

The Crystal Structure of a Complex Hydride of Platinum, $[\text{Pt}((\text{C}_2\text{H}_5)_3\text{P})_2\text{HBr}]$

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$[\text{Pt}((\text{C}_2\text{H}_5)_3\text{P})_2\text{HBr}]$ is orthorhombic, space group $Pn2_1a$; the unit cell has the dimensions

$$a = 14.76 \pm 0.04, \quad b = 8.92 \pm 0.03, \quad c = 13.87 \pm 0.04 \text{ \AA},$$

and contains four molecules (density observed 1.88 g.cm.^{-3} , calculated 1.86 g.cm.^{-3}). The [010] and [100] projections were studied, making systematic use of difference syntheses.

The molecule has a *trans* square-planar configuration and is therefore a complex hydride of Pt(II). The Pt-P bonds (2.26 Å) are shorter than expected, probably because they have partial double-bond character. The unusual length of the Pt-Br bond (2.56 Å) is in accord with the high chemical lability of the coordinated bromide ion. It may be attributed to an electrostatic *trans*-influence of the hydride ion, or to a *cis*-influence of the phosphorus atoms operating through the d_{xy} -type orbitals of the metal, or to a combination of these two effects.

The positions of the carbon atoms were not found accurately, but they appeared in partial difference maps sufficiently clearly to show that the principal plane of the molecule containing the platinum, bromine and phosphorus atoms is not a plane of symmetry.

1. Introduction

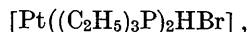
When the existence of stable volatile platinum hydride complexes was first reported from these laboratories (Chatt, Duncanson & Shaw, 1957) their stereochemistry was entirely unknown, and both the nature of the bond between the platinum and hydrogen atoms and the oxidation state of the platinum atom were uncertain. It was decided to determine the structure of one of these compounds by X-ray methods, since although the position of the important hydrogen atom could not be found experimentally, it was expected that it could be inferred once the positions of the heavier atoms were known.

Later work (Chatt, Duncanson & Shaw, 1958) on the dipole moments of a series of these hydrides suggested that the coordination round the platinum atom might be of the square planar type, in which case the platinum atom would presumably be formally divalent with the hydrogen present as a coordinated negative ion. This evidence, however, was not conclusive, and it seemed possible that these might be compounds of zero-valent platinum with trigonal pyramidal coordination round the platinum atom, with the hydrogen present in this case as a coordinated positive ion.

2. Experimental

Crystal specimens

The most suitable compound available was *bis*-triethylphosphine hydrobromoplatinum,



which crystallized from petroleum ether in large pris-

matic colourless crystals. A crystal with dimensions of about 0.01 mm. was used for recording the data for the [100] projection; the linear absorption coefficient for Cu radiation being 193 cm.^{-1} , absorption effects could be ignored.

No small equidimensional crystal could be found to give the data for the [010] projection, and a larger crystal was therefore used. It was carefully measured and found to be nearly cylindrical, length 0.3 mm. parallel to the *b*-axis, and diameter 0.115 mm. The absorption corrections of Bradley (1935) were applied to the intensities, assuming the crystal to be exactly cylindrical.

The compound is sensitive to X-rays, and this larger crystal showed signs of decomposition after the exposure needed to record the reflections with $\sin \theta \leq 0.95$ (40 hr., using a Philips sealed tube at 40 KVP and 20 m.a.). The smaller crystal was completely blackened after an exposure of 128 hr., when reflections with $\sin \theta \leq 0.8$ only had been recorded and there was considerable background. In view of these practical difficulties the structure was determined from the [100] and [010] projections only.

Crystal data

The crystals are orthorhombic, with the following unit-cell parameters:

$$a = 14.76 \pm 0.04, \quad b = 8.92 \pm 0.03, \quad c = 13.87 \pm 0.04 \text{ \AA}.$$

$$\text{Unit-cell volume} = 1826 \text{ \AA}^3.$$

$$\text{Density observed} = 1.88 \text{ g.cm.}^{-3}.$$

$$\text{Density calculated} = 1.86 \text{ g.cm.}^{-3}, \text{ assuming 4 molecules per unit cell.}$$

The unit-cell dimensions were obtained from rotation

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and Weissenberg photographs using Cu $K\alpha$ radiation (mean $\lambda=1.542 \text{ \AA}$), and the density by flotation in cadmium borotungstate solution.

The intensities of the reflections were estimated from Weissenberg photographs by visual comparison using the multiple-film method, and after correction for absorption as described above, were modified by the usual Lorentz and polarization factors. The scale factor needed to place the structure factors on the absolute scale, together with an average temperature factor for all atoms, was estimated by Wilson's method (1942).

The systematically absent reflections were: $0kl$ with $(k+l)$ odd, and $hk0$ with h odd. The two possible space groups are the centrosymmetric $Pnma$ and the non-centrosymmetric $Pn2_1a$. If the structure is centrosymmetrical, each molecule must lie on one of the mirror planes at $y=\frac{1}{4}$ and $\frac{3}{4}$. If there is no centre of symmetry and the space group is $Pn2_1a$, no molecular symmetry is required. The results of tests for piezo-electric properties were inconclusive and consequently did not assist in deciding between the two space groups.

An attempt was made to distinguish the centrosymmetrical space group $Pnma$ from the non-centrosymmetrical $Pn2_1a$ by the statistical test of Howells, Phillips & Rogers (1950). This gave no conclusive result, no doubt because the presence of the heavy atoms invalidates the statistical basis of this method. The structure analysis subsequently showed that the space group is $Pn2_1a$.

3. Determination of the structure

The method used in finding the structure was described in general terms in an earlier paper (Alderman, Owston & Rowe, 1960). It makes use of difference syntheses to remove the effects of the dominating heavy atoms and to find the positions of the light atoms with as few assumptions as possible. The atomic scattering factors were taken from the *Internationale Tabellen* (1935); as a convenient approximation the values for oxygen and fluorine were used for the $-\text{CH}_2-$ and $-\text{CH}_3$ groups respectively.

For both possible space groups the symmetry of the $[010]$ projection is the same, pgg . The position of the platinum atom was found from the Patterson synthesis $P(U, W)$, computed using the 252 observed $h0l$ reflections, and an electron-density synthesis was calculated, the structure factors being given the same phases as the contribution of the platinum atom (Fig. 1(a)). A study of this map suggested a provisional structure with the platinum, bromine and phosphorus atoms in the positions shown in Fig. 1(a), and with the light atoms symmetrically arranged about a plane of symmetry perpendicular to the $[010]$ axis, as required by the space group $Pnma$. This structure was used only for refining the scale factor between observed and calculated structure factors.

The parameters of the platinum and bromine atoms

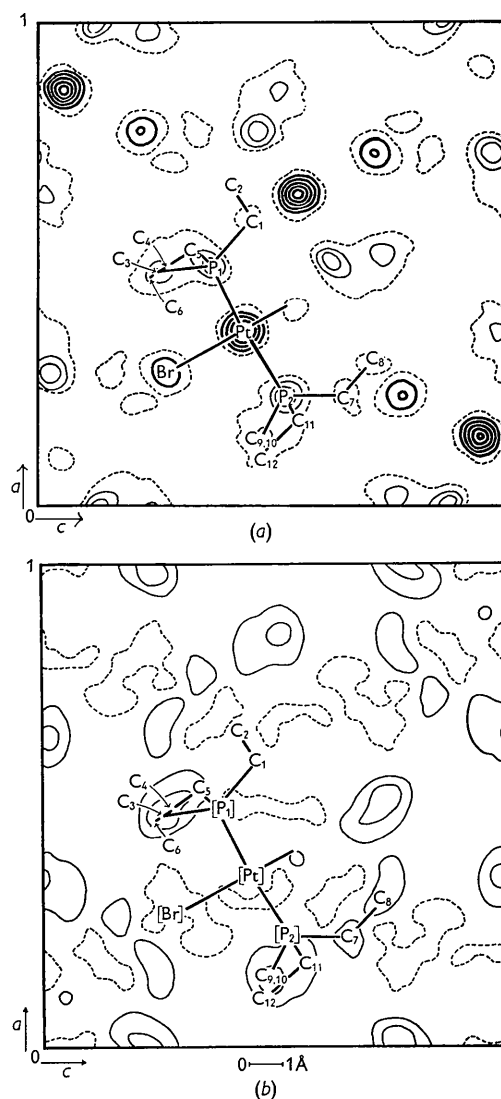


Fig. 1. Electron-density distribution projected down $[010]$, with the final non-centrosymmetrical structure. [The small peak near to the expected hydrogen atom position, *trans* to the bromine atom, is attributed to the C_1 atom of the neighbouring molecule with the platinum atom at $y=\frac{3}{4}$]. (a) q_0 synthesis, where the observed structure factors were given the same signs as the platinum atom contributions. Contours at every 20 $\text{e.}\text{\AA}^{-2}$ round platinum, 5 $\text{e.}\text{\AA}^{-2}$ elsewhere; 5 $\text{e.}\text{\AA}^{-2}$ contour broken. (b) $(q_0 - q'_c)$ synthesis, where the structure factors were given the same signs as the contributions of the platinum, bromine and phosphorus atoms, and q'_c represents the calculated electron-density distribution of these atoms. Contours at every 4 $\text{e.}\text{\AA}^{-2}$, zero contour broken.

were first refined, followed by those of the phosphorus atoms. The partial difference map obtained by subtracting the heavy atoms is shown in Fig. 1(b). Several attempts were made to place carbon atoms on the composite peaks in positions which gave reasonable bond lengths and angles. This could be done for the $-\text{CH}_2-$ groups, and a further partial difference map was

obtained, subtracting the heavy atoms and the six $-\text{CH}_2-$ groups. It was not possible, however, to find suitable positions for all six methyl groups, assuming the space group $Pnma$, since the resulting difference maps always showed negative areas round the assumed positions of at least some of the atoms.

It was therefore necessary to reconsider the symmetry. The platinum, bromine and phosphorus atoms in a given molecule must lie in or near a plane parallel to the (010) plane, since the Pt-Br and Pt-P distances in the projection are close to the expected bond lengths as given by the sums of the atomic radii. If the molecule is not exactly symmetrical about a plane parallel to the (010) plane, as is suggested by the difficulty in finding the positions of the light atoms, the symmetry $Pnma$ is only a pseudo-symmetry, and the true space group is $Pn2_1a$. It was found necessary to study the two projections simultaneously to decide whether or not this is so.

The Patterson projection $P(V, W)$, computed using the 59 observed $0kl$ reflections, showed a concentration

of vectors along $V=0, \frac{1}{2}$, implying that the structure consists of planar molecules separated by a distance of $b/2$. A possible platinum-platinum vector was confirmed by the z -coordinate already found for the platinum atom from the [010] projection. The y -coordinate of the platinum atom was assumed to be $\frac{1}{4}$, since this is required by the space group $Pnma$ and is permitted by $Pn2_1a$, in which the y -coordinate of any one atom can be chosen arbitrarily. The electron-density map (Fig. 2(a)) was calculated and a provisional structure was deduced compatible with that for the [010] projection and having the plane-group symmetry cm as required by the space group $Pnma$. The parameters of the four heavy atoms were refined, and it was confirmed that they are coplanar.

The partial difference map after subtracting these heavy atoms (Fig. 2(b)) represents the electron density of the six ethyl groups, with the mirror plane of symmetry imposed by the special positions of the heavy atoms. As in the [010] projection, positions could be found on this map for some of the $-\text{CH}_2-$ groups but not for the terminal methyl groups, strengthening the probability that the space group is $Pn2_1a$. The interpretation of the maps in Figs. 1(b) and 2(b) was therefore attempted, assuming this space group, and with the aid of models reasonable positions

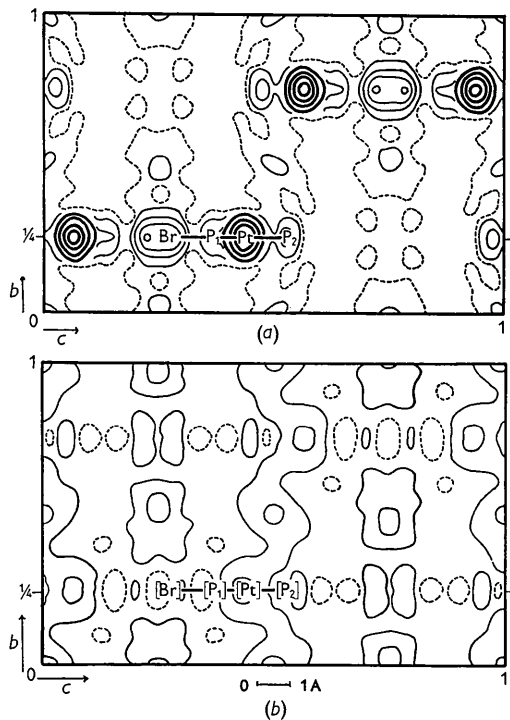


Fig. 2. Electron-density distribution projected down [100] with the heavy atom positions marked. The mirror planes at $y=\frac{1}{4}, \frac{3}{4}$ arise from the phases which were assumed; they are not required by the space group $Pn2_1a$. (a) q_0 synthesis, where the observed structure factors were given the same phases as the platinum atom contributions. Contours at every $25 \text{ e.}\text{\AA}^{-2}$ round platinum, $10 \text{ e.}\text{\AA}^{-2}$ elsewhere; $5 \text{ e.}\text{\AA}^{-2}$ contour broken. (b) $(q_0 - q'_c)$ synthesis, where the observed structure factors were given the same phases as the contributions of the platinum, bromine and phosphorus atoms, and q'_c represents the calculated electron density of these atoms. Contours at every $4 \text{ e.}\text{\AA}^{-2}$, zero contour broken.

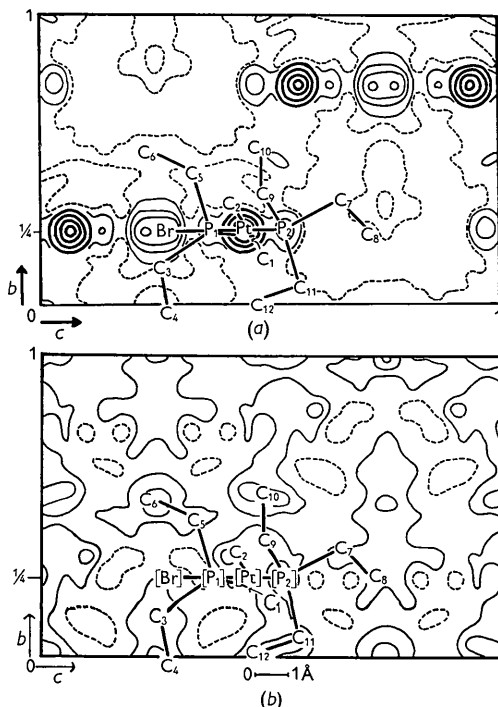


Fig. 3. Electron-density distribution projected down [100]. The phases were calculated from the final non-centrosymmetrical structure shown. The space group is $Pn2_1a$: the planes at $y=\frac{1}{4}, \frac{3}{4}$ are no longer symmetry planes. (a) q_0 synthesis. Contours at every $25 \text{ e.}\text{\AA}^{-2}$ round platinum, $10 \text{ e.}\text{\AA}^{-2}$ elsewhere; $5 \text{ e.}\text{\AA}^{-2}$ contour broken. (b) $(q_0 - q'_c)$ synthesis, where q'_c represents the calculated electron density of the platinum, bromine and phosphorus atoms. Contours at every $4 \text{ e.}\text{\AA}^{-2}$, zero contour broken.

Table 1. *Final atomic coordinates (decimal) and standard deviations (in Å)*

	<i>x</i>	<i>y</i>	<i>z</i>	[010] projection		[100] projection
				$\sigma(ax)$	$\sigma(cz)$	$\sigma(cz)$
Pt	0.1420	0.2500	0.0602	0.0046	0.0047	
Br	0.2255	0.2500	0.2221	0.0155	0.0145	0.0292
P ₁	0.003	0.250	0.128	0.034	0.030	0.056
P ₂	0.271	0.250	-0.027	0.034	0.037	0.052
C ₁	-0.094	0.233	0.040	0.121	0.103	0.223
C ₂	-0.162	0.333	0.083	0.120	0.112	0.339
C ₃	0.017	0.133	0.242	0.133	0.099	0.643
C ₄	0.008	-0.017	0.225	0.127	0.094	0.209
C ₅	-0.033	0.450	0.167	0.113	0.110	0.257
C ₆	0.025	0.516	0.258	0.116	0.100	0.140
C ₇	0.269	0.350	-0.154	0.119	0.106	0.337
C ₈	0.200	0.250	-0.217	0.125	0.103	0.223
C ₉	0.355	0.383	0.020	0.108	0.118	0.307
C ₁₀	0.355	0.533	0.020	0.109	0.123	0.251
C ₁₁	0.325	0.067	-0.058	0.126	0.100	0.444
C ₁₂	0.393	0.017	0.025	0.120	0.116	0.225
Mean C	—	—	—	0.120	0.107	0.300

Table 2. *Temperature factors*

Atom	Projection	
	[100]	[010]
Pt	exp $-[0.0183k^2 + 0.0046l^2]$	exp $-[0.0055h^2 + 0.0066l^2 + 0.0003hl]$
Br	exp $-[0.0361k^2 + 0.0088l^2]$	exp $-[0.0062h^2 + 0.0067l^2 - 0.0054hl]$
P	exp $-[3.54 (\sin^2 \theta)/\lambda^2]$	exp $-[5.00 (\sin^2 \theta)/\lambda^2]$
-CH ₂ -		exp $-[9.00 (\sin^2 \theta)/\lambda^2]$
-CH ₃		exp $-[11.00 (\sin^2 \theta)/\lambda^2]$

Table 3. *Bond lengths and angles, with standard deviations*

Bond	<i>d</i>	$\sigma(d)$	Angle	θ	$\sigma(\theta)$
Pt-Br	2.56 Å	0.04 Å	Br-Pt-P ₁	94.1°	2.0°
			Br-Pt-P ₂	93.7	2.0
Pt-P ₁	2.26	0.07	Pt-P ₁ -C ₁	112.5	9.0
Pt-P ₂	2.26	0.07	Pt-P ₁ -C ₃	104.1	20.2
P ₁ -C ₁	1.89	0.28	Pt-P ₁ -C ₅	111.4	9.4
P ₁ -C ₃	1.91	0.67	Pt-P ₂ -C ₇	117.6	11.3
P ₁ -C ₅	1.94	0.31	Pt-P ₂ -C ₉	135.8	11.4
P ₂ -C ₇	1.97	0.38	Pt-P ₂ -C ₁₁	119.9	14.9
P ₂ -C ₉	1.84	0.35	Mean Pt-P-C	116.9	12.7
P ₂ -C ₁₁	1.87	0.48			
Mean P-C	1.90	0.41	P ₁ -C ₁ -C ₂	101.9	21.0
C ₁ -C ₂	1.47	0.47	P ₁ -C ₃ -C ₄	112.5	41.6
C ₃ -C ₄	1.37	0.71	P ₁ -C ₅ -C ₆	113.8	18.0
C ₅ -C ₆	1.63	0.37	P ₂ -C ₇ -C ₈	103.6	21.5
C ₇ -C ₈	1.61	0.46	P ₂ -C ₉ -C ₁₀	130.1	26.7
C ₉ -C ₁₀	1.34	0.46	P ₂ -C ₁₁ -C ₁₂	110.6	27.9
C ₁₁ -C ₁₂	1.59	0.55	Mean P-C-C	112.1	26.1
Mean C-C	1.50	0.50			

were found for some of the -CH₂- groups. Difference maps with these atoms subtracted were then calculated for both projections, any necessary changes in their positions made, and the positions of further atoms deduced. Repeated application of this trial-and-error process, checked at every stage by difference maps, led to the structure shown in Figs. 1(b) and 3(b),

in which the heavy atoms remain in the plane $y = \frac{1}{4}$ but the ethyl groups are not symmetrically disposed about the plane.

As a further test of this structure, an electron-density map was calculated for the [100] projection (Fig. 3(a)), the phases given to the coefficients being those calculated from the structure of Fig. 3(b).

4. Results

The values found for the atomic parameters are listed in Tables 1 and 2, and the bond lengths and bond angles in Table 3 and Fig. 4. The standard deviations for the [010] projection were calculated from the diagonal elements only of the least-squares matrix, using the same system of weighting as in the previous paper (Alderman *et al.*, 1960). The standard deviations of the z -coordinates in the [100] projection were calculated in the same way, the values being approximately twice those found for the [010] projection because of the smaller number of reflections involved. The estimation of the standard deviations of the y -coordinates is less straightforward. For the atoms with a y -coordinate of $\frac{1}{4}$ the first derivative ($\partial A/\partial y$) is zero for all $0kl$ reflections, and the first-order approximations used in the least-squares method are inadequate. It has been assumed therefore, as the best available approximation, that for the [100] projection

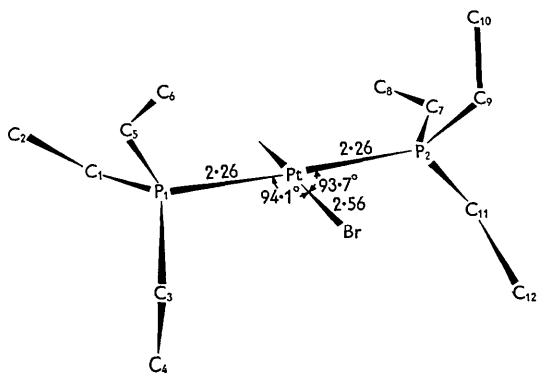


Fig. 4. Molecular configuration, with bond lengths (in Å) and angles.

the standard deviations of the y -coordinates are the same as for the z -coordinates (expressed in Ångström units). The values obtained by the least-squares method agree well with those estimated from Cruickshank's formulae (1949, 1954).

The final discrepancy factor $R = \Sigma |\Delta F| / \Sigma |F_o|$ was 0.126 for the $h0l$ and 0.112 for the $0kl$ reflections. The root-mean-square electron density in the final difference maps was 1.60 e.Å⁻² for the [010] and 1.75 e.Å⁻² for the [100] projection.

5. Discussion

The bromine and phosphorus atoms are, within experimental error, coplanar with the platinum atom, with P-Pt-Br angles of 94°. This excludes any type of trigonal pyramidal coordination round the platinum atom, and when considered together with the bond lengths and angles leaves little doubt that the four atoms, one bromine, two phosphorus and one hydrogen, are bound to platinum in a *trans* square-planar configuration (Fig. 4). It is then reasonable to conclude that the platinum atom is in the divalent state, with the hydrogen and bromine atoms present as coordinated hydride and bromide ions.

The Pt-P bonds are short (2.26 Å) compared with the sum of the atomic radii (2.42 Å), presumably because they have some double-bond character. The Pt-Br bond, on the other hand, is longer than expected (2.56 Å, compared with the radius sum, 2.43 Å), suggesting that the bond is more ionic than usual: this would agree with the high chemical lability of the bromine atom. There are two possible causes of the unusual length of the Pt-Br bond, and their relative importance cannot at present be decided.

Table 4. Observed and calculated structure factors

(a) [100] projection															
h	k	l	$ F_o $	$ F_c $	α°	h	k	l	$ F_o $	$ F_c $	α°				
0	0	0	—	492	0	12	47	47	357	9	29	23	4		
	2		164	148	0	14	47	39	182	11	50	57	3		
	4		203	197	0					13	50	64	0		
	6		171	159	180	0	3	1	187	168	350				
	8		123	121	180	3	160	170	8	0	6	0	176		
	10		102	120	180	5	174	179	3	2	72	86	165		
	12		50	45	180	7	<23	26	0	4	26	17	159		
	14		54	42	0	9	43	35	195	6	57	51	4		
						11	79	80	175	8	66	57	0		
						13	76	93	180	10	49	48	0		
0	1	1	245	253	182					12	31	24	357		
	3		166	203	193	0	4	0	333	339	4				
	5		225	247	183	2	113	103	15	0	7	1	7		
	7		<20	30	204	4	56	68	1	0	3	57	61	359	
	9		51	38	4	6	113	95	185	5	49	54	359		
	11		94	92	1	8	83	88	181	7	35	7	8		
	13		84	106	359	10	89	73	180	9	34	11	176		
						12	35	36	180						
0	2	0	416	397	183	0	5	1	99	90	182				
	2		143	126	184	3	104	110	171	2	45	44	2		
	4		55	48	139	5	90	73	176	4	38	11	19		
	6		146	146	350	7	27	26	159						
	8		116	128	2					0	8	0	359		
	10		84	94	357					2	45	44	2		
										4	38	11	19		
										0	9	1	34	23	177

Table 4 (cont.)

(b) [010] projection

<i>h k l</i>	$ F_o $	F_c	<i>h k l</i>	$ F_o $	F_c	<i>h k l</i>	$ F_o $	F_c	<i>h k l</i>	$ F_o $	F_c
0 0 0	—	492	0 0 4	179	192	10	14	-13	8	43	47
2	122	-148	1	125	-114	11	90	86	9	24	-15
4	59	-62	2	115	-89	12	34	33	10	< 12	8
6	121	146	3	164	-167	13	38	-36	11	21	21
8	190	184	4	27	20	14	< 12	2	12	22	-20
10	106	-93	5	207	220	15	< 10	-9	13	17	-15
12	37	-16	6	45	-39	16	< 9	-10			
14	88	80	7	36	-14	17	16	16	0 0 12	49	-36
16	20	-13	8	12	21				1	73	74
18	11	-13	9	114	-121	0 0 8	111	-104	2	< 14	8
			10	16	-16	1	67	47	3	< 14	7
1 0 1	164	192	11	41	31	2	60	54	4	< 14	-7
2	117	-118	12	24	-18	3	83	-72	5	41	-37
3	103	-118	13	62	51	4	152	155	6	< 14	-3
4	92	75	14	13	5	5	80	78	7	< 13	-11
5	70	-32	15	30	-25	6	49	-53	8	18	-16
6	22	-25	16	16	-12	7	33	-24	9	34	43
7	184	185	17	20	-20	8	40	-34	10	11	-1
8	< 12	2	18	< 7	1	9	< 14	-1	11	< 10	-11
9	24	-20				10	83	70	12	< 9	0
10	88	-78	1 0 5	60	-62	11	< 14	3	13	18	-21
11	81	-63	2	177	-188	12	< 13	20			
12	69	55	3	55	60	13	< 12	1	1 0 13	< 14	7
13	42	37	4	112	92	14	27	-29	2	52	60
14	27	-20	5	38	45	15	< 10	4	3	24	-21
15	38	40	6	70	53	16	< 8	6	4	25	-24
16	< 12	-9	7	90	-97				5	< 13	6
17	28	-26	8	70	-76	1 0 9	67	-61	6	29	-35
18	< 8	-1	9	34	20	2	35	35	7	< 12	-3
			10	81	-76	3	90	91	8	26	28
0 0 2	121	147	11	< 14	9	4	< 13	-4	9	11	4
1	126	-108	12	87	80	5	75	70	10	20	16
2	53	-58	13	< 14	-13	6	41	-44	11	12	-13
3	73	-79	14	< 13	-7	7	102	-101	12	18	-21
4	274	-359	15	24	-14	8	36	22			
5	152	145	16	24	-24	9	40	38	0 0 14	35	31
6	98	90	17	< 8	6	10	< 14	-5	1	23	27
7	< 11	23	18	< 6	5	11	35	39	2	13	-10
8	74	65				12	< 12	-7	3	19	18
9	105	-97	0 0 6	147	-152	13	< 11	-8	4	< 12	-16
10	95	-89	1	150	-153	14	< 10	-5	5	29	-31
11	37	21	2	41	29	15	19	-20	6	< 12	13
12	< 14	-8	3	11	-39	16	< 6	7	7	< 11	-1
13	44	43	4	85	70				8	14	11
14	40	-35	5	103	106	0 0 10	97	-98	9	19	24
15	34	-28	6	58	-71	1	24	23	10	8	-11
16	< 11	0	7	30	23	2	< 14	4			
17	16	11	8	51	-43	3	49	32	1 0 15	22	23
18	19	-20	9	90	-88	4	63	61	2	18	19
			10	50	45	5	73	-55	3	22	-22
1 0 3	43	-51	11	33	21	6	45	-41	4	< 11	-3
2	155	-159	12	21	11	7	< 14	-1	5	< 10	-2
3	77	-58	13	44	42	8	36	-38	6	20	-20
4	83	88	14	36	-30	9	32	28	7	23	25
5	112	-106	15	25	-23	10	31	34	8	16	17
6	159	165	16	< 10	5	11	19	-15			
7	85	68	17	< 8	-7	12	< 12	8	0 0 16	20	22
8	139	-120				13	18	-14	1	< 10	8
9	64	-54	1 0 7	101	-107	14	19	-22	2	10	8
10	29	-17	2	74	-47				3	< 9	-1
11	14	-19	3	170	192	1 0 11	48	-46	4	23	-27
12	63	56	4	46	56	2	69	75	5	< 8	-3
13	< 14	-10	5	< 12	4	3	44	34	6	19	22
14	13	14	6	55	48	4	31	-21			
15	22	18	7	53	-56	5	14	-10	1 0 17	14	16
16	37	-36	8	34	-25	6	54	-60	2	< 7	-2
17	21	-18	9	< 14	-12	7	28	-28	3	14	-19
18	7	8									

