RICE UNIVERSITY

The Crystal Structure of 2,6-Dichloro-4-diazinophenoxide

by

Cecil Travis Presley

A THESIS SUBMITTED
IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF

Doctor of Philosophy

Thesis Director's Signature:

Ronald L. Sass

Houston, Texas

September, 1967

ACKNOWLEDGMENTS

The author would like to express his most sincere gratitude to Dr. R. L. Sass, whose timely assistance was invaluable to the completion of this work. In addition, the author wishes to thank:

Maurice Dyke for writing the Fourier series summation programs;

The Robert A. Welch Foundation of Houston, Texas, for supporting this research;

The Common Research Computer Facility located in the Texas Medical Center and supported by United States Public Health Service Grant FR-00254 for the use of its 7094 computer.

The author is exceedingly grateful for the support given by Jerry Presley, as well as the financial support contributed by a National Defense Graduate Fellowship.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

EXPERIMENTAL

2,6-dichloro-4-diazinophenoxide, $N_2C_6H_2Cl_2O$, was prepared by Dr. James Perry by diazotizing the corresponding dichloro-aminophenol with acidic sodium nitrite at 0 degrees centigrade. The single crystals used in this study were obtained by recrystallization from hot solutions of ethyl acetate as needles exhibiting well defined faces and a high degree of optical clarity.

Shortly after the initial X-ray study had begun, it was observed that the crystals were being affected by extended periods of exposure to X-rays. The color of the crystals, initially a light orange, would change gradually into a deep red. This change was also observable on X-ray photographs as a broadening of the diffraction spots. It was presumed that this was some type of decomposition. For this reason, it was necessary to use several different crystals in the collection of the entire intensity record.

Equi-inclination Weissenberg photographs showed laue symmetry of 2/m with the following conditions for reflection:

$hk\ell$: no condition

$h0\ell$: $\ell=2n$

$0k0$: $k=2n$

These systematic absences imply that the crystals belong to the monoclinic space group $P2_1/c$.

The cell constants were measured manually, using the Philips PAILRED, by determining the omega angular displacements about the rotation

axis of the crystal necessary to produce all diffraction maxima on a given lattice row in reciprocal space. Monochromatized Molybdenum radiation was employed and both α_1 (λ= 0.70926 A°)and α_2 (λ= 0.71354 A°) wavelengths were used in the determination.

The recorded angular displacements were used in the following relation (1,p.35):

$$\frac{\lambda}{2}\,\frac{n}{d} = \sin(\omega + \alpha)$$

where: ω is an angular displacement

α is a phase angle

λ is a wavelength

n is the order of the diffracted beam

1/d is the desired reciprocal lattice spacing

The best values of the parameters in this relation were determined by choosing those values which minimize the sum of the squares of the residuals in the phase angles. The cell constants so obtained were:

a = 13.29	σ = .01
b = 10.36	σ = .01
c = 10.83	σ = .01
β = 103°4'	σ = .05

The density of 2,6-dichloro-4-diazinophenoxide was determined by floatation in a mixture of carbon tetrachloride and methylene chloride. The density of the floatation medium was found to be 1.72 g/cm^3. The density calculated for one molecule in the unit cell is 0.214 g/cm^3. This indicates that there are eight molecules per unit cell. Since $P2_1/c$ has

fourfold general positions, it appears that the asymmetric unit is composed of two molecules.

The intensity records for this compound were collected by two different techniques, both performed at room temperature. The first technique was that of Weissenberg equi-inclination photography. A standard Weissenberg camera, manufactured by the Charles Supper Company, was used with copper radiation filtered through nickel foil. The intensities were collected on Kodak Medical No-Screen X-ray film and were estimated visually by comparison with standard intensity exposures. These intensities were corrected for Lorentz, polarization, Tunnell (2,p.152) and spot size effects (3). No correction was attempted for absorption and extinction. In this manner data were collected with the a-axis as rotation axis for h=0,...,5 and with the b-axis as rotation axis for k=0,...,4. The data were then scaled by the method of Dickerson (4). This record contained 1419 intensities.

In the last stages of the investigation, counter equipment became available for use and another intensity record was collected. The instrument used was Philips Automatic-Indexing-Linear-Reciprocal lattice-Exploring-Diffractometer (PAILRED) which also employs equi-inclination geometry. Molybdenum radiation, monochromatized from the [111] face of silicon, was used. In this case the b-axis was aligned with the rotation axis. Intensities were measured by scanning omega (the angular displacement about the rotation axis) through a sufficient region to insure that the peak was well within the scan limits (usually a range of 3 degrees), and accumulating counts. Background was counted for a fixed time on both

sides of the omega scan limits. Intensities were not accepted if the relative counting statistical error $\Delta I/I$ exceeded .50.

$$\frac{\Delta I}{I} = \frac{(T+t^2B)^{1/2}}{T-tB}$$

where: T = total counts during omega scan

t = ratio of scan time to background counting time

B = total counts for background

These reflections were corrected with suitable Lorentz, polarization, and Tunnell factors (5). Because of the time element the collection of this intensity record was terminated after 871 reflections.

SOLUTION OF THE TRIAL STRUCTURE

The method used to obtain a trial structure for this compound was to solve the Patterson function for the positions of the atoms in the electron density function. It seems appropriate here to give a brief discussion of some of the properties of the Patterson function.

A Patterson function is a map of all the interatomic vectors of an electron density function translated to a common origin. The contribution to the magnitude of the Patterson from a given interatomic vector is proportional to the product of the atomic numbers of the atoms involved. Consequently, if one studies a molecule that has a few atoms which are considerably larger in atomic number than the others (such as is the case in this study), then one can expect the vectors involving the heavier atoms to be more prominent than most of the other vectors in the Patterson map. Hence, an attempt is made to relate the larger peaks in the Patterson to positions of the heavy atoms.

The first data to become available for analysis were the 0kl data. Consequently, the [100] projection of the Patterson function was calculated in hope of finding the peaks due to the four chlorine atoms.

Space group $P2_1/c$, shown in Figure 1, has fourfold general positions which are listed below (6):

$$x, \quad y, \quad z$$

$$\bar{x}, \quad \bar{y}, \quad \bar{z}$$

$$\bar{x}, \ 1/2+y, \ 1/2-z$$

$$x, \ 1/2-y, \ 1/2+z$$

Atoms located in the symmetry related positions above will produce

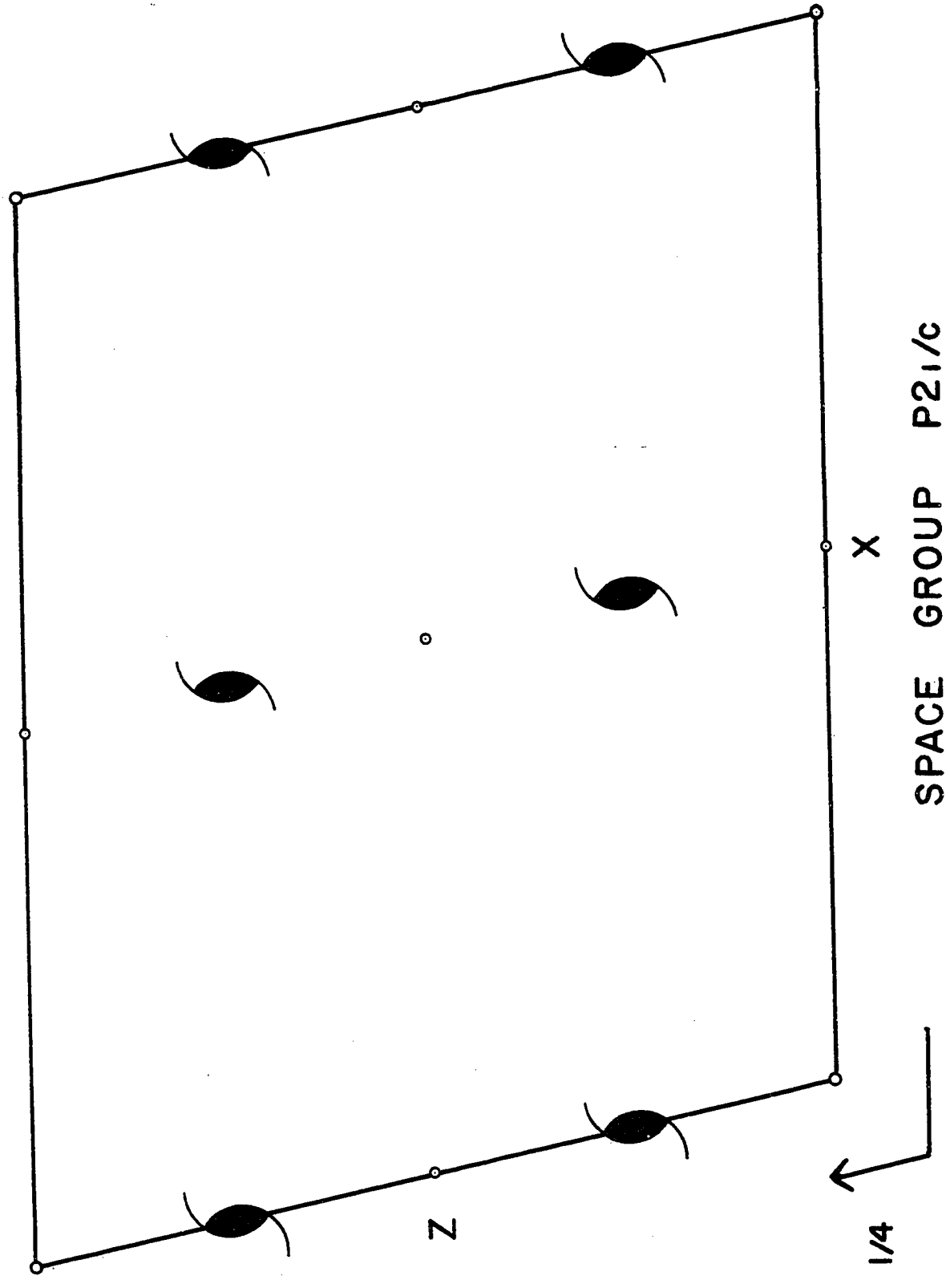


Figure 1

their maximum contributions to the Patterson function at the following positions:

COORDINATES	WEIGHT
(0, 0, 0)	4
$\pm$ (0, 1/2-2y, 1/2)	2
$\pm$ (2x, 1/2, 1/2+2z)	2
$\pm$ (2x, 2y, 2z)	1
$\pm$ (2x, $\overline{2y}$, 2z)	1

where weight is the number of times a particular peak occurs and $\pm$ indicates the presence of two peaks, each of which has the weight indicated and occupies positions related by inversion through the origin. These interactions are called Harker peaks.

The Harker line (0,1/2 $\pm$ 2y, 1/2) seemed to imply that there were only two types of chlorine coordinates in the y direction. Two chlorine atoms occupied positions near y=0 and the other two occupied positions near y=.25. If the atoms had occupied exactly those positions, pseudo-symmetry would have been introduced into the Patterson function. This feature would completely change the interpretation of the major peaks of the Patterson function.

Listed below are positions where peaks should occur in the Patterson function due to interactions between two atoms not related by symmetry.

COORDINATES	WEIGHT
$\pm$ ($\alpha_{1,2}$, $\beta_{1,2}$, $\gamma_{1,2}$,)	2
$\pm$ ($\alpha_{1,2}$, $\overline{\beta}_{1,2}$, $\gamma_{1,2}$,)	2

COORDINATES	WEIGHT
$\pm (a_{1,2}, b_{1,2}, c_{1,2})$	2
$\pm (a_{1,2}, \bar{b}_{1,2}, c_{1,2})$	2
$\pm (\alpha_{1,2}, 1/2+b_{1,2}, 1/2+\lambda_{1,2},)$	2
$\pm (\alpha_{1,2}, 1/2-b_{1,2}, 1/2+\gamma_{1,2})$	2
$\pm (a_{1,2}, 1/2+\beta_{1,2}, 1/2+c_{1,2})$	2
$\pm (a_{1,2}, 1/2-\beta_{1,2}, 1/2+c_{1,2})$	2

where $\alpha_{1,2} = x_1-x_2$, $\beta_{1,2} = y_1-y_2$, etc.

$a_{1,2} = x_1+x_2$, $b_{1,2} = y_1+y_2$, etc.

Six sets of peaks of this type will occur in the Patterson function corresponding to the six ways of pairing the four chlorine atoms. Furthermore, using the indication that the only two types of y coordinates which exist are y=0 and y=.25, values of b and β for each Patterson interaction can be calculated.

For the sake of tabulation, y coordinates were assigned as $y_1 = y_2 = .25$ and $y_3 = y_4 = 0$. Substitution of these values into the preceeding relationships obtains:

$$\beta_{1,2} = \beta_{3,4} = 0$$

$$b_{1,2} = .50 \qquad b_{3,4} = 0$$

$$b_{1,3} = b_{1,4} = b_{2,3} = b_{2,4} = .25$$

$$\beta_{1,3} = \beta_{1,4} = \beta_{2,3} = \beta_{2,4} = .25$$

It appeared that this type of interaction peak would exist at only three different values of the y coordinate in the Patterson function, namely y = 0, 1/4 and 1/2.

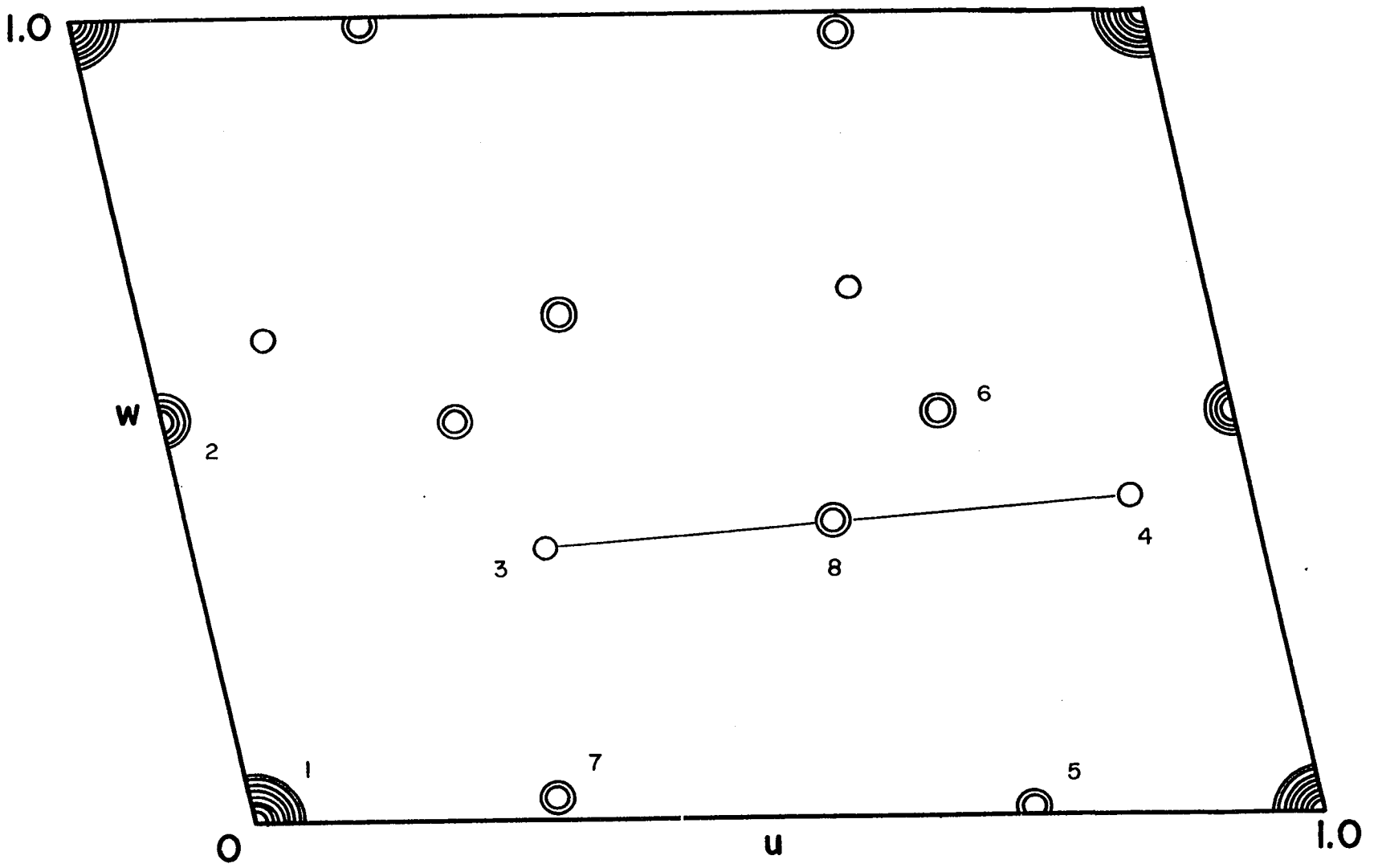
However, after synthesizing all three y sections from a partial data set, it appeared that the y = .25 section would be too complicated to interpret. The frequency with which this coordinate value occurred implied a very high density of peaks on this section. It was found that the heavy atom peaks were so populous as to induce enough false symmetry to effectively halve the translation period in the z direction. Resolution of the peaks in this region into their components seemed too unlikely, and so the analysis of this section was discontinued.

The y=0 and y=.5 sections of the Patterson function were synthesized again, this time with the full visual data set. They are shown in Figures 3 and 5 respectively.

The types of peaks which should occur on each section are listed in Table 1.

It can be noted from Table 1 that the most prominent peaks on both of the sections were due to interactions between independent chlorine atoms. The table also reveals that on the section y = 1/2, two of these larger peaks, namely 16 and 17, are related by a translation of 1/2 in the z direction. This situation occurs again relating peaks 5 and 6 on the y = 0 section. This same operation relates the smaller peaks 10 and 11 to peaks 12 and 13 respectively. Also a translation of z = 1/2 and y = 1/2 will take peaks 7 and 8 into peaks 18 and 19 in that order. Of the smaller peaks, 3 and 4 will move to the positions occupied by peaks 14 and 15 if these translations are applied.

These relations suggested that the use of image-seeking functions



P(u,0,w) Heavy Atom Patterson

Figure 2

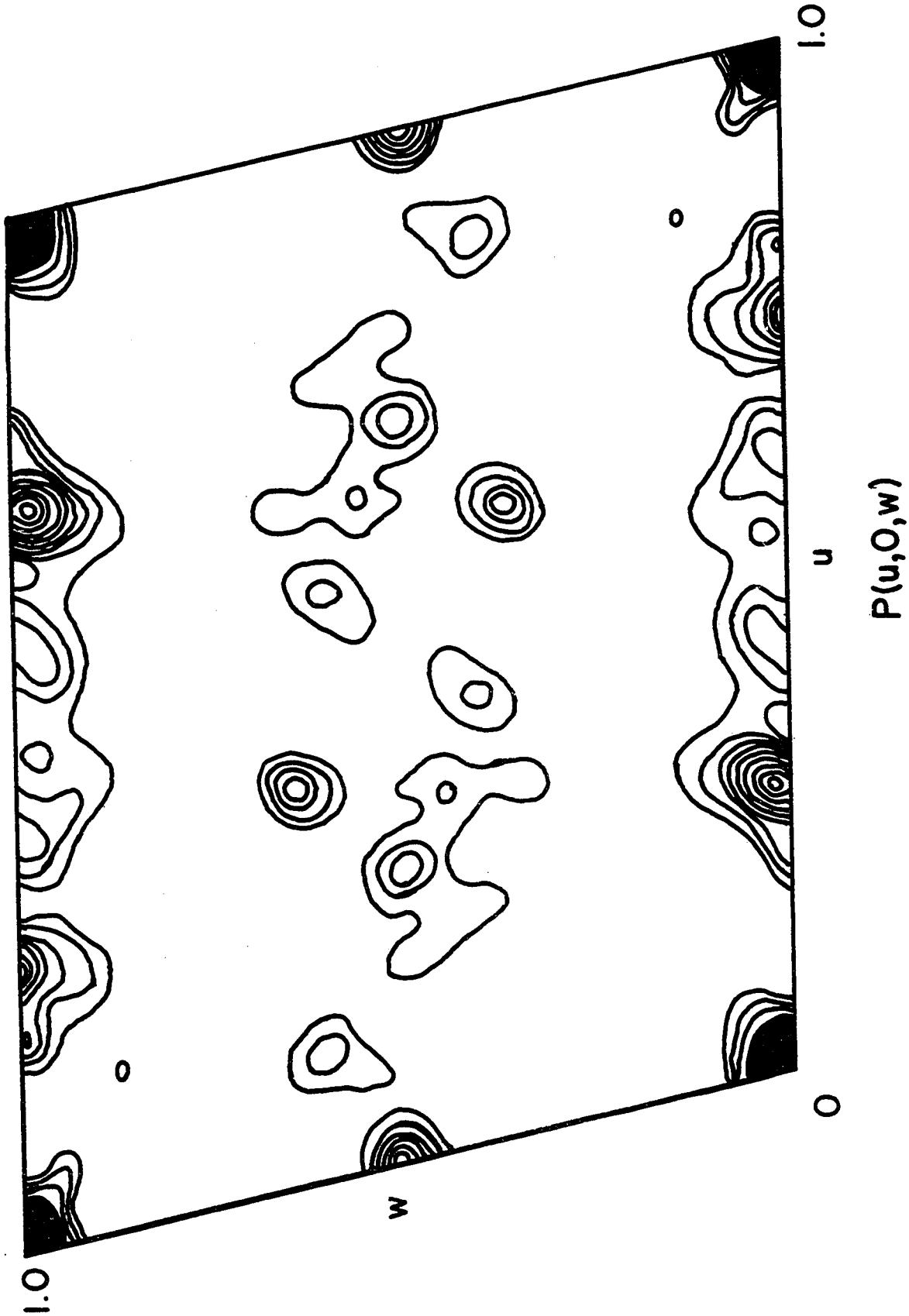


Figure 3

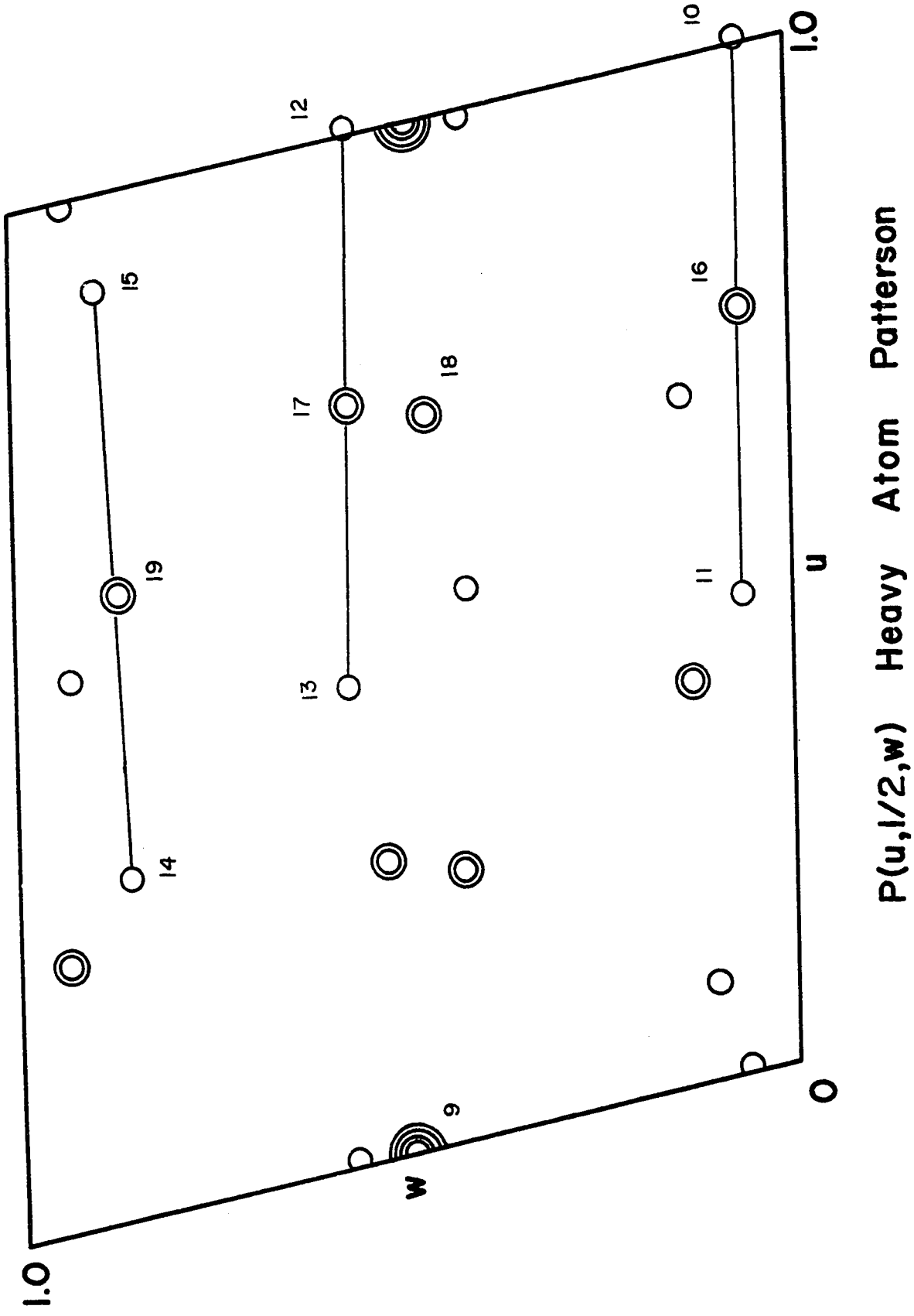

P(u,1/2,w) Heavy Atom Patterson
1.0
1.0
u
w
0
9
10
11
12
13
14
15
16
17
18
19

Figure 4

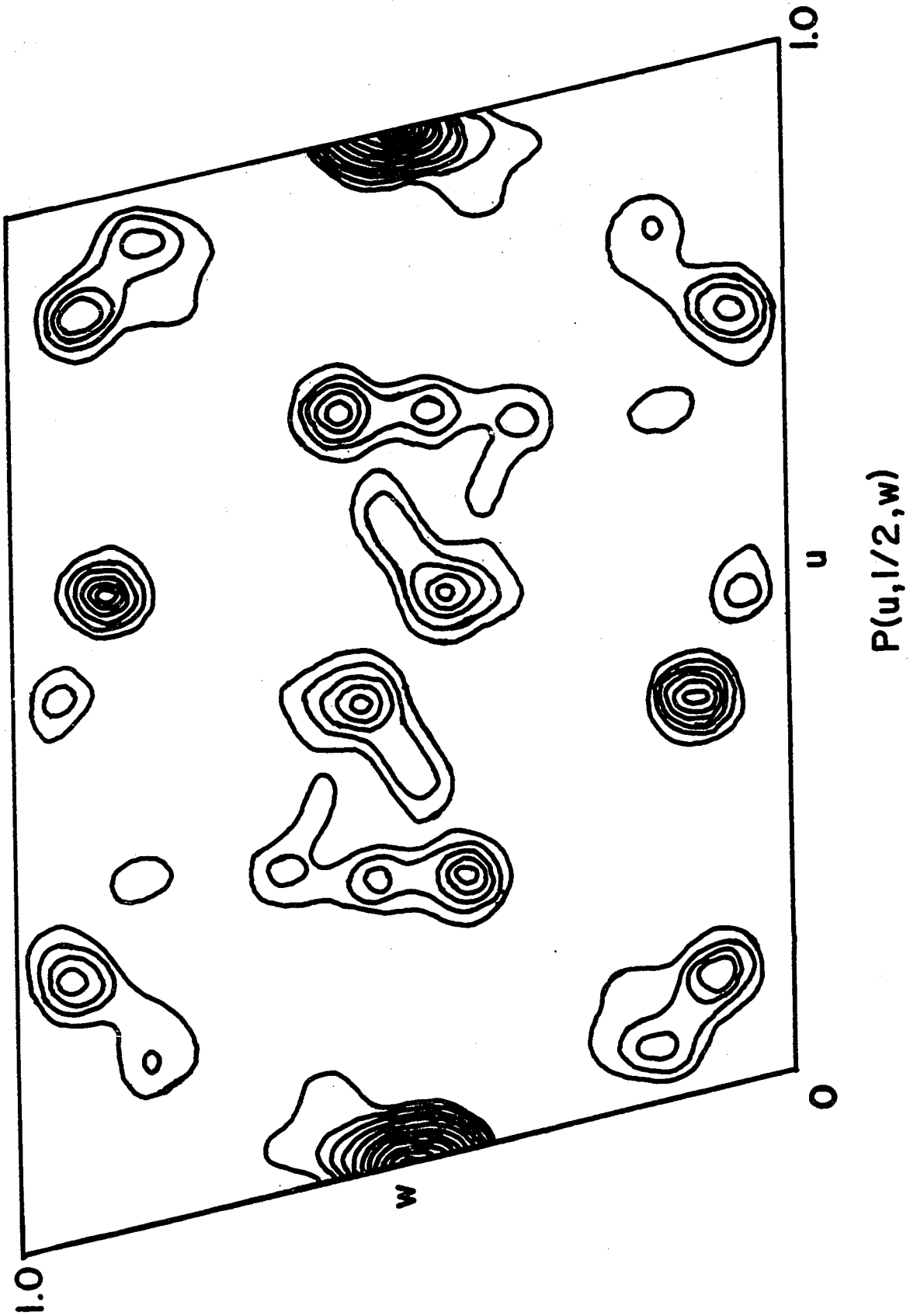


Figure 5

TABLE 1

HEAVY ATOM PATTERSON INTERACTIONS

LAYER	PEAK TYPE	COORDINATES X	COORDINATES Z	WEIGHT	PEAK NO.
Y = 0	Harker	0	0	16	1
		0	1/2	8	2
		$\pm 2x_3$	$2z_3$	2	3
		$\pm 2x_4$	$2z_4$	2	4
	Interaction	$\pm \alpha_{1,2}$	$\gamma_{1,2}$	4	5
		$\pm \alpha_{1,2}$	$1/2+\gamma_{1,2}$	4	6
		$\pm \alpha_{3,4}$	$\gamma_{3,4}$	4	7
		$\pm a_{3,4}$	$c_{3,4}$	4	8
Y = 1/2	Harker	0	1/2	8	9
		$\pm 2x_1$	$2z_1$	2	10
		$\pm 2x_2$	$2z_2$	2	11
		$\pm 2x_1$	$1/2+2z_1$	2	12
		$\pm 2x_2$	$1/2+2z_2$	2	13
		$\pm 2x_3$	$1/2+2z_3$	2	14
		$\pm 2x_4$	$1/2+2z_4$	2	15
	Interaction	$\pm a_{1,2}$	$c_{1,2}$	4	16
		$\pm a_{1,2}$	$1/2+c_{1,2}$	4	17
		$\pm \alpha_{3,4}$	$1/2+\gamma_{3,4}$	4	18
		$\pm a_{3,4}$	$1/2+c_{3,4}$	4	19

would prove profitable in locating these Patterson peaks. Image-seeking functions is the name given to a whole class of methods designed to resolve the Patterson, a function in vector space, into its fundamental components, the electron densities, which are functions in crystal space. One of these functions, the minimum function, was used here, but merely to simplify the Patterson, rather than obtain the electron density. The theory of image-seeking functions with several examples is discussed at length by Buerger (7, p. 239).

Minimum functions were constructed graphically for the sections of interest using the line images corresponding to the translations discussed in the preceeding paragraphs. However, since these line images are not contained in the electron density the resulting minimum functions, contrary to what is customary, will not resemble the electron density. Instead they do serve as simplifications of the Patterson sections in Figure 3 and 5. Figure 7 can be interpreted as the collection of peaks in the y = 1/2 section of the Patterson which also have peaks related to them by translations of 1/2 in the z direction. Figure 6 has an analogous interpretation for the y = 0 section except here the translation is y = 1/2 and z = 1/2.

It can be shown for this Patterson that the assignment of a peak of the type 2x, 2z leads to eight possible coordinates for the atom in question. If one is concerned with only a single atom, then it is immaterial which of these positions is chosen, since a choice of any given one only serves to fix an origin and one of two structural

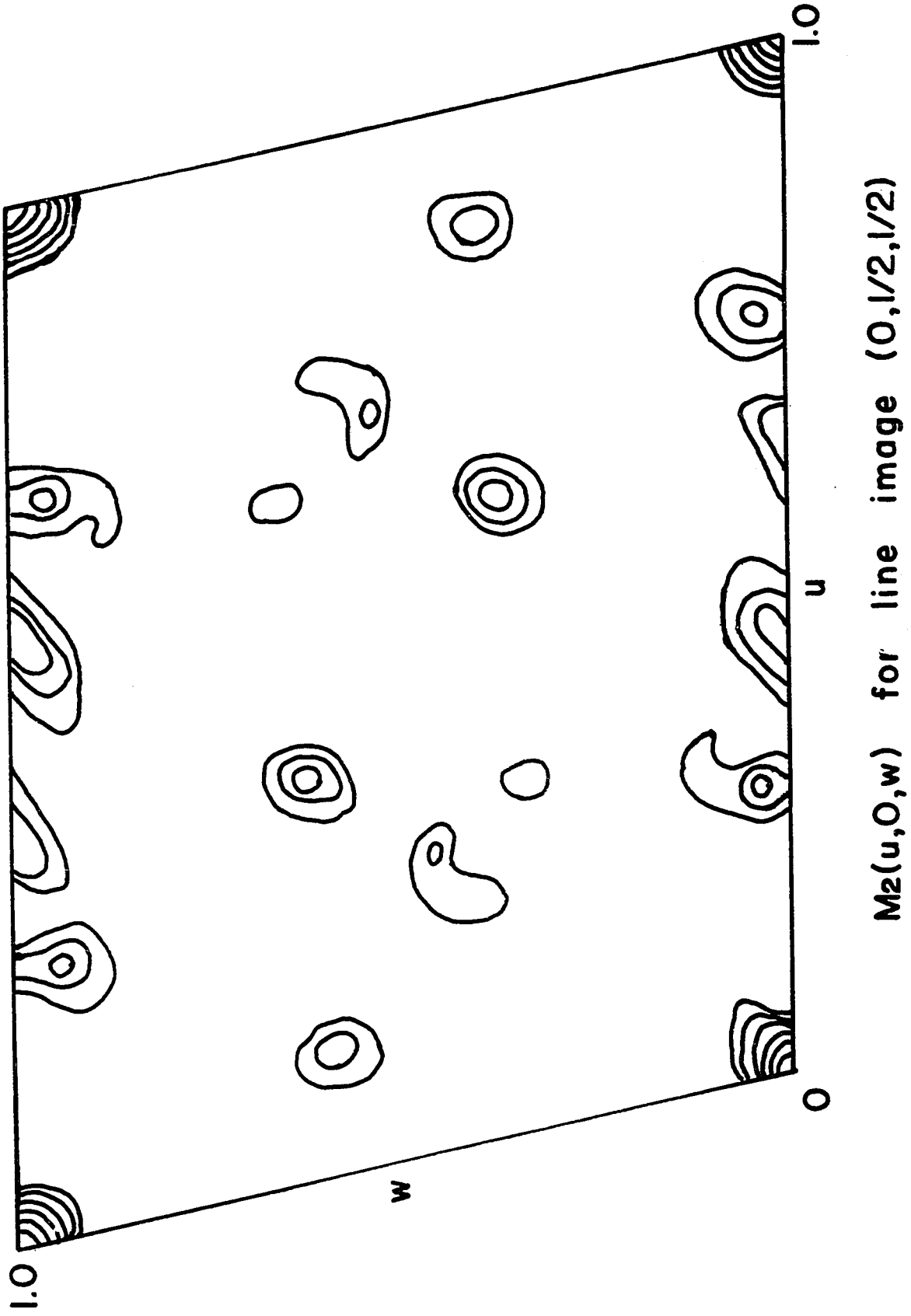


Figure 6

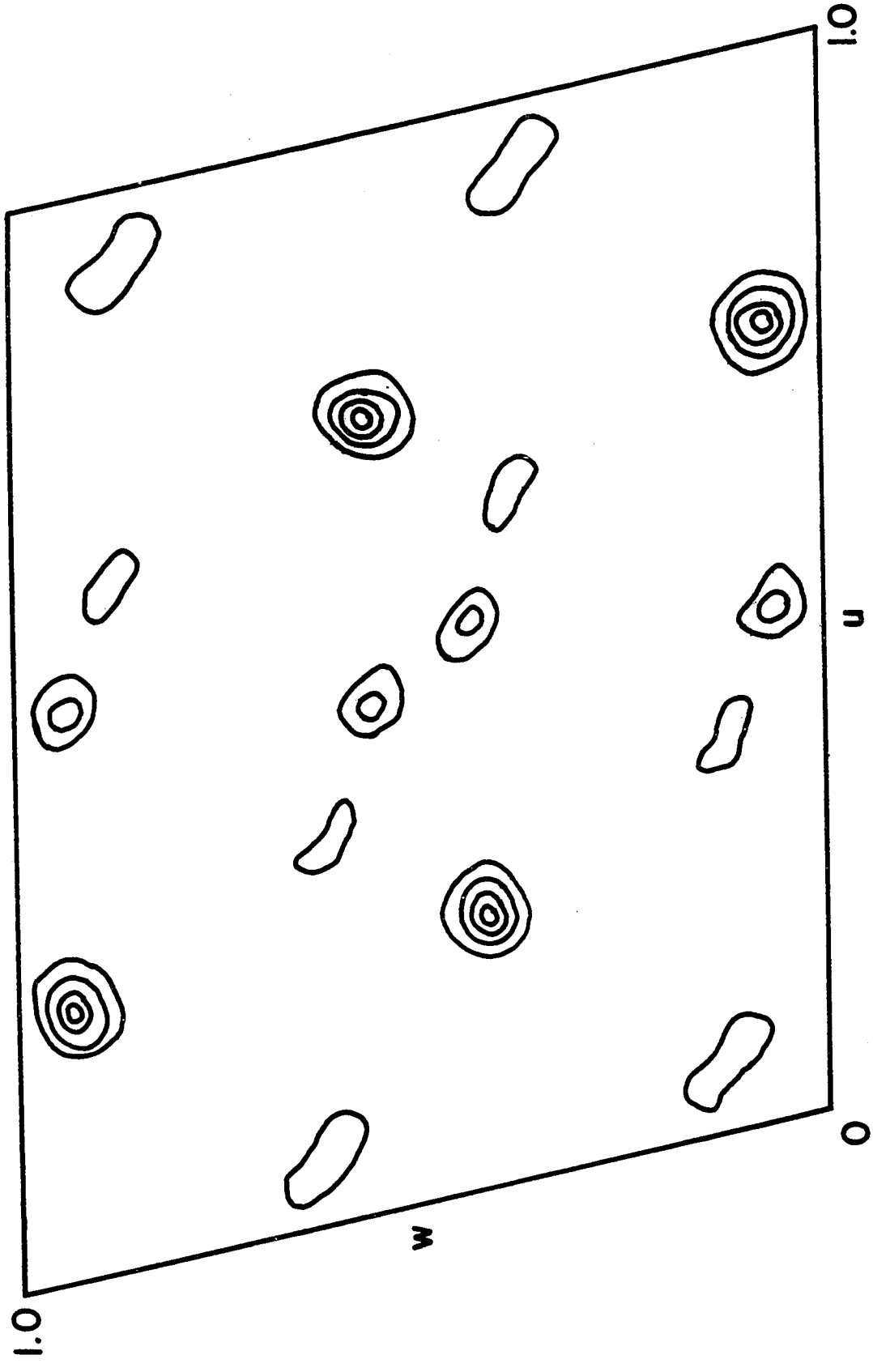


Figure 7

enantiomorphs. However once the decision is made for one atom, care must be taken to insure that the other positions are chosen to be consistent with this first choice (i.e. all atomic coordinates must be relative to the same origin and of the same enantiomorphic type). It would appear then that one must choose the correct structure from $(8)^3$ or 512 possibilities. However under further analysis it becomes obvious that the number of possibilities is much smaller.

Consider for example atoms 3 and 4. From peak 3 and 4 of Table 1 one can derive eight possible positions for each atom. Choosing to place atom 3 at a given position leaves eight choices for a position for atom 4. However, it can be shown that peak 8 must bisect a line connecting peaks 3 and 4. This relation can be used to unequivocally establish the proper atomic position for atom 4 relative to the choice for atom 3.

The same argument applies to atoms 1 and 2. However, notice that peaks due to atoms 1 and 2 are repeated by a translation of $z = 1/2$. This serves to double the number of possible positions. It appears then that one has two sets of parameters for atoms 1 and 2 which are indistinguishable.

The major problem arises when trying to relate parameters for atoms 3 and 4 to the parameters for atoms 1 and 2. All peaks arising from interactions between these sets occur on the $y = 1/4$ level of the Patterson. Since the $y = 1/4$ section was not resolved well enough to make assignments of individual peaks, there was no basis within the Patterson for establishing the relationship between atoms

1 and 3 for example. This undesirable situation is further complicated by the fact that atoms 1 and 2 were known to lie near points which are often nodes for the trignometric functions used in the structure factor calculation. An upshot of this is that small errors in these positions will make large differences in the calculated structure factors. This in turn has the effect of making least squares refinement from slightly incorrect positions more difficult if not impossible.

A trial structure was chosen for the four chlorine atoms which gave reasonably good agreement with the observed structure factors. The agreement was sufficiently good so that this structure was accepted as correct for these atoms. Using the full three-dimensional Patterson function in order to determine the approximate orientation of the molecules, several attempts were made to put the remainder of the molecules into the trial structure. All such trial structures failed to refine when least squares calculations were carried out.

Finally it was noted that if the data were divided into two groups depending on whether $k + 1$ was even or odd, the agreement of the data with the model was noticeably worse for large structure factors for which $k + 1$ was odd. This is indicative of an error in the assignment of atoms 3 and 4 with the wrong enantiomorphic choice for atoms 1 and 2. When this error was corrected a better trial structure was obtained. This trial structure was refined as indicated in the following section.

The agreement of this trial structure with the Patterson sections

which were used in its derivation can be seen by comparing Figures 2 and 4 with 3 and 5.

REFINEMENT

The refinement of this model was accomplished by least squares adjustment of the parameters X_i in order to minimize the function

$$\sum_j \left[\sqrt{\omega_j}(F_j^{obs} - F_j^{calc}) - \sqrt{\omega_j} \sum_i \left(\frac{\partial F_j^{calc}}{\partial x_i} \right) \Delta x_i \right]^2$$

The agreement of the calculated and observed structure factors was measured by the reliability index, R.

$$R = \frac{\sum_j | F_j^{obs} - F_j^{calc} |}{\sum_j | F_j^{obs} |}$$

The calculation was done with an IBM 7094 computer using the least squares analysis program ORFLS written by Busing, Levy, and Martin (8). The R index was reduced from 49% to 15% on the visual data set with isotropic refinement. Using data collected by the PAILRED this index dropped to 10.9%. Anisotropic temperature corrections were introduced into the visual data which further lowered the R index to 10.5%. The agreement between the calculated and observed structure factors at this level of refinement can be seen in Table 4.

The final positional parameters are shown in Table 2, and the final anisotropic thermal parameters are listed in Table 3. The bond distances and bond angles of interest were calculated with the IBM 7094 computer using the program ORFFE, written by Busing, Levy and Martin (9). The results are summarized in Tables 5 and 6.

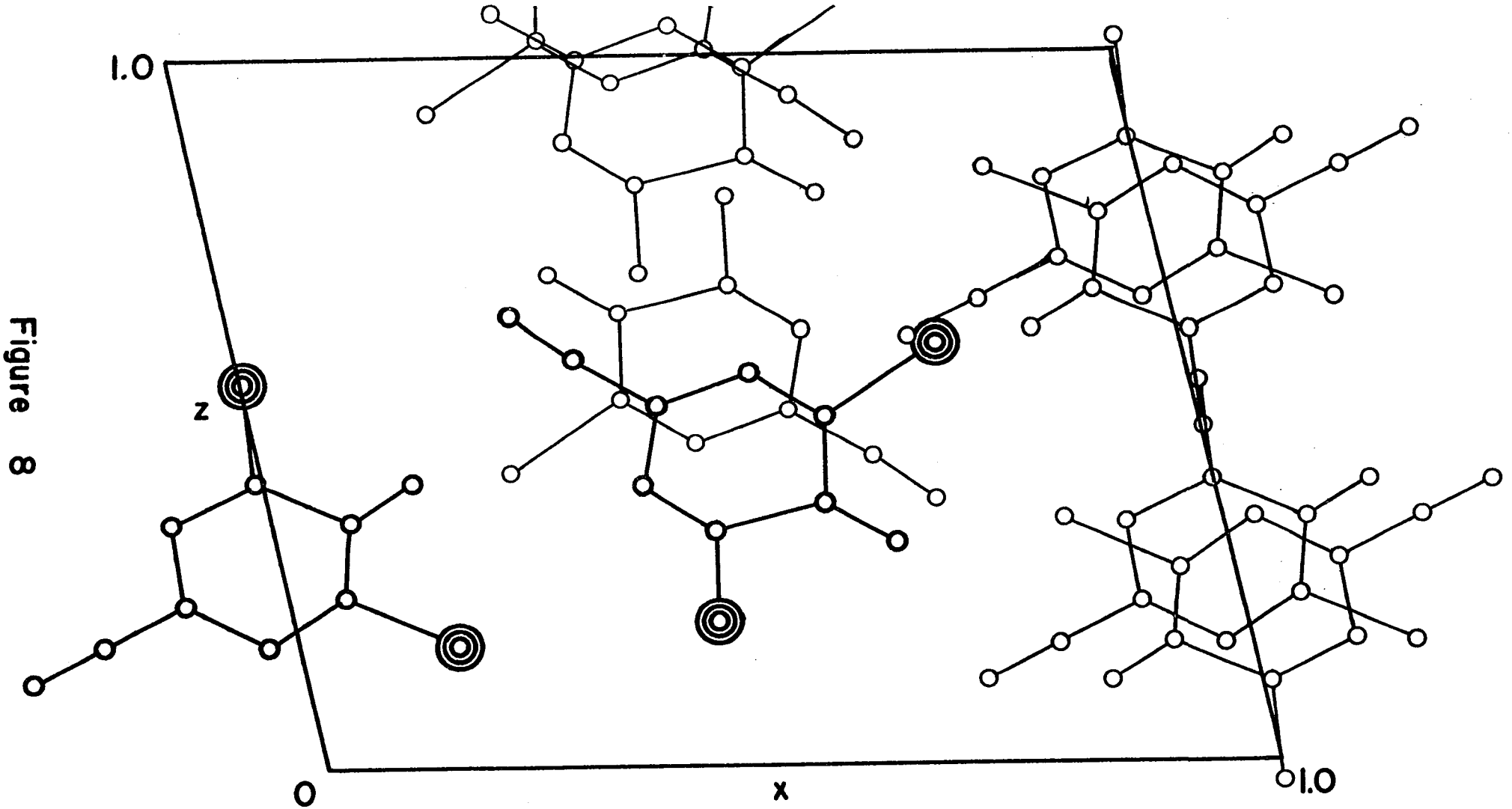


Figure 8

2,6-DICHLORO-4-DIAZINOPHENOXIDE [010] PROJECTION

TABLE 2

FINAL POSITIONAL PARAMETERS

	x	y	z
C1	-.0090 (10)	.1298	.3968 (12)
C2	.0822 (10)	.1035 (16)	.3468 (12)
C3	.0605 (10)	.0254 (17)	.2354 (13)
C4	-.0327 (10)	-.0224 (16)	.1745 (12)
C5	-.1175 (09)	.0144 (16)	.2296 (12)
C6	-.1071 (10)	.0879 (15)	.3385 (12)
O7	.1676 (07)	.1482 (13)	.3963 (10)
N8	-.2122 (09)	-.0333 (14)	.1690 (11)
N9	-.2880 (09)	-.0697 (17)	.1196 (13)
C11	.4764 (10)	.0734 (15)	.3320 (12)
C12	.5842 (11)	.1138 (15)	.3725 (11)
C13	.6063 (10)	.1966 (16)	.4823 (12)
C14	.5361 (10)	.2320 (15)	.5501 (12)
C15	.4324 (10)	.1853 (14)	.5025 (11)
C16	.4029 (10)	.1085 (15)	.3937 (11)
O17	.6491 (07)	.0783 (12)	.3174 (09)
N18	.3619 (08)	.2120 (13)	.5728 (10)
N19	.3033 (10)	.2333 (16)	.6278 (12)
CL1	.0086 (03)	.2176 (04)	.5337 (03)
CL2	.7342 (03)	.2439 (05)	.5372 (04)
CL3	.1678 (03)	-.0187 (05)	.1742 (04)
CL4	.4455 (03)	-.0213 (04)	.1997 (03)

TABLE 3

FINAL THERMAL PARAMETERS

ATOM	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C1	1.94(60)	2.30(79)	2.19(57)	-0.44(73)	1.69(202)	-0.48(56)
C2	1.69(58)	3.46(89)	2.22(56)	-0.20(76)	-1.01(205)	-0.37(61)
C3	1.77(58)	4.18(98)	2.69(60)	0.50(80)	1.95(207)	-0.98(68)
C4	1.78(57)	3.31(89)	2.41(58)	0.35(78)	1.86(198)	-0.11(62)
C5	1.07(50)	3.23(86)	2.18(55)	-0.48(71)	0.59(177)	-043(57)
C6	2.30(64)	2.65(79)	1.63(52)	0.60(73)	0.42(199)	-0.08(53)
07	1.75(45)	6.41(86)	4.08(54)	-1.21(60)	0.24(170)	-1.86(57)
N8	2.08(56)	4.83(90)	2.66(52)	0.29(70)	2.43(193)	-1.44(59)
N9	2.50(61)	6.86(116)	3.98(68)	-0.61(78)	1.55(234)	-3.02(72)
C11	1.31(52)	2.65(83)	1.93(52)	0.76(68)	-0.91(184)	-0.40(54)
C12	2.12(59)	3.11(84)	1.54(51)	1.02(76)	1.87(194)	0.34(56)
C13	1.84(60)	3.25(90)	1.99(54)	0.38(75)	0.08(194)	-0.90(59)
C14	1.75(57)	2.49(81)	2.43(57)	0.09(73)	0.53(200)	0.16(58)
C15	1.93(58)	2.06(75)	1.90(52)	0.71(70)	2.32(191)	-0.77(54)
C16	1.45(54)	2.67(77)	1.66(49)	-0.08(69)	-2.46(180)	-0.19(53)
017	2.06(44)	5.11(73)	2.76(44)	-0.91(55)	1.46(153)	-1.59(47)
N18	1.49(46)	4.14(81)	2.13(46)	-0.45(64)	-0.66(169)	-0.37(54)
N19	2.92(61)	6.14(108)	2.92(58)	0.39(80)	3.49(218)	-1.20(66)
CL1	2.74(17)	3.30(21)	2.30(14)	-0.32(21)	0.59(57)	-0.53(16)
CL2	1.60(15)	5.04(30)	4.01(18)	-1.26(21)	1.10(59)	-1.24(19)
CL3	1.81(15)	6.07(32)	3.23(17)	0.30(22)	3.74(56)	-0.94(19)
CL4	2.63(17)	4.14(24)	1.90(13)	-0.45(21)	0.61(51)	-0.83(15)

TABLE 4

Calculated and Observed Structure Factors

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
H=0				H=0			
0	2	2.68	2.33	5	1	4.82	- 6.04
0	4	20.97	- 12.73	5	3	77.61	- 76.78
0	6	42.82	43.46	5	4	51.73	- 53.74
0	8	18.63	- 17.72	5	5	13.46	14.23
0	12	6.99	- 4.78	5	10	13.05	- 18.02
1	1	22.70	- 21.53	5	11	8.19	11.82
1	2	11.81	12.38	6	0	13.09	- 12.47
1	3	37.22	- 46.16	6	1	41.95	41.86
1	4	49.37	- 61.77	6	2	28.92	- 26.98
1	5	37.82	44.02	6	3	30.31	28.47
1	6	36.97	- 40.95	6	4	5.79	6.16
1	7	20.23	- 19.14	6	7	15.44	14.86
1	8	22.78	- 20.92	6	10	8.32	11.23
1	10	11.86	- 11.68	6	11	4.49	7.54
1	13	8.68	- 3.05	7	1	32.64	32.19
2	0	22.10	22.65	7	2	9.59	10.18
2	1	30.47	- 32.50	7	5	27.67	27.91
2	2	127.43	-155.38	7	6	21.32	21.35
2	3	14.50	- 13.48	7	8	6.50	8.49
2	4	33.77	- 33.55	7	9	12.96	- 16.65
2	5	27.02	23.35	8	0	20.20	20.09
2	6	24.07	23.33	8	1	16.16	16.17
2	7	16.87	15.20	8	3	16.72	- 16.75
2	8	20.08	- 18.72	8	5	26.96	- 25.57
2	10	14.98	13.99	8	9	11.10	- 13.30
2	12	9.34	12.01	9	1	16.83	- 17.30
3	1	83.40	99.53	9	3	9.53	- 10.27
3	2	107.78	118.38	9	4	7.06	- 7.68
3	3	41.70	- 42.30	9	5	6.39	7.66
3	4	4.37	2.56	10	0	9.89	9.68
3	5	32.07	30.28	10	1	11.53	13.82
3	6	21.09	19.94	10	7	18.77	21.61
3	7	11.50	- 6.88	11	1	5.72	6.39
3	8	24.25	22.56	11	2	6.46	7.43
3	12	7.46	8.31	11	4	5.68	- 5.29
4	0	73.22	76.65	12	3	5.59	- 6.72
4	1	4.99	- 5.32	12	5	5.55	- 7.68
4	2	7.27	2.41	13	2	3.28	4.93
4	3	19.23	- 17.58	H=1			
4	4	46.71	42.99	0	-12	8.72	8.57
4	5	16.67	- 15.24	0	-10	30.78	- 30.90
4	6	20.72	18.31	0	- 4	44.65	- 44.74
4	7	10.21	- 8.21	0	- 2	3.67	- 1.77
4	8	23.90	- 22.29	0	2	11.67	- 12.77
4	9	13.57	- 12.23	0	4	42.79	44.51
4	10	6.70	- 6.53	0	6	5.70	- 3.28
4	11	8.28	7.95	0	8	7.92	6.41
4	12	5.73	- 6.04	0	10	6.95	- 7.22
				1	-13	4.34	7.60

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
	H=1				H=1		
1	-10	6.79	7.28	3	0	14.47	- 12.65
1	- 9	14.18	- 13.38	3	1	5.07	- 5.13
1	- 7	15.93	16.45	3	2	42.03	- 42.56
1	- 6	30.13	30.82	3	6	6.81	7.36
1	- 5	6.78	4.87	3	7	8.82	- 7.02
1	- 4	33.36	36.01	3	8	11.84	12.26
1	- 3	16.34	- 15.84	3	9	8.10	- 7.00
1	- 2	7.46	9.15	3	10	7.81	6.65
1	- 1	18.86	12.99	3	11	6.36	- 5.79
1	0	6.89	4.64	3	12	6.08	6.45
1	1	7.11	5.20	4	-12	4.54	5.07
1	2	25.36	21.64	4	-11	6.64	7.33
1	3	6.42	- 5.30	4	-10	14.70	- 15.00
1	4	30.47	33.02	4	- 9	6.87	7.96
1	5	3.76	2.67	4	- 6	7.09	- 6.85
1	6	4.22	- 4.13	4	- 5	28.81	27.67
1	8	18.67	- 19.19	4	- 4	6.49	- 7.40
1	10	16.29	- 17.21	4	- 3	5.89	5.50
1	12	6.11	- 10.60	4	- 2	14.50	15.95
1	13	7.83	5.19	4	- 1	13.31	- 14.45
2	-12	15.26	19.94	4	0	5.86	5.30
2	-10	11.68	- 13.65	4	1	11.70	- 12.19
2	- 8	20.72	19.87	4	2	18.53	- 17.10
2	- 7	17.11	- 16.41	4	3	21.52	18.44
2	- 6	25.61	23.58	4	4	28.19	24.77
2	- 5	8.80	- 8.50	4	5	10.34	- 10.23
2	- 4	16.32	- 15.25	4	6	19.54	18.98
2	- 3	4.07	4.83	4	7	12.67	- 11.37
2	- 2	7.27	6.33	4	8	16.16	16.60
2	- 1	5.75	8.42	4	9	9.60	- 9.56
2	0	15.84	12.07	4	11	7.89	- 9.53
2	2	16.67	- 14.71	5	-10	6.58	7.59
2	3	8.19	6.49	5	- 8	11.97	- 13.80
2	4	6.12	5.49	5	- 7	17.97	19.17
2	5	9.67	- 8.39	5	- 6	16.34	15.93
2	6	31.75	- 28.71	5	- 5	14.85	- 15.39
2	7	6.87	6.62	5	- 4	9.99	9.12
2	8	3.93	- 3.80	5	- 3	9.48	- 9.97
2	9	8.96	8.99	5	- 2	18.30	16.63
2	10	6.19	- 5.34	5	- 1	15.99	14.34
2	11	5.19	5.40	5	0	24.81	24.60
2	12	6.79	8.95	5	1	15.28	- 13.55
3	- 9	13.28	- 14.34	5	3	5.80	5.55
3	- 8	21.32	- 21.32	5	4	19.56	16.62
3	- 7	19.09	19.46	5	5	5.62	4.43
3	- 5	12.76	- 11.21	5	10	5.01	- 7.09
3	- 4	22.24	- 21.45	5	12	3.18	- 5.82
3	- 3	22.98	- 20.86	6	-12	3.04	5.42
3	- 2	24.86	- 24.27	6	- 9	8.08	- 8.59
3	- 1	16.94	16.95	6	- 8	7.93	8.54

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
H=1				H=1			
6	- 7	17.08	- 17.27	9	3	6.69	- 6.50
6	- 6	4.95	3.56	9	4	10.73	12.12
6	- 5	8.07	7.78	9	5	8.16	- 9.23
6	- 4	6.93	- 6.58	9	7	6.76	- 6.77
6	- 3	21.97	- 19.72	9	8	4.20	5.21
6	0	9.99	9.11	9	9	3.45	5.12
6	1	6.16	- 5.24	10	- 9	3.90	- 7.18
6	2	17.45	- 14.34	10	- 3	12.77	- 14.17
6	3	8.12	8.02	10	- 1	8.61	7.99
6	4	16.91	- 17.23	10	2	6.28	- 6.33
6	5	5.10	5.85	10	8	4.85	- 6.50
6	6	17.72	- 18.48	11	- 6	4.95	7.11
6	7	11.19	13.87	11	- 3	4.90	6.28
6	8	6.80	- 7.27	11	- 1	5.45	6.05
6	9	8.88	10.71	11	1	5.25	6.64
6	10	4.86	- 7.20	11	2	7.26	- 8.07
7	-11	5.26	- 7.29	11	5	5.65	- 5.87
7	-10	5.57	6.39	11	6	4.33	- 6.48
7	- 8	5.49	- 5.55	12	- 3	5.06	- 6.49
7	- 7	7.49	9.87	H=2			
7	- 6	8.39	9.29	0	-12	6.17	- 5.71
7	- 5	5.53	- 6.65	0	- 8	12.47	10.24
7	- 4	9.79	- 8.72	0	- 6	3.44	- 2.74
7	- 3	15.93	14.64	0	- 4	35.76	37.25
7	- 2	11.08	- 11.47	0	- 2	34.00	- 30.99
7	- 1	17.30	16.53	0	0	50.30	40.64
7	0	6.16	- 5.47	0	2	16.27	18.18
7	1	14.72	- 14.40	0	4	18.32	19.11
7	2	15.86	- 15.40	0	6	11.31	9.04
7	3	4.88	5.46	0	8	15.55	- 15.13
7	5	12.83	11.53	0	10	9.69	10.58
7	6	9.66	- 9.81	0	12	5.95	- 5.28
7	11	5.82	- 8.98	1	-12	5.41	6.70
8	- 8	19.11	20.56	1	-11	6.05	- 6.57
8	- 4	6.96	- 7.62	1	- 9	14.33	13.03
8	- 3	5.53	- 6.69	1	- 8	5.83	5.23
8	- 1	13.81	13.61	1	- 7	19.80	- 19.08
8	0	8.39	7.68	1	- 6	5.74	4.77
8	1	5.62	- 4.62	1	- 4	4.05	- 3.56
8	4	13.14	12.06	1	- 3	24.19	24.84
8	6	12.06	12.54	1	- 2	22.68	- 18.11
8	7	5.14	- 5.35	1	- 1	23.50	- 20.05
8	8	7.38	8.45	1	0	11.85	- 10.73
9	- 8	8.79	- 9.65	1	1	39.06	- 36.78
9	- 5	9.68	- 11.36	1	2	10.67	11.05
9	- 3	5.19	- 3.68	1	3	10.46	- 10.66
9	0	8.29	8.97	1	4	15.18	- 15.36
9	1	8.52	- 8.94	1	5	23.35	26.12
9	2	7.88	8.79	1	6	15.47	- 15.11
				1	7	22.71	- 22.87

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
H=2				H=2			
1	8	4.27	- 3.49	4	4	21.48	17.95
1	12	10.95	3.50	4	5	3.64	- 2.01
2	-12	4.65	- 4.61	4	7	7.45	- 5.69
2	-11	5.89	- 6.32	4	8	17.90	- 16.68
2	- 7	5.75	- 4.02	4	9	14.02	- 12.80
2	- 6	29.78	- 26.80	4	10	5.37	6.16
2	- 5	22.27	- 21.31	5	- 9	6.99	7.12
2	- 4	11.56	10.59	5	- 8	5.57	4.87
2	- 2	16.81	- 13.92	5	- 7	7.24	- 6.38
2	- 1	29.76	32.79	5	- 6	10.01	- 9.08
2	0	13.97	13.58	5	- 5	10.57	- 9.18
2	1	13.63	- 12.14	5	- 4	15.42	- 14.24
2	2	44.82	- 42.27	5	- 3	20.91	- 18.61
2	3	19.45	- 19.99	5	- 1	10.21	- 10.57
2	4	10.46	- 9.02	5	0	2.61	2.27
2	5	22.49	18.65	5	1	2.77	- 1.53
2	6	11.13	10.08	5	3	48.88	- 43.06
2	7	13.64	12.48	5	4	20.95	- 17.16
2	8	9.69	- 8.01	5	5	6.39	- 6.49
2	10	10.79	10.87	5	6	3.68	3.46
3	-11	8.94	- 9.19	5	11	3.92	6.76
3	- 9	11.39	10.66	6	-12	3.62	- 7.02
3	- 7	3.92	- 3.15	6	-11	5.54	- 7.91
3	- 6	3.80	- 4.16	6	- 9	5.06	- 5.91
3	- 5	13.99	11.97	6	- 7	4.67	- 4.71
3	- 4	8.59	9.77	6	- 6	7.34	- 6.83
3	- 3	7.59	7.69	6	- 5	9.23	- 7.99
3	- 2	13.38	13.58	6	- 4	4.45	4.19
3	- 1	6.27	4.76	6	- 2	3.59	- 1.63
3	0	8.21	- 8.80	6	- 1	8.80	- 8.29
3	1	35.78	36.70	6	0	13.15	12.21
3	2	18.95	16.33	6	2	5.48	5.53
3	3	15.30	13.94	6	3	14.01	12.04
3	5	26.74	24.92	6	4	14.88	15.10
3	6	10.27	6.94	6	5	10.62	10.06
3	7	10.65	- 9.75	6	7	11.83	10.76
3	8	6.18	3.66	6	10	6.36	9.57
3	9	3.91	4.58	6	11	5.56	10.08
3	11	6.15	4.92	7	-11	3.11	- 4.82
4	-13	3.24	7.12	7	- 8	5.99	5.22
4	-12	5.26	- 5.78	7	- 5	9.68	8.35
4	- 9	8.52	7.62	7	- 4	7.62	8.51
4	- 8	4.71	4.49	7	- 3	10.10	9.33
4	- 7	9.83	8.98	7	- 2	16.99	16.08
4	- 6	6.50	- 6.32	7	- 1	11.30	11.15
4	- 4	27.21	24.54	7	1	20.34	- 18.98
4	- 3	10.29	10.50	7	2	6.68	- 6.72
4	- 2	7.44	- 6.57	7	4	5.94	- 5.38
4	0	7.19	- 7.77	7	5	17.13	16.11
4	1	11.23	- 10.81	7	6	9.45	- 7.97
4	2	14.17	- 14.37	7	8	4.58	- 6.36
4	3	20.61	- 19.26	7	9	4.44	- 4.99

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
H=2				H=3			
7	10	3.48	- 4.89	1	-10	10.51	9.43
8	- 9	5.71	5.63	1	- 8	18.11	16.49
8	- 7	6.72	6.62	1	- 7	11.05	10.48
8	- 2	11.65	- 11.20	1	- 6	13.96	14.18
8	- 1	3.83	- 4.29	1	- 5	34.82	- 36.41
8	1	6.88	6.84	1	- 3	27.75	22.69
8	2	9.77	- 10.16	1	- 1	11.69	8.83
8	3	5.62	- 5.01	1	0	23.12	- 19.26
8	4	12.05	- 11.88	1	1	53.52	- 49.29
8	5	18.62	- 18.22	1	2	59.61	- 58.64
8	6	6.97	- 7.41	1	3	36.58	35.53
8	9	7.57	- 7.96	1	4	32.56	- 35.01
8	10	3.02	6.24	1	5	20.69	- 21.98
9	-10	3.53	- 6.30	1	6	14.21	- 12.46
9	- 6	5.06	- 5.16	1	8	8.72	- 8.50
9	- 5	3.97	- 3.78	1	9	12.56	13.70
9	- 3	6.22	- 5.49	2	-11	5.34	4.94
9	- 2	4.02	- 5.50	2	- 9	8.83	- 7.05
9	- 1	8.35	- 8.80	2	- 6	32.23	- 30.91
9	0	6.26	- 5.90	2	- 5	4.98	- 4.35
9	1	7.18	- 7.80	2	- 4	50.37	- 47.71
9	2	5.38	4.08	2	- 3	24.56	- 21.72
9	3	8.18	- 7.55	2	- 2	8.51	6.96
9	4	3.88	4.25	2	- 1	15.27	15.10
9	6	6.90	8.39	2	0	76.09	- 79.51
10	- 7	4.10	- 5.19	2	1	11.32	10.55
10	- 5	4.25	- 3.52	2	2	15.39	- 11.97
10	- 2	5.34	6.56	2	3	17.52	15.73
10	0	11.98	13.02	2	4	26.53	26.62
10	2	6.20	7.02	2	5	7.01	4.23
10	6	5.46	7.29	2	6	13.34	- 11.56
10	7	10.13	12.22	2	7	6.08	- 4.71
11	- 4	3.65	4.40	2	8	28.06	26.11
11	1	4.10	4.29	2	9	10.01	8.97
11	4	5.02	- 6.26	2	10	8.35	7.52
11	6	4.52	- 6.69	3	-12	9.32	- 8.78
12	- 2	2.97	- 3.98	3	-10	11.48	- 11.45
12	0	5.12	- 7.19	3	- 8	12.62	- 11.36
12	2	5.04	- 8.07	3	- 7	12.24	10.80
H=3				3	- 5	12.18	- 11.20
0	- 8	22.35	21.84	3	- 4	16.37	- 16.47
0	- 6	15.00	15.17	3	- 3	36.16	36.12
0	- 4	12.28	11.51	3	- 2	6.28	- 5.27
0	- 2	111.03	102.93	3	- 1	26.93	26.00
0	0	16.66	12.15	3	0	34.59	36.49
0	2	26.57	22.82	3	1	20.13	- 19.26
0	4	12.13	11.25	3	2	26.17	26.08
0	6	44.47	- 43.64	3	3	37.07	35.29
0	10	11.02	- 11.59	3	4	38.11	33.20
0	12	10.54	- 14.59	3	5	22.69	- 20.15

K	L	F_{obs}	F_{calc}
H=3			
3	6	11.07	9.50
3	7	10.73	- 7.54
3	9	8.74	6.78
3	10	10.15	9.99
3	11	10.85	- 13.22
4	-11	6.41	4.54
4	-10	7.14	-- 6.36
4	- 8	13.76	12.88
4	- 7	13.09	12.43
4	- 6	15.06	13.33
4	- 5	11.21	8.72
4	- 4	11.61	11.89
4	- 3	9.29	- 9.16
4	- 2	47.49	49.21
4	- 1	21.46	22.99
4	1	21.14	- 20.07
4	2	22.42	21.62
4	3	20.15	- 18.41
4	4	6.86	4.09
4	5	5.70	- 5.46
4	6	30.04	- 27.22
4	7	13.65	- 12.84
4	8	7.09	5.66
4	9	8.06	7.33
4	10	8.92	- 7.63
5	-10	8.13	9.70
5	- 6	16.47	- 14.78
5	- 5	25.42	- 22.86
5	- 4	15.28	- 14.64
5	- 3	8.93	- 6.84
5	- 1	20.39	- 9.02
5	1	36.30	- 33.26
5	2	36.08	- 32.45
5	3	21.16	20.47
5	6	10.52	- 10.26
5	8	8.03	- 9.15
5	9	11.50	15.21
6	- 9	8.72	- 8.54
6	- 6	10.35	- 10.28
6	- 4	9.91	- 7.75
6	- 3	25.21	- 23.52
6	- 1	14.47	14.00
6	0	32.03	- 30.81
6	3	12.56	12.87
6	4	11.48	10.09
6	5	16.70	16.79
6	9	6.83	- 9.09
7	- 8	10.98	- 11.24
7	- 3	19.18	18.04
7	- 2	9.10	10.20
7	- 1	14.84	13.27
7	0	14.80	13.84
7	3	16.80	17.40
7	4	16.18	15.90
7	5	7.82	- 5.86
7	7	9.10	- 10.92
8	- 8	7.69	8.32
8	- 7	11.30	11.27
8	- 5	12.31	10.94
8	- 1	10.96	11.79
8	1	13.69	- 12.86
8	3	10.89	- 10.26
8	7	11.47	- 13.37
9	- 4	9.65	- 10.26
9	- 1	13.48	- 14.47
9	1	12.47	- 13.29
9	2	9.48	- 10.02
10	5	9.37	12.07
11	- 6	6.79	8.76
H=4			
0	-12	18.88	- 20.19
0	-10	7.72	- 5.09
0	- 8	10.46	- 9.36
0	- 6	13.65	- 15.42
0	- 4	66.31	61.48
0	- 2	44.37	37.62
0	0	23.88	19.30
0	2	53.88	52.53
0	4	11.97	- 10.88
0	6	21.19	20.95
0	10	10.90	- 8.76
0	12	5.99	- 6.94
1	-12	11.82	12.71
1	-10	23.37	21.57
1	- 9	25.28	21.81
1	- 8	28.39	27.55
1	- 7	18.79	- 16.55
1	- 6	27.88	29.39
1	- 5	15.84	15.48
1	- 4	41.00	34.95
1	- 3	21.77	- 17.86
1	- 2	48.05	37.10
1	- 1	51.27	- 43.01
1	0	15.50	- 9.38
1	1	69.54	65.57
1	2	21.48	- 19.82
1	3	16.97	- 16.86
1	4	15.62	- 17.05
1	5	15.31	- 16.51
1	6	24.24	- 24.41
1	7	21.14	21.37
1	8	22.51	- 23.31

K	L	F_{obs}	F_{calc}	K	L	F_{obs}	F_{calc}
H=4				H=4			
1	9	15.19	- 16.76	4	- 2	11.20	9.61
1	10	10.64	- 9.59	4	- 1	25.07	- 25.96
1	11	10.34	10.74	4	0	35.54	37.71
2	-11	11.54	- 12.23	4	2	42.47	41.79
2	-10	17.07	16.88	4	3	9.39	9.44
2	- 9	12.95	- 13.10	4	4	11.40	- 9.73
2	- 8	6.99	6.80	4	5	29.74	- 26.34
2	- 6	16.88	- 15.57	4	6	29.00	24.81
2	- 5	4.81	- 2.53	4	8	9.84	- 7.62
2	- 3	3.26	5.34	4	9	8.62	- 6.97
2	- 2	70.83	- 68.63	4	10	10.27	- 11.57
2	- 1	44.81	- 41.16	5	- 9	8.55	10.09
2	0	46.68	- 45.02	5	- 8	14.58	15.13
2	1	10.15	9.31	5	- 7	16.85	- 16.66
2	2	17.44	14.17	5	- 6	20.90	- 19.94
2	3	25.73	23.72	5	- 4	21.29	19.14
2	4	46.70	- 43.01	5	- 2	15.52	13.94
2	5	4.85	- 5.30	5	- 1	42.45	- 37.07
2	7	14.50	12.87	5	0	29.49	- 27.55
2	10	7.76	7.39	5	4	12.77	- 12.69
2	12	8.63	12.17	5	5	11.26	- 11.43
3	-13	6.73	- 6.95	5	6	8.73	- 9.66
3	-11	8.89	- 7.80	5	7	11.21	11.28
3	-10	16.83	- 17.70	5	8	9.09	- 12.22
3	- 9	7.35	7.34	5	10	8.26	- 12.93
3	- 8	19.97	- 18.23	5	11	4.95	10.44
3	- 7	25.86	- 24.70	6	-10	10.46	11.95
3	- 6	29.54	- 29.36	6	- 9	9.94	- 10.32
3	- 5	20.54	18.35	6	- 7	10.48	- 9.25
3	- 4	38.32	- 37.85	6	- 6	13.08	- 12.23
3	- 3	27.41	27.80	6	- 5	33.49	- 31.50
3	- 1	25.23	- 23.31	6	- 3	13.17	11.89
3	0	4.71	- 3.04	6	- 2	36.11	- 31.59
3	1	29.30	27.64	6	0	20.82	- 18.81
3	2	18.27	17.70	6	3	24.17	22.87
3	3	9.57	- 8.96	6	4	33.88	- 30.70
3	5	14.51	- 11.01	6	7	12.89	14.02
3	4	19.67	17.46	7	-10	8.40	- 10.57
3	9	15.42	- 15.53	7	- 5	8.82	8.13
3	7	17.89	17.16	7	- 4	10.14	- 10.62
3	6	21.84	18.56	7	- 3	20.15	19.42
3	8	15.31	13.65	7	0	13.27	- 12.62
3	10	12.61	11.33	7	1	13.18	12.51
4	-12	14.42	- 19.24	7	2	23.38	23.19
4	-10	6.22	4.88	7	3	8.22	7.42
4	- 9	13.75	14.00	7	6	7.95	8.45
4	- 7	22.51	20.34	7	8	11.28	14.04
4	- 6	8.03	- 7.26	7	9	9.19	- 12.10
4	- 4	31.86	29.60	8	-10	5.72	6.28
4	- 3	30.08	29.97	8	- 7	13.94	15.20
				8	- 4	19.47	18.73

K	L	F_{obs}	F_{calc}
H=4			
8	- 3	18.72	19.03
8	0	8.88	7.80
8	1	14.00	- 16.29
8	2	17.86	18.90
8	5	13.20	- 13.90
8	6	8.02	8.98
8	9	6.54	- 9.92
9	- 8	6.92	7.49
9	- 3	9.29	- 9.76
9	- 2	8.94	10.53
9	- 1	12.37	- 13.66
9	0	10.03	- 11.87
9	2	12.79	14.23
9	3	7.93	- 8.21
9	7	6.21	6.95
10	- 5	9.92	- 12.25
10	0	9.10	- 10.58
10	7	8.69	13.80
11	0	9.51	- 13.09
11	1	7.94	10.40
H=5			
0	-10	31.45	29.33
0	- 8	38.28	- 38.80
0	- 4	5.41	4.03
0	- 2	12.63	- 10.40
0	0	38.64	34.64
0	2	52.66	53.28
0	4	6.80	6.80
0	6	11.98	11.31
0	8	22.66	23.27
1	-12	9.61	- 8.68
1	-11	15.29	14.39
1	-10	8.00	- 6.74
1	- 9	32.54	- 30.49
1	- 8	7.38	5.80
1	- 7	8.50	7.41
1	- 6	6.58	- 5.77
1	- 5	45.75	43.83
1	- 4	8.70	- 5.20
1	- 3	66.18	- 59.59
1	- 2	13.81	11.32
1	- 1	10.40	7.81
1	0	24.15	21.32
1	1	25.90	- 24.28
1	2	35.53	35.56
1	3	4.40	- 3.89
1	5	9.10	7.95
1	6	24.24	- 25.66
1	8	15.13	- 14.48
1	9	12.55	- 12.81
1	11	10.27	0.68

K	L	F_{obs}	F_{calc}
H=5			
2	-11	11.14	11.39
2	-10	16.18	15.17
2	- 8	25.73	- 24.77
2	- 7	23.31	- 21.52
2	- 6	35.92	36.07
2	- 5	25.33	37.66
2	- 4	5.56	5.33
2	- 3	20.88	20.15
2	- 2	22.61	- 23.61
2	- 1	8.03	- 7.06
2	0	3.95	- 2.98
2	1	3.78	- 2.81
2	2	16.88	- 15.45
2	3	20.47	- 18.07
2	4	39.64	- 38.86
2	5	20.11	18.90
2	6	17.37	- 17.11
2	10	8.07	- 8.11
2	11	5.48	- 6.42
2	7	24.18	24.07
3	-13	8.13	- 9.64
3	-12	9.57	- 9.99
3	-11	19.53	19.52
3	-10	8.59	7.50
3	- 9	7.39	- 5.79
3	- 8	19.99	19.54
3	- 6	18.21	- 17.78
3	- 5	22.98	21.89
3	- 4	7.44	- 5.52
3	- 3	17.91	- 17.36
3	- 1	44.24	46.27
3	0	44.43	- 43.24
3	1	28.11	26.23
3	2	6.34	- 4.73
3	3	9.32	7.96
3	4	11.22	8.48
3	7	16.36	- 13.69
3	10	10.76	- 11.84
3	11	6.21	- 5.43
4	-12	5.19	- 5.69
4	-11	4.80	5.56
4	- 8	30.33	- 30.17
4	- 7	18.77	- 16.28
4	- 6	30.17	29.38
4	- 5	14.91	12.63
4	- 4	16.55	- 15.93
4	- 2	25.33	- 26.52
4	- 1	13.56	- 10.72
4	1	19.90	19.33
4	2	9.92	9.15
4	3	22.57	- 22.54
4	4	29.18	27.19

K	L	F_{obs}	F_{calc}
H=5			
4	5	16.90	- 17.45
4	6	20.21	19.13
4	7	7.99	- 5.20
4	9	8.37	- 6.84
4	10	7.89	- 8.45
4	11	4.58	- 6.04
5	-11	9.58	9.98
5	- 7	13.84	- 11.60
5	- 6	13.20	- 13.80
5	- 4	32.30	27.61
5	- 3	26.97	- 24.72
5	- 2	25.38	21.99
5	- 1	7.58	- 7.37
5	0	6.03	- 1.60
5	1	10.02	- 8.77
5	2	15.57	15.22
5	3	25.08	- 22.97
5	4	6.64	7.49
5	5	30.48	- 30.23
6	- 9	10.94	9.35
6	- 6	33.00	29.31
6	- 4	10.94	- 7.79
6	- 3	6.98	- 4.79
6	- 2	18.16	15.53
6	- 1	22.05	- 18.35
6	0	27.19	24.37
6	1	17.45	15.61
6	2	17.85	- 15.83
6	3	10.79	10.67
6	4	10.98	- 10.79
6	5	9.52	8.60
7	-11	5.46	5.73
7	-10	11.11	11.43
7	- 8	9.45	- 8.10
7	- 6	11.98	- 10.53
7	- 5	16.22	14.76
7	- 4	12.02	12.00
7	- 2	15.23	- 11.65
7	0	15.53	- 14.95
7	1	17.80	16.63
7	2	13.41	- 12.60
7	3	24.15	23.64
7	4	20.96	- 20.59
8	- 9	6.61	6.75
8	- 8	9.24	- 7.52
8	- 4	25.70	- 22.79
8	- 3	16.10	13.52
8	2	8.03	- 8.87
8	7	9.38	- 12.60

K	L	F_{obs}	F_{calc}
H=5			
9	- 9	5.32	- 4.49
9	- 4	10.26	8.62
9	- 2	10.13	- 8.84
9	0	13.98	12.97
9	1	9.89	- 9.96
9	2	21.20	21.55
5	9	7.64	- 8.97
10	- 7	9.03	- 8.95
10	- 2	9.07	9.21
10	- 1	8.71	- 8.57
10	0	7.66	8.45
10	2	8.20	8.43
11	- 2	10.01	- 9.45
11	4	8.89	- 9.49
12	- 3	4.98	5.91
12	- 2	5.01	- 6.18
12	0	9.74	- 10.83
12	1	5.07	6.67
H=6			
0	-10	9.63	7.25
0	- 8	8.67	- 9.31
0	- 6	18.34	19.77
0	- 4	23.11	22.64
0	0	16.62	12.97
0	4	41.59	- 49.50
0	8	20.57	- 19.91
0	10	11.58	- 9.00
1	-10	18.48	14.26
1	- 8	21.74	21.89
1	- 7	9.13	- 8.25
1	- 6	9.14	7.05
1	- 5	13.00	11.81
1	- 4	9.36	- 8.49
1	- 3	6.22	- 4.23
1	- 2	19.61	- 12.31
1	- 1	21.52	- 14.22
1	0	25.77	- 18.54
1	1	23.19	21.31
1	2	15.79	- 17.33
1	3	11.45	- 14.27
1	4	12.06	- 13.58
1	7	31.11	34.23
1	9	18.50	- 18.94
2	-12	3.67	3.77
2	-10	5.73	3.64
2	- 9	14.27	- 12.24
2	- 8	21.15	- 19.19
2	- 7	7.59	- 5.80
2	- 6	9.90	- 8.66

K	L	F_{obs}	F_{calc}
H=6			
2	- 5	17.31	- 16.64
2	- 4	9.90	- 7.42
2	- 3	6.11	5.71
2	- 2	22.84	- 21.57
2	- 1	3.55	- 3.32
2	0	4.69	3.03
2	2	21.40	19.05
2	3	9.43	9.50
2	5	16.19	- 16.40
2	6	43.85	40.76
2	7	6.94	5.50
2	8	5.35	3.31
2	9	6.35	6.92
2	11	3.69	- 3.83
3	-12	7.88	- 7.69
3	- 8	7.66	- 6.81
3	- 7	10.86	- 11.17
3	- 6	12.93	- 13.80
3	- 5	14.24	14.51
3	- 4	8.56	- 6.83
3	- 2	19.34	- 20.05
3	0	9.99	9.11
3	1	23.11	- 21.25
3	2	29.09	26.43
3	3	17.90	- 14.66
3	5	27.49	- 22.87
3	7	14.65	10.29
3	9	20.57	- 16.96
3	10	10.29	- 9.42
4	-11	5.25	4.56
4	- 9	11.37	11.19
4	- 7	23.13	25.29
4	- 6	22.45	25.01
4	- 4	14.75	14.99
4	- 3	13.93	14.70
4	- 2	6.45	- 7.71
4	- 1	7.44	- 7.48
4	0	7.28	7.92
4	3	9.80	9.66
4	4	41.03	- 38.40
4	5	23.53	- 21.29
4	6	8.77	6.74
4	7	6.43	3.55
4	8	4.79	- 3.53
4	9	5.73	4.75
4	10	10.84	- 11.82
H=7			
0	-12	9.44	- 9.54
0	-10	16.07	13.26
0	- 8	6.63	7.38
0	- 6	26.29	29.13
0	- 4	8.26	- 5.92
0	- 2	51.57	48.64
0	0	52.17	54.58

K	L	F_{obs}	F_{calc}
H=7			
0	2	10.12	9.22
0	4	14.57	17.82
0	6	14.21	- 14.13
1	-12	8.64	6.14
1	-10	16.89	12.31
1	- 9	17.82	- 12.75
1	- 8	17.74	16.99
1	- 6	12.10	12.90
1	- 5	14.41	12.75
1	- 3	49.23	- 42.58
1	- 2	21.19	- 16.51
1	- 1	43.20	37.54
1	0	32.72	- 29.23
1	1	29.12	- 28.73
1	2	21.00	- 22.30
1	3	9.14	- 10.53
1	4	11.46	- 12.88
1	5	19.46	21.20
1	6	23.12	- 26.01
1	7	14.01	- 15.27
1	8	23.60	- 23.87
2	-12	3.52	3.22
2	-10	3.98	5.25
2	- 9	6.71	-- 5.66
2	- 8	24.01	- 22.56
2	- 7	15.20	- 12.88
2	- 5	10.41	9.50
2	- 4	40.39	- 40.94
2	- 3	8.63	6.02
2	- 2	22.46	- 22.26
2	0	21.48	- 18.86
2	1	21.13	21.63
2	2	37.53	- 35.01
2	3	12.95	- 12.29
2	4	9.44	8.38
2	5	7.12	5.83
2	6	12.73	- 12.46
2	7	16.21	16.16
2	10	9.66	13.82
3	-13	6.53	- 7.06
3	-12	11.98	- 11.76
3	-10	13.38	- 12.84
3	- 7	12.22	10.49
3	- 5	10.99	10.73
3	- 4	10.41	- 10.41
3	- 3	18.64	- 19.76
3	- 1	43.27	45.88
3	0	18.76	19.77
3	2	27.85	25.04
3	3	7.16	5.96
3	4	31.48	26.19
3	5	11.56	9.51
3	6	14.72	10.38
3	7	13.46	- 9.29

K	L	F_{obs}	F_{calc}
H=7			
3	9	8.65	7.90
3	10	16.75	18.48
4	-11	4.24	5.23
4	- 9	6.69	5.25
4	- 8	6.21	- 6.71
4	- 6	28.89	31.32
4	- 5	14.56	14.09
4	- 3	10.32	- 10.27
4	- 2	21.83	23.36
4	- 1	22.36	- 24.05
4	0	22.65	25.19
4	1	7.45	7.15
4	2	17.18	- 17.22
4	3	20.98	- 18.51
4	4	26.87	24.24
4	5	6.12	- 3.77
4	7	9.17	- 6.28
4	8	14.83	- 14.85
4	9	11.13	- 12.96
H=8			
0	-12	6.81	- 4.91
0	-10	15.53	- 13.96
0	- 8	12.62	15.31
0	- 6	11.71	- 12.11
0	- 2	43.72	41.09
0	2	32.14	38.75
0	4	22.56	23.97
0	6	4.63	- 5.86
0	8	11.94	- 12.01
0	10	10.35	- 10.31
1	-11	16.62	- 14.24
1	- 8	5.62	9.13
1	- 7	14.95	15.56
1	- 6	22.80	24.78
1	- 5	26.25	- 26.35
1	- 4	27.26	25.77
1	- 3	24.09	21.04
1	- 2	22.56	22.56
1	- 1	8.70	-- 66116
1	1	22.35	- 24.06
1	3	19.20	22.11
1	5	11.59	- 12.37
1	6	13.56	- 13.46
1	8	9.84	- 10.19
1	9	7.23	- 7.58
2	-12	3.64	- 3.83
2	-11	6.86	6.40
2	-10	8.30	- 7.23
2	- 9	9.29	- 8.87
2	- 8	26.31	24.10
2	- 6	10.64	- 9.38

K	L	F_{obs}	F_{calc}
H=8			
2	- 5	4.45	- 5.36
2	- 4	21.01	- 20.17
2	- 3	9.65	- 8.66
2	- 1	8.89	10.79
2	0	43.38	- 45.32
2	1	4.05	- 3.02
2	2	12.41	- 12.53
2	3	8.88	7.44
2	4	12.13	- 11.15
2	5	9.61	8.61
2	6	9.38	- 7.50
2	8	8.45	8.74
2	9	3.23	4.45
2	10	3.31	4.06
3	-11	11.08	- 11.54
3	-10	9.70	- 7.08
3	- 9	7.59	7.55
3	- 8	10.60	- 10.43
3	- 7	10.79	10.50
3	- 6	10.93	- 9.61
3	- 5	19.82	- 19.59
3	- 4	24.09	- 24.37
3	- 3	23.68	24.55
3	- 2	7.40	- 8.67
3	- 1	6.86	6.76
3	0	10.47	- 9.33
3	1	7.90	- 5.91
3	3	28.21	23.86
3	4	17.49	13.99
3	5	11.80	- 8.89
3	6	20.39	16.81
3	7	10.17	- 8.90
3	8	12.69	10.35
4	-11	7.58	8.77
4	-10	12.85	- 13.88
4	- 9	8.98	- 9.17
4	- 8	8.73	8.78
4	- 7	11.64	11.65
4	- 6	8.70	- 9.24
4	- 5	9.92	9.91
4	- 4	5.17	6.02
4	- 2	32.87	37.52
4	- 1	12.27	11.81
4	1	18.19	- 18.70
4	2	22.51	21.49
4	3	7.80	- 7.51
4	4	18.63	15.33
4	7	13.12	- 12.27
4	8	5.79	6.10
H=9			
0	-12	6.81	- 4.60

K	L	F_{obs}	F_{calc}
H=9			
0	- 8	10.12	- 12.64
0	- 4	13.85	15.87
0	- 2	9.53	- 7.32
0	2	7.72	- 8.84
0	4	10.85	11.77
0	6	6.54	5.95
1	- 9	16.15	15.05
1	- 7	11.71	- 14.06
1	- 4	7.18	6.19
1	- 3	10.05	7.19
1	- 2	9.54	7.23
1	- 1	13.13	- 11.21
1	0	13.87	12.96
1	7	10.92	- 10.21
1	10	3.49	- 3.74
2	-12	5.51	- 6.30
2	-11	8.47	- 8.75
2	-10	12.67	11.01
2	- 9	4.19	- 3.52
2	- 6	6.68	- 5.54
2	- 5	12.39	- 11.22
2	- 4	11.01	10.04
2	- 3	3.63	- 2.74
2	0	3.65	4.46
2	2	10.54	- 10.12
2	5	9.12	7.94
2	8	5.11	- 5.86
3	- 7	8.35	- 9.12
3	- 6	11.32	- 11.20
3	- 5	11.65	12.15
3	- 3	13.17	14.10
3	2	7.25	- 6.01
3	4	10.93	- 7.72
3	5	12.32	9.31
3	7	6.40	- 5.69
4	-12	5.22	- 8.43
4	-10	4.70	5.69
4	- 9	8.30	9.66
4	- 7	13.61	12.66
4	- 6	10.12	- 9.47
4	- 3	7.64	8.52
4	- 2	11.38	- 13.32
4	- 1	5.76	5.24
4	1	4.29	- 5.19
4	2	7.11	5.95
4	3	14.03	- 11.96
4	7	4.14	- 4.72
4	9	3.51	- 5.65

K	L	F_{obs}	F_{calc}
H=10			
0	-10	27.45	28.11
0	- 8	22.38	28.23
0	- 4	13.44	13.19
0	- 2	23.79	27.97
0	0	8.67	- 7.36
0	4	29.06	- 29.53
0	8	7.45	8.10
1	-11	6.33	5.65
1	-10	7.18	5.60
1	- 7	8.00	- 8.45
1	- 4	28.80	- 29.92
1	- 3	16.29	14.88
1	- 2	24.72	- 24.62
1	- 1	32.75	- 35.96
1	0	12.02	- 12.44
1	1	13.65	16.43
1	2	13.47	- 14.52
1	3	10.84	11.36
1	4	14.12	- 12.10
1	5	18.41	- 18.45
1	6	8.72	- 8.84
1	7	10.57	10.30
2	-10	9.35	- 7.03
2	- 8	9.19	- 8.81
2	- 7	5.89	- 4.36
2	- 6	30.15	- 28.87
2	- 4	6.74	- 5.34
2	- 3	23.07	21.41
2	0	15.98	- 15.22
2	1	10.52	- 10.53
2	2	26.52	- 27.51
2	3	11.76	10.82
2	5	5.98	6.44
2	6	3.68	3.37
2	8	10.49	- 14.23
3	-10	12.07	- 12.87
3	- 9	6.89	- 4.89
3	- 6	18.14	- 18.23
3	- 3	23.76	22.55
3	- 2	10.15	10.48
3	- 1	10.39	- 9.68
3	0	30.64	27.59
3	1	12.71	10.58
3	2	21.47	- 18.04
3	4	9.93	7.12
3	5	16.98	- 13.85
3	6	10.67	- 9.10
3	7	14.22	12.71

K	L	F_{obs}	F_{calc}
H=10			
3	8	4.94	4.80
4	-10	4.52	- 4.57
4	- 9	8.37	9.10
4	- 8	10.87	- 9.32
4	- 6	10.07	10.47
4	- 5	13.99	- 15.24
4	- 4	22.23	23.70
4	- 2	6.88	- 6.01
4	- 1	6.15	- 5.31
4	0	18.34	- 19.88
4	1	15.67	- 14.57
4	2	19.66	17.51
4	4	14.01	- 12.01
4	5	8.71	- 7.80
4	6	11.99	- 12.56
4	7	7.07	- 8.56
H=11			
0	-12	4.90	- 5.26
0	-10	12.26	12.08
0	- 8	11.12	- 13.75
0	- 4	35.96	43.33
0	0	34.14	41.88
0	2	19.30	22.58
0	4	19.34	- 20.16
0	6	11.94	11.17
0	8	16.98	- 20.05
1	-10	12.15	10.90
1	- 8	15.80	17.44
1	- 6	13.17	14.83
1	- 5	11.30	11.40
1	- 4	23.69	23.62
1	- 1	24.67	- 26.50
1	1	20.39	25.36
1	3	11.79	- 10.65
1	4	22.88	- 20.99
1	6	18.59	- 17.08
2	-11	7.24	- 9.22
2	-10	10.64	10.50
2	- 8	12.05	- 10.85
2	- 7	5.18	- 4.06
2	--6	6.91	- 5.89
2	- 5	8.81	- 6.97
2	- 2	56.60	- 56.92
2	- 1	8.60	- 6.57
2	0	11.80	- 10.92
2	3	5.53	6.03
2	4	18.32	- 22.02
2	5	5.36	6.73
2	6	13.76	18.59

K	L	F_{obs}	F_{calc}
H=11			
3	-11	5.18	6.36
3	-10	8.17	- 8.35
3	- 7	13.69	- 13.91
3	- 6	27.16	- 28.42
3	- 5	15.19	13.74
3	- 3	10.33	9.97
3	- 1	8.94	- 5.34
3	1	29.95	26.02
3	2	27.01	24.08
3	3	21.32	- 18.14
3	4	14.88	12.97
3	6	13.89	13.29
4	-10	6.78	9.27
4	- 9	9.42	12.01
4	- 8	5.23	- 4.77
4	- 7	9.79	10.34
4	- 4	24.44	28.56
4	- 3	14.23	13.89
4	-11	5.01	- 3.79
4	0	21.52	21.46
4	2	21.18	21.33
4	4	6.67	- 6.21
4	5	6.06	- 5.77
4	6	5.25	5.19
4	7	4.58	- 5.58
H=12			
0	-10	12.62	- 11.56
0	- 6	10.17	11.85
0	- 4	14.62	- 17.37
0	- 2	17.66	24.36
0	0	13.17	14.23
0	4	6.90	6.75
1	- 9	15.16	- 15.16
1	- 6	10.32	12.78
1	- 2	10.73	- 12.38
1	- 1	12.48	- 15.00
1	3	7.59	7.44
1	4	7.18	- 7.58
1	6	10.89	- 9.51
2	- 7	10.59	- 10.70
2	- 6	10.29	8.74
2	- 4	20.65	- 19.25
2	- 3	5.48	5.03
2	- 1	10.23	9.70
2	0	8.71	- 9.10
2	1	9.86	- 8.75
2	2	12.69	- 12.63
2	3	4.67	4.27
2	5	9.68	11.28

K	L	F_{obs}	F_{calc}
	H=12		
2	6	3.55	- 4.80
2	7	3.62	5.19
3	-11	5.07	7.14
3	- 9	9.12	- 10.10
3	- 8	9.86	- 9.21
3	- 7	12.88	12.78
3	- 2	15.16	- 13.71
3	- 1	16.35	14.58
3	0	12.61	11.27
3	6	5.08	4.95
4	-10	5.72	- 8.17
4	- 9	5.81	7.50
4	- 8	6.19	- 8.54
4	- 6	10.17	10.59
4	- 1	8.60	- 9.61
4	0	7.57	- 6.78
4	1	14.38	- 14.25
4	2	10.58	10.55
4	3	6.04	- 5.85
4	4	11.42	11.76
4	5	4.80	- 5.55
4	6	3.29	4.84
	H=13		
0	- 8	10.22	12.70
1	3	12.77	- 12.08
1	5	11.12	11.26
2	-10	5.76	- 6.50
2	- 7	4.20	- 4.05
2	- 6	5.07	- 4.81
2	- 2	8.84	8.24
2	3	7.00	- 8.68
2	6	4.86	8.12
3	- 7	8.58	7.95
3	- 6	5.98	4.96
3	- 5	6.61	- 7.26
3	5	6.02	5.42
4	- 8	5.35	6.56
4	- 4	4.88	- 4.82
4	1	10.35	11.53
4	2	4.98	- 5.26
	H=14		
0	- 8	9.53	12.46
0	- 6	11.67	15.30
0	- 2	18.71	22.53
0	2	11.26	- 11.11
1	- 2	10.47	- 13.04
1	- 1	11.66	12.48
1	0	15.89	- 17.16
1	1	14.41	- 13.64
1	2	17.40	- 16.03
1	3	9.15	8.94
1	4	6.66	- 8.34

K	L	F_{obs}	F_{calc}
	H=14		
2	-10	7.47	- 9.61
2	- 9	2.62	- 3.37
2	- 8	2.94	- 44335
2	- 6	6.86	- 6.54
2	- 5	4.02	3.15
2	- 4	22.88	- 23.05
2	- 3	8.55	- 6.81
2	- 1	5.74	5.48
2	0	6.49	- 6.38
2	1	5.90	5.82
2	4	13.44	17.91
3	- 7	11.52	11.26
3	- 2	8.30	6.33
3	--1	12.61	11.43
3	0	20.59	18.99
3	1	12.48	- 11.35
3	2	10.19	9.59
3	3	5.98	5.27
3	4	8.13	7.77
4	- 8	8.04	10.30
4	- 6	6.37	7.15
4	- 5	8.10	8.17
4	- 3	8.08	- 8.61
4	- 2	16.43	18.77
4	1	5.36	- 6.40
4	2	9.26	- 8.71
4	3	5.39	- 6.47
	H=15		
0	- 6	6.45	- 7.61
0	- 4	21.29	26.32
0	- 2	11.40	13.48
0	2	18.39	19.47
1	- 3	9.38	11.00
1	- 1	14.46	- 14.35
1	2	7.73	- 8.59
1	3	9.24	9.21
2	- 8	7.68	8.13
2	- 6	19.36	- 18.90
2	- 3	4.19	4.83
2	- 2	12.63	- 14.03
2	- 1	5.40	5.77
2	0	16.60	- 18.90
3	- 8	5.16	- 5.71
3	- 7	5.69	- 5.03
3	- 6	10.36	- 9.71
3	- 4	9.21	- 8.83
3	- 3	18.75	17.74
3	- 2	5.53	6.33
3	- 1	7.64	- 7.25
3	2	9.72	9.39
4	- 7	4.19	6.97
4	- 4	14.19	17.07

K	L	F_{obs}	F_{calc}
	H=15		
4	- 2	8.68	10.45
4	1	10.52	- 13.76
4	2	10.65	14.78
	H=16		
0	- 6	6.04	- 8.08
0	- 4	13.44	14.69
0	- 2	6.77	- 6.95
1	- 2	18.79	15.15
1	0	9.25	9.55
2	- 6	5.21	- 4.64
2	- 4	13.25	14.07
2	- 3	5.94	- 5.92
2	- 2	3.34	- 4.10
2	- 1	3.80	- 3.94
2	0	4.52	- 4.45
3	- 6	6.01	-- 4.91
3	- 5	11.84	11.35
3	- 4	5.50	- 5.54
3	0	11.69	- 10.71

TABLE 5

INTERATOMIC DISTANCES IN 2,6-DICHLORO-4-DIAZINOPHENOXIDE

BOND	BOND LENGTH IN A°	BOND	BOND LENGTH IN A°
C1 - C2	1.461 (18)	C11 - C12	1.462 (18)
C2 - C3	1.426 (20)	C12 - C13	1.442 (18)
C3 - C4	1.358 (18)	C13 - C14	1.362 (18)
C4 - C5	1.440 (18)	C14 - C15	1.442 (18)
C5 - C6	1.384 (18)	C15 - C16	1.402 (17)
C6 - C1	1.384 (18)	C16 - C11	1.353 (18)
C2 - O7	1.231 (16)	C12 - O17	1.211 (16)
C5 - N8	1.374 (16)	C15 - N18	1.362 (16)
N8 - N9	1.096 (15)	N18 - N19	1.103 (15)
C1 - CL1	1.709 (13)	C11 - CL4	1.707 (13)
C3 - CL3	1.764 (14)	C13 - CL2	1.740 (14)

where the notation 1.461 (18) indicates that the bond length is 1.461 A° with an estimated standard deviation of .018 A°.

TABLE 6

BOND ANGLES IN 2,6-DICHLORO-4-DIAZINOPHENOXIDE

Angle defined by three atoms with central atom at vertex

ATOMS			ANGLE IN DEGREES
C1	C2	C3	113.1 (1.2)
C2	C3	C4	127.3 (1.3)
C3	C4	C5	114.6 (1.3)
C4	C5	C6	124.2 (1.2)
C5	C6	C1	117.6 (1.2)
C6	C1	C2	123.2 (1.2)
C11	C12	C13	114.9 (1.2)
C12	C13	C14	124.6 (1.3)
C13	C14	C15	115.7 (1.2)
C14	C15	C16	123.6 (1.2)
C15	C16	C11	118.2 (1.2)
C16	C11	C12	122.9 (1.2)
C5	N8	N9	178.9 (1.2)
C15	N18	N19	178.6 (1.3)
O7	C2	C1	121.9 (1.3)
O7	C2	C3	125.0 (1.4)
O17	C12	C11	121.6 (1.3)
O17	C12	C13	123.5 (1.3)

TABLE 6

(continued)

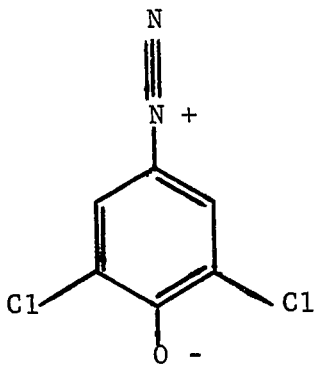
ATOMS			ANGLE IN DEGREES
CL1	C1	C2	117.3 (1.0)
CL1	C1	C6	119.5 (1.0)
CL2	C13	C12	116.7 (1.0)
CL2	C13	C14	118.5 (1.0)
CL3	C3	C2	116.0 (1.1)
CL3	C3	C4	116.7 (1.1)
CL4	C11	C12	116.7 (1.0)
CL4	C11	C16	120.3 (1.0)

DISCUSSION

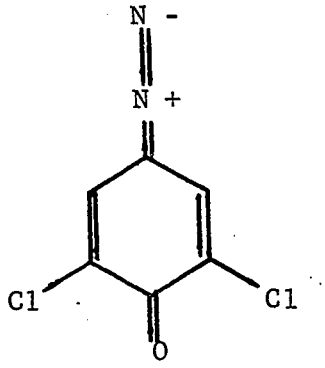
By comparing Tables 5 and 6 with the labeled asymmetric unit in Figure 9 the bond distances and angles may be related to the molecular model. The C-Cl bonds are all within the range of values found in the literature (10) for this type of bond in similar compounds.

The N-N bond distances agree rather well with the distances found in diazomethane and methyl azide. The C-N bonds and the corresponding bond in diazomethane agree within experimental error.

The six membered rings do not exhibit D_{6h} symmetry. However such asymmetry is not uncommon as a brief literature survey will show. Furthermore, in this molecule the possibility exists for resonance among the following structures:



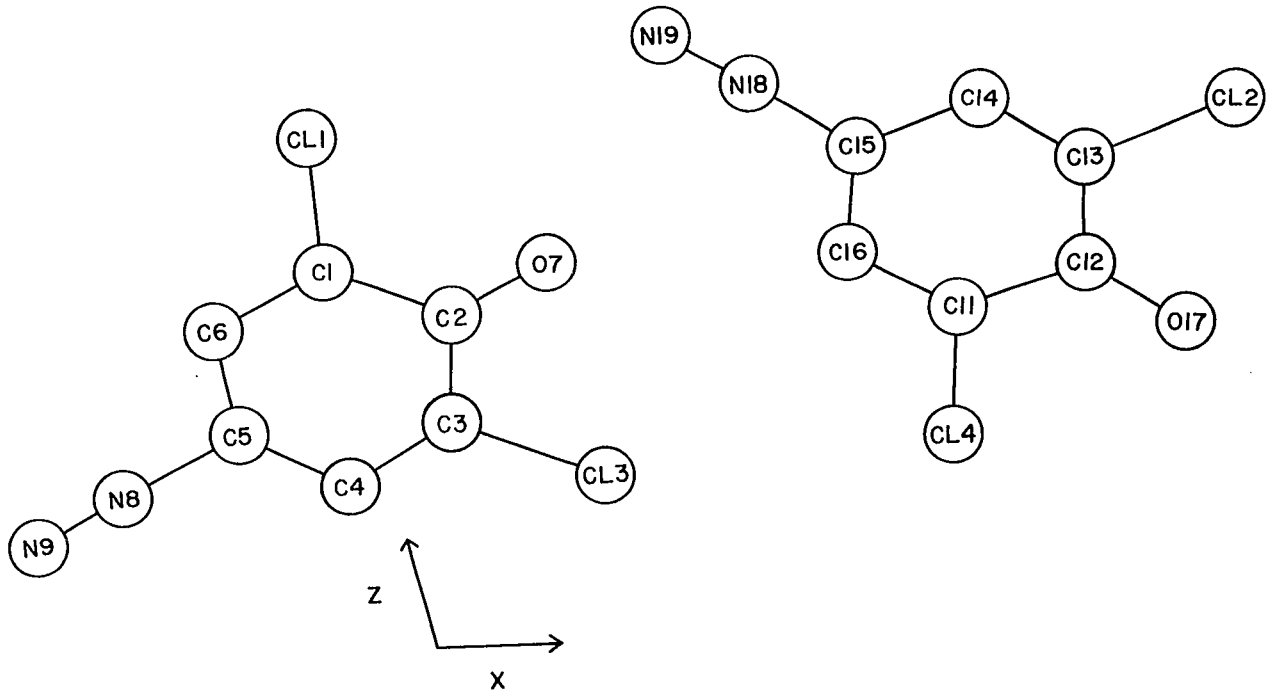
I



II

If structure II makes a significant contribution to the resonance hybrid, then the ring and the C-O bond should be expected to have quinoid character. The C-O bond is in good agreement with

Figure 9



Model of Asymmetric Unit - [010] Projection

similar bonds found in many quinone structures.

The average length observed for the bonds which would be double bonds in the quinoid structure is 1.364 A° whereas the average length observed for the other four bonds in the ring is 1.432 A°. This difference, if significant, would suggest that structure II makes a meaningful contribution to the resonance hybrid for this molecule.

There are no abnormally short intermolecular distances displayed in the crystal. The closest approach occurs between the diazo group and the oxygen atom on an adjacent molecule. This distance was calculated to be 2.90 A°, approximately the sum of the van der Waal's radii for these two atoms.

To determine the planarity of the rings, least squares planes were fitted to the six carbon atoms in each molecule. These results are displayed in Table 7. For the atoms used in determining the plane the deviations found in each molecule were judged to be within experimental error. However, when the distances of the other atoms from this plane were calculated significant deviations were found. For example in molecule I it appears that the oxygen atom as well as the chlorine atoms are slightly above and below the plane respectively, with the diazo group being approximately coplanar with the ring. In molecule II the oxygen and chlorine deviations were less and may not be significant in all cases. However, the diazo group shows a remarkable deviation from the plane of the ring.

TABLE 7

PLANARITY OF 2,6-DICHLORO-4-DIAZINOPHENOXIDE

ATOM	PLANE	Deviations from least squares planes fitted to atoms in the ring (in Angstroms)
C1	1	-.016
C2	1	.013
C3	1	.002
C4	1	-.015
C5	1	.013
C6	1	.003
O7	1	.073
N8	1	-.005
N9	1	.001
CL1	1	-.070
CL3	1	-.061
C11	2	-.001
C12	2	-.014
C13	2	-.017
C14	2	-.006
C15	2	-.009
C16	2	-.012
O17	2	-.059
N18	2	-.120
N19	2	-.200
CL2	2	-.043
CL4	2	.009

The equations of the planes in unit cell coordinates are:
Plane 1: $-.398\,x + 8.575\,y - 5.839\,z = -1.183$
Plane 2: $-1.075\,x + 8.437\,y - 5.865\,z = -1.839$

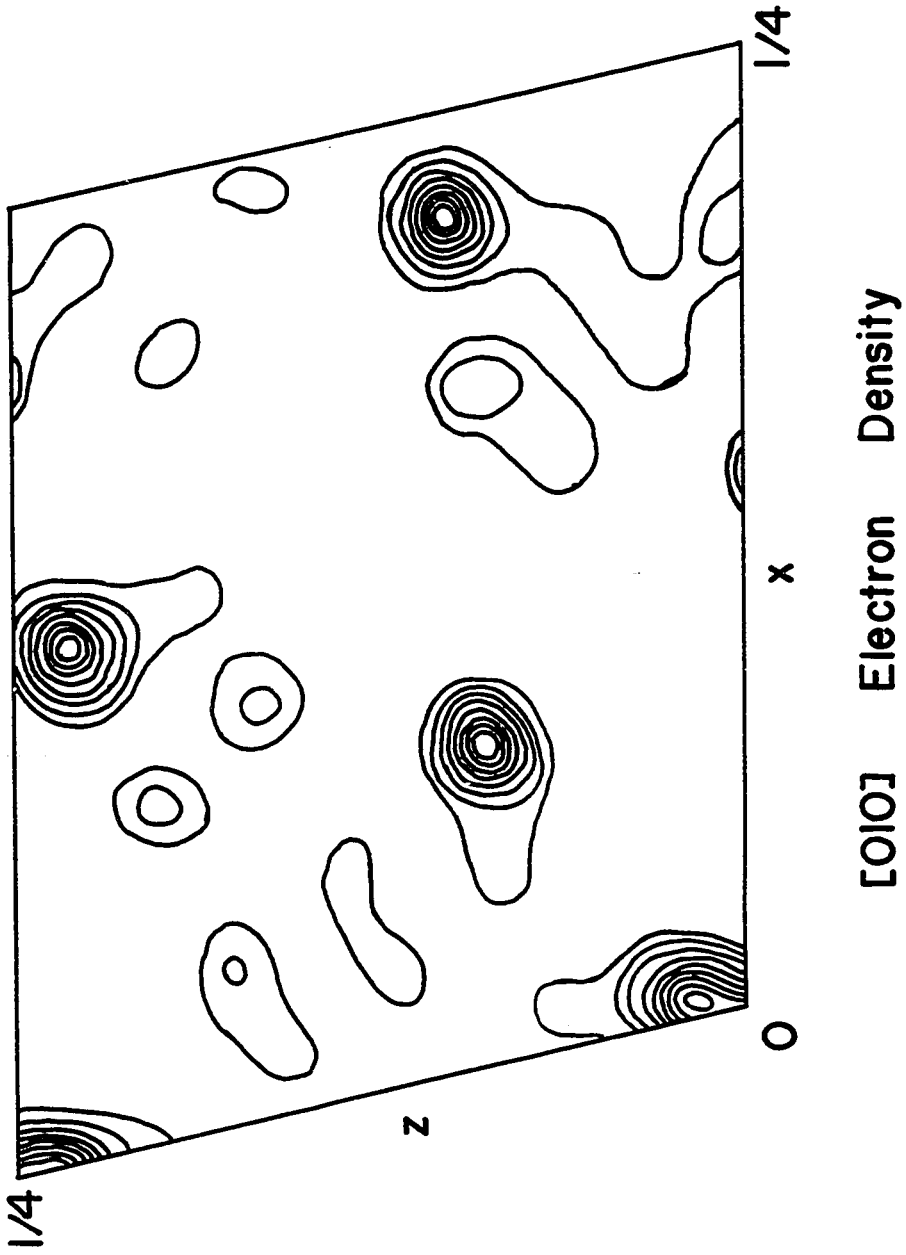


Figure 10

It is difficult to attribute this deviation to the nature of the bonding in this molecule because there is no apparent trend of this type in the other independent molecule. A reasonable alternative to this is the hypothesis that this deviation is due to packing forces in the crystal. Since the molecules are independent, they almost certainly exist in slightly different environments, and because of this could exhibit differences in configuration.