the final refinement are assumed to be the correct ones. The value N is defined as the percentage of correct signs minus the percentage of incorrect signs.

Conclusion

This work shows that despite smallness of the change in scattering factor caused by anomalous dispersion and unavoidable inaccuracy in the observations caused by background radiation, this effect may be used for phase determination, even with visually estimated data.

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The Crystal Structure of bis-(*N*-isopropylsalicylaldiminato)nickel(II)

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The crystal structure of bis-(*N*-isopropylsalicylaldiminato)nickel(II), $[OC_6H_4CHNC_3H_7]_2Ni$, has been determined from three-dimensional X-ray diffraction data with Fe $K\alpha$ radiation. Cell dimensions are $a=13{\cdot}219$, $b=19{\cdot}697$, $c=15{\cdot}140$ Å, $Z=8$. Space group, *Pbca*.

The structure consists of discrete molecules in which nickel(II) exhibits a tetrahedral coordination configuration.

Introduction

The structures of coordination compounds of nickel(II) are of considerable interest because the ion is found to adopt several different coordination configurations: planar (*e.g.* Stewart & Lingafelter, 1959), octahedral (*e.g.* Stewart, Lingafelter & Breazeale, 1961), and tetrahedral (*e.g.* Venanzi, 1958). Evidence has recently been given (Sacconi, Orioli, Paoletti & Ciampolini, 1962; Sacconi, Paoletti & Ciampolini, 1963) which indicates that the nickel(II) ion has a tetrahedral coordination configuration in bis-(*N*-isopropylsalicylaldiminato)nickel(II) $[OC_6H_4CHNC_3H_7]_2Ni$. We have now completed (Fox, Lingafelter, Orioli & Sacconi, 1963) the three-dimensional X-ray diffraction investigation of the structure.

Experimental

Two batches of bis-(*N*-isopropylsalicylaldiminato)-nickel(II) were used and found to be identical. One batch was prepared at the University of Florence by the reaction between bis(salicylaldehydato)nickel(II) and isopropylamine in methanol solution. The other batch was prepared by Dr H. F. Bauer at the University of California at Los Angeles by the reaction between nickel(II) chloride and the Schiff base *N*-isopropylsalicylaldimine in aqueous ethanol.

Cell dimensions were determined from a rotation and a zero-level Weissenberg photograph taken about the c axis with Fe $K\alpha$ radiation ($\lambda=1{\cdot}9373$ Å). For calibration of the films the rotation pattern of NaCl ($a_0=5{\cdot}6387$ Å) was superimposed on each diffraction pattern. The cell dimensions are:

$$a=13{\cdot}219\pm0{\cdot}006,\ b=19{\cdot}697\pm0{\cdot}008\ ,$$
$$c=15{\cdot}140\pm0{\cdot}018\ \text{Å}\ .$$

The cell contains eight molecules; observed density, 1·31 g.cm^{-3}, calculated 1·32 g.cm^{-3}. Systematic absences of $0kl$ for k odd, $h0l$ for l odd, and $hk0$ for h odd indicate the space group to be *Pbca*.

The crystal chosen for collection of intensities was a needle having dimensions $0{\cdot}22\times0{\cdot}14\times1{\cdot}22$ mm. Equiinclination Weissenberg photographs, *hk*0 through *hk*6, were taken on a Nonius integrating Weissenberg camera with Fe $K\alpha$ radiation. In order to obtain a satisfactory range of intensities, multiple films and a range of exposure times were used. Camera integration was in one direction only, and each diffraction spot in the linear response range of each film was then scanned normal to the direction of camera integration with a Moll type densitometer feeding into a Leeds & Northrup amplifier and recorder having a logarithmic slide wire. The areas under the densitometer tracings were measured with a planimeter and used as relative intensities. The several films for a given level were put on the same scale by comparing common spots. The seven levels were then scaled together using intensities from 0*kl* and *h*0*l* zero-level integrating Weissenberg photographs.

1282 unique reflections were examined, representing 56% of the reflections within the limiting (Fe) sphere. Of these, 979 had measurable intensities and 303 were too faint to be measured. The range of measured intensities is 1 to 4500.

Lorentz and polarization factors were applied and relative structure factors calculated with a data reduction program written for the IBM 709. No correction was made for absorption.

All calculations were carried out on an IBM 709 with a set of programs written (or adapted) at the University of Washington. The atomic scattering factors used were those of Viervoll & Øgrim (1949) for nickel, those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen, nitrogen and carbon, and those of McWeeny (1951) for hydrogen. No correction was made for anomalous dispersion.

Structure determination

Since there are eight molecules in the cell of space group *Pbca*, no atoms are required to be in special positions. Intensity data from the three axial zones were used to evaluate the three Patterson projections, $P(u, v)$, $P(v, w)$, and $P(u, w)$. The positional parameters of the nickel atom in the 8-fold general position were readily obtained from these projections. A three-dimensional Fourier synthesis was then calculated, using phases determined by the nickel atom only. This Fourier synthesis showed a number of peaks from which twelve (in addition to the large nickel peak) were selected to determine coordinates for a second cycle. (Three of this twelve were later found to be spurious.) A series of four Fourier syntheses, interpreted with the aid of a model, enabled the positions of all 25 non-hydrogen atoms to be determined, and resulted in a reduction of R to 20·8%. R is defined throughout as $\Sigma||F_o|-|F_c||/\Sigma|F_o|$, where the sums are over the unique, observed reflections only. A three-dimensional ΔF synthesis at this point gave no further significant improvement.

Further refinement was carried out using the full-matrix least-squares program of Busing & Levy (1959). The eight reflections, (020), (220), (112), (212), (023),

Table 1. *Positional parameters and their e.s.d.'s*

Atom	x/a	$\sigma_x \times 10^5$	y/b	$\sigma_y \times 10^5$	z/c	$\sigma_z \times 10^5$
C(1)	−0·0505	46	0·3012	32	0·2202	70
C(2)	−0·0491	49	0·3672	34	0·2555	74
C(3)	−0·1335	53	0·3956	36	0·2970	70
C(4)	−0·2220	53	0·3582	36	0·3052	69
C(5)	−0·2273	49	0·2932	32	0·2717	70
C(6)	−0·1433	42	0·2619	29	0·2289	60
C(7)	−0·1541	46	0·1940	31	0·1987	62
C(8)	−0·1182	55	0·0889	32	0·1337	80
C(9)	−0·1072	69	0·0773	33	0·0347	95
C(10)	−0·0541	57	0·0371	36	0·1819	75
C(11)	0·2106	45	0·1019	30	0·0859	79
C(12)	0·2785	52	0·0485	33	0·1142	66
C(13)	0·3441	53	0·0159	37	0·0545	87
C(14)	0·3435	55	0·0336	36	−0·0348	90
C(15)	0·2801	57	0·0831	35	−0·0676	71
C(16)	0·2125	42	0·1168	30	−0·0059	76
C(17)	0·1510	53	0·1720	30	−0·0451	69
C(18)	0·0396	54	0·2638	34	−0·0567	70
C(19)	0·0949	75	0·3302	38	−0·0327	84
C(20)	−0·0757	57	0·2701	37	−0·0392	74
N(1)	−0·0884	36	0·1587	24	0·1545	50
N(2)	0·0844	40	0·2077	24	−0·0019	55
O(1)	0·0300	29	0·2771	22	0·1822	43
O(2)	0·1525	30	0·1303	22	0·1439	45
Ni	0·0476	7	0·1939	5	0·1216	11

Shifts $\times 10^5$ during final cycle

$\bar{\delta}$		11		9		33
$\delta_{\max}$		23		36		75

Table 2. *Thermal parameters*

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·00415	0·00292	0·00378	0·00021	−0·00061	−0·00055
C(2)	0·00528	0·00339	0·00346	−0·00025	−0·00121	−0·00076
C(3)	0·00592	0·00346	0·00514	0·00047	−0·00014	−0·00232
C(4)	0·00612	0·00347	0·00419	0·00110	0·00063	−0·00118
C(5)	0·00461	0·00299	0·00514	0·00035	0·00056	−0·00008
C(6)	0·00394	0·00265	0·00162	−0·00008	−0·00025	−0·00007
C(7)	0·00385	0·00283	0·00216	−0·00019	−0·00054	−0·00008
C(8)	0·00704	0·00225	0·00355	−0·00056	0·00099	−0·00113
C(9)	0·01134	0·00211	0·00923	−0·00161	0·00034	−0·00141
C(10)	0·00816	0·00303	0·00595	0·00094	0·00135	0·00092
C(11)	0·00403	0·00223	0·00300	−0·00074	−0·00080	0·00043
C(12)	0·00530	0·00342	0·00155	−0·00041	−0·00019	0·00052
C(13)	0·00534	0·00305	0·00932	0·00035	0·00020	−0·00013
C(14)	0·00598	0·00306	0·00903	0·00086	0·00228	−0·00064
C(15)	0·00753	0·00284	0·00524	−0·00016	0·00370	−0·00045
C(16)	0·00392	0·00243	0·00099	−0·00044	0·00076	−0·00077
C(17)	0·00650	0·00213	0·00460	−0·00088	0·00122	−0·00025
C(18)	0·00806	0·00267	0·00369	0·00039	0·00080	−0·00016
C(19)	0·01201	0·00287	0·00929	−0·00014	−0·00089	0·00155
C(20)	0·00725	0·00325	0·00881	0·00146	−0·00093	0·00024
N(1)	0·00344	0·00229	0·00319	0·00041	−0·00016	−0·00023
N(2)	0·00487	0·00209	0·00311	0·00028	0·00153	−0·00072
O(1)	0·00350	0·00316	0·00337	−0·00043	0·00112	−0·00111
O(2)	0·00408	0·00333	0·00336	0·00082	0·00080	−0·00067
Ni	0·00404	0·00292	0·00403	0·00013	0·00065	−0·00075

Mean e.s.d.'s

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C	0·00055	0·00024	0·00140	0·00029	0·00064	0·00040
N	0·00035	0·00016	0·00087	0·00021	0·00043	0·00026
O	0·00029	0·00015	0·00071	0·00018	0·00034	0·00022
Ni	0·00006	0·00003	0·00023	0·00003	0·00008	0·00005

Shifts during final cycle

$\bar{\delta}$	0·00012	0·00006	0·00081	0·00007	0·00011	0·00011
$\delta_{\max}$	0·00049	0·00019	0·00335	0·00020	0·00035	0·00036

(114), (025), and (116) were omitted because of large secondary extinction effects. A modified Hughes weighting scheme was used: $\sqrt{w}=0$ for unobserved reflections for which $F_c \leq F_{\min}$, $w=1$ for unobserved reflections with $F_c > F_{\min}$ and for observed reflections with $F_o \leq 30$, $\sqrt{w}=30/F_o$ for observed reflections with $F_o > 30$. The function minimized was $\Sigma w(|F_o|-|F_c|)^2$. Five cycles of least-squares refinement with individual isotropic temperature factors reduced R to 10·5%. Introduction of the hydrogen atoms in calculated positions (assuming a C–H distance of 1·04 Å) reduced R to 9·3%. The hydrogen atoms were assigned isotropic thermal parameters, $B=4·5$ Å^2, and were not refined. The hydrogen atoms were clearly evident on a difference synthesis calculated at this point, but in the present case, as we have found in other cases, introduction of hydrogen atoms in calculated positions gave a slightly lower R than introduction of hydrogen atoms in the positions estimated from the difference synthesis. Three final cycles of least-squares refinement with individual anisotropic temperature factors reduced R to 5·7%.

Table 3. *Positional parameters of hydrogen atoms*

Atom	x/a	y/b	z/c
H(1)	0·0166	0·3960	0·2502
H(2)	−0·1298	0·4448	0·3223
H(3)	−0·2844	0·3794	0·3369
H(4)	−0·2940	0·2658	0·2797
H(5)	−0·2225	0·1700	0·2124
H(6)	−0·1933	0·0825	0·1532
H(7)	−0·1519	0·1134	0·0019
H(8)	−0·1315	0·0290	0·0176
H(9)	−0·0322	0·0836	0·0156
H(10)	−0·0611	0·0442	0·2489
H(11)	0·0213	0·0429	0·1627
H(12)	−0·0780	−0·0117	0·1647
H(13)	0·2808	0·0346	0·1810
H(14)	0·3924	−0·0229	0·0740
H(15)	0·3927	0·0101	−0·0772
H(16)	0·2807	0·0958	−0·1348
H(17)	0·1617	0·1829	−0·1107
H(18)	0·0515	0·2533	−0·1225
H(19)	0·1717	0·3261	−0·0435
H(20)	0·0827	0·3410	0·0348
H(21)	0·0655	0·3699	−0·0698
H(22)	−0·1121	0·2255	−0·0549
H(23)	−0·1042	0·3098	−0.0766
H(24)	−0·0870	0·2809	0·0280

Each of these cycles was carried out in two steps, since the total number of parameters exceeds the capacity of the computer. In the first step, positional and thermal parameters of the atoms C(1) through

Table 4. *Observed and calculated structure factors*

Columns are k, $10|F_o|$, and $10F_c$. Unobserved reflections are marked with *. Reflections omitted because of secondary extinction are marked with E

0,K,0		
2	1456E	-3151
4	90	-64
6	327	320
8	1056	-1120
10	1222	1333
12	780	-710
14	218	-218
16	126	108
18	500	-490
2,K,0		
0	1127	1304
1	59	-51
2	1802E	-2748
3	1429	1730
4	1515	1717
5	1595	1828
6	703	686
7	433	-439
8	1040	-1092
9	675	645
10	554	537
11	481	-434
12	397	-330
13	362	334
14	89*	58
15	492	473
16	278	251
17	151	-121
18	301	-271
19	107	-101
4,K,0		
0	602	-614
1	1429	-1551
2	564	-542
3	1594	1697
4	254	249
5	917	-884
6	333	-294
7	1187	-1222
8	338	-336
9	1166	1168
10	241	254
11	761	-687
12	617	-572
13	89*	89
14	146	132
15	89	87
16	165	140
17	426	-412
18	59*	-47
6,K,0		
0	816	-713
1	1460	-1578
2	600	582
3	902	838
4	72*	96
5	459	-390
6	77*	-35
7	252	193
8	649	625
9	988	956
10	145	-134
11	663	-586
12	89*	6
13	415	-387
14	257	-247
15	223	224
16	171	143
17	217	-199
8,K,0		
0	935	-925
1	1085	-1055
2	831	789
3	420	422
4	297	-274
5	213	193
6	607	-589
7	431	-357
8	524	480
9	410	390
10	403	-366
11	313	293
12	455	439
13	244	235
14	66*	27
15	56*	-58
16	219	-231
10,K,0		
0	1216	-1315
1	89*	-79
2	837	792
3	370	334
4	481	-445
5	87*	-17
6	543	-498
7	83*	-8
8	466	431
9	126	-128
10	424	-414
11	83	-95
12	413	403
13	124	-113
12,K,0		
0	733	-729
1	118	-122
2	326	323
3	72	109
4	67*	94
5	373	-361
6	172	172
7	55*	39
8	257	251
0,K,1		
2	707	-806
4	40*	56
6	904	-926
8	800	-753
10	881	844
12	758	-796
14	456	448
16	83*	-114
18	68*	18
1,K,1		
1	924	1044
2	1110	-1233
3	1356	-1673
4	232	-210
5	141	162
6	513	-541
7	620	-654
8	147	142
9	294	-252
10	500	-464
11	807	815
12	468	-462
13	716	-749
14	259	250
15	616	623
16	83*	26
17	329	-314
18	68*	-29
19	55*	16
2,K,1		
1	202	-181
2	656	-637
3	1140	1192
4	795	817
5	173	142
6	688	-714
7	59*	56
8	129	-104
9	629	-590
10	432	428
11	95	68
12	359	-362
13	489	455
14	208	190
15	338	-326
16	174	-180
17	75*	-70
18	66*	34
19	105	113
3,K,1		
1	165	161
2	792	742
3	1162	1120
4	1835	2084
5	1226	1248
6	1184	-1224
7	189	-202
8	262	266
9	472	-469
10	294	-258
11	294	279
12	391	-359
13	431	-391
14	659	657
15	255	237
16	375	-357
17	216	-210
18	62*	7
19	48*	-59
4,K,1		
1	124	120
2	730	696
3	878	841
4	565	-516
5	1163	-1172
6	396	-363
7	517	516
8	104	-96
9	232	213
10	169	162
11	663	-676
12	297	-303
13	682	701
14	86*	95
15	576	-554
16	337	327
17	68*	3
18	57*	-47
19	137	126
5,K,1		
1	323	-304
2	568	-556
3	424	-409
4	685	691
5	120	-106
6	716	-687
7	563	556
8	1031	1023
9	141	132
10	545	521
11	86*	8
12	587	-563
13	279	-254
14	580	569
15	112	-103
16	277	-287
17	219	235
18	138	150
6,K,1		
1	415	-393
2	67*	56
3	1299	1348
4	71*	4
5	777	-752
6	354	329
7	328	315
8	81*	39
9	139	108
10	172	-152
11	746	-716
12	86*	-49
13	450	417
14	234	234
15	547	-546
16	240	-240
17	286	284
7,K,1		
1	252	-226
2	891	-851
3	136	126
4	539	506
5	518	-506
6	269	-263
7	84*	54
8	191	-181
9	286	-247
10	417	407
11	86*	-94
12	504	-483
13	371	361
14	182	191
15	154	-161
16	207	-222
8,K,1		
1	320	-305
2	374	354
3	388	394
4	443	-427
5	85*	-92
6	508	501
7	125	-103
8	153	-121
9	184	195
10	85*	-45
11	82*	-30
12	215	221
13	205	191
14	64*	-80
15	107	-109
9,K,1		
1	86*	-23
2	180	-167
3	544	532
4	230	225
5	597	-583
6	87*	-57
7	266	251
8	508	-511
9	350	336
10	310	307
11	217	-217
12	173	-172
13	330	323
14	95	96
10,K,1		
1	159	-150
2	180	171
3	254	259
4	393	-377
5	84*	-63
6	398	378
7	81*	-66
8	78*	-3
9	74*	-15
10	93	-100
11	111	-107
12	340	342
11,K,1		
1	144	-162
2	153	125
3	477	469
4	194	-167
5	269	-262
6	273	278
7	70*	65
8	66*	15
9	60*	-34
10	100	82
11	277	-295
12,K,1		
1	206	200
2	68*	-17
3	434	-439
4	65*	-43
5	112	108
6	259	280
7	147	-147
8	49	-73
13,K,1		
1	154	-160
2	309	338
3	46*	-68
4	171	-183
0,K,2		
0	469	542
2	794	915
4	270	306
6	141	-137
8	295	-291
10	216	214
12	517	497
14	198	-165
16	119	-115
18	131	152
1,K,2		
0	774	-754
1	1713E	-2486
2	36*	51
3	123	106
4	153	132
5	279	280
6	184	-210
7	1140	-1219
8	411	-395
9	1221	1280
10	176	-182
11	793	-792
12	715	712
13	231	219
14	101*	-7
15	151	120
16	224	-230
17	380	-368
18	77*	-8
19	393	409
2,K,2		
0	362	-347
1	1668E	-2006
2	1285	-1300
3	625	599
4	654	630
5	1041	1068
6	64*	-14
7	253	-243
8	186	-170
9	351	324
10	87*	-75
11	134	-116
12	105	72
13	100*	-3
14	247	232
15	265	251
16	94*	33
17	128	104
18	74*	-49
19	59*	79
3,K,2		
0	108	112
1	732	-734
2	1223	1238
3	968	1007
4	445	-423
5	70	-30
6	238	-242
7	1292	-1393
8	563	600
9	640	641
10	984	-1010
11	506	-468
12	444	432
13	107	104
14	100*	-18
15	228	205
16	449	-440
17	109	-114
18	274	282
4,K,2		
0	926	-923
1	60*	-18
2	163	151
3	288	-251
4	488	-489
5	70*	-33
6	727	-711
7	667	-654
8	434	406
9	187	-196
10	93*	42
11	159	-162
12	160	152
13	101*	76
14	227	-240
15	134	-115
16	162	150
17	77*	-83
18	64*	4
5,K,2		
0	1381	-1453
1	149	-132
2	1432	1489
3	287	281
4	559	-548
5	108	-99
6	508	-489
7	356	326
8	1381	1444
9	286	277
10	856	-880
11	298	-299
12	305	285
13	100*	-56
14	134	130
15	90*	89
16	258	-248
17	70*	11
18	267	280
6,K,2		
0	334	-327
1	310	-308
2	722	729
3	479	-444
4	134	-123
5	268	227
6	659	586
7	100	-76
8	95*	43
9	208	-211
10	178	-182
11	372	-377
12	100*	25
13	253	245
14	119	-105
15	84*	42
16	73*	-57
17	127	-141
7,K,2		
0	1233	-1289
1	359	328
2	794	766
3	89*	-73
4	131	131
5	222	-213
6	421	-406
7	413	418
8	602	599
9	617	-641
10	627	-643
11	200	196
12	96*	73
13	312	326
14	89	-82
15	74*	-37
16	154	-160
8,K,2		
0	189	-193
1	95*	100
2	299	286
3	214	214
4	142	-112
5	191	-188
6	254	259
7	101*	84
8	178	-158
9	167	172
10	98*	-94
11	339	340
12	89*	-6
13	129	-118
14	72*	-12
15	141	160
9,K,2		
0	625	-615
1	810	827
2	305	305
3	430	-451
4	104	99
5	183	-172
6	349	-347
7	771	786
8	184	169
9	346	-335
10	148	-151
11	481	475
12	153	145
13	314	-316
14	94	-97
10,K,2		
0	497	499
1	109	-106
2	305	-289
3	108	-82
4	130	-139
5	97*	17
6	193	-192
7	171	175
8	89*	26
9	140	-146
10	150	143
11	85	-83
12	131	-152
11,K,2		
0	93*	-25
1	608	603
2	92*	50
3	349	-339
4	118	-113
5	87*	-15
6	84*	2
7	257	274
8	81	67
9	558	-583
10	60*	35
11	347	408
12,K,2		
0	181	213
1	376	334
2	104	-95
3	153	-143
4	172	179
5	70*	-4
6	71	72
7	60*	-59

Table 4 (*cont.*)

	12,K,2	
8	51*	-18
	13,K,2	
0	371	417
1	316	321
2	179	-175
3	174	-185
	0,K,3	
2	1612E	-2873
4	1337	1620
6	825	-912
8	1014	1103
10	467	-443
12	260	-276
14	340	346
16	205	-213
18	63	-50
	1,K,3	
1	1403E	-1550
2	984	-976
3	882	949
4	164	155
5	635	-665
6	51*	-23
7	407	-437
8	103	112
9	476	509
10	259	-276
11	406	-403
12	79*	-88
13	487	506
14	245	-239
15	81*	-18
16	76*	0
17	70*	58
18	139	150
19	169	163
	2,K,3	
1	366	311
2	1056	-1003
3	412	389
4	1460	1462
5	547	-550
6	738	-765
7	219	218
8	461	491
9	126	111
10	339	354
11	174	-144
12	317	-304
13	496	520
14	285	256
15	243	-221
16	229	-232
17	71	80
18	178	-187
19	45*	13
	3,K,3	
1	490	458
2	554	478
3	47*	-54
4	691	-706
5	353	-353
6	321	-280
7	239	259
8	86	-94
9	387	408
10	418	-404
11	259	-287
12	479	431
13	241	253
14	456	-465
15	79*	-6
16	216	211
17	150	-157
18	262	268
	4,K,3	
1	118	-112
2	272	-241
3	480	447
4	1003	960
5	1428	-1414
6	775	-734
7	494	468
8	72	-62
9	88	107
10	80	-86
11	200	-204
12	199	-180
13	362	363
14	443	443
15	154	-173
16	70*	-10
17	63	-71
18	91	109
	5,K,3	
1	353	368
2	308	306
3	262	-258
4	511	-517
5	250	-207
6	933	910
7	270	240
8	74*	-23
9	77*	-9
10	765	-767
11	82*	-64
12	923	876
13	81*	74
14	355	-351
15	332	-345
16	134	119
17	160	177
18	40*	54
	6,K,3	
1	545	-551
2	495	465
3	553	529
4	588	-548
5	397	-373
6	443	429
7	824	792
8	79*	30
9	249	-222
10	256	-232
11	650	-662
12	81*	83
13	91	105
14	197	-186
15	159	140
16	57*	14
17	45*	31
	7,K,3	
1	217	-195
2	591	566
3	440	-423
4	785	-755
5	594	606
6	101	102
7	86	-95
8	194	-182
9	364	-333
10	257	-237
11	125	120
12	291	288
13	295	271
14	286	-283
15	100	-123
16	153	182
	8,K,3	
1	165	-158
2	368	355
3	474	444
4	754	-722
5	672	-653
6	418	376
7	429	403
8	107	-98
9	81*	83
10	79*	-60
11	153	168
12	188	221
13	291	301
14	284	-304
15	244	-271
	9,K,3	
1	431	434
2	283	288
3	245	-222
4	338	-330
5	478	457
6	82*	-44
7	217	-215
8	79*	-32
9	76*	74
10	72*	36
11	67*	41
12	61*	-2
13	431	-448
14	144	-139
	10,K,3	
1	81*	46
2	372	368
3	80*	65
4	639	-611
5	78*	-60
6	399	375
7	74*	55
8	247	-248
9	74	-77
10	157	160
11	98	-97
12	127	125
	11,K,3	
1	198	189
2	158	-170
3	306	-296
4	71*	46
5	306	304
6	236	239
7	258	-261
8	201	213
9	53*	83
10	75	-107
	12,K,3	
1	62*	-4
2	484	448
3	60*	-22
4	338	-367
5	56	-65
6	292	311
7	47	-46
	13,K,3	
1	41*	-41
2	67	-91
	0,K,4	
0	174	-154
2	225	-220
4	466	478
6	712	-686
8	1012	1102
10	512	-524
12	129*	98
14	181	193
16	293	-299
18	379	337
	1,K,4	
0	780	736
1	1591E	1829
2	51*	12
3	1294	-1314
4	822	-857
5	313	306
6	94*	-110
7	293	300
8	102*	55
9	364	386
10	212	-211
11	124*	-111
12	130*	5
13	133*	99
14	133*	123
15	129*	-14
16	121*	-11
17	123*	-116
18	93*	-6
19	123	125
	2,K,4	
0	898	-868
1	1221	1155
2	1199	1205
3	396	-417
4	389	-388
5	238	216
6	799	-807
7	97*	40
8	725	758
9	150	-155
10	743	-790
11	597	589
12	180	188
13	193	-195
14	177	201
15	265	-261
16	319	-287
17	474	468
18	247	254
19	199	-203
	3,K,4	
0	509	-476
1	224	208
2	158	126
3	312	286
4	153	-165
5	841	795
6	441	-436
7	593	-507
8	110*	130
9	117*	67
10	123*	56
11	129*	69
12	163	153
13	133*	-74
14	221	-177
15	157	130
16	115*	66
17	237	-236
18	171	208
	4,K,4	
0	753	-723
1	1028	983
2	363	362
3	415	-398
4	915	-869
5	97*	-20
6	104*	-16
7	647	653
8	679	695
9	839	-871
10	207	-190
11	510	509
12	133*	-22
13	158	-191
14	199	204
15	445	-414
16	109*	-65
17	417	409
18	72*	68
	5,K,4	
0	96*	-9
1	358	-316
2	552	-551
3	195	-178
4	603	547
5	362	361
6	290	-287
7	118*	-79
8	270	266
9	128*	31
10	131*	-10
11	133*	94
12	146*	182
13	143*	-134
14	282	281
15	113*	-74
16	100*	53
17	82*	55
	6,K,4	
0	108*	42
1	1263	1231
2	297	283
3	759	-768
4	163	143
5	536	-500
6	148	153
7	828	857
8	262	-235
9	1144	-1111
10	133*	-74
11	519	534
12	284	276
13	150	-97
14	279	-274
15	103*	-46
16	87*	-5
17	247	316
	7,K,4	
0	120*	34
1	329	328
2	330	-325
3	123*	23
4	176	146
5	180	-156
6	183	-161
7	145*	-89
8	321	316
9	188	-164
10	312	-325
11	128*	45
12	123*	81
13	114*	-3
14	103*	3
15	97*	93
16	67*	-92
	8,K,4	
0	430	450
1	1192	1235
2	515	-465
3	131*	-61
4	243	225
5	243	-224
6	307	321
7	509	469
8	490	-494
9	219	-205
10	288	309
11	379	382
12	111*	-33
13	163	-152
14	127	-163
15	189	-187
	9,K,4	
0	351	364
1	545	-497
2	147*	-162
3	133*	-64
4	133*	-78
5	132*	-60
6	144*	-143
7	129*	45
8	170	191
9	120*	76
10	125*	-97
11	105*	-86
12	94*	51
13	79*	-44
	10,K,4	
0	499	483
1	271	256
2	482	-471
3	281	-286
4	127*	-27
5	128*	-122
6	312	280
7	147	144
8	425	-455
9	185	-155
10	430	475
11	83*	64
12	222	-263
	11,K,4	
0	219	222
1	117*	17
2	160	-161
3	189	202
4	279	266
5	224	-221
6	103*	-64
7	97*	-51
8	89*	18
9	79*	-74
10	64*	46
	12,K,4	
0	377	436
1	422	-470
2	276	-312
3	148	186
4	245	271
5	110	98
6	114	135
7	65*	-87
	0,K,5	
2	1320E	2101
4	903	-912
6	342	344
8	266	274
10	260	-268
12	465	491
14	498	-578
16	262	246
18	111	-122
	1,K,5	
1	161	153
2	1144	1244
3	1026	1148
4	177	-178
5	1082	-1202
6	256	276
7	676	701
8	103*	-21
9	157	-159
10	248	-237
11	324	-306
12	181	162
13	227	240
14	184	-144
15	230	-206
16	114*	132
17	101*	-5
18	121	-140
	2,K,5	
1	59*	-88
2	341	373
3	240	-217
4	688	-647
5	310	296
6	91*	-30
7	123	105
8	352	335
9	113*	58
10	313	-338
11	191	196
12	561	613
13	277	-321
14	337	-366
15	449	414
16	238	203
17	178	141
18	79*	-68
	3,K,5	
1	633	-598
2	314	302
3	1203	1228
4	513	-506
5	918	-896
6	148	190
7	615	638
8	130	113
9	226	-213
10	177	-186
11	257	-280
12	396	426
13	396	430
14	241	-262
15	418	-394
16	107*	2
17	92*	62
18	155	148
	4,K,5	
1	85*	-90
2	208	-197
3	106	73
4	95*	40
5	412	412
6	469	455
7	1038	-1050
8	117*	-20
9	122*	-35
10	283	-242
11	457	470
12	243	259
13	370	-396
14	303	-307
15	200	187
16	241	215
17	98	-82
	5,K,5	
1	98*	92
2	673	619
3	102*	24
4	561	-498
5	271	-276
6	858	863
7	311	294
8	123*	-39
9	237	-228
10	129*	-66
11	152	-150
12	128*	80
13	124*	16
14	116*	-118
15	105*	5
16	107	96
17	71*	102
	6,K,5	
1	262	-226
2	111*	-7
3	368	-349
4	240	227
5	560	507
6	330	-281

Table 4 (*cont.*)

	6,K,5	
7	177	−204
8	128*	49
9	225	−208
10	130*	70
11	435	424
12	164	−140
13	241	−243
14	107*	26
15	94*	81
16	193	−177
	7,K,5	
1	190	−177
2	429	378
3	299	−287
4	761	−730
5	294	266
6	698	703
7	240	−207
8	347	−361
9	129*	2
10	127*	56
11	567	524
12	240	270
13	216	−212
14	298	−291
15	78*	48
	8,K,5	
1	543	479
2	338	−307
3	584	−536
4	451	428
5	189	182
6	281	−280
7	185	155
8	127*	−128
9	248	−255
10	123	134
11	379	449
12	215	−242
13	161	−189
14	240	287
	9,K,5	
1	374	361
2	404	379
3	611	−563
4	501	−458
5	520	476
6	366	365
7	314	−335
8	303	−309
9	113*	75
10	106*	88
11	96*	47
12	99	133
13	195	−251
	10,K,5	
1	124*	−95
2	559	−530
3	122*	23
4	515	495
5	117*	−11
6	253	−253
7	260	290
8	103*	50
9	213	−243
10	85*	54
11	72*	86
	11,K,5	
1	110*	1
2	108*	4
3	419	−415
4	104*	34
5	533	582
6	94*	87
7	334	−368
8	93	−84
9	66*	98
	12,K,5	
1	86*	−80
2	141	−152
3	238	260
4	145	145
5	151	−162
	0,K,6	
0	553	789
2	42*	−48
4	457	−445
6	430	408
8	91*	64
10	194	−208
12	186	−205
14	107*	84
16	164	−168
18	131	155
	1,K,6	
0	1003	1041
1	1389E	1606
2	294	−296
3	395	−387
4	114	110
5	820	−879
6	308	317
7	483	492
8	634	−685
9	559	−665
10	103*	−36
11	350	382
12	166	−165
13	161	157
14	236	−233
15	227	−216
16	275	286
17	283	241
18	123	−107
	2,K,6	
0	380	365
1	345	−315
2	57*	36
3	411	402
4	532	584
5	75*	−3
6	284	−262
7	266	269
8	373	−403
9	103	−131
10	105*	68
11	184	−200
12	172	192
13	213	259
14	106*	8
15	99*	45
16	352	−327
17	91	−107
18	80	97
	3,K,6	
0	1249	1233
1	419	384
2	1150	−1133
3	834	−776
4	261	234
5	485	492
6	795	778
7	583	625
8	798	−870
9	496	−569
10	537	592
11	114	146
12	304	−336
13	108*	−62
14	104*	−40
15	245	−237
16	412	387
17	264	261
	4,K,6	
0	768	−687
1	77*	−29
2	78*	15
3	270	285
4	431	430
5	89*	70
6	94*	−20
7	98*	36
8	260	246
9	185	209
10	175	−207
11	114	−174
12	109*	55
13	106*	94
14	100*	−75
15	91*	−59
16	79*	53
17	62*	42
	5,K,6	
0	754	703
1	205	−191
2	225	−208
3	94	−117
4	246	249
5	181	−169
6	285	237
7	104*	−12
8	846	−904
9	109*	44
10	925	872
11	114	105
12	218	−210
13	174	−161
14	194	−192
15	83*	−22
16	277	273
	6,K,6	
0	124	−126
1	220	−181
2	141	137
3	160	−161
4	146	−108
5	124	89
6	171	−188
7	109*	30
8	110*	66
9	110*	72
10	109*	100
11	107*	−41
12	192	−192
13	145	125
14	85*	29
15	73*	−32
16	77	106
	7,K,6	
0	1233	1232
1	640	−624
2	760	−730
3	782	749
4	108*	−11
5	109*	62
6	426	398
7	234	−246
8	549	−595
9	242	218
10	549	528
11	206	−190
12	275	−244
13	152	133
14	172	−170
15	55*	49
	8,K,6	
0	110*	13
1	110*	−63
2	110*	4
3	110*	−91
4	150	−156
5	202	192
6	110*	−59
7	224	204
8	106*	48
9	339	−327
10	97*	93
11	257	251
12	81*	−59
13	70*	−77
14	51*	42
	9,K,6	
0	480	476
1	484	−438
2	497	−458
3	213	207
4	111	105
5	106*	62
6	272	267
7	389	−361
8	241	−216
9	376	329
10	94*	13
11	125	−112
12	91	−69
	10,K,6	
0	275	261
1	102*	−72
2	299	−263
3	100*	20
4	98*	24
5	95*	−72
6	92*	45
7	87*	35
8	81*	5
9	74*	−77
10	63*	3
11	46*	35
	11,K,6	
0	308	−307
1	454	−443
2	224	225
3	113	115
4	82*	15
5	269	246
6	73*	−22
7	238	−208
8	114	114
	12,K,6	
0	272	279
1	125	103
2	186	−180
3	59*	88
4	53*	−28

C(10), N(1), O(1) and Ni were refined: in the sceond step the parameters of atoms C(11) through C(20), N(2), O(2), and Ni were refined. The standard deviations estimated by the Busing–Levy program were therefore multiplied by the factor 1·0657 (from $\sqrt{[(1021-117)/(1021-225)]}$). The mean and maximum shifts of parameters during the final cycle are given in Tables 1 and 2.

The final values of the parameters and their estimated standard deviations (from the Busing–Levy program) are given in Tables 1 to 3. The list of observed and calculated structure factors is given in Table 4.

Discussion

The crystal consists of discrete molecules. Bond distances and angles are shown in Figs. 1 and 2 and are listed along with their estimated standard deviations in Tables 5 and 6.

A most important result of the structure determination is the confirmation of the tetrahedral coordination configuration of the nickel(II) ion in this paramagnetic chelate compound. The coordination tetrahedron is, of course, considerably distorted because the intrachelate angles N(1)–Ni–O(1) and N(2)–Ni–O(2) are fixed at about 94° by the requirements of the salicylaldimine groups. The planes of the Ni–N(1)–O(1) and Ni–N(2)–O(2) groups are found to make an angle of 81·5° with each other. Equations for the several group planes are given in Table 7.

Table 5. *Intramolecular distances and their e.s.d.'s*

C(1)–C(2)	1·406 Å	0·010	C(11)–C(12)	1·448 Å	0·010
C(2)–C(3)	1·398	0·011	C(12)–C(13)	1·407	0·013
C(3)–C(4)	1·388	0·010	C(13)–C(14)	1·396	0·019
C(4)–C(5)	1·379	0·010	C(14)–C(15)	1·379	0·011
C(5)–C(6)	1·425	0·010	C(15)–C(16)	1·452	0·012
C(6)–C(1)	1·456	0·008	C(16)–C(11)	1·421	0·016
C(6)–C(7)	1·422	0·009	C(16)–C(17)	1·481	0·010
C(1)–O(1)	1·300	0·009	C(11)–O(2)	1·294	0·011
C(7)–N(1)	1·298	0·009	C(17)–N(2)	1·303	0·010
N(1)–C(8)	1·464	0·008	N(2)–C(18)	1·503	0·010
C(8)–C(9)	1·524	0·019	C(18)–C(19)	1·542	0·011
C(8)–C(10)	1·514	0·012	C(18)–C(20)	1·552	0·011
N(1)–Ni	1·990	0·005	N(2)–Ni	1·950	0·009
O(1)–Ni	1·894	0·005	O(2)–Ni	1·898	0·004
C(9)–C(7)	3·439	0·017	C(19)–C(17)	3·209	0·011
C(9)–O(2)	3·950	0·013	C(19)–O(1)	3·523	0·016
C(9)–N(2)	3·650	0·011	C(20)–N(1)	3·665	0·014
C(10)–C(7)	3·371	0·011	C(20)–C(17)	3·567	0·011
C(10)–O(2)	3·339	0·010	C(20)–O(1)	3·635	0·014

The Ni–O distance (mean value, 1·90 Å) is about

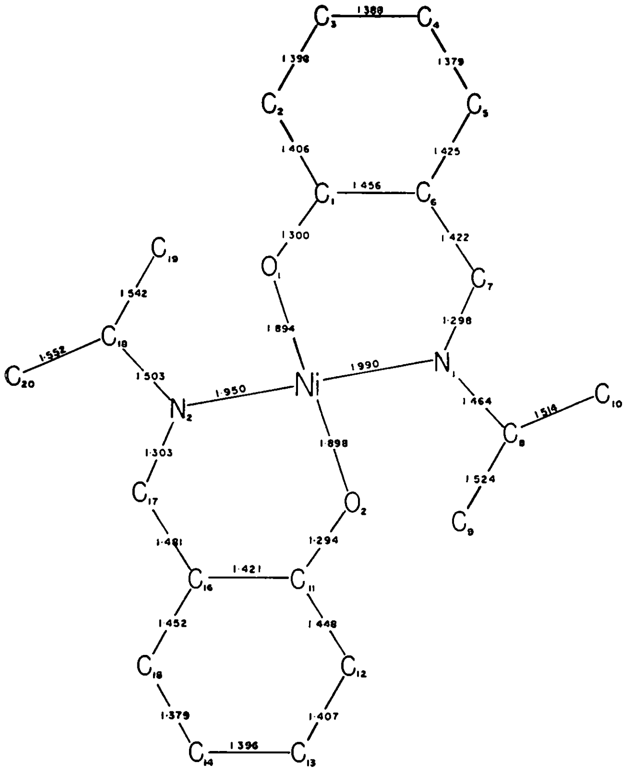

Fig. 1. Bond lengths.

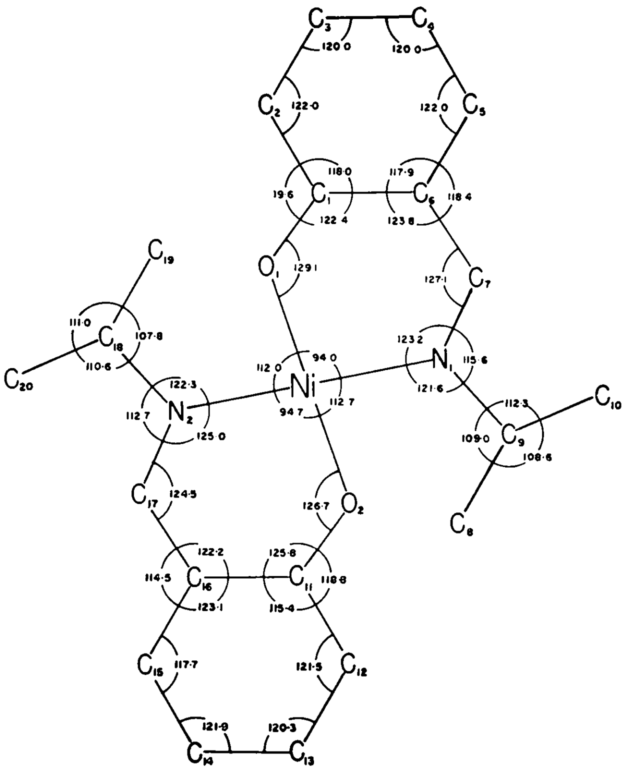

Fig. 2. Bond angles.

Table 6. *Bond angles and their e.s.d.'s*

C(5)–C(6)–C(7)	118·4°	0·8	C(15)–C(16)–C(17)	114·5°	1·0
C(1)–C(6)–C(5)	117·9	0·8	C(11)–C(16)–C(15)	123·1	1·2
C(6)–C(5)–C(4)	122·0	0·9	C(16)–C(15)–C(14)	117·7	1·1
C(5)–C(4)–C(3)	120·0	0·9	C(15)–C(14)–C(13)	121·9	1·4
C(4)–C(3)–C(2)	120·0	0·9	C(14)–C(13)–C(12)	120·3	1·3
C(3)–C(2)–C(1)	122·0	0·9	C(13)–C(12)–C(11)	121·5	1·1
C(2)–C(1)–C(6)	118·8	0·8	C(12)–C(11)–C(16)	115·4	1·0
C(2)–C(1)–O(1)	119·6	0·8	C(12)–C(11)–O(2)	118·8	1·1
C(1)–C(6)–C(7)	123·8	0·8	C(11)–C(16)–C(17)	122·2	1·1
C(6)–C(7)–N(1)	127·1	0·9	C(16)–C(17)–N(2)	124·5	1·0
C(6)–C(1)–O(1)	122·4	0·8	C(16)–C(11)–O(2)	125·8	1·2
C(7)–N(1)–Ni	123·2	0·6	C(17)–N(2)–Ni	125·0	0·7
C(8)–N(1)–Ni	121·1	0·5	C(18)–N(2)–Ni	122·3	0·6
C(7)–N(1)–C(8)	115·6	0·7	C(17)–N(2)–C(18)	112·7	0·9
N(1)–C(8)–C(9)	109·0	1·0	N(2)–C(18)–C(19)	107·8	0·8
N(1)–C(8)–C(10)	112·3	0·8	N(2)–C(18)–C(20)	110·6	0·8
C(9)–C(8)–C(10)	108·6	1·0	C(19)–C(18)–C(20)	111·0	0·8
C(1)–O(1)–Ni	129·1	0·6	C(11)–O(2)–Ni	126·7	0·8
N(1)–Ni–N(2)	120·9	0·5	O(1)–Ni–O(2)	125·1	0·4
N(1)–Ni–O(1)	94·0	0·2	O(1)–Ni–N(2)	112·0	0·4
N(1)–Ni–O(2)	112·7	0·3	O(2)–Ni–N(2)	94·7	0·3

Table 7. *Coefficients of least-squares plane equations, $Ax+By+Cz+1=0$*

	A	B	C
Salicylaldimine No. 1	−0·5523	0·6218	−1·5158
Coordination No. 1	−1·8663	1·9690	−3·9902
Isopropyl No. 1	5·1555	4·1894	−0·1385
Salicylaldimine No. 2	−0·1986	−0·1906	−0·0501
Coordination No. 2	−0·1635	−0·2003	−0·0717
Isopropyl No. 2	0·0546	−0·1355	0·3779

0·1 Å shorter than the Ni–N distance (mean value, 1·97 Å), as has been found in both bis-(*N*-methylsalicylaldiminato)-nickel(II) (Frasson, Panattoni & Sacconi, 1959) and -copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961). The Ni–O distance of 1·90 Å is intermediate between the 1·80 Å distance found in bis-(*N*-methylsalicylaldiminato)-nickel(II), which has a planar coordination configuration, and the 2·02 Å distance found in diaquabis-salicylaldehydato)nickel(II), which has an octahedral coordination configuration (Stewart *et al.*, 1961). The Ni–N distance of 1·97 Å is also greater than the corresponding distance, 1·90 Å, found in the planar *N*-methyl compound, but no comparable value is available for the octahedral coordination configuration.

Table 8. *Distances from least squares planes of salicylaldimine residues*

C(1)	0·0030 Å	C(11)	−0·0020 Å
C(2)	−0·0052	C(12)	0·0007
C(3)	0·0005	C(13)	−0·0154
C(4)	0·0007	C(14)	−0·0040
C(5)	0·0085	C(15)	0·0147
C(6)	0·0012	C(16)	0·0293
C(7)	−0·0309	C(17)	−0·0282
N(1)	0·0277	N(2)	0·0004
O(1)	−0·0024	O(2)	0·0046
Ni	0·1400	Ni	0·1959

The bond distances and angles in the salicylaldimine groups appear to be normal. These groups, furthermore, show no significant deviation from planarity, as indicated by the distances listed in Table 8. It is apparent, however, that the nickel atom is not in the plane of the salicylaldimine group, lying 0·14 Å out of the plane in one case and 0·20 Å in the other. The planes of the two salicylaldimine groups form an angle of 82° with each other.

The bond distances and angles in the isopropyl groups are normal. The plane of each isopropyl group is nearly normal to the plane of the salicylaldimine group to which it is attached.

The contact distances between non-bonded atoms all appear to be reasonable. Distances between the methyl groups of the isopropyl and other atoms within the same molecule are listed in Table 5 and range from 3·209 Å upwards. Contact distances between molecules are listed in Table 9, in which is given the closest neighbor of each atom in the molecule. These distances range upwards from 3·30 Å.

Table 9. *Closest contacts of neighboring molecules*

Atom	Distance	Closest atom	Molecule position
C(1)	3·80 Å	C(18)	$x, \frac{1}{2}-y, \frac{1}{2}+z$
C(2)	3·74	C(10)	$\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$
C(3)	3·65	C(9)	$x, \frac{1}{2}-y, \frac{1}{2}+z$
C(4)	3·65	O(1)	$-\frac{1}{2}+x, y, \frac{1}{2}-z$
C(5)	3·30	O(1)	$-\frac{1}{2}+x, y, \frac{1}{2}-z$
C(6)	3·68	C(20)	$x, \frac{1}{2}-y, \frac{1}{2}+z$
C(7)	3·71	O(2)	$-\frac{1}{2}+x, y, \frac{1}{2}-z$
C(8)	4·12	C(14)	$\bar{x}, \bar{y}, \bar{z}$
C(9)	3·65	C(3)	$x, \frac{1}{2}-y, -\frac{1}{2}+z$
C(10)	3·74	C(2)	$\bar{x}, -\frac{1}{2}+y, \frac{1}{2}-z$
N(1)	4·48	C(12)	$-\frac{1}{2}+x, y, \frac{1}{2}-z$
O(1)	3·30	C(5)	$\frac{1}{2}+x, y, \frac{1}{2}-z$
Ni	3·91	C(5)	$\frac{1}{2}+x, y, \frac{1}{2}-z$
C(11)	3·85	C(20)	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
C(12)	3·81	C(10)	$\frac{1}{2}+x, y, \frac{1}{2}-z$
C(13)	3·87	C(9)	$\bar{x}, \bar{y}, \bar{z}$
C(14)	3·81	C(9)	$\bar{x}, \bar{y}, \bar{z}$
C(15)	3·68	C(3)	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
C(16)	3·64	C(20)	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
C(17)	3·85	C(5)	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
C(18)	3·80	C(1)	$x, \frac{1}{2}-y, -\frac{1}{2}+z$
C(19)	3·97	C(13)	$\frac{1}{2}-x, \frac{1}{2}+y, z$
C(20)	3·64	C(16)	$-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
N(2)	4·34	C(2)	$x, \frac{1}{2}-y, -\frac{1}{2}+z$
O(2)	3·71	C(7)	$\frac{1}{2}+x, y, \frac{1}{2}-z$

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