
O(1)	obs	14·2	148	111	122	41	18	27
	calc	14·2	146	117	121	39	19	27
O(2)	obs	15·2	172	131	120	43	40	21
	calc	15·4	171	135	121	43	38	23
O(3)	obs	14·4	160	118	108	46	24	20
	calc	14·7	158	124	108	46	25	21
O(4)	obs	13·6	140	101	97	36	23	7
	calc	13·7	139	106	99	36	22	9
O(5)	obs	14·0	144	121	105	36	35	17
	calc	14·1	145	125	106	38	33	19
O(6)	obs	12·5	120	102	102	43	14	21
	calc	12·7	120	105	102	42	16	21
N(1)	obs	14·5	167	151	118	58	16	28
	calc	14·7	166	152	121	56	18	29
N(2)	obs	13·6	142	131	113	35	23	17
	calc	13·9	144	132	115	34	23	19
e.s.d.'s		0·3	4	3	3	2	2	2

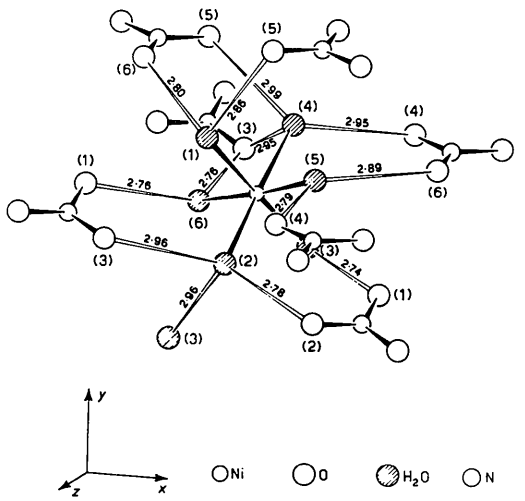

Fig. 3. Hydrogen bonds radiating from water molecules of the hexaquonickel cation. Bond lengths are in Å.

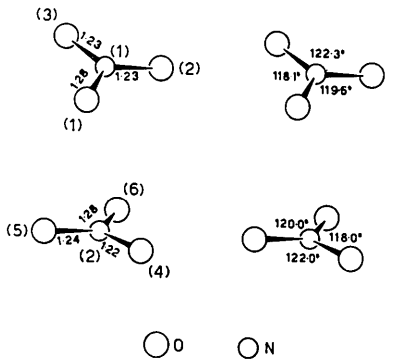

Fig. 4. Nitratio anions. Bond lengths are in Å.

five transitions, three of which should be second order and two polymorphic transitions. This means that with a fixed central atom there exist several possible structures, differing slightly in energy, which can be trans-

Table 6. *Main interatomic distances and angles*

E.s.d.'s are given in parentheses.

Hexaquonickel cation			
Ni——$H_2O(1)$	2·082 (11) Å	$H_2O(1)$–Ni—$H_2O(2)$	92·3 (4)°
Ni——$H_2O(2)$	2·078 (11)	$H_2O(1)$–Ni—$H_2O(3)$	178·6 (3)
Ni——$H_2O(3)$	2·058 (11)	$H_2O(1)$–Ni—$H_2O(4)$	91·0 (4)
Ni——$H_2O(4)$	2·095 (11)	$H_2O(1)$–Ni—$H_2O(5)$	85·8 (4)
Ni——$H_2O(5)$	2·045 (12)	$H_2O(1)$–Ni—$H_2O(6)$	91·4 (4)
Ni——$H_2O(6)$	2·021 (11)	$H_2O(2)$–Ni—$H_2O(3)$	88·4 (4)
		$H_2O(2)$–Ni—$H_2O(4)$	176·5 (4)
		$H_2O(2)$–Ni—$H_2O(5)$	91·9 (4)
		$H_2O(2)$–Ni—$H_2O(6)$	90·7 (3)
		$H_2O(3)$–Ni—$H_2O(4)$	88·3 (4)
		$H_2O(3)$–Ni—$H_2O(5)$	93·2 (3)
		$H_2O(3)$–Ni—$H_2O(6)$	89·5 (3)
		$H_2O(4)$–Ni—$H_2O(5)$	87·5 (3)
		$H_2O(4)$–Ni—$H_2O(6)$	90·1 (3)
		$H_2O(5)$–Ni—$H_2O(6)$	176·2 (3)
Nitrato anions			
N(1)——O(1)	1·285 (15) Å	O(1)——N(1)–O(2)	119·6 (9)
N(1)——O(2)	1·226 (15)	O(1)——N(1)–O(3)	118·1 (9)
N(1)——O(3)	1·229 (15)	O(2)——N(1)–O(3)	122·3 (9)
N(2)——O(4)	1·220 (15)	O(4)——N(2)–O(5)	122·0 (9)
N(2)——O(5)	1·242 (15)	O(4)——N(2)–O(6)	118·0 (9)
N(2)——O(6)	1·282 (16)	O(5)——N(2)–O(6)	120·0 (9)
Hydrogen bonds			
$H_2O(1)$–$O(5)$	2·862 (18) Å		
$H_2O(1)$–$O(6^{i})$	2·800 (20)		
$H_2O(2)$–$O(3^{ii})$	2·958 (21)		
$H_2O(2)$–$O(2^{iii})$	2·779 (18)		
$H_2O(2)$–$H_2O(3^{iv})$	2·955 (17)		
$H_2O(3)$–$O(1^{iii})$	2·744 (17)		
$H_2O(4)$–$O(3^{i})$	2·953 (16)		
$H_2O(4)$–$O(4^{v})$	2·951 (21)		
$H_2O(4)$–$O(5^{i})$	2·987 (18)		
$H_2O(5)$–$O(4^{iii})$	2·789 (15)		
$H_2O(5)$–$O(6^{v})$	2·889 (21)		
$H_2O(6)$–$O(1^{ii})$	2·762 (17)		
$H_2O(6)$–$O(3^{i})$	2·764 (15)		

Asymmetric units

i	$-x$,	$1-y$,	$-z$
ii	$-x$,	$1-y$,	$1-z$
iii	$1-x$,	$1-y$,	$1-z$
iv	$-x$,	$-y$,	$-z$
v	$1-x$,	$1-y$,	$-z$

formed into one another by changing the temperature. By lowering or increasing the temperature one should cause differences in the energy state of the nitrato groups or of the water molecules in a way similar to that operated by changing the central metal atom.

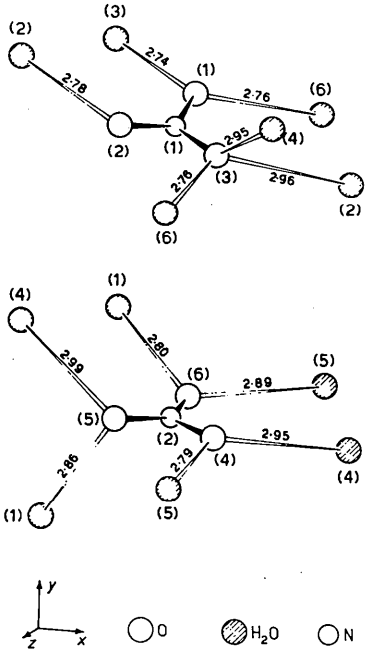

Fig. 5. Hydrogen bonds pointing towards oxygen atoms of nitrato anions.

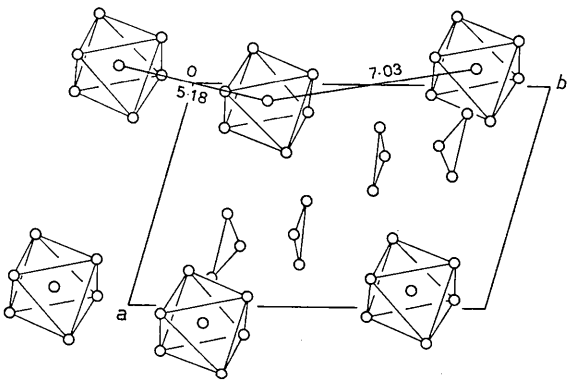

Fig. 6. Packing of cations and anions, viewed down [001].

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The Structure of Phragmalin : An X-ray Analysis of Phragmalin Iodoacetate

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The molecular structure of phragmalin, the methyl ester of a $C_{28}H_{34}O_{11}$ terpenoid constituent of the tree *Entandrophragma caudatum* (a member of the Meliaceae family), has been determined by means of an X-ray study of phragmalin iodoacetate. The crystals of phragmalin iodoacetate are orthorhombic with space group $P2_12_12_1$ and 4 molecules in the unit cell of dimensions $a = 15{\cdot}70$, $b = 19{\cdot}59$ and $c = 11{\cdot}25$ Å. An orthoester linkage spanning a cyclohexane ring results in an atomic arrangement which is unique for this class of natural product.

Introduction

The major alkaloid isolated from the tree *Entandrophragma caudatum* of the plant family Meliaceae was found to be an ester of nicotinic acid (Baarschers, 1967). On alkaline hydrolysis of the *Entandrophragma* alkaloid, the products isobutyric acid and nicotinic acid were readily identified. A larger fragment, the acid $C_{28}H_{34}O_{11}$, was converted into its methyl ester which was subsequently named 'phragmalin' (Baarschers, 1967).

Several compounds showing a marked resemblance to limonin (II) (Arnott, Davie, Robertson, Sim & Watson, 1961) have in recent years been found to occur