Acta Cryst. (1967). **23**, 127

The Crystal Structures of the Tetragonal and Monoclinic Forms of Bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium

By Prem C. Jain and E. C. Lingafelter

Department of Chemistry, University of Washington, Seattle, Washington 98105, *U.S.A.*

(*Received* 9 *September* 1966 *and in revised form* 28 *November* 1966)

Bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium crystallizes from ethanol solution in two crystalline forms, tetragonal and monoclinic. The monoclinic form is isomorphous with bis-(*N*-isopropyl-3-methylsalicylaldiminato)nickel. The crystal structures of both forms have been determined from three-dimensional X-ray diffraction data. The cell dimensions for the tetragonal form are: $a_0 = 18{\cdot}178$, $c_0 = 12{\cdot}879$ Å; and for the monoclinic form are: $a_0 = 11{\cdot}297$, $b_0 = 9{\cdot}755$, $c_0 = 9{\cdot}844$ Å, $\beta = 107°51'$. The space group for the tetragonal form is $I4_1/a$ with eight molecules per cell and for the monoclinic form is $P2_1/c$ with two molecules per cell. The palladium atoms have trans-planar coordination in both cases. The bond distances and angles in the chelate are similar to those reported for other salicylaldimine complexes, but differences in packing cause significant bending of the nearly planar molecules.

Introduction

In an attempt to understand the structural reasons for the variations in coordination configuration in a series of substituted salicylaldimine chelates of nickel(II), we have undertaken the investigation of the crystal structures of a number of salicylaldimine chelates (Fox, Orioli, Lingafelter & Sacconi, 1964; Braun & Lingafelter, 1966, 1967*a*,*b*). The palladium(II) ion has been selected as an ion whose coordination configuration would probably be planar in all cases (Sacconi, Ciampolini, Maggio & Del Re, 1960). We have now completed the structural investigation of the two forms of bis-(*N*-isopropyl-3-methylsalicylaldiminato) palladium.

Experimental

Bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium was prepared by the method of Sacconi, Paoletti & Del Re (1957), using 3-methylsalicylaldehyde which was prepared by the method of Duff (1941). Purification and recrystallization were carried out from ethyl alcohol by slow evaporation. The crystals are yellow in color and two forms of crystals are easily separable under the microscope. The crystals of the tetragonal form are rectangular with pyramidal ends while those of the monoclinic form are rhomb-shaped plates. Well-defined crystals of the two forms were chosen for study. They had approximate dimensions $0{\cdot}09 \times 0{\cdot}10 \times 0{\cdot}12$ mm and $0{\cdot}14 \times 0{\cdot}15 \times 0{\cdot}03$ mm, respectively. All measurements were made with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å) on a Picker Single Crystal Diffractometer equipped with pulse-height analyser and a General Electric Single Crystal Orienter (Furnas, 1957). The cell constants and their estimated standard deviations were obtained by least-squares fit of 21 θ values in the case of the tetragonal form and of 10 θ values in the case of the monoclinic form. The final values are: tetragonal, $a_0 = 18{\cdot}178(1)$, $c_0 = 12{\cdot}879(2)$ Å; monoclinic, $a_0 = 11{\cdot}297(1)$, $b_0 = 9{\cdot}755(1)$, $c_0 = 9{\cdot}844(1)$ Å, $\beta = 107°51\,(1)'$. The systematic absence of hkl for $h+k+l \neq 2n$, $00l$ for $l \neq 4n$, and $hk0$ for $h \neq 2n$ and $k \neq 2n$ identified the space group $I4_1/a$ for the tetragonal form, while the systematic absence of $0k0$ for k odd and $h0l$ for l odd identified the space group $P2_1/c$ for the monoclinic form. The crystal densities were found to be $1{\cdot}40$ g.cm^{-3} and $1{\cdot}45$ g.cm^{-3}, respectively. The calculated densities, assuming eight molecules in the tetragonal cell and two molecules in the monoclinic cell, are $1{\cdot}429$ g.cm^{-3} and $1{\cdot}473$ g.cm^{-3}, respectively. The linear absorption coefficient for Mo $K\alpha$ radiation is $8{\cdot}76$ cm^{-1}.

All calculations were carried out on an IBM 7094 computer with the programs written or adapted by Stewart (1964). Diffractometer settings for each of the possible diffraction maxima were calculated for the ω–2θ scan method using the recommended formula of Alexander & Smith (1964): scan range $= A + B \tan \theta$, with A set equal to $1{\cdot}4$ and B equal to $1{\cdot}00$.

At the beginning and at the end of each six-hour interval of data collection, the intensities of four standard reflections were measured. The reciprocal of the mean value of these eight intensity measurements was then used as a relative scale factor for all intensities measured during that interval. These scale factors showed no systematic variation with time and the maximum variation from the scale factor for the initial interval was 0·5% for the tetragonal crystal data and 3·5% for the monoclinic crystal data.

Intensities were measured for 1847 reflections, lying in the range $0 < 2\theta < 50°$, for the tetragonal crystal and 1284 reflections, lying in the range $0 < 2\theta < 45°$, for the monoclinic crystal. 563 of the reflections from the tetragonal crystal and 495 of the reflections from the monoclinic crystal gave net intensities less than twice their standard deviations (calculated from counting statistics) and were assigned intensity values of $2\sigma_I$ and treated as 'unobserved'.

Lorentz and polarization factors were applied and relative structure factors were calculated. No corrections were made for absorption or for anomalous dispersion.

Structure determination

Tetragonal form

From a survey of the intensity distribution in the case of the tetragonal form it may be seen that, in general, only reflections with $h+k+l=4n$ and h, k and l all even or with $h+k$ and l both odd are strong. This suggests that the palladium atoms lie in positions (*c*) or (*d*) (*International Tables for X-ray Crystallography*, 1965), and (*c*) can be chosen without loss of generality. For all calculations the origin was chosen at a center of symmetry.

An initial structure factor calculation, carried out by placing the palladium atom of the asymmetric unit at the origin, gave R equal to 0·31. R is defined throughout as $\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$, with summation over the unique, observed reflections only. The atomic scattering factors used were those of Thomas & Umeda (1957) for palladium, those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen and carbon, and those from Table 2 of Stewart, Davidson & Simpson (1965) for hydrogen.

A three-dimensional Fourier synthesis, using phases determined by palladium alone, was calculated. On the Fourier map the nitrogen atom was located easily. Another structure factor calculation based on palladium and nitrogen brought R to 0·28. A Fourier synthesis phased on these atoms revealed the positions of the oxygen atom and carbon atoms C(1), C(2) and C(3). Another three-dimensional Fourier synthesis phased on these atoms enabled the positions of all the remaining non-hydrogen atoms to be located. During all stages of these structure factor and Fourier calculations an overall isotropic temperature factor, $B=2{\cdot}72$, was used.

Further refinement was carried out with the full-matrix least-squares program of Busing & Levy (1959). The function minimized was $\Sigma\,w(|F_o|-|F_c|)^2$. Four cycles of least-squares refinement with individual isotropic temperature factors and with all $w=1$ (except that, in each cycle, unobserved reflections with $F_c<F_o$ were given $w=0$) reduced R to 0·063. Three more cycles of least-squares refinement with anisotropic temperature factors reduced R to 0·049.

At this stage, a ΔF synthesis showed the positions of all hydrogen atoms. The hydrogen atom coordinates thus obtained were refined by three cycles of least-squares refinement, treating the non-hydrogen atoms as fixed.

A further series of four cycles of combined refinement of both non-hydrogen and hydrogen atoms with mixed temperature factors (individual anisotropic tem-

Table 1. *Tetragonal form*

Positional parameters ($\times 10^4$) and thermal parameters ($\times 10^2$)

The thermal parameters are of the form $\exp\left(-\frac{1}{4}\sum_{i=1}^{3}\sum_{j=1}^{3} B_{ij}h_ih_ja_i^*a_j^*\right)$.

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Pd	2500	2500	2500	279 (2)	273 (2)	303 (2)	28 (2)	12 (2)	26 (2)
O	2927 (1)	2557 (2)	1081 (2)	321 (13)	356 (14)	298 (14)	104 (12)	58 (11)	60 (11)
N	1821 (2)	3362 (2)	2197 (3)	311 (16)	274 (15)	327 (19)	104 (12)	9 (14)	26 (13)
C(1)	2096 (2)	3453 (3)	0343 (3)	346 (20)	271 (18)	338 (21)	40 (15)	1 (16)	−16 (16)
C(2)	2687 (2)	2940 (2)	0291 (3)	315 (19)	263 (18)	306 (21)	−30 (15)	−9 (14)	29 (14)
C(3)	3041 (2)	2838 (2)	−0673 (3)	392 (22)	304 (21)	398 (24)	−21 (17)	74 (19)	14 (18)
C(4)	2798 (3)	3205 (3)	−1530 (4)	617 (31)	521 (28)	268 (24)	28 (23)	65 (22)	65 (21)
C(5)	2207 (3)	3696 (3)	−1494 (4)	653 (33)	535 (30)	383 (27)	140 (25)	−8 (26)	121 (24)
C(6)	1871 (3)	3810 (3)	−0567 (4)	508 (27)	415 (25)	311 (24)	89 (20)	−26 (20)	63 (19)
C(7)	1731 (2)	3631 (2)	1279 (3)	337 (21)	274 (20)	398 (24)	107 (17)	−39 (18)	52 (18)
C(8)	1380 (3)	3700 (3)	3046 (4)	453 (26)	435 (24)	311 (23)	169 (20)	103 (20)	56 (42)
C(9)	1452 (5)	4527 (4)	3072 (6)	1009 (56)	496 (32)	467 (36)	245 (34)	131 (32)	−054 (28)
C(10)	0606 (5)	3419 (7)	2969 (9)	526 (41)	1163 (73)	772 (59)	11 (41)	252 (41)	−144 (50)
C(11)	3682 (3)	2313 (4)	−0732 (5)	505 (30)	567 (34)	470 (31)	66 (25)	207 (26)	94 (26)

Positional parameters ($\times 10^3$) and isotropic thermal parameters

				B
H(4)	296 (2)	312 (2)	−213 (3)	2·7 (1)
H(5)	203 (3)	396 (3)	−203 (4)	5·8 (1)
H(6)	147 (3)	416 (3)	−049 (4)	4·8 (1)
H(7)	138 (2)	399 (2)	122 (3)	2·5 (1)
H(8)	157 (2)	351 (2)	364 (3)	2·3 (1)
H(91)	127 (4)	470 (3)	372 (5)	9·0 (2)
H(92)	206 (4)	470 (4)	306 (6)	10·4 (2)
H(93)	125 (4)	472 (4)	253 (6)	8·6 (2)
H(101)	043 (3)	354 (3)	335 (4)	3·7 (2)
H(102)	032 (4)	351 (4)	226 (6)	9·9 (3)
H(103)	051 (4)	288 (5)	274 (8)	14·5 (3)
H(111)	391 (3)	235 (3)	−136 (4)	4·7 (1)
H(112)	415 (3)	249 (3)	−019 (5)	6·3 (1)
H(113)	355 (2)	184 (2)	−056 (4)	3·7 (1)

Table 2. *Observed and calculated structure factors for the tetragonal form*

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

H,0,0		
2	807	-858
4	2301	2271
6	319	196
8	2445	2449
10	1037	1046
12	1044	1073
14	291*	231
16	1282	1311
18	281*	-24
20	785	748
H,2,0		
2	6239	6273
4	2115	-2148
6	3846	3845
8	2047	2027
10	1311	1305
12	205*	5
14	1117	1153
16	224*	-13
18	1044	1030
20	253*	15
H,4,0		
2	174*	-135
4	2933	2922
6	673	551
8	2938	2929
10	169*	267
12	1335	1357
14	339	343
16	1070	1099
18	244*	-9
20	866	867
H,6,0		
2	1730	1725
4	2040	2025
6	2638	2580
8	591	595
10	1667	1687
12	300*	278
14	1171	1183
16	236*	180
18	1104	1071
20	258*	162
H,8,0		
2	570	550
4	2257	2260
6	1845	1775
8	1534	1552
10	209*	-25
12	1124	1145
14	233*	-34
16	1118	1124
18	253*	139
H,10,0		
2	2424	2411
4	357	341
6	1285	1291
8	209*	50
10	1255	1252
12	352	-258
14	987	981
16	242*	91
18	768	767
H,12,0		
2	519	-508
4	1836	1811
6	273*	-201
8	1036	1036
10	228*	-222
12	878	899
14	244*	31
16	792	778
H,14,0		
2	1402	1437
4	223*	77
6	1216	1252
8	231*	-156
10	937	905
12	242*	-6
14	727	731
16	259*	-3
H,16,0		
2	229*	-18
4	1058	1061
6	234*	74
8	1124	1107
10	248*	-34
12	774	749
14	258*	-7
H,18,0		
2	946	934
4	247*	18
6	1081	1049
8	251*	74
10	789	737
H,20,0		
2	259*	-81
4	958	941
6	259*	163
H,0,1		
1	2291	2208
3	1413	1376
5	1459	1433
7	1066	1070
9	1414	1422
11	1853	1875
13	714	730
15	540	524
17	632	580
19	583	561
21	389	354
H,1,1		
2	1737	1733
4	1814	-1796
6	1307	1285
8	486	-547
10	956	938
12	1098	-1076
14	767	785
16	582	-575
18	538	578
20	302	-302
H,2,1		
1	2508	2477
3	1098	-1095
5	2494	2478
7	1872	1861
9	1818	1805
11	1123	1110
13	930	920
15	784	786
17	472	494
19	576	566
21	500	428
H,3,1		
2	3195	-3179
4	3243	3230
6	1936	-1923
8	493	460
10	786	-771
12	614	581
14	561	-528
16	596	573
18	510	-472
20	443	441
H,4,1		
1	830	-869
3	4591	4620
5	180*	133
7	2151	2117
9	1239	1248
11	913	904
13	1108	1125
15	351	357
17	608	624
19	502	495
21	460	491
H,5,1		
2	2424	2397
4	1412	-1401
6	2109	2100
8	2010	-2027
10	358	372
12	525	-530
14	517	539
16	399	-380
18	489	-485
20	308	-331
H,6,1		
1	1160	1132
3	367	394
5	1864	1843
7	851	856
9	1268	1268
11	1029	1044
13	480	421
15	553	544
17	496	455
19	459	511
H,7,1		
2	179*	-5
4	2094	2053
6	1212	-1231
8	1196	1211
10	789	-802
12	833	818
14	475	-430
16	421	449
18	252*	-200
20	431	374
H,8,1		
1	1068	1065
3	1047	1023
5	1308	1341
7	755	739
9	855	808
11	818	831
13	553	519
15	469	464
17	579	594
19	448	409
H,9,1		
2	1809	1782
4	190*	-57
6	636	625
8	513	-559
10	919	945
12	417	-395
14	611	606
16	251*	-220
18	448	430
H,10,1		
1	1613	1547
3	820	830
5	674	679
7	1104	1080
9	495	455
11	680	672
13	544	571
15	630	621
17	331	370
19	367	353
H,11,1		
2	874	-882
4	1098	1127
6	514	-507
8	249	213
10	311	-366
12	512	483
14	361	-384
16	601	582
18	276	-315
H,12,1		
1	782	771
3	1020	1046
5	674	684
7	541	514
9	853	862
11	97*	377
13	608	656
15	325	400
17	427	343
H,13,1		
2	812	837
4	1041	-1032
6	779	783
8	579	-559
10	229*	129
12	455	-471
14	526	518
16	325	-350
H,14,1		
1	807	817
3	740	765
5	532	530
7	402	481
9	399	432
11	646	638
13	459	447
15	410	365
H,15,1		
2	718	-742
4	505	542
6	763	-739
8	425	417
10	610	-610
12	340	367
14	261*	-244
H,16,1		
1	817	815
3	448	433
5	358	345
7	469	434
9	554	550
11	550	536
13	342	328
H,17,1		
2	438	484
4	460	-423
6	325	333
8	603	-640
10	386	321
12	344	-295
H,18,1		
1	401	337
3	402	415
5	419	476
7	472	529
9	485	492
11	276	310
H,19,1		
2	471	-374
4	407	383
6	389	-424
8	255*	195
10	346	-321
H,20,1		
1	442	448
3	393	343
5	460	451
7	445	427
H,21,1		
2	446	445
4	381	-376
H,0,2		
2	1658	1685
4	282	-294
6	1741	1703
8	186*	-105
10	2795	2768
12	210*	2
14	1650	1669
16	253*	13
18	948	924
20	274*	-38
H,1,2		
1	158*	-25
3	176*	40
5	291	-223
7	376	-368
9	258	-206
11	272	-278
13	217*	-69
15	228*	-8
17	234*	14
19	249*	125
21	256*	-78
H,2,2		
2	1306	-1306
4	2726	2735
6	182*	-135
8	2081	2065
10	196*	73
12	2039	2045
14	229*	124
16	1370	1382
18	244*	-16
20	830	773
H,3,2		
1	321	315
3	832	-874
5	861	836
7	781	765
9	375	399
11	205*	-169
13	304	-253
15	381	387
17	290	212
19	245*	-30
21	262*	119
H,4,2		
2	3291	3282
4	180*	-8
6	2626	2608
8	185*	49
10	1455	1473
12	264	178
14	1461	1486
16	230*	11
18	1053	1075
20	258*	-107
H,5,2		
1	307	312
3	393	381
5	1348	1325
7	191*	212
9	492	461
11	210*	119
13	278	189
15	260	203
17	243*	126
19	251*	199
H,6,2		
2	180*	-79
4	2619	2624
6	713	-675
8	2366	2363
10	203*	-47
12	1367	1388
14	231*	-31
16	1010	1052
18	249*	82
20	806	745
H,7,2		
1	344	368
3	810	781
5	731	-729
7	1092	1083
9	367	-389
11	218*	129
13	363	389
15	235*	-95
17	246*	223
19	251*	81
H,8,2		
2	1904	1912
4	191*	-209
6	2535	2562
8	669	-683
10	1801	1819
12	246	-188
14	1229	1232
16	294	306
18	730	730
H,9,2		
1	372	361
3	191*	-67
5	196*	-206
7	420	-400
9	439	443
11	224*	-105
13	237*	-141
15	247*	203
17	245*	-5
19	266*	220
H,10,2		
2	195*	-7
4	2030	2050
6	204*	115
8	1647	1686
10	325	-338
12	1410	1439
14	256*	169
16	984	1009
18	258*	69
H,11,2		
1	212	134
3	249	-229
5	209*	-159
7	213*	96
9	225*	186
11	231*	-2
13	241*	-45
15	253*	-1
17	261*	233
H,12,2		
2	1988	1993
4	210*	-39
6	1626	1637
8	233	239
10	1008	1042
12	238*	170
14	1191	1229
16	256*	-45
H,13,2		
1	232	211
3	222*	-336
5	221*	29
7	229*	143
9	231*	-47
11	246*	145
13	246*	-188
15	272*	150
H,14,2		
2	222*	16
4	1520	1524
6	229*	-195
8	936	944
10	410	349
12	975	982
14	251*	160
16	607	579
H,15,2		
1	369	-348
3	241	211
5	233*	-61
7	235*	95
9	241*	-79
11	253*	-261
13	267	183
15	263*	-140
H,16,2		
2	1152	1204
4	235*	-226
6	909	893
8	241*	57
10	929	928
12	336	310
14	570	474
H,17,2		
1	241*	-108
3	236*	-35
5	240*	-0
7	287	-207
9	251*	-39
11	254*	-28
H,18,2		
2	245*	-80
4	721	705
6	246*	39
8	919	884
10	256*	175
H,19,2		
1	250*	-56
3	251*	-74
5	267*	-73
7	257*	-110
9	257*	-40
H,20,2		
2	546	566
4	255*	80
6	708	632
H,21,2		
1	260*	107
3	279	-108
H,0,3		
1	3019	3039
3	873	898
5	1000	974
7	952	957
9	1059	1067
11	461	460
13	703	635
15	951	925
17	241*	99
19	406	336
21	246*	194
H,1,3		
2	1917	-1916
4	453	414
6	673	-704
8	1060	1054
10	740	-770
12	891	901
14	773	-766
16	724	775
18	824	-821
20	381	316
H,2,3		
1	868	889
3	1045	1010
5	879	870
7	1318	1311
9	935	940
11	499	514
13	560	554
15	437	436
17	423	429
19	250*	25
21	409	384
H,3,3		
2	554	-537
4	792	-782
6	1681	1680
8	766	-784
10	1200	1167
12	910	-909
14	919	925
16	682	-673
18	441	418
20	535	-467
H,4,3		
1	750	750
3	862	842
5	850	860
7	1134	1150
9	779	795
11	1043	1035
13	859	846
15	425	382
17	349	347
19	435	386
H,5,3		
2	1151	-1185
4	1522	1511
6	702	-727
8	1244	1264
10	1120	-1118
12	1235	1237
14	359	-344
16	436	-498
18	576	-575
20	261*	320
H,6,3		
1	1414	1399
3	1038	999
5	1259	1263
7	1107	1097
9	1260	1266
11	708	741
13	947	975
15	624	589
17	317	462
19	547	543
H,7,3		
2	1455	1466
4	741	-770
6	820	787
8	1281	-1273
10	1149	1149
12	391	-377
14	781	861
16	627	-629
18	386	383
H,8,3		
1	1053	1063
3	1689	1664
5	1395	1399
7	1204	1215
9	962	960
11	909	933
13	480	443
15	818	841
17	596	574
19	313	357
H,9,3		
2	529	-533
4	838	846
6	1066	-1045
8	1045	1066
10	889	-880
12	1045	1069
14	624	-609
16	338	451
18	341	-273
H,10,3		
1	1178	1179
3	1165	1186
5	873	855
7	871	874
9	627	630
11	754	840
13	630	653
15	633	643
17	581	613
H,11,3		
2	835	844
4	794	-818
6	583	600
8	814	-835
10	1167	1226
12	694	-723
14	535	505
16	300	-287
18	335	300
H,12,3		
1	679	701
3	833	874
5	922	934
7	283	325
9	715	725
11	499	499
13	870	889
15	477	523
17	492	438
H,13,3		
2	937	-939
4	477	-447
6	505	-507
8	752	699
10	555	-567
12	551	559
14	465	-404
16	404	352
H,14,3		
1	814	770
3	976	953
5	544	527
7	553	615
9	406	459
11	759	728
13	612	597
15	509	513
H,15,3		
2	582	574
4	537	-556
6	611	631
8	536	-485
10	253	341
12	386	-358
14	312	314
H,16,3		
1	584	514
3	410	426
5	524	528
7	477	484
9	611	620
11	634	635
13	404	327
H,17,3		
2	534	-562
4	628	653
6	662	-636
8	427	458
10	429	-409
12	265*	265
H,18,3		
1	332	350
3	452	475
5	324	315
7	479	493
9	425	506
11	287*	270
H,19,3		
2	299	256
4	501	-471
6	381	405
8	552	-467
H,20,3		
1	471	417
3	263*	151
5	562	404
H,21,3		
2	266*	-237
H,0,4		
2	186*	207
4	2395	2423
6	683	-611
8	1618	1635
10	375	-321
12	1695	1651
14	301	-218
16	969	984
18	252*	-165
20	799	757
H,1,4		
1	1060	1065
3	595	577
5	863	861
7	676	-632
9	347	383
11	379	381
13	409	-334
15	234*	-175
17	238*	21
19	251*	54
H,2,4		
2	2239	2217
4	780	-233
6	2260	2262
8	460	-400
10	2007	2021
12	218*	-96
14	992	1021
16	286	-287
18	686	710
20	259*	186
H,3,4		
1	422	-382
3	184*	8
5	145*	-111
7	561	580
9	411	-376
11	213*	61
13	227*	-152
15	277	-254
17	244*	-12
19	253*	-63
H,4,4		
2	185*	-53
4	2406	2414
6	663	-630
8	2248	2258
10	207	-44
12	1596	1604
14	282	-381
16	640	614
18	250*	156
20	662	675
H,5,4		
1	253	-209
3	782	774
5	346	337
7	653	-643
9	207*	117
11	548	-540
13	231*	35
15	239*	-166
17	270	-238
19	257*	144
H,6,4		
2	2148	2138
4	343	-353
6	2249	2264
8	203*	96
10	1847	1871
12	248*	-102
14	817	830
16	245*	-72
18	803	824
H,7,4		
1	188*	-44
3	320	322
5	200	-185
7	202*	149
9	214*	87
11	237*	206
13	233*	-135
15	275	-203
17	257*	73
19	256*	-142
H,8,4		
2	568	546
4	1877	1923
6	318	259
8	1933	1965
10	221*	132
12	965	978
14	238*	-126
16	954	965
18	260*	162
H,9,4		
1	197*	-86
3	294	292
5	205*	-101
7	83*	-11
9	421	449
11	461	431
13	283	-153
15	268	-100
17	256*	28
H,10,4		
2	1563	1603
4	676	647
6	1759	1784
8	298	287
10	1307	1315
12	231*	43
14	977	992
16	281	236
18	674	666
H,11,4		
1	210*	126
3	310	-351
5	221*	-111
7	226*	207
9	232*	203
11	233*	85
13	241*	10
15	266*	-7
17	263*	13
H,12,4		
2	418	362
4	1730	1756
6	362	375
8	1327	1342
10	237*	26
12	1129	1174
14	254*	232
16	794	785
H,13,4		
1	227*	-157
3	812	-747
5	227*	38
7	235*	189
9	280	203
11	243*	-12
13	255*	-110
15	263*	152
H,14,4		
2	1621	1665
4	770	145
6	1335	1324
8	235*	-17
10	1094	1121
12	259*	182
14	896	955
H,15,4		
1	428	-424
3	248	-180
5	238*	-182
7	441	343
9	244*	-19
11	262*	-42
13	267*	230
H,16,4		
2	241*	239
4	1124	1130
6	243*	-168
8	1046	1070
10	251*	155
12	860	894
H,17,4		
1	246*	-144
3	240*	-51
5	249*	92
7	253*	-62
9	256*	102
11	265*	61
H,18,4		
2	775	779
4	248*	-27
6	877	867
8	256*	-10
10	869	858
H,19,4		
1	258*	-104
3	252*	-2
5	279*	116
7	261*	28
H,20,4		
2	264*	-44
4	628	598
H,0,5		
1	1110	1090
3	1002	1006
5	1172	1175
7	198*	47
9	981	1021
11	1183	1179
13	528	569
15	663	620
17	252*	283
19	486	483
H,1,5		
2	1237	1236
4	2653	-2583
6	1494	1480
8	1309	-1268
10	1367	1363
12	677	-720
14	342	299
16	242*	-219
18	419	403
20	261*	-123
H,2,5		
1	1433	1409
3	1551	1558
5	264	292
7	669	633
9	704	705
11	1124	1107
13	797	836
15	252	282
17	596	603
19	489	479
H,3,5		
2	2425	-2371
4	601	591
6	1161	-1145
8	1302	1302
10	937	-940
12	377	398
14	504	-531
16	608	580
18	253*	-75
20	270*	291
H,4,5		
1	2046	2011
3	837	861
5	907	964
7	919	936
9	984	986
11	510	552
13	252	244
15	563	552
17	518	509
19	503	487
H,5,5		
2	193*	302
4	945	-923
6	940	954
8	826	-822
10	295*	161
12	901	-893
14	671	707
16	352	-248
18	401	422
H,6,5		
1	1484	1449
3	880	925
5	709	752
7	1159	1237
9	939	972
11	372	346
13	236*	133
15	381	367
17	613	590
19	404	427
H,7,5		
2	635	-676
4	909	879
6	1249	-1255
8	385	349
10	1039	-1041
12	389	431
14	414	-405
16	409	394
18	423	-362
H,8,5		
1	1033	1004
3	519	544
5	1359	1352
7	1141	1152
9	662	668
11	304	168
13	390	375
15	616	566
17	483	464
H,9,5		
2	736	735
4	946	-941
6	559	564
8	1253	-1266
10	238*	244
12	588	-563
14	440	374
16	268*	-337
18	285*	179
H,10,5		
1	472	464
3	1300	1294
5	1186	1215
7	1267	1286
9	445	459
11	318	265
13	510	480
15	614	621
17	402	379
H,11,5		
2	743	-737
4	634	635
6	978	-984
8	280	288
10	538	-596
12	491	435
14	425	-418
16	309	278
H,12,5		
1	1001	1013
3	604	631
5	1513	1544
7	962	946
9	453	467
11	394	353
13	615	627
15	457	441
H,13,5		
2	879	880
4	755	-779
6	619	614
8	390	-466
10	485	445
12	401	-381
14	357	381
H,14,5		
1	634	681
3	1033	1024
5	556	613
7	[illegible]	724
9	[illegible]	387
11	578	555
13	559	500
H,15,5		
2	418	-424
4	858	845
6	570	-511
8	475	517
10	285*	-292
12	383	376
H,16,5		
1	370	377
3	669	612
5	640	662
7	414	289
9	456	511
11	399	492
H,17,5		
2	508	556
4	290	-327
6	565	476
8	302	-296
10	488	428
H,18,5		
1	297	259
3	496	474
5	348	386
7	410	411
9	434	396
H,19,5		
2	265*	-270
4	272	311
6	264*	-188
H,20,5		
1	506	445
3	268*	251
H,0,6		
2	2688	2672
4	741	723
6	2072	2115
8	495	459
10	1364	1396
12	318	263
14	1292	1295
16	253*	-219
18	813	832
H,1,6		
1	194*	153
3	848	-779
5	650	-627
7	230	238
9	213*	146
11	228*	165
13	233*	-23
15	241*	-98
17	335	242
19	265*	134
H,2,6		
2	196*	135
4	2190	2175
6	512	473
8	1799	1814
10	386	311
12	1303	1297
14	237*	15
16	991	999
18	258*	-183
H,3,6		
1	516	-493
3	1301	-1227
5	196*	-69
7	575	531
9	275*	152
11	331	-388
13	321	-353
15	290	245
17	258*	264
19	264*	93
H,4,6		
2	2359	2351
4	220	-219
6	2074	2073
8	214*	150
10	1833	1859
12	239*	255
14	996	1007
16	244*	0
18	817	800
H,5,6		
1	530	-513
3	443	-378
5	255	54
7	210*	154
9	226*	-233
11	540	-547
13	262	134
15	260*	182
17	277*	124
19	264*	55
H,6,6		
2	684	-659
4	2060	2077
6	276*	176
8	1830	1831
10	231*	130
12	1395	1411
14	244*	227
16	830	876
18	284*	41
H,7,6		
1	302	-349
3	209*	-122
5	208*	-26
7	432	-366
9	540	-617
11	390	-349
13	245*	203
15	256*	184
17	258*	14
H,8,6		
2	1920	1925
4	215*	176
6	1426	1416
8	228*	104
10	1396	1429
12	241*	161
14	1049	1071
16	272*	21
18	732	690
H,9,6		
1	450	-428
3	376	336
5	221*	-77
7	322	-374
9	686	-688
11	239*	-13
13	248*	14
15	288*	79
17	267*	-105
H,10,6		
2	214*	-47
4	1240	1260
6	227*	-180
8	1012	1033
10	236*	75
12	1053	1091
14	253*	31
16	675	682
H,11,6		
1	693	678
3	224*	-163
5	231*	84
7	417	-441
9	238*	-216
11	244*	13
13	249*	111
15	263*	-88
H,12,6		
2	1313	1335
4	236*	-173
6	651	651
8	322	-168
10	1027	1017
12	284*	63
14	682	650
H,13,6		
1	233*	155
3	430	441
5	328	-260
7	251*	-95
9	245*	-46
11	255*	170
13	256*	-34
H,14,6		
2	234*	-145
4	865	850
6	259	-156
8	749	786
10	252*	54
12	782	733
H,15,6		
1	244*	102
3	272*	287
5	247*	172
7	287	-196
9	284*	148
11	252*	9
H,16,6		
2	901	944
4	246*	-185
6	814	815
8	253*	51
10	728	700
H,17,6		
1	252*	149
3	251*	115
5	253*	-46
7	261*	106
9	263*	83
H,18,6		
2	275*	32
4	839	753
6	287*	-36
8	717	667
H,19,6		
1	265*	225
3	263*	-62
5	264*	66
H,0,7		
1	1465	1477
3	1075	1105
5	879	887
7	1476	1459
9	940	935
11	553	555
13	785	804
15	767	774
17	516	474
19	316	363
H,1,7		
2	901	-875
4	1129	1117
6	783	-773
8	627	592
10	233*	-169
12	481	-436
14	815	-807
16	527	542
18	339	-274
H,2,7		
1	1450	1465
3	1185	1177
5	1313	1309
7	842	842
9	1176	1168
11	853	832
13	673	662
15	677	671
17	547	502
19	462	373
H,3,7		
2	1061	1056
4	826	-851
6	1000	1007
8	375	-452
10	524	486
12	687	-677
14	303	269
16	515	-521
18	653	600
H,4,7		
1	1005	1003
3	682	674
5	1065	1069
7	1311	1283
9	662	659
11	937	949
13	625	624
15	752	752
17	461	519
H,5,7		
2	890	-862
4	1241	1228
6	757	-713
8	726	715
10	812	-787
12	248	270
14	482	-462
16	451	449
18	361	-297
H,6,7		
1	593	570
3	1096	1123
5	994	988
7	625	655
9	682	668
11	526	537
13	829	807
15	657	651
17	457	401
H,7,7		
2	1274	1265
4	680	-677
6	766	816
8	605	-590
10	606	625
12	585	-552
14	366	403
16	266*	-276
H,8,7		
1	739	707
3	680	686
5	829	828
7	365	476
9	418	429
11	465	524
13	724	719
15	497	474
17	464	319
H,9,7		
2	797	-773
4	964	1036
6	673	-685
8	796	798
10	388	-350
12	606	612
14	461	-435
16	287*	422
H,10,7		
1	640	660
3	748	764
5	332	312
7	271	276
9	550	524
11	625	628
13	516	474
15	290	273
H,11,7		
2	757	777
4	796	-774
6	676	683
8	420	-482
10	653	668
12	259*	-326
14	467	422
H,12,7		
1	515	488
3	667	691
5	238*	139
7	325	348
9	337	386
11	497	508
13	321	105
H,13,7		
2	931	-945
4	244*	228
6	429	-435
8	566	583
10	324	-280
12	265*	265
14	318	-224
H,14,7		
1	636	577
3	240*	141
5	475	435
7	292*	233
9	502	498
11	353	357
13	332	173
H,15,7		
2	351	329
4	699	-651
6	388	360
8	259*	-180
10	266*	251
H,16,7		
1	376	395
3	257	222
5	256*	269
7	543	545
9	276*	263
H,17,7		
2	627	-579
4	391	294
6	271	-303
8	420	326
H,18,7		
1	372	367
3	310	320
5	362	304
H,19,7		
2	268*	229
H,0,8		
2	215*	10
4	2115	2131
6	249*	-114
8	1458	1491
10	250*	178
12	1399	1404
14	254*	-40
16	693	711
18	278*	236
H,1,8		
1	211*	-137
3	217*	-82
5	220*	94
7	245	217
9	237*	124
11	242	210
13	242*	-2
15	250*	-163
17	262*	22
H,2,8		
2	2009	2026
4	219*	-134
6	1637	1651
8	230*	109
10	1412	1401
12	243*	216
14	991	994
16	258*	180
18	789	760
H,3,8		
1	213*	-47
3	216*	-139
5	226	252
7	232*	209
9	235*	163
11	260	196
13	241*	126
15	258*	53
17	266*	-42
H,4,8		
2	217*	-1
4	1713	1754
6	224*	42
8	1377	1384
10	240*	263
12	828	806
14	249*	139
16	962	963
H,5,8		
1	216*	70
3	292	268
5	363	337
7	232*	128
9	238*	162
11	239*	-30
13	279	337
15	256*	138
17	265*	-11
H,6,8		
2	1492	1489
4	222*	-68
6	1648	1660
8	237*	-9
10	906	907
12	256*	-56
14	845	838
16	261*	192
H,7,8		
1	224*	101
3	410	476
5	229*	62
7	234*	73
9	234*	-5
11	397	406
13	254*	71
15	272*	139
H,8,8		
2	228*	-162
4	1440	1483
6	235*	-131
8	1035	1011
10	300	-141
12	957	959
14	261*	158
16	556	486
H,9,8		
1	295	213
3	233*	134
5	233*	-29
7	237*	-10
9	238*	-15
11	249*	211
13	265*	274
15	267*	-23
H,10,8		
2	1377	1415
4	234*	-50
6	1152	1181
8	240*	78
10	704	711
12	253*	157
14	696	726
H,11,8		
1	241*	4
3	237*	-40
5	236*	-19
7	302	-293
9	252*	291
11	255*	44
13	261*	135
H,12,8		
2	240*	17
4	1058	1074
6	241*	55
8	859	843
10	307	254
12	587	547
H,13,8		
1	244*	-137
3	245*	-176
5	251*	-96
7	253*	194
9	255*	-62
11	262*	212
H,14,8		
2	877	898
4	250*	-142
6	829	865
8	260*	130
10	734	637
H,15,8		
1	256*	-25
3	252*	-122
5	254*	-3
7	259*	61
9	263*	52
H,16,8		
2	254*	-115
4	697	702
6	260*	110
8	645	615
H,17,8		
1	356	-304
3	262*	86
5	263*	-79
H,18,8		
2	703	626
H,0,9		
1	919	921
3	973	976
5	757	724
7	896	894
9	472	414
11	599	570
13	470	383
15	274*	79
17	432	387
H,1,9		
2	890	908
4	811	-829
6	756	764
8	487	-499
10	578	576
12	507	-486
14	398	398
16	381	-375
H,2,9		
1	923	904
3	1069	1061
5	554	552
7	685	661
9	622	625
11	392	447
13	423	374
15	259*	205
17	278	208
H,3,9		
2	838	-847
4	921	937
6	492	-470
8	967	998
10	577	-560
12	284*	375
14	308	-291
16	411	462
H,4,9		
1	845	836
3	431	468
5	818	812
7	673	693
9	797	790
11	489	465
13	270	157
15	362	392
H,5,9		
2	927	944
4	435	-420
6	890	900
8	464	-466
10	718	692
12	400	-368
14	442	443
16	267*	-173
H,6,9		
1	589	638
3	506	492
5	1146	1123
7	568	614
9	611	626
11	581	590
13	378	348
15	423	396
H,7,9		
2	328	-438
4	494	494
6	238*	-188
8	475	461
10	514	-472
12	618	607
14	781*	-214
H,8,9		
1	578	609
3	981	969
5	385	413
7	923	963
9	477	430
11	395	470
13	625	665
15	770*	130
H,9,9		
2	555	596
4	369	-391
6	367	261
8	250*	-222
10	404	365
12	297*	-282
14	389	404
H,10,9		
1	490	502
3	744	746
5	610	636
7	253*	301
9	715	703
11	408	465
13	326	269
H,11,9		
2	476	-490
4	353	347
6	475	-441
8	429	423
10	260*	-153
12	325	332
H,12,9		
1	628	573
3	425	483
5	442	407
7	432	423
9	488	482
11	437	416
H,13,9		
2	423	334
4	459	-470
6	404	413
8	316	-335
10	536	531
H,14,9		
1	601	602
3	293	252
5	454	415
7	545	498
9	374	356
H,15,9		
2	411	-352
4	319	302
6	423	-353
8	432	438
H,16,9		
1	385	281
3	494	473
5	478	421
H,17,9		
2	268*	141
H,0,10		
2	1632	1623
4	376	-435
6	1172	1172
8	251*	-6
10	757	731
12	260*	-100
14	849	791
16	270*	17
H,1,10		
1	237*	-25
3	314	-261
5	244*	-77
7	243*	199
9	248*	94
11	257*	-296
13	257*	20
15	259*	101
H,2,10		
2	240*	149
4	1140	1131
6	281	-240
8	1157	1163
10	246*	-118
12	755	710
14	265*	-113
16	634	656
H,3,10		
1	636	-654
3	623	602
5	241*	1
7	245*	-34
9	241*	-29
11	248*	-165
13	273*	-35
15	284*	-26
H,4,10		
2	898	891
4	241*	75
6	1160	1145
8	259*	-96
10	1017	1032
12	256*	-185
14	778	751
H,5,10		
1	247*	314
3	376	-285
5	545	522
7	248*	-150
9	331	-268
11	255*	42
13	262*	-112
15	275*	133
H,6,10		
2	240*	179
4	945	974
6	385	386
8	894	930
10	253*	-202
12	922	825
14	265*	35
H,7,10		
1	243*	202
3	313	351
5	244*	-111
7	248*	51
9	251*	-64
11	276*	-53
13	264*	141
H,8,10		
2	1127	1164
4	282	260
6	984	1008
8	250*	87
10	754	755
12	262*	5
14	783	753
H,9,10		
1	251*	154
3	299	298
5	247*	-8
7	267*	54
9	254*	-3
11	261*	79
13	268*	16
H,10,10		
2	245*	141
4	1160	1187
6	258*	-82
8	999	1001
10	269*	37
12	752	713
H,11,10		
1	274*	117
3	264*	22
5	253*	141
7	265*	-251
9	263*	209
11	268*	-30
H,12,10		
2	863	894
4	274*	149
6	953	981
8	259*	-98
10	886	871
H,13,10		
1	264*	15
3	259*	-107
5	256*	-131
7	262*	152
9	274*	-272
H,14,10		
2	281*	42
4	813	745
6	266*	209
8	871	833
H,15,10		
1	267*	17
3	263*	-116
5	280*	-53
H,16,10		
2	668	617
H,0,11		
1	260*	-263
3	793	836
5	391	444
7	423	502
9	447	358
11	423	368
13	535	440
H,1,11		
2	367	-375
4	324	342
6	378	-450
8	718	701
10	631	-662
12	561	567
14	427	-410
H,2,11		
1	242*	-32
3	624	678
5	711	659
7	249*	134
9	486	437
11	586	520
13	420	420
H,3,11		
2	249*	177
4	362	-316
6	545	574
8	647	-658
10	534	482
12	435	-434
14	404	452
H,4,11		
1	783	796
3	329	232
5	521	516
7	304	346
9	403	323
11	502	579
13	271*	275
H,5,11		
2	649	-649
4	811	827
6	627	-661
8	298	367
10	562	-533
12	465	481
14	409	-357
H,6,11		
1	624	669
3	431	431
5	304	285
7	450	348
9	362	430
11	266*	270
13	431	409
H,7,11		
2	770	778
4	700	-711
6	684	-672
8	661	-676
10	411	434
12	358	-420
H,8,11		
1	344	347
3	675	626
5	252*	143
7	410	367
9	344	425
11	448	443
H,9,11		
2	338	-351
4	586	620
6	731	-745
8	398	362
10	447	-402
H,10,11		
1	559	569
3	372	282
5	607	568
7	387	339
9	384	454
11	496	460
H,11,11		
2	308	331
4	327	-413
6	284	319
8	385	-447
10	275*	232
H,12,11		
1	566	604
3	490	440
5	268*	278
7	486	540
H,13,11		
2	432	-380
4	291	303
6	271*	-332
H,14,11		
1	443	441
3	297*	269
5	396	365
H,0,12		
2	262*	96
4	1189	1205
6	292*	-2
8	875	900
10	264*	-125
12	644	683
H,1,12		
1	250*	117
3	255*	-232
5	262*	306
7	260*	3
9	259*	130
11	270*	6
13	272*	7
H,2,12		
2	979	944
4	305	313
6	778	763
8	272	-243
10	824	805
12	270*	9
H,3,12		
1	302	-311
3	411	-417
5	438	-372
7	302	327
9	287*	-46
11	265*	-0
H,4,12		
2	250*	83
4	740	766
6	272*	67
8	658	673
10	260*	-15
12	692	654
H,5,12		
1	254*	143
3	274*	-281
5	255*	-113
7	261*	-101
9	266*	218
11	271*	-22
H,6,12		
2	1104	1121
4	256*	-67
6	726	710
8	263*	94
10	680	708
H,7,12		
1	258*	-133
3	259*	-191
5	257*	-25
7	254*	-20
9	267*	-25
11	274*	7
H,8,12		
2	254*	-37
4	895	882
6	263*	-75
8	629	755
10	267*	-5
H,9,12		
1	265*	5
3	287*	-314
5	285*	-120
7	267*	110
9	291*	-128
H,10,12		
2	647	709
4	264*	-71
6	775	814
8	267*	-60
H,11,12		
1	338	-216
3	260*	51
5	273*	-184
7	272*	1
H,12,12		
2	264*	-28
4	610	574
H,13,12		
1	277*	-23
H,0,13		
1	608	600
3	448	463
5	557	524
7	465	510
9	283*	334
11	275*	155
H,1,13		
2	484	473
4	527	-476
6	547	523
8	349	-302
10	327	261
H,2,13		
1	681	705
3	552	522
5	468	440
7	497	504
9	457	326
11	351	253
H,3,13		
2	272	-353
4	350	347
6	347	-396
8	365	362
10	346	-345
H,4,13		
1	492	494
3	486	486
5	558	566
7	349	256
9	414	444
H,5,13		
2	458	379
4	419	-370
6	269*	143
8	308	-349
10	389	316
H,6,13		
1	440	385
3	475	497
5	278*	304
7	571	591
9	274*	144
H,7,13		
2	564	-551
4	285	260
6	273*	-260
8	273*	146
H,8,13		
1	514	451
3	287	281
5	436	425
7	277	211
H,9,13		
2	410	348
4	278*	-341
6	349	329
H,10,13		
1	273*	148
3	347	370
5	280*	235
H,11,13		
2	280	-218
H,0,14		
2	741	745
4	274*	27
6	789	812
8	280*	-22
H,1,14		
1	265*	-53
3	273*	145
5	269*	-30
7	273*	15
H,2,14		
2	269*	57
4	900	901
6	274*	-15
8	698	664
H,3,14		
1	271*	-66
3	270*	112
5	272*	75
7	279*	-80
H,4,14		
2	874	864
4	275*	-5
6	722	685
H,5,14		
1	266*	31
3	270*	33
5	272*	2
H,6,14		
2	267*	-13
4	651	671
6	273*	-70
H,7,14		
1	270*	24
3	271*	17
H,8,14		
2	619	610
H,0,15		
1	457	342
3	316	366
H,1,15		
2	466	-438
H,2,15		
1	407	327
3	284*	353
H,3,15		
2	281*	292

perature factors for non-hydrogen and isotropic temperature factors for hydrogen atoms) brought R to 0·038.

Finally a sequence of four cycles of refinement was carried out using a set of weights based on the mean ΔF as a function of F_o. These weights were determined as follows (Wiesner & Lingafelter, 1966): from a plot of ΔF *versus* F_o, using the structure factors at $R=0{\cdot}049$, expressions were obtained giving ΔF as a linear function of F_o for several ranges of F_o. Weights were then set equal to $1/(\Delta F)^2$. Thus:

$\sqrt{w}=0{\cdot}0$
for unobserved reflections with $F_c < F_{\min}$.

$\sqrt{w}=1/(8{\cdot}65-0{\cdot}0883\ F_o)$
for unobserved reflections with $F_c \geq F_{\min}$ and for observed reflections with $F_o \leq 60$.

$\sqrt{w}=1/(4{\cdot}10-0{\cdot}0125\ F_o)$
for observed reflections with $60 < F_o \leq 120$.

$\sqrt{w}=1/2{\cdot}60$
for observed reflections with $F_o > 120$.

This sequence of cycles resulted in no change in R, and only small changes in parameters, but the weighting scheme is believed to represent a reasonable reflection of the probable accuracy of the structure factors. In the final cycle the average parameter shift was $0{\cdot}02\sigma$ and the maximum was $0{\cdot}10\sigma$ for non-hydrogen and $0{\cdot}26\sigma$ for hydrogen atoms.

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

Monoclinic form

The initial structure factor calculation was carried out with the parameters from the isomorphous nickel compound (Braun & Lingafelter, 1966), giving a value of $R=0{\cdot}119$. Refinement was carried out in exactly the same manner as for the tetragonal form after location of the hydrogen atoms. The weighting scheme used for the final sequence of cycles was

$\sqrt{w}=0{\cdot}0$
for unobserved reflections with $F_c < F_{\min}$.

$\sqrt{w}=1/(3{\cdot}33-0{\cdot}0583\ F_o)$
for unobserved reflections with $F_c \geq F_{\min}$ and for observed reflections with $F_o \leq 28$.

$\sqrt{w}=1/1{\cdot}70$
for observed reflections with $28 < F_o \leq 60$.

$\sqrt{w}=1/(-2{\cdot}59+0{\cdot}714\ F_o)$
for observed reflections with $60 < F_o \leq 95$.

$\sqrt{w}=1/4{\cdot}20$
for observed reflections with $F_o > 95$,

and gave a final R value of 0·052. In the final cycle the average parameter shift was $0{\cdot}06\sigma$ and the maximum was $0{\cdot}48\sigma$ for non-hydrogen and $0{\cdot}59\sigma$ for hydrogen atoms.

Table 3. *Monoclinic form*

Positional parameters ($\times 10^4$) and thermal parameters ($\times 10^2$)

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Pd	0000	5000	5000	338 (4)	422 (4)	292 (3)	12 (7)	111 (3)	−31 (8)
O	1827 (4)	5002 (11)	5955 (5)	263 (24)	819 (39)	391 (25)	10 (57)	83 (20)	−308 (52)
N	−0310 (7)	5797 (9)	6754 (8)	349 (39)	622 (45)	312 (33)	−2 (39)	111 (31)	−16 (33)
C(1)	1876 (9)	6327 (10)	7998 (10)	421 (55)	327 (49)	368 (42)	14 (43)	120 (39)	−23 (38)
C(2)	2446 (8)	5633 (9)	7133 (9)	278 (45)	391 (43)	306 (37)	−69 (35)	34 (35)	51 (33)
C(3)	3749 (9)	5512 (9)	7531 (11)	424 (52)	356 (52)	469 (46)	−84 (36)	126 (42)	−16 (35)
C(4)	4432 (12)	6207 (14)	8735 (13)	369 (64)	691 (75)	547 (63)	−139 (57)	136 (53)	−4 (54)
C(5)	3901 (14)	6933 (16)	9563 (15)	589 (83)	789 (84)	468 (59)	−262 (66)	103 (59)	−115 (59)
C(6)	2635 (13)	7025 (13)	9230 (13)	477 (83)	620 (73)	421 (53)	−121 (55)	106 (52)	−95 (50)
C(7)	0559 (10)	6360 (11)	7778 (11)	476 (61)	497 (57)	410 (48)	28 (46)	241 (44)	−109 (43)
C(8)	−1600 (10)	5846 (14)	6867 (10)	431 (56)	883 (76)	339 (45)	102 (55)	219 (40)	−137 (50)
C(9)	−1662 (19)	5321 (46)	8231 (21)	489 (94)	1759 (27)	602 (91)	−11 (162)	153 (81)	173 (148)
C(10)	−2128 (16)	7295 (21)	6568 (23)	351 (80)	1098 (143)	1098 (122)	106 (88)	107 (85)	−358 (96)
C(11)	4357 (12)	4685 (15)	6641 (15)	535 (61)	640 (123)	671 (65)	62 (62)	202 (53)	33 (59)

Positional parameters ($\times 10^3$) and isotropic thermal parameters

H(4)	519 (9)	614 (9)	878 (9)	3·3 (2)
H(5)	431 (12)	721 (14)	1029 (13)	7·5 (4)
H(6)	229 (9)	738 (10)	983 (9)	3·5 (2)
H(7)	034 (8)	676 (9)	860 (10)	4·6 (2)
H(8)	−196 (7)	497 (13)	604 (9)	5·6 (2)
H(91)	−251 (11)	535 (11)	832 (11)	7·9 (3)
H(92)	−132 (10)	479 (16)	860 (12)	0·2 (4)
H(93)	−110 (13)	596 (16)	907 (15)	8·9 (4)
H(101)	−284 (12)	736 (14)	670 (13)	7·8 (4)
H(102)	−159 (7)	785 (7)	713 (6)	0·2 (1)
H(103)	−172 (11)	745 (12)	565 (12)	6·7 (3)
H(111)	414 (11)	539 (13)	568 (14)	9·7 (4)
H(112)	550 (19)	466 (22)	702 (19)	17·7 (6)
H(113)	405 (8)	363 (11)	666 (9)	4·2 (2)

Table 4. *Observed and calculated structure factors for the monoclinic form*

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

H,0,0		
1	1214	1077
2	429	430
3	140	137
4	752	742
5	1297	1299
6	511	524
7	470	455
8	211	218
9	178	163
10	129	154
H,1,0		
1	291	285
2	406	-412
3	159	141
4	76*	55
5	198	187
6	84*	17
7	113	-113
8	97*	16
9	101*	13
10	102*	-63
H,2,0		
0	1398	1392
1	932	891
2	976	864
3	398	396
4	476	474
5	719	717
6	631	648
7	475	475
8	271	258
9	252	225
10	174	176
H,3,0		
1	125	134
2	77*	-83
3	126	-146
4	82*	-101
5	86*	48
6	95*	74
7	101*	-57
8	164	-152
9	107*	-34
10	107*	-2
H,4,0		
0	690	690
1	516	493
2	221	229
3	408	395
4	474	450
5	533	577
6	417	411
7	317	310
8	328	318
9	231	228
H,5,0		
1	133	-101
2	206	-203
3	116	-120
4	118	107
5	102*	-68
6	106*	-14
7	194	-185
8	105*	-54
9	116	96
H,6,0		
0	386	378
1	384	371
2	404	398
3	326	313
4	293	274
5	333	320
6	289	275
7	263	261
8	143	132
H,7,0		
1	111	61
2	97*	-21
3	100*	-12
4	104*	57
5	106*	-33
6	107*	51
7	111*	-63
H,8,0		
0	272	265
1	318	310
2	265	277
3	118	128
4	249	217
5	239	215
H,9,0		
1	102*	16
2	104*	73
3	107*	40
H,1,1		
0	1068	1020
1	661	633
-1	1401	1399
2	1180	1135
-2	240	243
3	541	531
-3	760	734
4	789	839
-4	535	516
5	507	511
-5	978	972
6	451	471
-6	724	705
7	455	448
-7	331	330
8	336	351
-8	272	208
9	301	297
-9	210	232
10	256	274
-10	221	220
H,2,1		
0	1066	1159
1	76*	59
-1	647	664
2	78*	68
-2	156	-158
3	217	-216
-3	77*	118
4	178	194
-4	77*	17
5	84*	65
-5	336	331
6	89*	-2
-6	84*	54
7	97*	-67
-7	91*	-12
8	102*	-61
-8	97*	25
9	103*	-56
-9	101*	-71
-10	102*	-74
H,3,1		
0	1190	1261
1	633	614
-1	472	479
2	803	781
-2	398	407
3	568	558
-3	441	447
4	627	609
-4	507	510
5	643	634
-5	635	637
6	374	369
-6	409	404
7	425	427
-7	354	342
8	746	273
-8	198	195
9	201	187
-9	229	239
-10	218	248
H,4,1		
0	225	237
1	293	288
-1	77*	-89
2	138	-124
-2	79*	46
3	83*	-39
-3	102	122
4	87*	-32
-4	85*	-4
5	99	88
-5	88*	37
6	99*	88
-6	95*	40
7	127	-94
-7	122	66
8	104*	88
-8	104*	72
9	106*	-34
-9	103*	-47
H,5,1		
0	469	483
1	484	467
-1	481	509
2	464	454
-2	518	521
3	240	231
-3	337	352
4	414	387
-4	443	437
5	443	413
-5	377	376
6	349	365
-6	394	392
7	219	209
-7	303	305
8	105*	56
-8	240	241
-9	270	271
H,6,1		
0	89*	-27
1	125	-91
-1	87*	-20
2	93*	-71
-2	204	-197
3	103	120
-3	94*	93
4	100*	-14
-4	99*	30
5	103*	-0
-5	102*	14
6	105*	-40
-6	104*	-37
7	105*	19
-7	106*	-99
-8	146	138
H,7,1		
0	356	348
1	276	270
-1	302	331
2	174	146
-2	391	391
3	231	219
-3	366	370
4	313	289
-4	243	234
5	210	226
-5	296	289
6	124	176
-6	290	288
-7	340	333
H,8,1		
0	101*	-31
1	101*	27
-1	103*	-28
2	102*	-8
-2	103*	-24
3	103*	-8
-3	105*	36
4	105*	-20
-4	121	-55
5	108*	9
-5	106*	-14
-6	108*	-21
H,9,1		
0	218	186
1	266	238
-1	159	190
2	183	143
-2	220	236
3	159	169
-3	216	188
H,10,1		
0	105*	-7
H,11,1		
0	171	126
H,10,1		
1	107*	-12
H,11,1		
1	223	171
H,0,2		
0	68*	-57
1	638	604
-1	212	190
2	686	671
-2	837	793
3	775	749
-3	914	816
4	690	681
-4	454	431
5	553	559
-5	166	160
6	455	462
-6	303	319
7	393	408
-7	468	479
8	314	337
-8	388	394
9	296	317
-9	284	297
-10	310	314
-11	164	200
H,1,2		
0	225	-226
1	434	419
-1	166	138
2	223	-235
-2	75	29
3	76*	-33
-3	75*	-23
4	81*	-42
-4	157	153
5	84*	21
-5	78*	52
6	91*	54
-6	162	160
7	98*	-88
-7	87*	-20
8	102*	64
-8	94*	-42
9	103*	43
-9	102	10
-10	101*	4
H,2,2		
0	210	208
1	282	259
-1	1467	1612
2	598	567
-2	812	862
3	643	631
-3	395	382
4	849	849
-4	284	282
5	387	386
-5	481	474
6	424	430
-6	645	635
7	294	293
-7	396	394
8	163	181
-8	254	245
9	296	309
-9	271	282
-10	269	272
H,3,2		
0	76*	-85
1	78*	-51
-1	491	484
2	111	120
-2	166	-187
3	293	298
-3	119	135
4	87*	41
-4	81*	59
5	92*	-83
-5	84*	27
6	128	-77
-6	127	124
7	136	136
-7	155	-126
8	140	159
-8	99*	45
-10	103*	-78
9	107*	-71
-9	102*	-50
H,4,2		
0	748	792
1	552	565
-1	877	949
2	401	380
-2	676	657
3	278	294
-3	650	673
4	654	623
-4	309	337
5	416	425
-5	511	525
6	360	370
-6	431	429
7	105*	101
-7	484	470
8	106*	136
-8	314	309
-9	171	169
-10	164	172
H,5,2		
0	163	183
1	87*	110
-1	84*	41
2	160	168
-2	223	-236
3	93*	14
-3	89*	-68
4	98*	22
-4	93*	80
5	102*	70
-5	96*	19
6	104*	14
-6	101*	-39
7	116	113
-7	213	-194
8	107*	4
-8	106*	-64
-9	107*	45
H,6,2		
0	522	517
1	330	309
-1	490	455
2	241	235
-2	437	434
3	336	379
-3	410	412
4	404	378
-4	290	288
5	360	338
-5	348	353
6	200	213
-6	351	335
7	115	135
-7	389	367
-8	274	284
H,7,2		
0	99*	-47
1	99*	-3
-1	99*	-25
2	100*	26
-2	99*	-22
3	104*	-15
-3	102*	-29
4	106*	-25
-4	104*	30
5	105*	-10
-5	106*	-60
6	107*	-10
-6	105*	2
-7	108*	-62
H,8,2		
0	266	268
1	219	232
-1	234	227
2	223	205
-2	219	224
3	248	231
-3	249	262
4	235	187
-4	261	258
-5	199	194
-6	205	193
H,9,2		
0	105*	-60
1	106*	-60
-1	104*	19
2	107*	-51
-2	105*	-9
-3	106*	40
H,10,2		
0	239	220
H,11,2		
0	113*	-41
H,10,2		
1	141	140
H,11,2		
1	112*	29
H,10,2		
2	117*	101
H,11,2		
2	114*	-13
H,1,3		
0	497	470
1	662	627
-1	791	769
2	452	442
-2	731	737
3	496	466
-3	529	525
4	590	583
-4	244	232
5	415	402
-5	400	375
6	449	463
-6	485	480
7	158	163
-7	567	561
8	160	180
-8	420	410
-9	232	218
-10	263	275
-11	248	248
H,2,3		
0	163	-150
1	77*	-64
-1	437	-463
2	227	214
-2	135	-134
3	125	143
-3	274	-267
4	86*	-55
-4	110	57
5	92*	2
-5	198	-188
6	99*	16
-6	147	-147
7	227	221
-7	125	-107
8	103*	35
-8	96*	-70
-9	100*	58
-10	101*	-4
H,3,3		
0	452	477
1	558	545
-1	585	626
2	149	152
-2	801	853
3	563	565
-3	632	640
4	436	434
-4	335	345
5	423	427
-5	375	373
6	239	272
-6	557	546
7	105*	121
-7	593	605
8	283	299
-8	449	454
-9	166	167
-10	222	225
H,4,3		
0	280	-290
1	86*	88
-1	84*	38
2	88*	82
-2	158	169
3	106	91
-3	311	-314
4	96*	-13
-4	87*	25
5	107*	-66
-5	102	-75
6	103*	64
-6	96*	41
7	104*	46
-7	127	-109
-8	222	-228
-9	101*	26
-10	105*	25
H,5,3		
0	362	390
1	410	393
-1	747	806
2	400	376
-2	471	501
3	490	485
-3	377	393
4	387	366
-4	359	372
5	270	260
-5	359	353
6	236	234
-6	510	498
7	245	251
-7	356	335
-8	337	339
-9	193	176
H,6,3		
0	106*	14
1	98*	6
-1	135	160
2	126	-61
-2	95*	-18
3	105*	-6
-3	105*	-24
4	105*	32
-4	100*	9
5	105*	-15
-5	102*	-20
6	111*	-9
-6	106*	15
-7	105*	-21
-8	105*	-33
H,7,3		
0	444	452
1	336	336
-1	382	383
2	295	285
-2	253	249
3	263	262
-3	242	267
4	327	311
-4	276	267
5	295	273
-5	259	281
-6	235	229
-7	178	176
H,8,3		
0	105*	-74
1	103*	10
-1	105*	-16
2	101*	-15
-2	105*	14
3	107*	-1
-3	105*	-40
-4	105*	29
-5	105*	-14
H,9,3		
0	225	223
1	116	124
-1	279	237
2	197	197
-2	173	157
3	161	159
H,10,3		
0	109*	-1
H,11,3		
0	117*	76
H,10,3		
1	109*	17
H,11,3		
1	115*	48
H,10,3		
2	111*	-17
H,11,3		
2	126	93
H,10,3		
3	112*	-4
H,0,4		
0	440	455
1	554	545
-1	685	687
2	167	173
-2	1157	1119
3	642	639
-3	1113	1072
4	375	373
-4	464	453
5	418	421
-5	411	380
6	144	131
-6	645	631
7	100*	-9
-7	645	636
8	280	306
-8	598	585
-9	200	176
-10	256	240
-11	245	237
H,1,4		
0	238	-223
1	177	160
-1	174	-164
2	125	140
-2	78	81
3	85*	-59
-3	124	-125
4	91*	-62
-4	101	-96
5	96*	-23
-5	87*	-36
6	132	154
-6	118	-129
-10	101*	31
7	102*	-26
-7	91*	-46
8	109	-79
-8	145	-127
-9	100*	14
H,2,4		
0	491	461
1	449	435
-1	573	570
2	465	449
-2	517	533
3	563	558
-3	775	805
4	408	410
-4	656	664
5	352	359
-5	367	381
6	181	184
-6	447	446
7	152	197
-7	383	382
-8	557	569
-9	349	337
-10	210	187
H,3,4		
0	83*	41
1	85*	-29
-1	272	-292
2	99	-73
-2	123	-139
3	92*	-54
-3	85*	-104
4	96*	-12
-4	144	-142
5	101*	22
-5	88*	24
6	104*	34
-6	166	-164
7	104*	-48
-7	98*	-52
-8	127	-140
-9	107	-69
-10	104*	82
H,4,4		
0	340	360
1	401	401
-1	192	221
2	579	575
-2	461	529
3	490	494
-3	432	470
4	340	331
-4	381	402
5	288	284
-5	312	302
6	249	239
-6	307	320
7	284	274
-7	351	350
-8	296	307
-9	248	252
H,5,4		
0	155	-142
1	97*	-38
-1	213	-242
2	98*	-43
-2	120	142
3	101*	-16
-3	94*	-22
4	130	-127
-4	97*	-15
5	104*	-19
-5	101*	-67
6	106*	17
-6	103*	-47
-7	105*	79
-8	103*	-1
H,5,4		
-9	107*	-14
H,6,4		
0	285	304
1	308	325
-1	345	359
2	350	345
-2	395	428
3	381	367
-3	278	287
4	288	282
-4	247	309
5	208	211
-5	260	267
-6	267	276
-7	223	216
-8	143	137
H,7,4		
0	106*	-14
1	106*	20
-1	105*	-39
2	105*	-44
-2	130	93
3	106*	16
-3	122	-90
4	110*	-51
-4	108*	7
5	109*	6
-5	106*	-6
-6	106*	19
-7	108*	19
H,8,4		
0	123	150
1	160	177
-1	339	363
2	195	207
-2	279	271
3	257	232
-3	195	199
4	217	209
-4	190	200
5	144	99
-5	190	182
H,9,4		
0	131	104
1	110*	31
-1	110*	-2
2	110*	-8
3	112*	-15
4	113*	2
5	115*	32
H,10,4		
0	139	79
H,11,4		
0	118*	13
H,10,4		
1	120	125
-1	145	130
H,11,4		
-1	117*	19
H,10,4		
2	155	118
3	121*	109
H,1,5		
0	545	539
1	209	230
-1	686	679
2	501	478
-2	826	819
3	522	532
-3	690	696
4	406	419
-4	727	734
5	295	293
-5	294	298
6	102*	81
-6	500	480
7	104*	282
-7	520	513
-8	595	584
-9	468	480
-10	103*	108
H,2,5		
0	88	50
1	150	-141
-1	83*	-16
2	204	-197
-2	105	86
3	96*	-39
-3	210	210
4	100*	-48
-4	105	-75
5	104*	50
-5	89*	-56
6	122	-138
-6	93*	26
7	106*	-111
-7	97*	61
-8	136	108
-9	143	-87
-10	101*	-11
H,3,5		
0	394	407
1	467	465
-1	454	469
2	455	451
-2	587	602
3	460	436
-3	546	555
4	343	348
-4	396	418
5	308	307
-5	271	295
6	188	207
-6	304	318
-7	320	329
-8	315	312
-9	286	288
-10	196	204
H,4,5		
0	96*	-28
1	97*	-61
-1	100	-104
2	99*	-62
-2	93*	-41
3	102*	-96
-3	94*	11
4	103*	-62
-4	94*	-27
5	104*	25
-5	96*	-14
6	107*	3
-6	101*	-53
-7	102*	10
-8	104*	74
-9	105*	54
H,5,5		
0	236	253
1	231	256
-1	332	377
2	345	349
-2	286	314
3	346	353
-3	395	425
4	307	306
-4	295	335
5	187	185
-5	246	239
6	177	155
-6	190	198
-7	180	186
-8	266	250
H,6,5		
0	105*	105
1	104*	-18
-1	176	-212
2	105*	40
-2	108	-119
3	104*	-66
-3	104*	36
4	108	-33
-4	105*	-80
5	109*	42
-5	106*	16
6	110*	-6
-6	108*	-80
-7	107*	8
H,7,5		
0	106*	83
1	169	209
-1	107*	100
2	242	245
-2	350	379
3	236	232
-3	287	305
4	120	121
-4	199	205
5	114*	121
-5	145	151
6	166	149
-6	133	164
H,8,5		
0	107*	56
1	108*	-16
-1	108*	9
2	110*	25
-2	108*	0
3	112*	-15
-3	108*	-33
4	113*	-5
5	115*	5
6	117*	3
H,9,5		
0	142	116
1	116*	132
-1	114*	113
2	178	139
3	182	156
4	118*	102
H,10,5		
0	116*	-2
1	117*	-12
-1	116*	78
2	117*	-4
H,0,6		
0	495	493
1	624	608
-1	418	419
2	599	599
-2	495	477
3	396	393
-3	324	303
4	313	314
-4	517	506
5	187	206
-5	634	629
6	358	335
-6	342	334
7	261	290
-7	390	371
8	110*	184
-8	166	160
-9	402	389
-10	302	303
H,1,6		
0	92*	-38
1	154	-146
-1	105	122
2	95*	6
-2	87*	39
3	99*	-28
-3	87*	-68
4	102*	13
-4	90*	60
5	138	-72
-5	91*	29
6	105*	-22
-6	95*	72
7	108*	36
-7	99*	92
8	108*	-18
-8	115	68
-9	104*	96
-10	103*	-60
H,2,6		
0	452	451
1	390	388
-1	492	509
2	501	488
-2	461	475
3	397	385
-3	457	457
4	305	318
-4	521	515
5	175	175
-5	379	381
6	210	191
-6	336	340
7	271	288
-7	378	382
8	160	164
-8	287	305
-9	315	299
-10	185	152
H,3,6		
0	130	117
1	126	-105
-1	96*	13
2	101*	-60
-2	95*	-28
3	108*	-113
-3	127	77
4	100*	35
-4	95*	18
5	104*	32
-5	97*	10
6	119	-69
-6	106*	35
7	109*	-28
-7	115	105
-8	170	117
-9	106*	69
H,4,6		
0	264	273
1	269	271
-1	343	357
2	286	265
-2	489	510
3	315	314
-3	377	401
4	204	231
-4	399	425
5	145	165
-5	272	277
6	111*	114
-6	297	306
7	127	139
-7	294	312
-8	271	283
-9	278	269
H,5,6		
0	133	112
1	104*	-28
-1	136	149
2	103*	-42
-2	105*	-109
3	105*	-56
-3	113	-40
4	108*	63
-4	104*	-54
5	106*	31
-5	105*	15
6	110*	-41
-6	105*	39
7	112*	-19
-7	105*	-25
-8	115	-30
H,6,6		
0	166	187
1	195	221
-1	107	130
2	236	221
-2	249	277
3	178	194
-3	318	324
4	113	139
-4	249	263
5	186	163
-5	143	170
6	149	123
-6	146	162
-7	223	232
H,7,6		
0	108*	-5
1	109*	-4
-1	106*	61
2	109*	-16
-2	107*	-52
3	110*	-32
-3	107*	-22
4	111*	26
-4	108*	-20
5	114*	3
-5	106*	4
6	116*	2
H,8,6		
0	180	180
1	163	159
-1	112*	104
2	190	173
3	147	115
4	157	115
H,9,6		
0	127	-44
1	113*	-31
-1	115*	-65
2	116*	10
3	119*	-15
H,10,6		
0	123*	109
-1	125	95
H,1,7		
0	325	340
1	479	477
-1	272	263
2	454	463
-2	359	362
3	376	361
-3	465	448
4	234	207
-4	428	417
H,2,7		
-4	98*	-62
H,1,7		
5	161	179
-5	444	434
6	231	236
-6	247	237
7	171	203
-7	172	167
-8	145	154
-9	208	182
H,2,7		
0	115	75
1	118	111
-1	118	64
2	107*	8
-2	117	-119
3	102*	-26
-3	99*	-71
4	104*	25
5	105*	13
-5	107*	104
6	109*	57
-6	102*	40
7	111*	23
-7	102*	-20
-8	103*	-56
-9	103*	-38
H,3,7		
0	205	201
1	299	314
-1	178	176
2	379	380
-2	426	420
3	271	278
-3	388	403
4	181	154
-4	380	400
5	127	115
-5	432	419
6	210	209
-6	223	216
7	131	164
-7	282	297
-8	238	243
H,4,7		
0	105*	7
1	108	19
-1	184	206
2	104*	-0
-2	104*	-18
3	106*	15
-3	147	-100
4	106*	43
-4	104*	37
5	110*	-72
-5	106*	61
6	112*	18
-6	104*	62
7	114*	40
-7	105*	-7
-8	108*	-59
H,5,7		
0	292	287
1	187	203
-1	154	198
2	263	240
-2	211	207
3	142	152
-3	336	363
4	191	175
-4	314	331
5	127	124
-5	289	268
6	116*	101
-6	189	181
-7	254	260
H,6,7		
0	106*	-14
1	109*	-38
-1	105*	41
2	109*	-56
-2	107*	60
3	111*	-5
-3	106*	-30
4	119	19
-4	107*	-14
5	115*	-10
-5	109*	-9
H,7,7		
0	188	205
1	158	181
-1	232	239
2	144	149
3	128	135
4	119*	136
H,8,7		
0	114*	-41
1	115*	-76
-1	112*	-13
2	115*	-28
3	117*	-22
H,9,7		
0	121*	138
1	121*	124
-1	120*	130
H,0,8		
0	177	185
1	235	235
-1	196	184
2	300	320
-2	409	411
3	237	229
-3	483	464
4	144	154
-4	362	348
5	155	156
-5	300	278
6	167	168
-6	212	175
-7	253	238
-8	290	285
H,1,8		
0	103*	31
1	101*	32
-1	102*	80
2	101*	6
-2	100*	-15
3	104*	48
-3	138	-67
4	104*	37
-4	102*	39
5	107*	-6
-5	102*	-23
6	110*	0
-6	102*	8
-7	102*	-79
-8	104*	-70
H,2,8		
0	262	286
1	275	270
-1	184	186
2	268	283
-2	238	224
3	191	184
-3	444	436
4	202	170
-4	291	285
5	193	187
-5	277	273
6	157	140
-6	242	223
-7	215	207
-8	247	274
H,3,8		
0	103*	-16
1	105*	66
-1	107*	-3
2	106*	32
-2	103*	95
3	119	73
-3	103*	-53
4	109*	-29
-4	104*	-67
5	110*	6
-5	102*	-5
6	113*	35
-6	103*	35
-7	106*	-52
H,4,8		
0	217	260
1	318	309
-1	236	266
2	244	231
-2	169	176
3	166	167
-3	257	263
4	114*	140
-4	279	264
5	116*	145
-5	289	300
-6	217	218
H,5,8		
0	107*	-69
1	109*	-13
-1	109*	-60
2	111*	10
-2	106*	10
3	112*	28
-3	106*	-5
4	112*	-21
-4	108*	-31
5	115*	-28
H,6,8		
0	195	204
1	207	207
-1	244	248
2	163	170
-2	207	222
3	122	152
4	126	107
H,7,8		
0	113*	-54
1	115*	-29
-1	112*	-6
2	116*	-23
-2	112*	-7
H,8,8		
0	124*	136
1	120*	107
-1	151	146
-2	144	139
H,1,9		
0	236	229
1	239	232
-1	247	234
2	145	157
-2	134	117
3	108*	93
-3	325	312
4	200	153
-4	313	291
5	115*	151
-5	269	249
-6	248	259
H,2,9		
0	106*	-64
1	105*	-49
-1	120	-102
2	107*	30
-2	105*	71
3	109*	11
-3	105*	75
4	110*	-22
-4	103*	-37
5	114*	2
-5	103*	-40
-6	104*	-53
H,3,9		
0	290	272
1	252	246
-1	291	296
2	175	168
-2	192	219
3	156	145
-3	176	177
4	131	141
-4	261	266
-5	240	251
H,4,9		
0	110*	-47
1	111*	-44
-1	108*	-55
2	110*	-2
-2	107*	-37
3	112*	6
4	114*	10
H,5,9		
0	220	234
1	216	212
-1	174	182
2	184	178
-2	177	169
3	140	125
H,6,9		
0	113*	-21
1	114*	5
-1	111*	-39
2	116*	-11
-2	111*	8
H,7,9		
0	122*	111
-1	158	120
-2	117*	119
H,0,10		
0	266	251
1	229	223
-1	315	305
2	111*	93
-2	144	172
3	171	135
H,1,10		
0	109*	-33
1	108*	-15
-1	106*	-35
2	110*	-23
-2	106*	-86
3	110*	-30
H,2,10		
0	246	230
1	220	201
-1	212	219
2	164	122
-2	181	189
3	114*	109
H,3,10		
0	111*	-63
1	111*	-32
-1	109*	-52
2	112*	-32
-2	109*	-47
H,4,10		
0	166	154
1	205	159
-1	127	164
-2	157	150
H,5,10		
0	115*	3
-1	113*	-7
-2	111*	5
H,6,10		
-2	117*	104
H,1,11		
0	196	211
-1	249	217
-2	257	241
H,2,11		
0	113*	10
-1	110*	10
-2	111*	12
H,3,11		
-1	153	149
-2	116*	130
H,4,11		
-2	115*	-23

The final values of the parameters and their estimated standard deviations are given in Table 3 and observed and calculated structure factors in Table 4.

Discussion

The centrosymmetric molecules of bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium are similar in configuration to those of the nickel compound and the 3-ethyl-palladium compound (Braun & Lingafelter, 1966, 1967*b*), but there are a number of significant differences in details, particularly in the case of the tetragonal form. The packing of the molecules of the two forms of bis-(*N*-isopropyl-3-methylsalicylaldiminato)-palladium are shown in Figs. 1 and 2.

Table 5. *Bond distances between carbon and hydrogen atoms*

	Tetragonal form	Monoclinic form
C(4)—H(4)	0·84 (4) Å	0·85 (10) Å
C(5)—H(5)	0·89 (5)	0·77 (12)
C(6)—H(6)	0·97 (5)	0·87 (11)
C(7)—H(7)	0·92 (4)	1·00 (10)
C(8)—H(8)	0·91 (4)	1·16 (11)
C(9)—H(91)	0·95 (7)	0·99 (13)
C(9)—H(92)	1·14 (7)	0·68 (14)
C(9)—H(93)	0·86 (7)	1·08 (14)
C(10)-H(101)	0·63 (6)	0·85 (15)
C(10)-H(102)	1·06 (8)	0·88 (7)
C(10)-H(103)	1·03 (8)	1·15 (13)
C(11)-H(111)	0·91 (5)	1·14 (13)
C(11)-H(112)	1·15 (5)	1·23 (20)
C(11)-H(113)	0·92 (4)	1·09 (10)

Fig. 3 shows the bond lengths and angles between the non-hydrogen atoms for both forms. The two sets of values agree well within twice the standard deviation. Also they agree well with those reported for similar compounds (Lingafelter & Braun, 1966; Braun & Lingafelter, 1966, 1967*a*,*b*). The bond distances involving the hydrogen atoms are given in Table 5.

Although the bond distances and angles show very little variation, considerable differences in the deviations from planarity are observed. Table 6 lists the deviations of selected atoms from the least-squares plane defined by the benzene ring in six recently studied *N*-isopropylsalicylaldimine chelates. The significant deviations from planarity are seen to be in the same direction for all compounds but to vary markedly in magnitude between compounds. Thus the oxygen atom is in all cases displaced from the plane in the same direction as the metal atom, while C(7) and N are displaced in the opposite direction. The considerable differences between the two forms of the bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium can only be due to intermolecular forces and it therefore seems likely

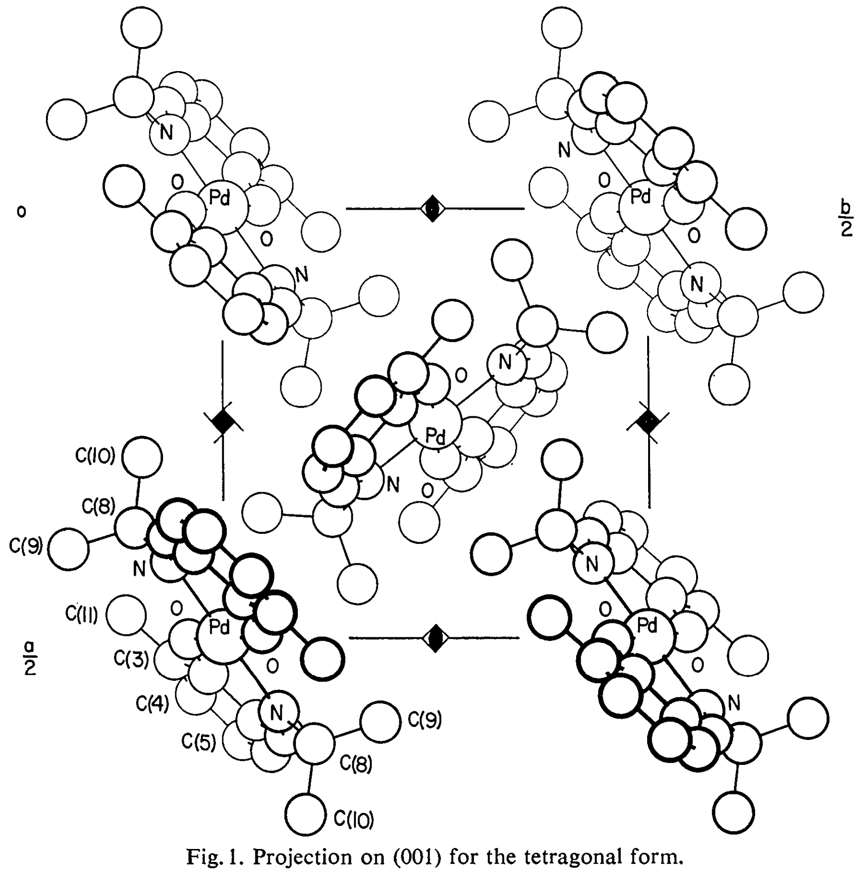

Fig. 1. Projection on (001) for the tetragonal form.

that all of the differences in deviations from planarity between different molecules may be due to inter- and intra-molecular steric effects, rather than special metal ion differences. This flexibility of these molecules has been pointed out previously (Cheeseman, Hall & Waters, 1965; Lingafelter, 1966; Boyko, Hall, Kinloch & Waters, 1966). Examination of intermolecular contact distances shows none which require comment. Comparison of intra-molecular contact distances with those in the nickel compound show that the steric interactions which inhibit the formation of the planar form of bis-(*N*-isopropylsalicylaldiminato)nickel compounds are somewhat less unfavorable in the palladium chelate than in the nickel chelate.

Table 6. *Distances of selected atoms from least-squares benzene plane.* (Å)

Atom	(*a*)	(*b*)	(*c*)	(*d*)	(*e*)	(*f*)	(*g*)	(*h*)
Metal	−0·256	−0·212	−0·294	−0·312	−0·220	−0·210	−0·139	−0·159
O	−0·052	−0·057	−0·039	−0·066	−0·062	0·021	0·001	0·010
N	0·049	0·201	0·187	0·021	0·055	0·024	−0·021	0·047
C(7)	0·073	0·145	0·170	0·061	0·083	0·007	0·040	0·068
C(8)	0·217	0·495	0·564	0·269	0·256	−0·020	0·074	0·251
C(9)	1·427	1·695	1·930	1·693	1·633	1·036	1·351	1·730
C(10)	−1·078	−0·723	−0·536	−0·669	−0·809	−1·402	−1·112	−0·690
C(11)	0·021	0·054	0·051	0·087	−0·013	−0·013		
*	0·008	0·017	0·008	0·012	0·013	0·003	0·008	0·007

(*a*) Bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium, tetragonal form.
(*b*) Bis-(*N*-isopropyl-3-methylsalicylaldiminato)palladium, monoclinic form.
(*c*) Bis-(*N*-isopropyl-3-methylsalicylaldiminato)nickel (Braun & Lingafelter, 1966).
(*d*) Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)nickel, ring I (Braun & Lingafelter, 1967*a*).
(*e*) Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)nickel, ring II (Braun & Lingafelter, 1967*a*).
(*f*) Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)palladium, (Braun & Lingafelter, 1967*b*).
(*g*) Bis-(*N*-isopropylsalicylaldiminato)nickel, ring I (Fox, Orioli, Lingafelter & Sacconi, 1964).
(*h*) Bis-(*N*-isopropylsalicylaldiminato)nickel, ring II (Fox, Orioli, Lingafelter & Sacconi, 1964).

* Root-mean-square deviations for the carbon atoms of the benzene ring.

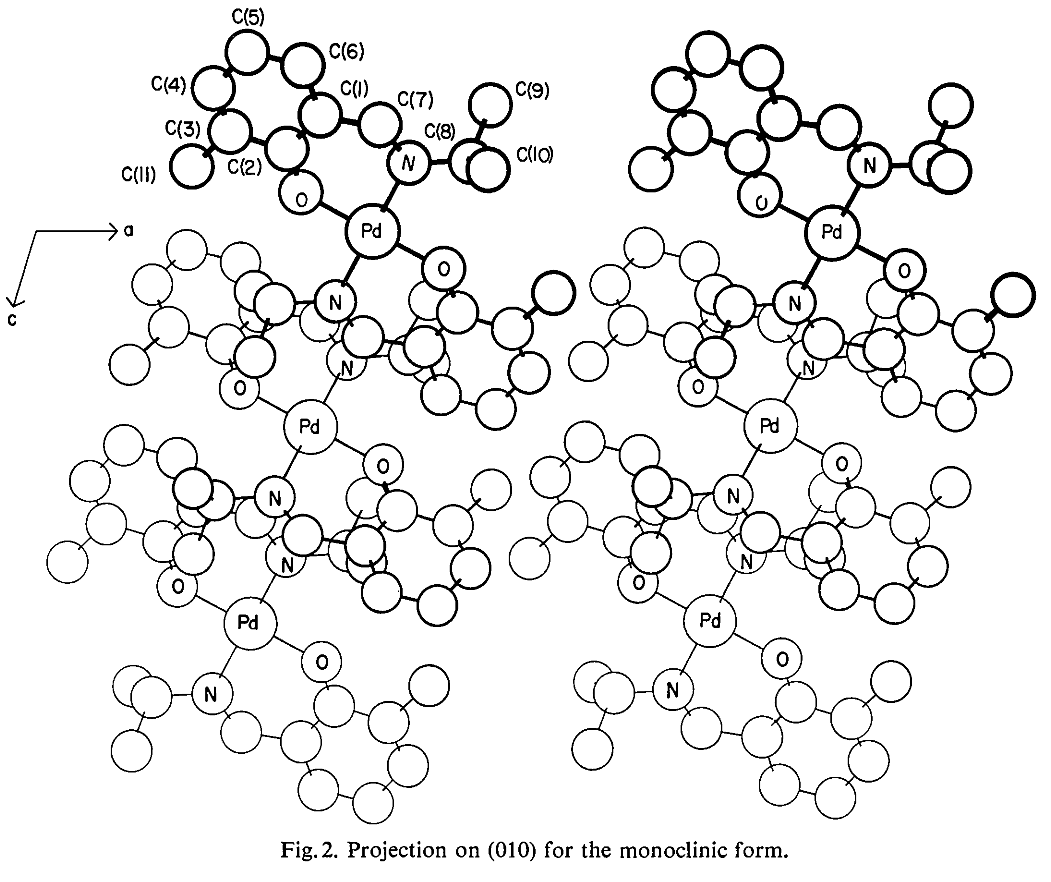

Fig. 2. Projection on (010) for the monoclinic form.

This study has been supported in part by the U.S. Public Health Service under Research Grant GM-10842 and in part by the U.S. National Science Foundation. The authors express their gratitude to Dr R. L. Braun for the crystal samples and for helpful discussions. One of us (P.C.J.) is thankful to the Kurukshetra University, Kurukshetra, India for the grant of leave of absence.

References

ALEXANDER, L. E. & SMITH, G. S. (1964). *Acta Cryst.* **17**, 1195.

BERGHUIS, J., HAANAPPEL, IJ. M., POTTERS, M., LOOPSTRA, B. O., MACGILLAVRY, C. H. & VEENENDAAL, A. L. (1955). *Acta Cryst.* **8**, 478.

BOYKO, E. R., HALL, D., KINLOCH, M. E. & WATERS, T. N. (1966). *Acta Cryst.* **21**, 614.

BRAUN, R. L. & LINGAFELTER, E. C. (1966). *Acta Cryst.* **21**, 546.

BRAUN, R. L. & LINGAFELTER, E. C. (1967*a*). *Acta Cryst.* **22**, 780.

BRAUN, R. L. & LINGAFELTER, E. C. (1967*b*). *Acta Cryst.* **22**, 787.

BUSING, W. R. & LEVY, H. A. (1959). Oak Ridge National Laboratories 59-4-37, Oak Ridge, Tennessee.

CHEESEMAN, T. P., HALL, D. & WATERS, T. N. (1965). *Nature, Lond.* **205**, 494.

DUFF, J. C. (1941). *J. Chem. Soc.* 547.

FOX, M. R., ORIOLI, P. L., LINGAFELTER, E. C. & SACCONI, L. (1964). *Acta Cryst.* **17**, 1159.

FURNAS, T. (1957). Single Crystal Orienter Manual: Milwaukee, General Electric Company, X-ray Department.

International Tables for X-ray Crystallography (1965). Vol. I, 2nd Ed., p. 178. Birmingham: Kynoch Press.

LINGAFELTER, E. C., (1966). *Coordin. Chem. Rev.* **1**, 151.

LINGAFELTER, E. C. & BRAUN, R. L. (1966). *J. Amer. Chem. Soc.* **88**, 2951.

SACCONI, L., PAOLETTI, P. & DEL RE, G. (1957). *J. Amer. Chem. Soc.* **79**, 4062.

SACCONI, L., CIAMPOLINI, M., MAGGIO, F. & DEL RE, G. (1960). *J. Amer. Chem. Soc.* **82**, 815.

STEWART, J. M. (1964). *Crystal Structure Calculation System for the IBM* 709, 7090, 7094. Technical Report TR-64-6, Computer Science Center, Univ. of Maryland and Research Computer Laboratory, Univ. of Washington.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

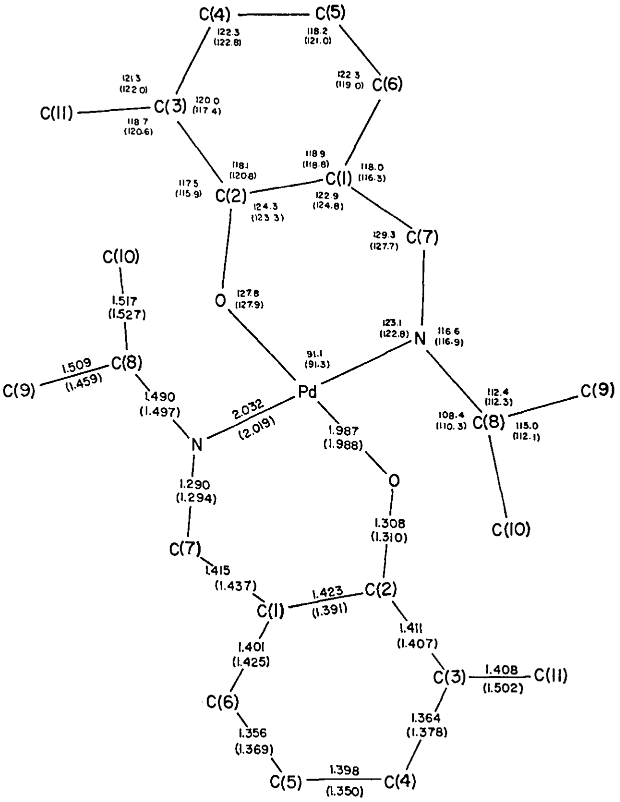

Fig. 3. Interatomic distances and angles between non-hydrogen atoms for the tetragonal and monoclinic (in parentheses) forms. Average values of the estimated standard deviations in the tetragonal form are: Pd–O, Pd–N, 0·003; O–C, 0·005; N–C, 0·006; C–C, 0·007; O–Pd–N, 0·1; Pd–O–C, 0·2; Pd–N–C, 0·3; N–C–C, O–C–C, C–N–C, C–C–C, 0·4. Average values of the estimated standard deviation in the monoclinic form are: Pd–O, 0·004; Pd–N, 0·008; O–C, 0·010, N–C, 0·013; C–C, 0·018; O–Pd–N, 0·3; Pd–O–C, 0·6; Pd–N–C, 0·7; N–C–C, 1·1; O–C–C, 0·8; C–N–C, 0·9; C–C–C, 1·1.

THOMAS, L. H. & UMEDA, K. (1957). *J. Chem. Phys.* **26**, 239.

WIESNER, J. R. & LINGAFELTER, E. C. (1966). *Inorg. Chem.* **5**, 1770.