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The Crystal Stucture of Bis-(*N*-isopropyl-3-methylsalicylaldiminato)nickel

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The crystal structure of bis(*N*-isopropyl-3-methylsalicylaldiminato)nickel, $Ni(C_{11}H_{14}NO)_2$, has been determined from three-dimensional integrating Weissenberg data. The cell has dimensions $a=11\cdot209$, $b=9\cdot979$, $c=9\cdot520$ Å, $\beta=107°31'$, space group $P2_1/c$, and contains two molecules.

The nickel atoms show planar coordination. Comparison of the molecular structure with that of bis(*N*-isopropylsalicylaldiminato)nickel shows the steric factors which cause the tetrahedral configuration to appear for *N*-sec-alkyl substituted salicylaldimine chelates, but gives no clue as to the reason for the planar configuration of the 3-methyl chelate.

Introduction

The crystal structures of chelates of nickel(II) and ring-substituted salicylaldimines are currently of considerable interest. Holm & Swaminathan (1963) have determined that for 3-substituted bis(*N*-isopropylsalicylaldiminato)nickel the magnetic moments are 3·28, 0 and 3·30 B.M. for the substituents hydrogen, methyl and ethyl, indicating the coordination configurations tetrahedral, planar and tetrahedral, respectively. The coordination configuration of the 3-hydrogen chelate has indeed been shown to be tetrahedral (Fox, Orioli, Lingafelter & Sacconi, 1964). We have now completed the structural determination of the 3-methyl chelate by three-dimensional X-ray diffraction techniques.

Experimental

The bis(*N*-isopropyl-3-methylsalicylaldiminato)nickel was prepared by the method of Sacconi, Paoletti & Del Re (1957) using 3-methylsalicylaldehyde, which was prepared according to the general procedure of the

Duff reaction (Duff, 1941). Final purification was accomplished by recrystallization from isopropyl alcohol. The crystal used for determination of cell dimensions and collection of the intensity data was a well-defined rhomb, tabular on (100), of approximate dimensions $0{\cdot}24 \times 0{\cdot}21 \times 0{\cdot}14$ mm.

Cell dimensions were measured on a Picker X-ray diffractometer equipped with a General Electric goniostat, using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å). The cell dimensions with their standard deviations are:

$$a = 11{\cdot}209 \pm 0{\cdot}002 \text{ Å}$$
$$b = 9{\cdot}979 \pm 0{\cdot}004 \qquad \beta = 107°31' \pm 4'$$
$$c = 9{\cdot}520 \pm 0{\cdot}004$$

Systematic absences of $0k0$ for k odd and $h0l$ for l odd identified the space group as $P2_1/c$. The cell contains two molecules: measured density $1{\cdot}35$ g.cm^{-3}, calculated density $1{\cdot}345$ g.cm^{-3}.

Equi-inclination Weissenberg photographs, $h0l$ through $h8l$, were taken with Cu $K\alpha$ radiation on a Nonius integrating camera, with integration in one direction only. In order to obtain a satisfactory range of intensities, a 60-hour exposure of one 4-film pack and a 3-hour exposure of another 4-film pack were made. 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 scaled together by comparing common spots. The nine levels were scaled together, using relative intensities from the $hk1$ and $hk2$ integrated Weissenberg photographs. Of the 1904 unique reflections examined (82% of the reflections within the limiting Cu $K\alpha$ sphere) 1448 had measurable intensities and 456 were too faint to be measured. The range of observed intensities was 1 to 10,500.

Structure determination

All calculations were carried out on an IBM 7094 computer with a set of programs written or adapted by Stewart (1964). 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 from Table 2 of Stewart, Davidson & Simpson (SDS, 1965) or McWeeny (1951) for hydrogen. Lorentz and polarization factors were applied and relative structure factors (F_{rel}) were calculated. No correction was made for absorption or for anomalous dispersion. A preliminary overall scale factor and overall temperature factor were estimated from the statistics of the normalized structure factors.

Since there are two molecules in the unit cell of space group $P2_1/c$ the nickel atoms are required to be at centers of symmetry. A three-dimensional Fourier synthesis was calculated, using phases determined by the nickel atoms at $(0,0,0)$ and $(0,\frac{1}{2},\frac{1}{2})$. Such a Fourier synthesis shows mirror planes in $(x,0,z)$ and $(x,\frac{1}{2},z)$. The oxygen atom was located nearly on a mirror plane and a nitrogen atom was arbitrarily selected from a pair of mirror-related positions. Another three-dimensional Fourier synthesis phased on nickel, oxygen, and nitrogen still retained the mirror plane for all carbon atoms. However, the correct y coordinates for 8 of the 11 carbon atoms could be selected on the basis of the planarity of the salicylaldimine residue. Another three-dimensional Fourier synthesis enabled the positions of all 14 non-hydrogen atoms to be determined, and resulted in a reduction of R to $0{\cdot}14$. R is defined throughout as $\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$, where the sums are over the unique, observed reflections only.

Further refinement was carried out with the full-matrix least-squares program of Busing & Levy (1959) as adapted in the UW crystallographic calculations system (Stewart, 1964). The function minimized was $\Sigma\, w(|F_o|-|F_c|)^2$. A modified Hughes weighting scheme was used: $w=0$ for unobserved reflections for which $F_c \le F_{\text{min}}$, $w=1$ for unobserved reflections for which $F_c > F_{\text{min}}$ and for observed reflections with $F_{\text{rel}} \le 8$, and $\sqrt{w} = 8/F_{\text{rel}}$ for observed reflections with $F_{\text{rel}} > 8$. Three cycles of least-squares refinement with individual isotropic temperature factors reduced R to $0{\cdot}11$.

A three-dimensional ΔF synthesis calculated at this stage clearly revealed the position of at least one hydrogen atom on each of the three terminal methyl groups. Introduction of these observed hydrogen atoms and the remaining calculated hydrogen atoms (assuming a C–H distance of $1{\cdot}01$ Å for trigonal and $1{\cdot}05$ Å for tetrahedral) reduced R to $0{\cdot}093$. After three cycles of refinement of the non-hydrogen atoms with individual anisotropic temperature factors, examination of the structure factors showed no evidence of secondary extinction. However, re-examination of the Weissenberg photographs showed that the 100 reflection was partially cut by the beam stop and that the 200 reflection could not be estimated accurately because of the strong white radiation from the 100 reflection. Therefore, these two reflections were omitted in further calculations. One cycle of refinement of level-to-level scale factors followed by two cycles of refinement with individual anisotropic temperature factors reduced R to $0{\cdot}055$.

A parallel refinement beginning at $R=0{\cdot}14$ was carried out, using a weighting scheme based on the plot of ΔF *vs* F_o: $w=0$ for unobserved reflections for which $F_c \le F_{\text{min}}$ and $\sqrt{w} = 1{\cdot}07/(0{\cdot}05F_{\text{rel}} + 1{\cdot}07)$ for unobserved reflections for which $F_c > F_{\text{min}}$ and for all observed reflections. Reduction of R to $0{\cdot}055$ was achieved with this weighting scheme in the same steps as with the Hughes weighting scheme. Comparison of the 39 positional parameters for the 13 non-hydrogen atoms showed that only 10 parameters had differences between these two weighting schemes greater than the standard error (σ), 3 greater than $1{\cdot}5\sigma$ and none greater than 2σ. Further least-squares refinement was then carried out only with the modified Hughes weighting scheme.

At this stage a structure factor calculation omitting hydrogen atoms ($R=0{\cdot}071$) was followed by a three-dimensional ΔF synthesis. From this ΔF synthesis the 14 independent hydrogen atoms were clearly located. A structure factor calculation including these hydrogen atoms gave an R of 0·052. Three cycles of least-squares refinement of the hydrogen coordinates and individual isotropic temperature factors, using all reflections and Hughes weights, with fixed contribution from the non-hydrogen atoms, were done in parallel calculations, using SDS scattering factors in one calculation and McWeeny scattering factors in another calculation. The hydrogen positional parameters after these parallel refinements were essentially identical. However, the hydrogen temperature factors were more reasonable using SDS scattering factors. Further least-squares refinement of the hydrogen parameters was therefore restricted to the use of SDS scattering factors.

The refinement was completed, reducing R to the final value of 0·048, by doing 12 cycles of least-squares refinement, alternating between 2 cycles on the non-hydrogen atoms with individual anisotropic temperature factors and 2 cycles on the hydrogen atoms with individual isotropic temperature factors. The large number of cycles was required because during the first 8 cycles the applied shifts in the least-squares program were inadvertently made too small by a factor of two. During the final 4 cycles of hydrogen atom refinement unit weights were used and reflections with $\sin\theta/\lambda > 0{\cdot}48$ were omitted. Estimated standard deviations were obtained by refining hydrogen atoms and non-hydrogen atoms simultaneously in two additional cycles of full-matrix least squares, one cycle using all data to obtain standard deviations for non-hydrogen atoms and one cycle using limited data to obtain standard deviations for hydrogen atoms.

The final values of the parameters and their estimated standard deviations are given in Tables 1 and 2. The list of observed and calculated structure factors is given in Table 3.

Discussion

The crystal consists of discrete molecules. Fig. 1 shows the projection of atomic coordinates on (010). Since the nickel atom is at a center of symmetry, it is required to have planar coordination. Tables 4 and 5 show least-squares equations for selected planes within the mol-

Table 1. *Parameters of non-hydrogen atoms and their estimated standard deviations*

	Positional parameter × 10^4			Thermal parameter × 10^2					
	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	0	5000	5000	403 (2)	383 (3)	337 (2)	24 (2)	127 (2)	−16 (2)
O	1685 (2)	4917 (2)	5967 (2)	345 (7)	419 (12)	302 (7)	34 (7)	96 (6)	−56 (7)
N	−349 (2)	5779 (3)	6678 (2)	363 (9)	372 (13)	312 (8)	29 (9)	128 (7)	−41 (9)
C(1)	1804 (3)	6381 (3)	7982 (3)	432 (11)	303 (15)	308 (10)	−19 (10)	116 (9)	−15 (9)
C(2)	2355 (3)	5577 (3)	7125 (3)	402 (12)	261 (13)	285 (10)	−17 (9)	101 (8)	43 (8)
C(3)	3672 (3)	5479 (4)	7538 (3)	393 (12)	416 (16)	367 (12)	−32 (10)	97 (9)	51 (10)
C(4)	4381 (3)	6200 (4)	8737 (4)	416 (13)	499 (19)	421 (13)	−96 (12)	43 (11)	17 (12)
C(5)	3842 (4)	7014 (4)	9561 (4)	537 (16)	453 (19)	428 (14)	−149 (14)	44 (12)	−59 (13)
C(6)	2577 (4)	7100 (4)	9123 (4)	563 (16)	380 (18)	398 (13)	−82 (13)	109 (12)	−101 (11)
C(7)	491 (3)	6362 (3)	7728 (3)	453 (12)	350 (18)	348 (11)	4 (10)	154 (10)	−91 (10)
C(8)	−1645 (3)	5797 (4)	6832 (3)	360 (12)	686 (22)	365 (12)	36 (13)	133 (10)	−133 (13)
C(9)	−1648 (4)	5174 (7)	8305 (5)	517 (18)	1155 (41)	483 (17)	−107 (23)	260 (15)	−70 (22)
C(10)	−2203 (5)	7168 (6)	6557 (7)	613 (23)	728 (32)	807 (29)	222 (21)	148 (21)	−197 (24)
C(11)	4287 (3)	4589 (6)	6692 (5)	348 (13)	717 (27)	526 (18)	51 (14)	120 (12)	−43 (15)

Final shift/error for positional parameters: 0·04 (average), 0·16 (maximum)
Final shift/error for thermal parameters: 0·15 (average), 0·48 (maximum)

Anisotropic thermal factors are of the form: $\exp\left[-\frac{1}{4}\sum_{i=1}^{3}\sum_{j=1}^{3} B_{ij}h_ih_ja_i^*a_j^*\right]$

Table 2. *Parameters of hydrogen atoms and their estimated standard deviations*

	Positional parameter × 10^3			Thermal parameter		Positional parameter × 10^3			Thermal parameter
	x/a	y/b	z/c	B		x/a	y/b	z/c	B
H(4)	527 (5)	606 (5)	900 (5)	5 (1)	H(93)	−132 (7)	591 (8)	894 (8)	9 (2)
H(5)	444 (5)	754 (6)	1042 (6)	6 (1)	H(101)	−306 (8)	715 (8)	658 (8)	11 (2)
H(6)	210 (4)	761 (5)	973 (5)	5 (1)	H(102)	−177 (6)	781 (7)	738 (7)	8 (2)
H(7)	28 (3)	691 (4)	849 (4)	3 (1)	H(103)	−194 (8)	758 (9)	558 (9)	13 (3)
H(8)	−215 (4)	518 (4)	612 (5)	3 (1)	H(111)	412 (6)	482 (5)	559 (8)	7 (2)
H(91)	−248 (7)	504 (5)	820 (8)	8 (2)	H(112)	518 (6)	446 (6)	699 (7)	7 (2)
H(92)	−109 (8)	431 (9)	825 (9)	11 (2)	H(113)	398 (5)	372 (7)	655 (6)	7 (2)

Final shift/error for positional parameter: 0·08 (average), 0·43 (maximum)
Final shift/error for thermal parameter: 0·06 (average), 0·43 (maximum)

Table 3. *Observed and calculated structure factors*

Columns are *h*, $10F_o$ and $10F_c$. Unobserved reflections are marked with an asterisk.

H,0,0		
3	186	-147
4	352	358
5	1025	1019
6	324	327
7	255	267
8	59	58
9	20	-6
10	35	-43
11	98	102
12	90	95
13	44	42
H,0,2		
0	424	-397
1	283	288
2	308	325
3	377	384
4	400	412
5	344	350
6	290	291
7	237	241
8	192	190
9	168	167
10	162	165
11	107	114
12	66	66
-1	185	-163
-2	515	531
-3	549	569
-4	71	92
-5	244	-200
-6	46	-10
-7	282	284
-8	265	279
-9	120	118
-10	158	167
-11	57	53
-12	68	59
-13	91	86
-14	46	50
H,0,4		
0	120	138
1	248	270
2	92	-58
3	375	394
4	155	160
5	246	239
6	59	60
7	204	-206
8	115	117
9	53	56
10	102	102
11	29	33
-1	324	330
-2	802	793
-3	790	782
-4	250	258
-5	85	110
-6	336	337
-7	345	349
-8	416	406
-9	132	130
-10	93	90
-11	119	113
-12	45	41
-13	89	92
-14	48	48
H,0,6		
0	250	257
1	368	353
2	373	372
3	183	182
4	174	168
5	35	36
6	165	167
7	172	170
8	73	76
9	67	67
-1	208	214
-2	219	229
-3	88	89
-4	269	275
-5	506	491
-6	131	136
-7	161	149
-8	59	-65
-9	176	168
-10	225	233
-11	33	36
-12	79	75
-13	14*	12
-14	52	50
H,0,8		
0	52	50
1	105	109
2	205	195
3	131	129
4	39	46
5	33	34
6	75	72
7	27	32
-1	20*	29
-2	202	198
-3	272	274
-4	153	152
-5	102	106
-6	45	36
-7	73	73
-8	132	137
-9	107	111
-10	58	66
-11	53	44
-12	30	27
-13	9*	20
H,0,10		
0	102	103
1	99	105
2	15*	-23
3	34	36
4	44	45
-1	149	146
-2	37	38
-3	92	-102
-4	146	153
-5	125	129
-6	140	147
-7	76	76
-8	27	30
-9	91	91
-10	75	74
-11	79	88
H,0,12		
-2	50	64
-3	74	89
-4	53	59
-5	73	68
-6	83	97
H,1,0		
1	265	279
2	325	-319
3	155	159
4	21	34
5	115	115
6	49	43
7	88	-72
8	25*	14
9	27*	26
10	42	-48
11	26*	-11
12	23*	14
13	17*	-7
H,1,1		
0	703	712
1	294	318
2	779	809
3	130	154
4	498	522
5	307	307
6	254	259
7	296	283
8	161	167
9	122	114
10	79	77
11	105	100
12	60	55
13	70	64
-1	968	1030
-2	102	-80
-3	458	469
-4	149	184
-5	535	561
-6	451	431
-7	160	164
-8	79	77
-9	76	78
-10	108	93
-11	35	33
-12	110	115
-13	19*	19
H,1,2		
0	245	-232
1	397	388
2	254	-256
3	29	-29
4	19*	-2
5	21*	-10
6	48	46
7	124	-123
8	27*	34
9	27*	29
10	26*	14
11	23*	-20
12	18*	-11
-1	173	157
-2	93	74
-3	19	11
-4	186	179
-5	18*	-11
-6	125	111
-7	22*	-4
-8	24*	-15
-9	26*	19
-10	27*	6
-11	45	-49
-12	24*	-14
-13	20*	4
-14	13*	-9
H,1,3		
0	121	136
1	344	350
2	154	182
3	206	212
4	345	349
5	217	210
6	311	328
7	58	61
8	27*	26
9	125	125
10	108	107
11	113	108
12	23	21
-1	391	399
-2	435	466
-3	274	309
-4	37	-16
-5	55	69
-6	156	171
-7	309	309
-8	278	286
-9	78	79
-10	111	111
-11	110	104
-12	80	79
-13	105	101
-14	58	56
H,1,4		
0	227	-218
1	179	164
2	193	182
3	23	-29
4	27	-27
5	39	-44
6	152	157
7	27*	24
8	62	-61
9	25*	23
10	21*	2
11	28	24
-1	168	-158
-2	87	95
-3	131	-109
-4	108	-107
-5	26	-32
-6	122	-104
-7	28	-17
-8	133	-121
-9	27*	-19
-10	27*	27
-11	27*	-4
-12	24*	22
-13	21*	-18
-14	14*	6
H,1,5		
0	274	277
1	80	-67
2	208	204
3	290	292
4	216	217
5	177	176
6	103	-99
7	90	91
8	81	76
9	64	61
10	56	58
-1	399	390
-2	549	558
-3	438	417
-4	528	516
-5	118	125
-6	225	231
-7	265	276
-8	313	309
-9	376	370
-10	27	29
-11	53	47
-12	46	38
-13	84	70
-14	86	81
H,1,6		
0	81	-77
1	168	-160
2	25*	13
3	26*	-22
4	27*	13
5	82	-87
6	50	-49
7	41	36
8	27	-25
9	17*	11
-1	101	105
-2	68	40
-3	52	-51
-4	83	68
-5	50	44
-6	52	49
-7	65	64
-8	52	45
-9	114	111
-10	27*	-12
-11	26*	-22
-12	23*	18
-13	18*	8
-14	11*	14
H,1,7		
0	172	161
1	331	317
2	308	304
3	226	228
4	90	90
5	45	38
6	121	112
7	105	108
8	77	75
-1	66	66
-2	151	140
-3	247	249
-4	210	223
-5	267	273
-6	75	76
-7	27*	-20
-8	27*	-6
-9	27*	7
-10	189	180
-11	111	106
-12	21*	19
-13	16*	-16
H,1,8		
0	66	63
1	49	44
2	27*	-8
3	40	39
4	37	36
5	23*	10
6	20*	3
7	21	20
-1	88	95
-2	27*	-7
-3	78	-79
-4	45	41
-5	27*	-17
-6	27*	17
-7	54	-54
-8	46	-52
-9	39	-43
-10	49	-51
-11	22*	7
-12	18*	-11
-13	12*	-7
H,1,9		
0	86	83
1	118	110
2	52	49
3	23*	-17
4	37	40
5	58	54
-1	89	88
-2	61	-61
-3	113	111
-4	147	139
-5	99	101
-6	149	145
-7	87	85
-8	138	133
-9	105	98
-10	80	77
-11	95	89
-12	98	93
H,1,10		
0	35	-33
1	22*	-4
2	20*	-11
3	33	-32
4	13*	5
-1	31	-37
-2	88	-89
-3	25*	27
-4	31	38
-5	47	-43
-6	24*	-30
-7	24*	-14
-8	33	37
-9	21	31
-10	17*	5
-11	13*	11
H,1,11		
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-2	145	152
-3	50	43
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-6	117	118
-7	123	127
-8	48	49
-9	27	23
H,2,0		
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2	513	541
3	51	60
4	108	119
5	450	442
6	429	447
7	294	311
8	77	73
9	67	66
10	24*	21
11	103	97
12	99	103
13	54	48
H,2,1		
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1	27	22
2	17	23
3	197	-198
4	207	207
5	124	109
6	20*	8
7	78	-80
8	112	-109
9	55	-63
10	73	-73
11	22*	17
12	32	-28
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-3	150	150
-4	14*	10
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-6	127	118
-7	27	-23
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-9	63	-67
-10	79	-81
-11	23*	-19
-12	21*	3
-13	16*	-4
H,2,2		
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1	76	-42
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3	321	350
4	569	602
5	176	183
6	243	248
7	199	197
8	35	22
9	129	127
10	125	131
11	99	99
12	53	49
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-2	613	617
-3	120	136
-4	19	11
-5	120	133
-6	344	354
-7	220	228
-8	140	143
-9	132	137
-10	139	129
-11	24*	16
-12	77	72
-13	65	62
-14	54	54
H,2,3		
0	152	-142
1	16	-14
2	225	206
3	170	163
4	60	-58
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6	33	-28
7	201	207
8	84	83
9	24	32
10	31	32
11	17*	-15
12	33	31
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-2	95	-101
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-5	192	-193
-6	178	-177
-7	99	-105
-8	95	-96
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-11	24*	35
-12	35	-39
-13	18*	-6
-14	12*	0
H,2,4		
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1	123	144
2	168	177
3	331	329
4	221	225
5	194	203
6	47	41
7	41	27
8	103	100
9	83	79
10	106	104
11	25	30
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-2	258	257
-3	563	575
-4	478	482
-5	112	111
-6	166	160
-7	129	123
-8	387	388
-9	266	267
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-11	105	101
-12	50	49
-13	93	86
-14	64	58
H,2,5		
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1	133	-128
2	158	-151
3	22*	-11
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6	97	-98
7	110	-111
8	22*	-8
9	19*	-11
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-2	88	94
-3	222	203
-4	74	-62
-5	86	-81
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-9	46	-41
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-11	23*	8
-12	21*	3
-13	17*	11
-14	11*	0
H,2,6		
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1	186	184
2	299	301
3	193	190
4	180	178
5	51	51
6	33	22
7	156	163
8	65	65
9	41	39
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-2	217	222
-3	238	230
-4	309	312
-5	252	248
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-7	181	173
-8	79	74
-9	129	121
-10	100	87
-11	73	66
-12	74	70
-13	16*	17
H,2,7		
0	54	47
1	109	111
2	24*	0
3	46	-40
4	24*	16
5	23*	-11
6	61	66
7	18*	17
8	24	19
-1	85	80
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-4	33	-29
-5	120	122
-6	64	64
-7	31	-28
-8	54	-49
-9	49	-46
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-12	18*	12
-13	14*	4
H,2,8		
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1	125	119
2	157	154
3	86	80
4	63	58
5	85	81
6	61	54
7	46	44
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-2	47	38
-3	248	257
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-6	99	97
-7	47	46
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-9	67	62
-10	107	105
-11	104	109
-12	36	32
H,2,9		
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1	23*	-25
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3	20*	23
4	18*	-17
5	14*	5
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-2	74	75
-3	94	89
-4	24*	-24
-5	42	-37
-6	55	-53
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-8	22*	-0
-9	21*	-8
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-11	16*	-22
-12	29	-27
H,2,10		
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1	97	98
2	23	26
3	18	16
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-3	53	44
-4	127	125
-5	130	130
-6	133	135
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-8	57	52
-9	66	64
-10	73	72
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H,2,11		
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-5	18*	-3
-6	17*	-17
-7	16*	4
-8	14*	-15
-9	20	16
H,2,12		
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H,3,0		
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2	70	-70
3	186	-174
4	31	-23
5	39	38
6	51	45
7	52	-61
8	168	-173
9	29*	-18
10	29*	16
11	27*	11
12	22*	-10
13	33	-35
H,3,1		
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1	311	337
2	512	542
3	290	321
4	276	287
5	433	424
6	209	217
7	302	310
8	157	165
9	43	44
10	47	46
11	84	91
12	81	72
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-2	118	147
-3	141	172
-4	184	202
-5	361	367
-6	194	201
-7	151	154
-8	48	49
-9	87	87
-10	109	107
-11	98	97
-12	73	75
-13	34	33
H,3,2		
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1	21	-13
2	80	61
3	261	262
4	88	83
5	45	-41
6	70	-67
7	55	58
8	153	151
9	77	-85
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-4	78	79
-5	20*	-1
-6	155	148
-7	146	-145
-8	27*	8
-9	29*	-28
-10	55	-59
-11	28*	-13
-12	25*	-22
-13	20*	-14
-14	5*	-28
H,3,3		
0	195	221
1	267	279
2	163	-140
3	298	295
4	240	240
5	223	240
6	156	155
7	55	-48
8	117	121
9	94	97
10	96	105
11	57	58
-1	345	353
-2	607	615
-3	437	419
-4	128	149
-5	96	106
-6	254	264
-7	385	382
-8	361	355
-9	69	64
-10	88	85
-11	51	51
-12	59	54
-13	118	110
H,3,4		
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1	40	-43
2	22*	0
3	24*	9
4	26*	19
5	28*	14
6	29*	21
7	29*	-2
8	28*	22
9	37	42
10	21*	12
11	13*	16
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-2	145	-146
-3	37	-28
-4	155	-142
-5	22*	7
-6	198	-199
-7	46	-55
-8	82	-79
-9	64	-67
-10	47	51
-11	38	38
-12	25*	-7
-13	33	-32
H,3,5		
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1	258	242
2	260	249
3	268	252
4	216	206
5	185	192
6	94	94
7	28*	27
8	80	81
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10	55	56
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-2	386	368
-3	353	358
-4	218	226
-5	103	104
-6	113	109
-7	131	139
-8	129	133
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-10	92	93
-11	42	45
-12	51	46
-13	36	38
H,3,6		
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1	92	-87
2	49	-57
3	136	-137
4	29*	-5
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6	80	-74
7	34	-35
8	22*	-14
9	16*	-0
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-5	35	34
-6	49	43
-7	118	102
-8	120	116
-9	63	68
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-11	26*	-20
-12	28	31
-13	35	27
H,3,7		
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1	157	152
2	240	247
3	161	159
4	37	40
5	34	-39
6	82	82
7	88	82
8	66	63
-1	28*	-11
-2	235	216
-3	229	232
-4	227	218
-5	293	280
-6	74	66
-7	103	102
-8	65	67
-9	168	155
-10	206	205
-11	71	69
-12	31	31
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H,3,8		
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1	101	108
2	41	39
3	82	85
4	40	-45
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6	49	50
7	12*	6
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-2	129	125
-3	32	-29
-4	35	-36
-5	29*	-0
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-7	36	-35
-8	81	-85
-9	100	-105
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-11	22*	17
-12	53	-51
H,3,9		
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1	136	133
2	58	56
3	46	44
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5	65	67
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-5	124	120
-6	163	157
-7	72	78
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-9	35	36
-10	21*	12
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H,3,10		
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3	16*	-12
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-2	45	-46
-3	26*	-19
-4	26*	-13
-5	46	-47
-6	49	-48
-7	24*	-22
-8	22*	11
-9	36	38
-10	16*	6
-11	5*	-12
H,3,11		
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1	44	55
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-5	79	82
-6	74	77
-7	64	65
-8	53	58
H,4,0		
1	234	262
2	82	-66
3	132	143
4	189	205
5	291	312
6	227	236
7	152	155
8	206	210
9	98	96
10	92	92
11	83	86
12	73	77
13	55	64
H,4,1		
0	163	148
1	277	269
2	144	-140
3	64	-62
4	17*	-0
5	79	69
6	115	114
7	155	-153
8	25*	26
9	25*	0
10	49	-57
11	21*	19
12	22	-25
-1	28	-16
-2	70	67
-3	171	174
-4	28	-29
-5	26	30
-6	48	53
-7	55	52
-8	76	76
-9	43	-44
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-11	23*	-11
-12	19*	-2
-13	14*	11
H,4,2		
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1	295	299
2	124	150
3	34	54
4	435	433
5	288	293
6	223	233
7	25*	-19
8	31	-30
9	58	52
10	59	66
11	94	103
12	12*	16
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-2	518	512
-3	495	477
-4	56	66
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-6	225	226
-7	361	360
-8	221	222
-9	30	27
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-11	23*	7
-12	87	92
-13	49	52
H,4,3		
0	304	-293
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2	119	107
3	165	160
4	33	34
5	76	-72
6	47	44
7	75	73
8	25*	5
9	23*	30
10	20*	19
11	15*	-7
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-3	359	-329
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-5	86	-94
-6	36	25
-7	44	-47
-8	228	-232
-9	25*	10
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-11	24*	-16
-12	38	-42
-13	25	-25
H,4,4		
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1	182	188
2	348	362
3	280	300
4	157	150
5	155	158
6	115	109
7	138	130
8	124	126
9	118	116
10	45	46
-1	83	-65
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-3	334	337
-4	220	219
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-6	66	67
-7	144	143
-8	155	142
-9	166	161
-10	109	113
-11	102	105
-12	20*	24
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H,4,5		
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1	59	-59
2	44	-36
3	89	-77
4	46	-50
5	25*	-3
6	25*	4
7	41	-38
8	21*	-26
9	41	38
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-2	107	-94
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-4	70	21
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-6	77	-73
-7	24*	-17
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-9	80	82
-10	25*	4
-11	23*	-1
-12	20	31
-13	15*	5
H,4,6		
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1	129	118
2	147	134
3	199	193
4	108	105
5	65	62
6	23*	3
7	36	42
8	37	40
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-2	327	331
-3	193	182
-4	250	246
-5	145	149
-6	154	154
-7	148	144
-8	102	99
-9	147	143
-10	87	85
-11	43	45
-12	22	26
-13	39	42
H,4,7		
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1	48	51
2	25*	1
3	25*	-3
4	38	50
5	67	-72
6	20*	16
7	29	34
8	8*	-22
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-3	80	-82
-4	54	58
-5	66	71
-6	79	86
-7	25*	9
-8	41	-43
-9	31	-27
-10	22*	-19
-11	28	-33
-12	16*	11
H,4,8		
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1	181	176
2	97	102
3	75	69
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6	93	105
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-2	28	25
-3	93	99
-4	117	124
-5	161	167
-6	114	115
-7	34	31
-8	82	88
-9	71	75
-10	122	121
-11	71	74
-12	53	53
H,4,9		
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1	22*	-14
2	21*	18
3	19*	12
4	16*	1
5	18	24
-1	41	-44
-2	34	-39
-3	24*	7
-4	24*	-25
-5	79	-84
-6	33	-34
-7	36	-37
-8	21*	-10
-9	20*	-16
-10	38	-44
-11	29	29
H,4,10		
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1	79	77
2	64	67
3	28	32
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-3	81	85
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-5	116	117
-6	78	78
-7	59	71
-8	42	47
-9	46	48
-10	39	43
H,4,11		
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-1	14*	13
-2	15*	-19
-3	16*	-3
-4	23	25
-5	16*	-4
-6	15*	-23
-7	13*	15
-8	26	29
H,5,0		
1	119	-115
2	235	-243
3	180	-191
4	142	138
5	47	-42
6	24*	-13
7	199	-202
8	126	-132
9	85	87
10	26*	-8
11	22*	27
12	18	-24
H,5,1		
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1	249	263
2	205	222
3	17*	6
4	167	173
5	252	256
6	200	200
7	115	116
8	37	-47
9	96	92
10	86	87
11	61	62
12	36	42
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-2	324	324
-3	123	136
-4	246	251
-5	158	160
-6	238	237
-7	203	202
-8	120	125
-9	169	170
-10	47	50
-11	66	76
-12	66	74
H,5,2		
0	145	147
1	126	127
2	182	169
3	89	74
4	48	43
5	93	90
6	26*	18
7	88	89
8	43	46
9	26*	-32
10	23*	-4
11	18*	-16
-1	83	78
-2	201	-184
-3	107	-98
-4	107	92
-5	21*	8
-6	23*	-17
-7	208	-202
-8	128	-124
-9	42	42
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-11	24*	-8
-12	41	-43
-13	13*	-16
H,5,3		
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1	194	198
2	189	191
3	267	256
4	233	220
5	150	149
6	136	141
7	120	121
8	48	45
9	24*	27
10	63	75
11	32	35
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-3	227	231
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-6	321	321
-7	192	198
-8	200	204
-9	75	85
-10	27*	15
-11	34	36
-12	59	61
-13	60	65
H,5,4		
0	201	-196
1	58	-54
2	23*	-0
3	43	42
4	116	-117
5	43	-53
6	27*	-10
7	27*	18
8	24*	-15
9	37	39
10	27	33
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-3	21*	8
-4	22*	11
-5	62	-67
-6	81	-78
-7	77	79
-8	27*	26
-9	27*	14
-10	26*	7
-11	24*	14
-12	20*	-14
-13	14*	3
H,5,5		
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1	60	63
2	226	221
3	243	235
4	231	228
5	79	74
6	39	42
7	86	88
8	83	88
9	60	62
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-3	286	270
-4	173	159
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-6	34	32
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-8	122	126
-9	122	121
-10	76	75
-11	45	48
-12	19*	18
-13	12*	17
H,5,6		
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1	26*	1
2	27*	-21
3	65	-60
4	52	56
5	31	28
6	24*	-13
7	21*	-10
8	16*	-8
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-2	127	-117
-3	41	-37
-4	26*	-18
-5	26*	24
-6	54	61
-7	27*	-20
-8	27*	-10
-9	26	-33
-10	33	-32
-11	22*	-18
-12	37	43
H,5,7		
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1	44	62
2	125	121
3	40	39
4	83	80
5	31	28
6	20*	-4
7	52	55
-1	31	28
-2	40	26
-3	235	218
-4	236	216
-5	170	165
-6	56	52
-7	106	101
-8	146	145
-9	145	152
-10	95	106
-11	71	76
-12	49	53
H,5,8		
0	50	-46
1	26*	16
2	25*	18
3	29	32
4	24	-22
5	44	-46
6	22	25
-1	48	-44
-2	27*	14
-3	27*	2
-4	27*	0
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-6	27*	-9
-7	26*	-24
-8	25*	-9
-9	23*	-10
-10	35	40
-11	16*	1
H,5,9		
0	164	166
1	126	129
2	98	96
3	31	36
4	16	15
-1	90	83
-2	84	78
-3	98	97
-4	94	94
-5	115	119
-6	151	155
-7	79	83
-8	22*	23
-9	19*	-11
-10	29	31
H,5,10		
0	29	31
1	22	-25
2	12*	-10
-1	20*	4
-2	21*	-6
-3	21*	4
-4	21*	-14
-5	63	-68
-6	37	-39
-7	19*	22
-8	17*	-2
-9	13*	-2
H,5,11		
-2	30	35
-3	32	33
-4	17	23
-5	23	27
-6	39	43
H,6,0		
1	227	239
2	178	169
3	118	117
4	82	90
5	177	178
6	190	188
7	164	161
8	60	61
9	24*	27
10	80	85
11	68	74
12	60	65
H,6,1		
0	20	23
1	138	-132
2	134	-134
3	124	113
4	47	28
5	22*	14
6	61	-52
7	25*	-25
8	103	109
9	24*	-7
10	21*	15
11	16*	-5
-1	68	74
-2	251	-245
-3	65	66
-4	38	44
-5	20*	11
-6	22*	-4
-7	157	-154
-8	151	154
-9	25	33
-10	23*	-20
-11	20*	-1
-12	17	-22
H,6,2		
0	355	328
1	108	106
2	54	60
3	154	152
4	226	217
5	218	217
6	99	104
7	25*	18
8	44	47
9	36	37
10	57	59
11	42	46
-1	386	369
-2	344	335
-3	241	248
-4	128	124
-5	181	175
-6	198	197
-7	261	256
-8	188	202
-9	102	108
-10	41	43
-11	39	33
-12	65	65
H,6,3		
0	30	18
1	18*	10
2	20*	-10
3	25	22
4	57	69
5	24*	-1
6	38	-35
7	25*	-3
8	23*	-7
9	34	-38
10	29	33
-1	141	138
-2	17*	-10
-3	41	37
-4	19*	26
-5	44	-41
-6	23*	-11
-7	24*	-8
-8	25*	-13
-9	41	-44
-10	23*	-28
-11	21*	-19
-12	16*	-10
H,6,4		
0	172	176
1	169	165
2	226	221
3	265	254
4	187	192
5	111	113
6	73	73
7	87	86
8	80	79
9	54	61
-1	244	230
-2	303	290
-3	188	179
-4	165	163
-5	125	125
-6	96	100
-7	83	79
-8	42	46
-9	65	65
-10	61	65
-11	26	24
-12	16*	12
H,6,5		
0	94	-88
1	31	-31
2	73	74
3	60	-61
4	49	-53
5	33	41
6	23*	-0
7	21*	17
8	29	33
-1	263	-260
-2	138	-128
-3	118	119
-4	71	-67
-5	24*	14
-6	96	-99
-7	25*	6
-8	25*	25
-9	24*	-21
-10	22*	14
-11	36	44
-12	15*	16
H,6,6		
0	44	39
1	102	97
2	98	101
3	97	97
4	54	51
5	72	67
6	36	38
7	36	37
-1	24*	-17
-2	86	82
-3	186	175
-4	173	169
-5	57	52
-6	51	36
-7	68	77
-8	146	140
-9	129	136
-10	85	84
-11	61	68
-12	45	49
H,6,7		
0	25*	23
1	25*	2
2	28	-32
3	23*	5
4	22*	3
5	19*	-6
6	28	-33
-1	43	48
-2	68	75
-3	29	-34
-4	25*	6
-5	25*	32
-6	49	55
-7	24*	26
-8	32	-38
-9	22*	-29
-10	19*	2
-11	24	-25
H,6,8		
0	106	105
1	106	103
2	69	71
3	63	68
4	31	38
5	20	18
-1	136	135
-2	141	134
-3	105	104
-4	124	125
-5	158	154
-6	121	130
-7	98	103
-8	61	61
-9	59	61
-10	56	68
H,6,9		
0	20*	-4
1	19*	25
2	17*	-9
3	13*	16
-1	52	-53
-2	22*	15
-3	22*	-18
-4	60	-65
-5	50	-56
-6	21*	0
-7	19*	-22
-8	18*	-6
-9	22	-27
H,6,10		
0	59	65
1	79	86
-1	27	34
-2	17*	19
-3	31	35
-4	38	51
-5	61	65
-6	53	62
-7	14*	21
H,7,0		
1	31	26
2	39	-37
3	44	-38
4	82	85
5	38	-28
6	77	77
7	52	-57
8	29*	-4
9	43	48
10	23*	-10
11	40	39
H,7,1		
0	200	222
1	86	93
2	48	-49
3	73	75
4	130	134
5	120	122
6	73	68
7	34	-41
8	39	43
9	58	55
10	54	60
11	60	64
-1	201	211
-2	272	289
-3	245	265
-4	90	88
-5	146	142
-6	185	188
-7	250	265
-8	195	204
-9	28*	14
-10	53	49
-11	77	71
H,7,2		
0	51	-47
1	59	-58
2	21*	8
3	24	31
4	26*	27
5	28*	0
6	43	-51
7	29*	-10
8	27*	30
9	24*	3
10	18*	-2
-1	17	-11
-2	18*	-13
-3	26	-21
-4	41	46
-5	65	-67
-6	27*	-22
-7	48	-51
-8	29*	-26
-9	63	67
-10	25*	-17
-11	21*	-4
H,7,3		
0	380	400
1	235	222
2	144	139
3	131	128
4	206	211
5	210	220
6	107	106
7	63	64
8	64	64
9	46	36
10	10*	8
-1	259	250
-2	153	150
-3	121	125
-4	113	112
-5	192	183
-6	105	106
-7	96	94
-8	36	38
-9	40	44
-10	52	51
-11	21*	14
-12	33	37
H,7,4		
0	35	-32
1	57	57
2	27*	-20
3	34	27
4	54	-60
5	29*	-4
6	28*	21
7	25*	1
8	21*	-18
9	14*	18
-1	88	-72
-2	109	110
-3	55	-51
-4	26*	28
-5	27*	-0
-6	28*	27
-7	41	44
-8	29*	-26
-9	27*	-8
-10	25*	4
-11	20*	22
-12	12*	-9
H,7,5		
0	74	-81
1	87	91
2	155	147
3	167	164
4	49	42
5	37	39
6	64	65
7	28	28
8	51	56
-1	52	-59
-2	273	278
-3	255	241
-4	90	92
-5	28*	13
-6	29*	13
-7	121	129
-8	129	128
-9	97	97
-10	87	86
-11	39	35
H,7,6		
0	29*	6
1	34	37
2	29*	12
3	28*	-22
4	27*	26
5	24*	0
6	21*	13
7	14*	-2
-1	71	76
-2	98	-99
-3	29*	-5
-4	29*	9
-5	29*	5
-6	29*	9
-7	62	-66
-8	27*	-25
-9	25*	-17
-10	22*	-20
-11	27	-24
H,7,7		
0	89	85
1	105	103
2	57	58
3	69	71
4	49	53
5	47	43
-1	130	132
-2	105	107
-3	53	47
-4	126	124
-5	107	109
-6	105	110
-7	62	60
-8	45	39
-9	76	80
-10	60	59
H,7,8		
0	42	-52
1	24*	-12
2	23*	-12
3	20*	2
4	15*	-0
-1	26*	-8
-2	27*	0
-3	32	-23
-4	36	-35
-5	26*	-10
-6	25*	-16
-7	24*	-2
-8	22*	7
-9	18*	-3
-10	12*	11
H,7,9		
0	51	48
1	84	85
2	66	76
-1	57	57
-2	44	45
-3	30	34
-4	79	88
-5	129	144
-6	75	88
-7	26	28
-8	16*	18
H,7,10		
-2	14*	3
-3	16*	7
-4	16*	9
-5	15*	-17
H,8,0		
1	203	203
2	175	172
3	14*	12
4	95	87
5	118	112
6	170	168
7	112	116
8	29	-29
9	42	40
10	53	49
H,8,1		
0	37	-38
1	46	49
2	24	-34
3	15	-6
4	17*	-6
5	24	30
6	35	40
7	49	-53
8	18*	4
9	15*	18
10	11*	9
-1	48	-46
-2	12*	-0
-3	33	34
-4	67	-68
-5	41	-45
-6	19*	-19
-7	19*	-23
-8	19*	0
-9	51	-55
-10	14*	5
H,8,2		
0	203	182
1	102	101
2	53	51
3	120	110
4	102	101
5	158	154
6	67	68
7	36	41
8	40	41
9	55	50
-1	171	159
-2	163	147
-3	132	125
-4	166	170
-5	77	82
-6	121	119
-7	99	97
-8	153	159
-9	122	124
-10	15*	-0
-11	33	41
H,8,3		
0	145	-149
1	16*	-15
2	17*	-0
3	18*	15
4	21	-11
5	42	-52
6	18*	-4
7	17*	3
8	14*	-12
9	9*	6
-1	15*	7
-2	65	72
-3	36	-35
-4	17*	13
-5	18*	-10
-6	19*	17
-7	19*	28
-8	19*	-21
-9	42	53
-10	35	41
-11	10*	-9
H,8,4		
0	53	46
1	107	92
2	147	143
3	150	154
4	161	162
5	31	31
6	49	51
7	59	60
8	28	26
-1	282	276
-2	190	186
-3	146	142
-4	100	92
-5	77	71
-6	101	109
-7	64	66
-8	56	59
-9	28	27
-10	39	40
-11	10*	7
H,8,5		
0	63	62
1	19*	21
2	44	52
3	19*	10
4	18*	5
5	17*	-1
6	15*	16
7	11*	7
-1	24	29
-2	24	11
-3	19*	-8
-4	19*	5
-5	51	55
-6	19*	-6
-7	19*	-26
-8	39	-44
-9	16*	-19
-10	30	-29
H,8,6		
0	54	53
1	63	60
2	101	104
3	38	40
4	53	55
5	43	45
6	46	49
-1	19*	8
-2	19*	-12
-3	151	150
-4	73	74
-5	46	49
-6	29	24
-7	41	33
-8	102	105
-9	65	72
-10	56	60
H,8,7		
0	37	-36
1	17*	-5
2	16*	-8
3	15*	-19
4	12*	10
-1	18*	-3
-2	73	-80
-3	67	-75
-4	18*	13
-5	21	-21
-6	24	-24
-7	30	-38
-8	15*	-18
-9	12*	5
-10	5*	2
H,8,8		
0	94	93
1	33	35
2	53	53
3	37	42
-1	85	88
-2	78	83
-3	62	66
-4	112	119
-5	121	128
-6	71	75
-7	64	68
-8	38	39
H,8,9		
-1	17	26
-2	22	27

ecule and the angles between these planes. Distances of various atoms from these least squares planes are shown in Table 6. The nickel atom is found to be significantly (0·381 Å) out of the salicylaldimine plane, as is the case in many salicylaldiminate complexes.

Table 4. *Coefficients of least-squares plane equations*

$A(x/a)+B(y/b)+C(z/c)=D$

	A	*B*	*C*	*D*
Coordination plane	2·571	9·119	−3·699	2·710
Benzene plane	2·304	7·757	−5·987	0·591
Salicylaldimine plane	1·831	7·879	−5·838	0·639
Isopropyl plane	8·995	4·004	1·709	2·009

Table 5. *Angles between least-squares planes*

Plane 1	Plane 2	Angle
Coordination	Benzene	16·9°
Coordination	Salicylaldimine	16·8
Coordination	Isopropyl	66·3
Salicylaldimine	Benzene	2·5
Salicylaldimine	Isopropyl	79·2
Benzene	Isopropyl	77·6

Table 6. *Distances from least-squares planes*

The planes are determined by the atoms marked with*

	Coordination plane $\sigma=0{\cdot}0$	Benzene plane $\sigma=0{\cdot}008$ Å	Salicylaldimine plane $\sigma=0{\cdot}050$ Å
Ni	0·0 Å*	−0·294 Å	−0·381 Å
O	0·0*	−0·039	−0·060*
N	0·0*	0·187	0·049*
C(1)	−0·620	0·005*	−0·058*
C(2)	−0·345	−0·012*	−0·026*
C(3)	−0·443	0·008*	0·050*
C(4)	−0·838	0·003*	0·053*
C(5)	−1·137	−0·010*	−0·008*
C(6)	−1·019	0·006*	−0·048*
C(7)	−0·359	0·170	0·049*
C(8)	0·374	0·564	0·361
C(9)	1·487	1·930	1·713
C(10)	−0·835	−0·536	−0·777
C(11)	−0·101	0·051	0·146

Bond lengths and angles are shown in Fig. 2 and are listed along with their estimated standard deviations in Tables 7 and 8. The nine bond lengths of the salicylaldimine residue agree, within 0·01 Å, with the average values for eight salicylaldiminate complexes determined by three-dimensional X-ray analysis and agree, within 0·02 Å, with the values calculated by the Hückel molecular orbital method (Lingafelter & Braun, 1966).

The Ni–O and Ni–N distances of 1·837 Å and 1·920 Å in this planar complex are shorter than the corresponding average distances of 1·896 Å and 1·970 Å in the related tetrahedral complex bis(*N*-isopropylsalicylaldminato)nickel (Fox, Orioli, Lingafelter & Sacconi, 1964). This shortening of the nickel coordination bond distances in going from tetrahedral to planar coordination may be a simple consequence of the greater strength of the σ bonds in the planar complex, which according to Pauling (1960) is 'essentially covalent', than in the tetrahedral complex, which is 'essentially ionic'. In addition, however, the shortening of these bond distances may be partially due to an increased amount of d_π-p_π interaction in the Ni–O and Ni–N bonds in the planar complex.

There is good agreement between the Ni–O and Ni–N bond distances in this planar complex and the corresponding distances in other nickel salicylaldimine chelates having planar coordination configuration. For comparison, these two bond distances are 1·825 and 1·908 Å in bis(*N*-phenylsalicylaldiminato)nickel (Chastain & Lingafelter, to be published), 1·851 and 1·922 Å in the α(orthorhombic) form of bis(*N*-methylsalicylaldiminato)nickel (Fox & Lingafelter, to be published), and 1·80 and 1·90 Å in the β(monoclinic) form of bis-(*N*-methylsalicylaldiminato)nickel (Frasson, Panattoni & Sacconi, 1959).

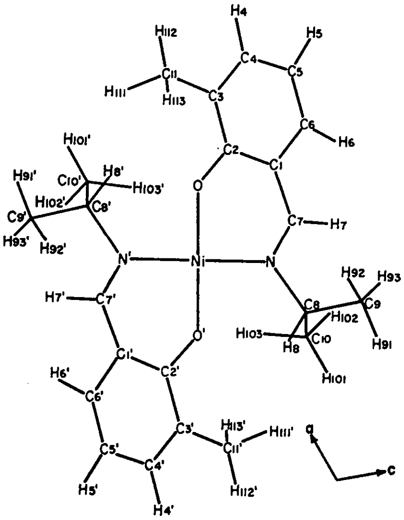

Fig. 1. Projection on (010).

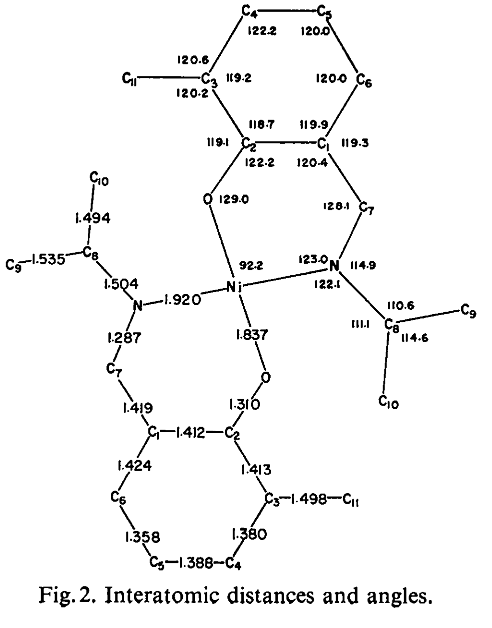

Fig. 2. Interatomic distances and angles.

The chelate 'bite' distance (*i.e.* the distance between the oxygen atom and nitrogen atom in the chelate ring) is 2·707 Å in this planar complex. As in the other three planar complexes mentioned above, this distance is shorter than the 2·836 Å observed in tetrahedral bis(*N*-isopropylsalicylaldiminato)nickel (Fox, Orioli, Lingafelter & Sacconi, 1964) which in turn is shorter than the 2·913 Å observed in octahedral diaquobis(salicylaldehydato)nickel (Stewart, Lingafelter & Breazeale, 1961).

Table 9 shows the intermolecular distance from each atom to the nearest heavy atom and also to the nearest hydrogen atom. These distances are normal, the closest intermolecular contacts being 2·4 Å for H---H, 2·61 Å for O---H, 3·73 Å for N---H, 2·85 Å for C(sp^2)---H, 3·13 Å for C(sp^3)---H, 3·382 Å for C(sp^2)---C(sp^2), 3·647 Å for C(sp^2)---C(sp^3), and > 5·0 Å for Ni---Ni.

It has been suggested that the tendency toward formation of tetrahedrally coordinated species when sec-alkyl groups are substituted on the nitrogen atom of salicylaldimine is due to steric effects (Sacconi, Paoletti & Ciampolini, 1963). Examination of the non-bonded intramolecular distances, listed in Table 10, shows that this is clearly the case. There are several close contacts which could be relieved by distortion from planar coordination. The distance of 2·20 Å between H(8) and O′ is considerably shorter than 2·6 Å, the sum of the van der Waals radii. That an attempt has been made partially to relieve this close contact is evident from the bond angle C(9)–C(8)–H(8) of 103°. However, in so doing, H(8) is now only 2·2 Å from H(92) and 2·1 Å from H(91), both shorter than 2·4 Å, the sum of the van der Waals radii. In addition, H(93) has been pushed to within 2·2 Å of H(7). Unfortunately, the corresponding distances in the related tetrahedral bis(*N*-isopropylsalicylaldiminato)nickel are not known accurately for comparison, since the hydrogen positions in that structure were not experimentally determined. However, intramolecular distances based upon calculated hydrogen positions do not show the close contacts evident in the planar complex. For example, the two distances in the tetrahedral complex which correspond to the

Table 7. *Bond distances and their e.s.d.'s*

	Distance	e.s.d.		Distance	e.s.d.
Ni —O	1·837 Å	0·002 Å	C(4)—H(4)	0·96 Å	0·05 Å
Ni —N	1·920	0·003	C(5)—H(5)	1·04	0·05
O——C(2)	1·310	0·003	C(6)—H(6)	0·98	0·06
N——C(7)	1·287	0·004	C(7)—H(7)	0·99	0·05
N——C(8)	1·504	0·004	C(8)—H(8)	0·97	0·04
C(1)–C(2)	1·412	0·004	C(9)—H(91)	0·92	0·08
C(1)–C(6)	1·424	0·004	C(9)—H(92)	1·07	0·09
C(1)–C(7)	1·419	0·004	C(9)—H(93)	0·96	0·08
C(2)–C(3)	1·413	0·004	C(10)–H(101)	0·97	0·09
C(3)–C(4)	1·380	0·005	C(10)–H(102)	1·02	0·06
C(3)–C(11)	1·498	0·006	C(10)–H(103)	1·14	0·10
C(4)–C(5)	1·388	0·006	C(11)–H(111)	1·04	0·07
C(5)–C(6)	1·358	0·005	C(11)–H(112)	0·97	0·07
C(8)–C(9)	1·535	0·006	C(11)–H(113)	0·93	0·07
C(8)–C(10)	1·494	0·007			

Table 8. *Bond angles and their e.s.d.'s, in degrees*

Apex	End	End	Angle	e.s.d.	Apex	End	End	Angle	e.s.d.
Ni	O	N	92·2°	0·1°	C(4)	C(3)	H(4)	116°	3°
O	Ni	C(2)	129·9	0·2	C(4)	C(5)	H(4)	122	3
N	Ni	C(7)	123·0	0·2	C(5)	C(4)	H(5)	117	3
N	Ni	C(8)	122·1	0·2	C(5)	C(6)	H(5)	123	3
N	C(7)	C(8)	114·9	0·3	C(6)	C(1)	H(6)	113	3
C(1)	C(2)	C(6)	119·9	0·3	C(6)	C(5)	H(6)	127	3
C(1)	C(2)	C(7)	120·4	0·3	C(7)	N	H(7)	123	2
C(1)	C(6)	C(7)	119·3	0·3	C(7)	C(1)	H(7)	109	2
C(2)	O	C(1)	122·2	0·3	C(8)	N	H(8)	108	3
C(2)	O	C(3)	119·1	0·3	C(8)	C(9)	H(8)	103	3
C(2)	C(1)	C(3)	118·7	0·3	C(8)	C(10)	H(8)	109	3
C(3)	C(2)	C(4)	119·2	0·3	C(9)	C(8)	H(91)	104	4
C(3)	C(2)	C(11)	120·2	0·3	C(9)	C(8)	H(92)	97	5
C(3)	C(4)	C(11)	120·6	0·3	C(9)	C(8)	H(93)	99	5
C(4)	C(3)	C(5)	122·2	0·3	C(10)	C(8)	H(101)	110	5
C(5)	C(4)	C(6)	120·0	0·3	C(10)	C(8)	H(102)	112	4
C(6)	C(1)	C(5)	120·0	0·4	C(10)	C(8)	H(103)	106	5
C(7)	N	C(1)	128·1	0·3	C(11)	C(3)	H(111)	117	4
C(8)	N	C(9)	110·6	0·3	C(11)	C(3)	H(112)	121	4
C(8)	N	C(10)	111·1	0·4	C(11)	C(3)	H(113)	115	4
C(8)	C(9)	C(10)	114·6	0·4					

H(8)---O′ contact are both greater than 4·6 Å. In addition, closer intramolecular contacts in the planar complex than in the tetrahedral complex are illustrated by comparing the distances from the isopropyl carbon atoms to atom C(7) and atom O′. The important distances to be compared here for bis(*N*-isopropyl-3-methylsalicylaldiminato)nickel and bis(*N*-isopropyl-salicylaldiminato)nickel are, respectively: 2·871 and 3·324 Å for C(9)---C(7), 2·997 and 3·469 Å for C(10)---C(7), and 2·745 and 3·651 Å for C(8)---O′. The C(9)---O′ and C(10)---O′ contacts appear to be normal in both complexes, although the C(10)---O′ distance of 3·358 Å in the planar complex is very slightly less than the sum of the van der Waals radii for the methyl group and oxygen, 3·4 Å.

There is some slight evidence of intramolecular steric hindrance between the 3-methyl group and the *N*-isopropyl group. The closest C---C contact is the C(10)---C(11′) distance of 3·708 Å. This is probably not a seriously close contact, however, since the minimum C---H distances are 3·17 Å between C(10) and H(111′) and 3·16 Å between C(10) and H(113′), practically within the sum of the van der Waals radii, 3·2 Å, and since the minimum H---H distances are 2·8 Å between

Table 10. *Intramolecular distances*

The primed atoms are related to the unprimed atoms by the center of symmetry.

	C(9)	H(91)	H(92)	H(93)
C(7)	2·871 Å	3·73 Å	2·85 Å	2·66 Å
H(7)	2·74	3·6	3·0	2·2
H(8)	1·99	2·1	2·2	2·7
O′	4·055	4·32	3·95	4·64
C(11′)	4·793	4·51	> 5·0	> 5·0
H(111′)	3·90	3·5	4·3	4·6
H(112′)	> 5·0	5·0	> 5·0	> 5·0
H(113′)	4·71	4·5	> 5·0	> 5·0
	C(10)	H(101)	H(102)	H(103)
C(7)	2·997	3·88	2·85	3·12
H(7)	2·85	3·6	2·4	3·2
H(8)	2·03	2·3	2·9	2·5
O′	3·358	3·84	4·22	2·95
C(11′)	3·708	3·47	4·71	3·59
H(111′)	3·17	2·8	4·2	3·4
H(112′)	4·29	3·9	> 5·0	4·2
H(113′)	3·16	3·0	4·1	2·9
	C(11)	H(111)	H(112)	H(113)
O	2·807	2·87	3·77	2·74
H(4)	2·60	3·4	2·5	3·3
	C(8′)	H(8′)	N	
O	2·745	2·20	2·707	

Table 9. *Closest intermolecular contacts*

Key for molecule positions

1.	x	y	z	8.	$1-x$	$1-y$	$2-z$
2.	$-x$	$1-y$	$2-z$	9.	$1-x$	$\frac{1}{2}+y$	$\frac{3}{2}-z$
3.	$-x$	$\frac{1}{2}+y$	$\frac{3}{2}-z$	10.	$1-x$	$y-\frac{1}{2}$	$\frac{3}{2}-z$
4.	$-x$	$y-\frac{1}{2}$	$\frac{3}{2}-z$	11.	$1+x$	y	z
5.	x	$\frac{3}{2}-y$	$\frac{1}{2}+z$	12.	$x-1$	y	z
6.	x	$\frac{3}{2}-y$	$z-\frac{1}{2}$	13.	$x-1$	$\frac{3}{2}-y$	$z-\frac{1}{2}$
7.	$1-x$	$1-y$	$1-z$				

Atom	Nearest non-hydrogen	Molecule position	Distance	Nearest hydrogen	Molecule position	Distance
Ni	C(6)	4	4·311 Å	H(6)	4	3·41 Å
O	C(10)	4	3·550	H(102)	4	2·61
N	C(6)	4	4·378	H(102)	4	3·73
C(1)	C(9)	2	3·913	H(92)	3	3·16
C(2)	C(10)	4	3·648	H(6)	6	2·85
C(3)	C(5)	6	3·828	H(5)	6	3·12
C(4)	C(4)	8	3·382	H(4)	8	3·06
C(5)	C(11)	9	3·732	H(112)	9	3·22
C(6)	C(9)	2	3·647	H(8)	3	3·11
C(7)	C(9)	2	3·917	H(92)	3	3·22
C(8)	C(6)	4	3·882	H(6)	4	3·48
C(9)	C(6)	2	3·647	H(103)	5	3·20
C(10)	O	3	3·550	H(113)	3	3·43
C(11)	C(5)	10	3·732	H(5)	6	3·13
H(4)	C(4)	8	3·06	H(113)	9	2·9
H(5)	C(3)	5	3·12	H(111)	5	2·7
H(6)	O	5	2·82	H(8)	3	2·7
H(7)	Ni	5	3·45	H(92)	3	3·2
H(8)	C(6)	4	3·11	H(6)	4	2·7
H(91)	C(6)	2	3·28	H(112)	12	2·6
H(92)	C(1)	4	3·16	H(6)	2	3·1
H(93)	C(10)	5	3·51	H(103)	5	2·4
H(101)	C(11)	3	3·45	H(5)	13	2·7
H(102)	O	3	2·61	H(113)	3	3·1
H(103)	C(9)	6	3·20	H(93)	6	2·4
H(111)	C(11)	7	3·25	H(111)	7	2·6
H(112)	C(5)	10	3·22	H(91)	11	2·6
H(113)	C(4)	10	3·17	H(101)	4	2·8

H(101) and H(111′) and 2·9 Å between H(103) and H(113′), well above the sum of the van der Waals radii, 2·4 Å. Although metal chelate scale models (Holm & Swaminathan, 1963) have indicated unfavorable interaction in the planar structure between the *N*-isopropyl group and the 3-substituent of the adjacent benzene ring, this interaction is evidently somewhat alleviated by the 'step' of about 0·76 Å (see Table 6) between the two salicylaldimine residues and possibly by distortion of the bond angle Ni–N–C(8) to 122·1°.

It is still not clear why bis(*N*-isopropyl-3-methylsalicylaldiminato)nickel is planar in the solid. Our attempts to understand this in terms of molecular volumes in the solid or in terms of total crystal packing energy have so far been unsuccessful. Further details of intra- and intermolecular contacts in related planar and tetrahedral complexes may lead to an explanation. We are proceeding to determine the crystal structures of bis(*N*-isopropylsalicylaldiminato)palladium, bis(*N*-isopropyl-3-methylsalicylaldiminato)palladium, bis(*N*-isopropyl-3-ethylsalicylaldiminato)palladium, bis(*N*-isopropyl-3-methylsalicylaldiminato)zinc, and bis(*N*-isopropyl-3-ethylsalicylaldiminato)nickel.

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Structure Cristalline de l'Acide Dodécanedioïque, COOH[CH$_2$]$_{10}$COOH Caractères Structuraux des Diacides Aliphatiques Saturés a Nombre Pair de Carbones

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(*Reçu le* 9 *février* 1966)

The first part of this paper gives results on the structure of dodecanedioic acid. In the second part, the authors discuss their crystallographic results on various compounds of this family of diacids, with particular reference to the effect of hydrogen atoms on the parameters of carbon atoms and the C–C bonds, distances and angles in the carboxylic group, the location of hydrogen atoms of the bond, and the importance of intermolecular forces.

La structure de l'acide dodécanedioïque, s'apparente directement à celles des acides adipique, subérique et sébacique (Housty & Hospital, 1964, 1965*a*, *b*); nous avons pu la déterminer par analyse directe des projections de la fonction de Patterson.

Nous allons diviser cette communication en deux parties principales: tout d'abord nous donnerons les résultats concernant la structure cristalline de l'acide dodécanedioïque, puis nous ferons une étude critique des résultats concernant la série des diacides pairs.

I. STRUCTURE CRISTALLINE DE L'ACIDE DODÉCANEDIOÏQUE

Maille et groupe spatial

Nous avons obtenu des monocristaux par évaporation très lente d'une solution d'acide dodécanedioïque dans l'acide formique.

Ce produit cristallise dans le système monoclinique avec les paramètres cristallins suivants: