THE CRYSTAL STRUCTURE

of

DIGLYCINE HYDROBROMIDE

by

(Mrs.) ELSA PAULINE BARNEY

A.B., Syracuse University
(1944)

SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

at the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
(1947)

Signature of author.....
Department of Geology, May 23, 1947

Certified by.....
Thesis Supervisor

.....
Chairman, Department Committee on Graduate Students

The Crystal Structure of Diglycine Hydrobromide

Table of Contents

Introduction

Due to the possibilities of the use of diglycine compounds in glycine separations and identifications and their usefullness as therapeutic agents it would seem desirable to obtain information on the structure of one such compound. By X-ray analysis the arrangement of the atoms in the crystal unit may be determined and the properties of the crystal may be accounted for in terms of this arrangement. A crystal such as diglycine[1] hydrobromide affords an excellent example of the use of a heavy atom in determining the phase constants which cannot be ascertained from experimental observations.

Briefly the steps in the structure determination consisted of the following:

I. Determination of the unit cell and space group.

2. Determination of the atomic positions.

Literature and History of the Problem

As far as is known no previous work has been done on the crystal structure of diglycine hydrobromide. The method of preparation of diglycine hydrobromide, diglycine hydrochloride

---

(I) Refers to two glycine molecules, not glycylglycine.

and diglycine hydriodide has been reported by Walter S. Frost.[2] Previous to this diglycine hydrochloride was reported by K. Kraut[3] and F. Hartmann.

Material

Crystals and powder of diglycine hydrobromide ($C_4H_{11}BrN_2O_4$) were presented by Dr. W. S. Frost of the Burnham Soluble Iodine Co, Auburndale, Mass. The material had been made by evaporation of water solutions of glycine and monoglycine hydrobromide and glycine and hydrobromic acid in theoretical quantities. Suitable crystals for x-ray work could not be found in this material so that crystals were grown by evaporation of a water solution of the diglycine hydrobromide powder. The crystals are stable in the dry condition, very soluble in water yielding free hydrobromic acid on dissolving in water and are insoluble in alcohol and ether. The average melting point in I63° - I65°.

The orthorhombic crystals are elongated parallel to the c axis and are almost equidimensional in cross section. The typical habit of the crystals is shown in Figure I.

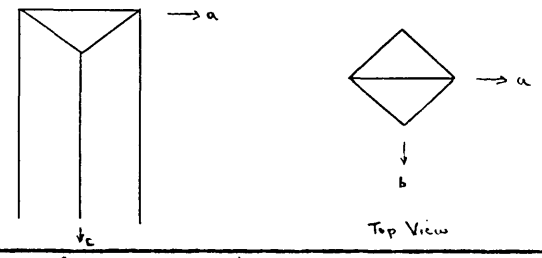


Figure I.

(2) Walter S. Frost. Bis (Amino-Acid) Derivatives. I. Diglycine Halogen Addition Products. J.A.C.S. 64 (I942) I286.

(3) K.Kraut and F. Hartmann. Ann I33 (I865) I0I.

Experimental Method of Investigation

The colorless crystals of diglycine hydrobromide were investigated by means of the De Jong, Weissenberg, precession and rotation x-ray methods. X-ray work with the De Jong method was done by M. J. Buerger. For the determination of parameters the Weissenberg method was used for the hko and hol reflections using CuK$_\alpha$ radiation. The precession method was used for the okl reflections using MoK$_\alpha$ radiation. The Dawton[4,4c] method was used to determine the intensities.

The Unit Cell

A c-axis rotation photograph gave c = 5.40 Å. Measurements from a c-axis Weissenberg photograph gave a = 8.21 Å and b = 18.42 Å.

The number of molecules per cell is 4 and may be calculated as,

$$Z = \frac{\text{Volume (density)}}{\text{Formula weight } (1.66 \times 10^{-24})}$$

$$= \frac{(5.40)(18.42)(8.21) \times 10^{-24} \ (1.941)}{231.02 \ (1.66 \times 10^{-24})}$$

$$= 4.12$$

---

(4) Dawton, R.H.V.N. The Integration of Large Numbers of X-ray Crystal Reflections. Proc. Phys. Soc. 50 (1938) 919-925.

(4)$_2$ Klein, G.E. The Crystal Structure of Nepheline. Thesis M.I.T. (1947)

The Space Group

The space group $P_{2_1 2_1 2_1}$ was determined by M.J. Buerger using the Weissenberg photographs. Characteristic absences noted indicating this space group are:

hoo	where h odd
oko	where k odd
ool	where l odd

Reflection	Condition for non-extinction	Interpretation of Extinction	Symbol of symmetry element
hoo	h = 2n	[00$\bar{1}$] screw axis, component $a/2$	2_1
oko	k = 2n	[0$\bar{1}$0] screw axis, component $b/2$	2_1
ool	l = 2n	[00$\bar{1}$] screw axis, component $c/2$	2_1

Only general positions occur in this space group. The equi-points with the conventional origin[6] of this space group are:

$$xyz;\ \tfrac{1}{2}-x,\ \bar{y},\ \tfrac{1}{2}+z;\ \tfrac{1}{2}+x,\ \tfrac{1}{2}-y,\ \bar{z};\ \bar{x},\ \tfrac{1}{2}+y,\ \tfrac{1}{2}-z.$$

---

(5) M.J.Buerger. X-Ray Crystallography, John Wiley and Sons Inc. (1942) 83.

(6) Internationale Tabellen zur bestimmung Kristallstrukturen. I (Gebruder Borntraeger, Berlin (1935).

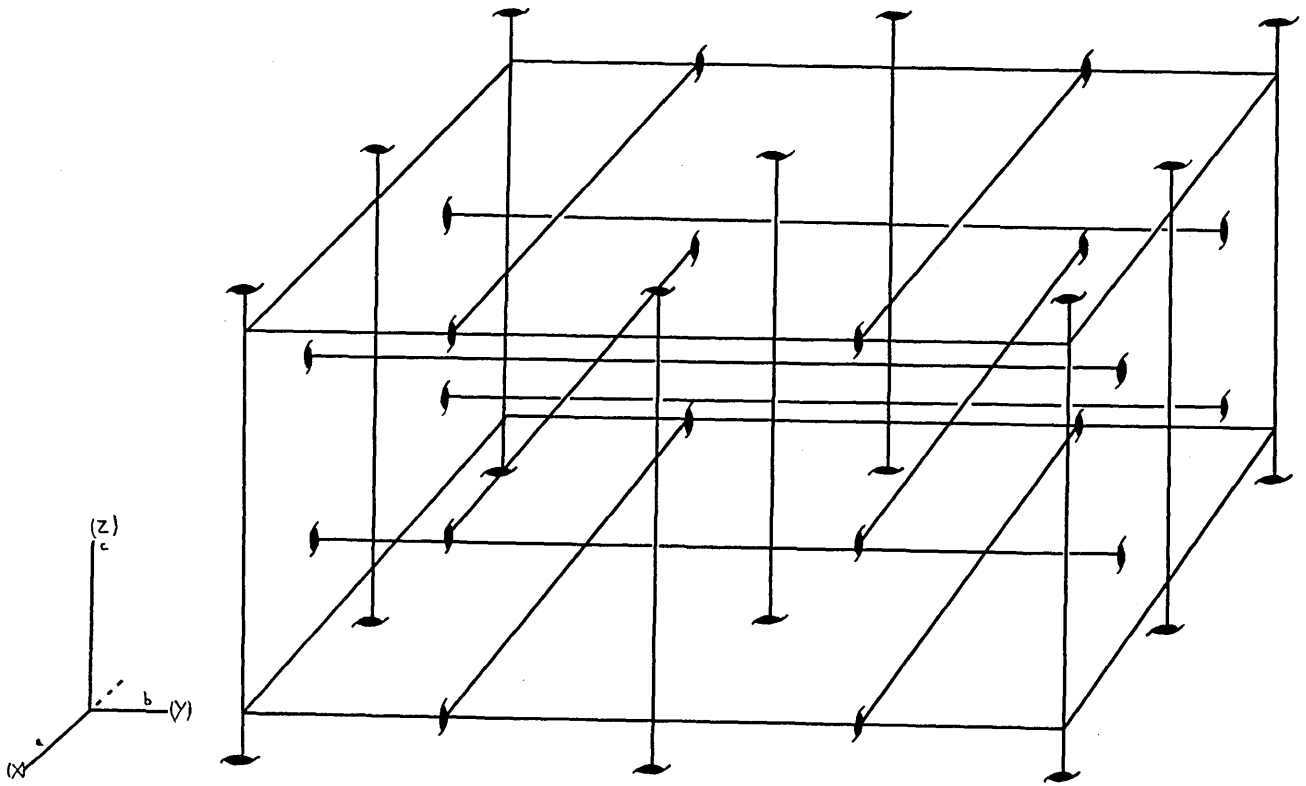
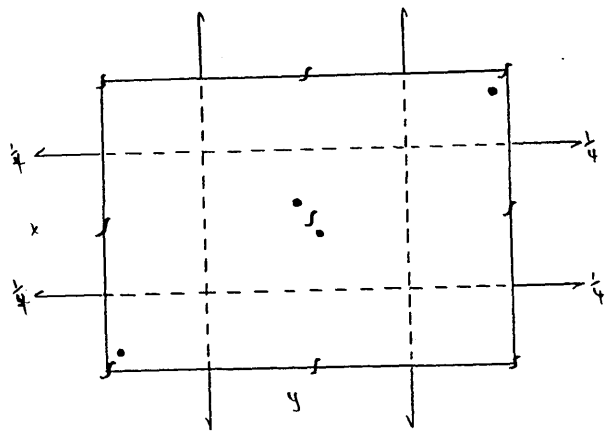
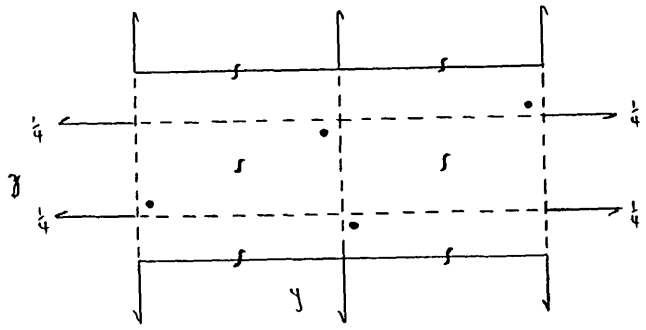


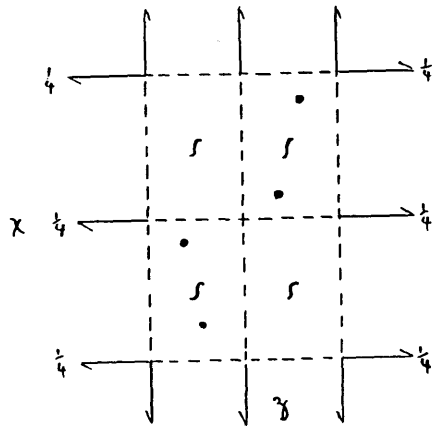
Figure 2. Perspective view of space group $P_{2_1 2_1 2_1}$. The symmetry elements consist only of horizontal and vertical 2-fold screw axes.



$x, y;\ \frac{1}{2}+x,\ \frac{1}{2}-y;\ \frac{1}{2}-x,\ \frac{1}{2}+y\ ;\ \bar{x}, \bar{y}.$



$y, z;\ \frac{1}{2}-y, \bar{z}\ ;\ \frac{1}{2}+y, \frac{1}{2}-z;\quad \bar{y}, \frac{1}{2}+z.$



$x, z;\ \frac{1}{2}-x, \frac{1}{2}-z;\ \bar{x}, \frac{1}{2}+z\ ;\ \frac{1}{2}+x, \bar{z}.$

Equi-points. $x, y, z;\ \frac{1}{2}+x, \frac{1}{2}-y, \bar{z};\ \frac{1}{2}-x, \frac{1}{2}+y;\ \frac{1}{2}-z;\ \bar{x}, \bar{y}, \frac{1}{2}+z.$

Figure 3

However, for this work the origin was taken on a two fold screw axis because for the prism zone reflections the B terms of the structure factor vanish. In general simplification of the structure results if the origin of the coordinates to which the positions of the atoms are referred is taken as a symmetry center of the space group. With this origin the equi-points are

$$x,y,z;\ \tfrac{1}{2}+x,\ \tfrac{1}{2}-y,\ \bar{z};\ \tfrac{1}{2}-x,\ \tfrac{1}{2}+y,\ \tfrac{1}{2}-z;\ \bar{x},\ \bar{y},\ \tfrac{1}{2}+z.$$

The space group symmetry elements are shown in Figure 2. Figure 3 indicates the derivation of the equi-points of the general position as used in this work.

Determination of Parameters

First Patterson Projection (xyo).

Initially F^2 values from a De Jong photograph taken by M.J. Buerger were used to obtain a Patterson projection (xyo). The F^2 values were obtained directly as the intensities which are usually obtained experimentally were corrected for by means of a mechanical cam. The Patterson and Tunell[7] method was used for all the Patterson projections for this crystal. The $|F|^2$ values for the various hko reflections used in this summation are given in Table I.

---

(7) E. L. Patterson and G. Tunell. A Method For The Summation of The Fourier Series Used in The X-ray Analysis of Crystal Structure. Amer. Mineral. 27 (1942) 655-679.

Table I.

De Jong $|F|^2$ and F values.

Reflection	Observed F^2	10 F^2 = F^2 used in series	F signs based on Br.
020	.2	2	I4
040	I.2	I2	34
060	.2	2	I4
080	4.0	40	-64
0I00	I.I	II	-34
0I20	8.4	84	-92
0I40	5.4	54	-74
II0	7.6	76	87
I20	o.o	0	0
I30	.5	5	22
I40	I.2	I2	-35
I50	5.I	5I	7I
I60	6.6	66	-8I
I70	0.0	0	0
I80	8.5	85	-92
I90	I.2	I2	-35
II00	I0.6	I06	-I03
III0	2.0	20	-32
II20	7.8	78	-88
II30	.5	5	-22
II40	0.0	0	0
II50	4.I	4I	-64
200	.4	4	-20
2I0	I.2	I2	-35
220	5.7	57	-75
230	5.7	57	-75
240	I.I	II	-33
250	I.8	I8	-42
260	I.8	I8	-42
270	I5.8	I58	-I26
280	2.9	29	54
290	7.5	75	-87
2I00	8.5	85	92
2II0	I.6	I6	-28
2I20	.I	I	I0
2I30	3.9	39	-62
2I40	5.3	53	73
2I50	I.I	II	33
3I0	II.7	II7	-I08
320	.4	4	20
330	5.3	53	-73
340	I.3	I3	36
350	3.2	32	-57

Table I (cont.-)

De Jong $|F|^2$ and F values.

Reflection	Observed F^2	$\|G\|F^2$ = F^2 useful series	F signs based on Br
360	.8	8	28
370	.9	9	-30
380	.4	4	20
390	.3	3	I7
3I00	4.2	42	65
3II0	7.I	7I	84
3I20	.3	3	I7
3I30	28.0	280	I67
400	.2	2	-I4
4I0	3.9	39	62
420	.5	5	-22
430	.3	3	I7
440	.4	4	-20
450	8.0	80	89
460	.9	9	-30
470	I3.0	I30	I44
480	.4	4	20
490	9.7	97	98
4I00	0	0	0
4II0	I3.6	I36	II7
4I20	.8	8	28
5I0	5.7	57	75
520	.7	7	~~28~~ 17
530	II.8	II8	~~84~~ 109
540	2.9	29	~~I09~~ 54
550	.4	4	~~54~~ 20
560	9.9	99	99
570	0	0	0
580	5.0	50	7I
590	I.I	II	-33
5I00	2.2	22	47
5II0	4.9	49	-70
600	5.4	54	74
6I0	.4	4	-20
620	6.0	60	77
630	0	0	0
640	4.5	45	67
650	0	0	0
660	.8	8	28
670	5.I	5I	-7I

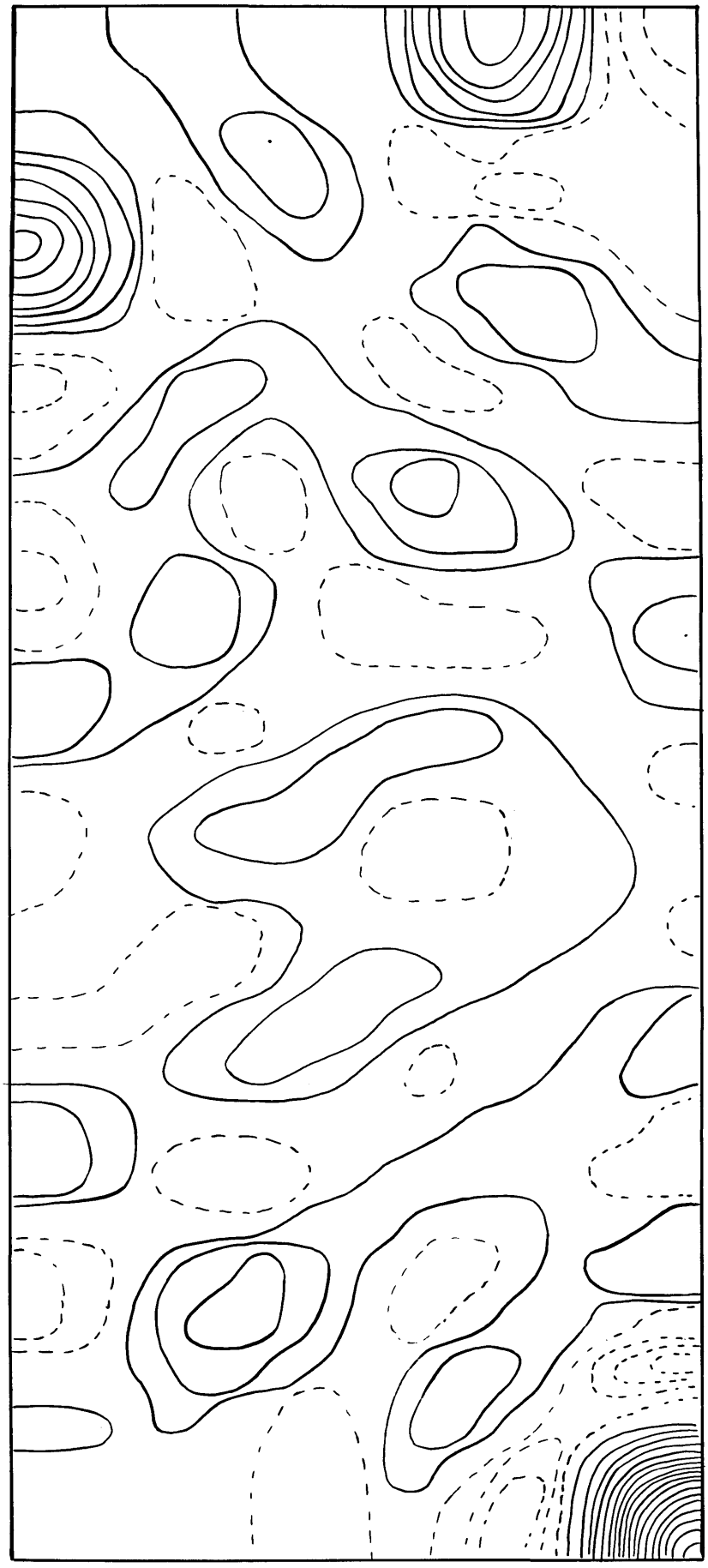
The prism zones of this space group project as the plane group C''_{2v} which is shown in figure 3. Pertinent data for the interpretation of the Patterson projection of this plane group follows.[8]

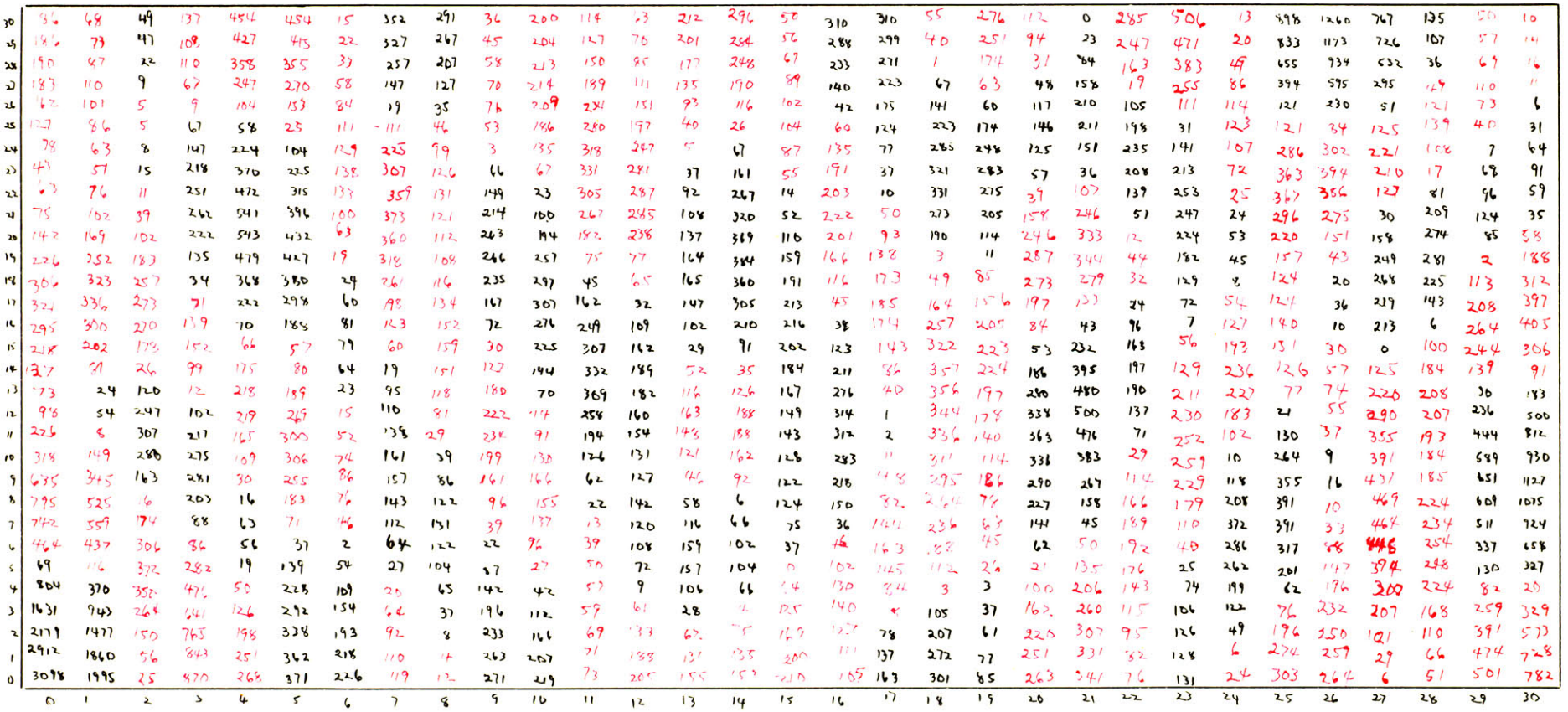
Group	No. Equi-pts	Coordinates of equi-points	Fourier Series Data	*F^2 Series Data		
				Group	Wt.	Rep. Pts.
C''_{2v}	2	(a) 0 0; $\frac{1}{2}$ $\frac{1}{2}$ (b) $\frac{1}{2}$ 0; 0 $\frac{1}{2}$	$(hk) = (-1)^{h+k}(\bar{h}k)$ $= (\bar{h}\bar{k}) = (-1)^{h+k}(h\bar{k})$ $h0 = 0$, h odd $0k = 0$, k odd	C'_{2v}	$2Z_r^2$	$2x_r + \frac{1}{2}, \frac{1}{2}$; $\frac{1}{2}, 2y_r + \frac{1}{2}$.
					Z_r^2	$2x_r, 2y_r$.
	4	(c) $x, y; \frac{1}{2}-x, y+\frac{1}{2}$; $\bar{x}, \bar{y}$; $\frac{1}{2}+x, \frac{1}{2}-y$.			$2Z_rZ_s$	$x_r - x_s, y_r - y_s$; $x_r - x_s + \frac{1}{2}$, $y_r + y_s + \frac{1}{2}$; $x_r + x_s, y_r + y_s$; $x_r + x_s + \frac{1}{2}$, $y_r - y_s + \frac{1}{2}$.

The Patterson projection depends on F^2 and not on F. Thus it is independent of the phase constants. The distance from the origin to a peak corresponds to an interatomic distance in the crystals. The Patterson projection (xyo) of one fourth of the cell (the motif) of diglycine hydrobromide obtained is given in Figure 4. In view of the high atomic weight of bromine in the molecule undoubtedly the intense peaks 1127, 1260 and 543 represent the Br-Br interatomic distances. These points occur at the points $2x\ \frac{1}{2}$, $\frac{1}{2}$; $\frac{1}{2}$, $2y\ \frac{1}{2}$; and 2x, 2y, respectively. With information from the above table the x and y parameters of the heavy bromine atom were determined as shown in the following table
table.

---

(8) A. L. Patterson. Tabulated Data For The Seventeen Plane Groups. Zeits. Krist. vol. 90 (1935) 517-542.




X
Y
Figure 4
1" = 1Å

Peak	$2x+\frac{1}{2}$	$2y+\frac{1}{2}$	$2x$	$2y$	x	y
543			$\frac{20.5}{60}$	$\frac{4.35}{60}$	.171	.036
II27	$\frac{51.35}{60}$				.178	
I260		$\frac{34.1}{60}$				.0342

Preliminary Electron Density $\rho(xyo)$

In view of the high atomic scattering power (f) of bromine compared with that of the other atoms oxygen, nitrogen and carbon in the molecule it was assumed that the heavy bromine determined the phases. Since in projection the crystal is centrosymmetrical the phases will be either o or π i.e. + or −.

For the space group $P_{2_1 2_1 2_1}$, D_2^4, choosing the origin at a symmetry center the components of the structure factor for an atom in the general position are as follows:

$$A = 4\{\cos 2\pi(hx+ky+lz) + \cos 2\pi(h[\tfrac{1}{2}+x]+k[\tfrac{1}{2}-y]-lz) + \cos 2\pi(h[\tfrac{1}{2}-x]+k[\tfrac{1}{2}+y]+l[\tfrac{1}{2}-z]) + \cos 2\pi(-hx-ky+l[\tfrac{1}{2}+z])\}.$$

$$B = 4\{\sin 2\pi(hx+ky+lz) + \sin 2\pi(h[\tfrac{1}{2}+x]+k[\tfrac{1}{2}-y]-lz) + \sin 2\pi(h[\tfrac{1}{2}-x]+k[\tfrac{1}{2}+y]+l[\tfrac{1}{2}-z]) + \sin 2\pi(-hx-ky+l[\tfrac{1}{2}+z])\}.$$

For the various reflections these may be reduced to:

Refl.	A	A	B
	$h+k=2n$	$h+k=2n+1$	
$h\bar{k}o$	$4\cos 2\pi hx\cos 2\pi ky$	$-4\sin 2\pi hx\sin 2\pi ky$	0
$\bar{h}ko$	$4\cos 2\pi kx\cos 2\pi ky$	$4\sin 2\pi hx\sin 2\pi ky$	0
	$k+l=2n$	$k+l=2n+1$	
$ok\bar{l}$	$4\cos 2\pi ky\cos 2\pi lz$	$-4\sin 2\pi ky\sin 2\pi lz$	0
$o\bar{k}l$	$4\cos 2\pi ky\cos 2\pi lz$	$4\sin 2\pi ky\sin 2\pi lz$	0
	$h+l=2n$	$h+l=2n+1$	
$h\bar{o}l$	$4\cos 2\pi hx\cos 2\pi lz$	$-4\sin 2\pi hx\sin 2\pi lz$	0
$\bar{h}ol$	$4\cos 2\pi hx\cos 2\pi lz$	$4\sin 2\pi hx\sin 2\pi lz$	0

Structure Factor Calculations

Equi-points: $x, y;\ \frac{1}{2}+x, \frac{1}{2}-y;\ \frac{1}{2}-x, \frac{1}{2}+y;\ \bar{x}\,\bar{y}$.

hk0

$A = A_1 + A_2 + A_3 + A_4 \qquad A_1 = \cos 2\pi(hx+ky+lz)$

$A = \cos 2\pi(hx+ky) + \cos 2\pi\left[h\left(\tfrac{1}{2}+x\right)+k\left(\tfrac{1}{2}-y\right)\right] + \cos 2\pi\left[h\left(\tfrac{1}{2}-x\right)+k\left(\tfrac{1}{2}+y\right)\right] + \cos 2\pi(-hx-ky).$

$\cos -A = \cos A \qquad \cos A + \cos B = 2\cos\tfrac{1}{2}(A+B)\cos\tfrac{1}{2}(A-B)$

$A = 2\cos 2\pi(hx+ky) + 2\cos 2\pi\tfrac{1}{2}\left(\tfrac{h}{2}+hx+\tfrac{k}{2}-ky+\tfrac{h}{2}-hx+\tfrac{k}{2}+ky\right]\cos 2\pi\tfrac{1}{2}\left[\tfrac{h}{2}+hx+\tfrac{k}{2}-ky-\tfrac{h}{2}+hx-\tfrac{k}{2}-ky\right].$

$A = 2\cos 2\pi(hx+ky) + 2\cos 2\pi\left(\tfrac{h}{2}+\tfrac{k}{2}\right)\cos 2\pi(hx-ky).$

if $h+k = 2n \qquad \cos\pi(h+k) = +1$

$A = 2\cos 2\pi(hx+ky) + \cos 2\pi(hx-ky)$

$A = 4\cos 2\pi\tfrac{1}{2}(hx+ky+hx-ky)\cos 2\pi\tfrac{1}{2}(hx+ky-hx+ky).$

$A = 4\cos 2\pi hx \cos 2\pi ky.$

if $h+k = 2n+1 \qquad \cos\pi(h+k) = -1$

$A = 2\cos 2\pi(hx+ky) - 2\cos 2\pi(hx-ky)$

$\cos A - \cos B = -2\sin\tfrac{1}{2}(A+B)\sin\tfrac{1}{2}(A-B)$

$A = -4\sin 2\pi\tfrac{1}{2}(hx+ky+hx-ky)\sin 2\pi\tfrac{1}{2}(hx+ky-hx+ky)$

$A = -4\sin 2\pi hx \sin 2\pi ky$

$B = B_1 + B_2 + B_3 + B_4 \qquad B_1 = \sin 2\pi(hx+ky+lz)$

$B = \sin 2\pi(hx+ky) + \sin 2\pi\left[h\left(\tfrac{1}{2}+x\right)+k\left(\tfrac{1}{2}-y\right)\right] + \sin 2\pi\left[h\left(\tfrac{1}{2}-x\right)+k\left(\tfrac{1}{2}+y\right)\right] + \sin 2\pi(-hx-ky).$

$\sin -A = -\sin A \quad \therefore \sin 2\pi(-hx-ky) = -\sin 2\pi(hx+ky)$

$\sin x + \sin y = 2\sin\tfrac{1}{2}(x+y)\cos\tfrac{1}{2}(x-y)$

$B = 2\sin 2\pi\tfrac{1}{2}\left[h\left(\tfrac{1}{2}+x\right)+k\left(\tfrac{1}{2}-y\right)+h\left(\tfrac{1}{2}-x\right)+k\left(\tfrac{1}{2}+y\right)\right]\cos 2\pi\tfrac{1}{2}\left[h\left(\tfrac{1}{2}+x\right)+k\left(\tfrac{1}{2}-y\right)-h\left(\tfrac{1}{2}-x\right)-k\left(\tfrac{1}{2}+y\right)\right].$

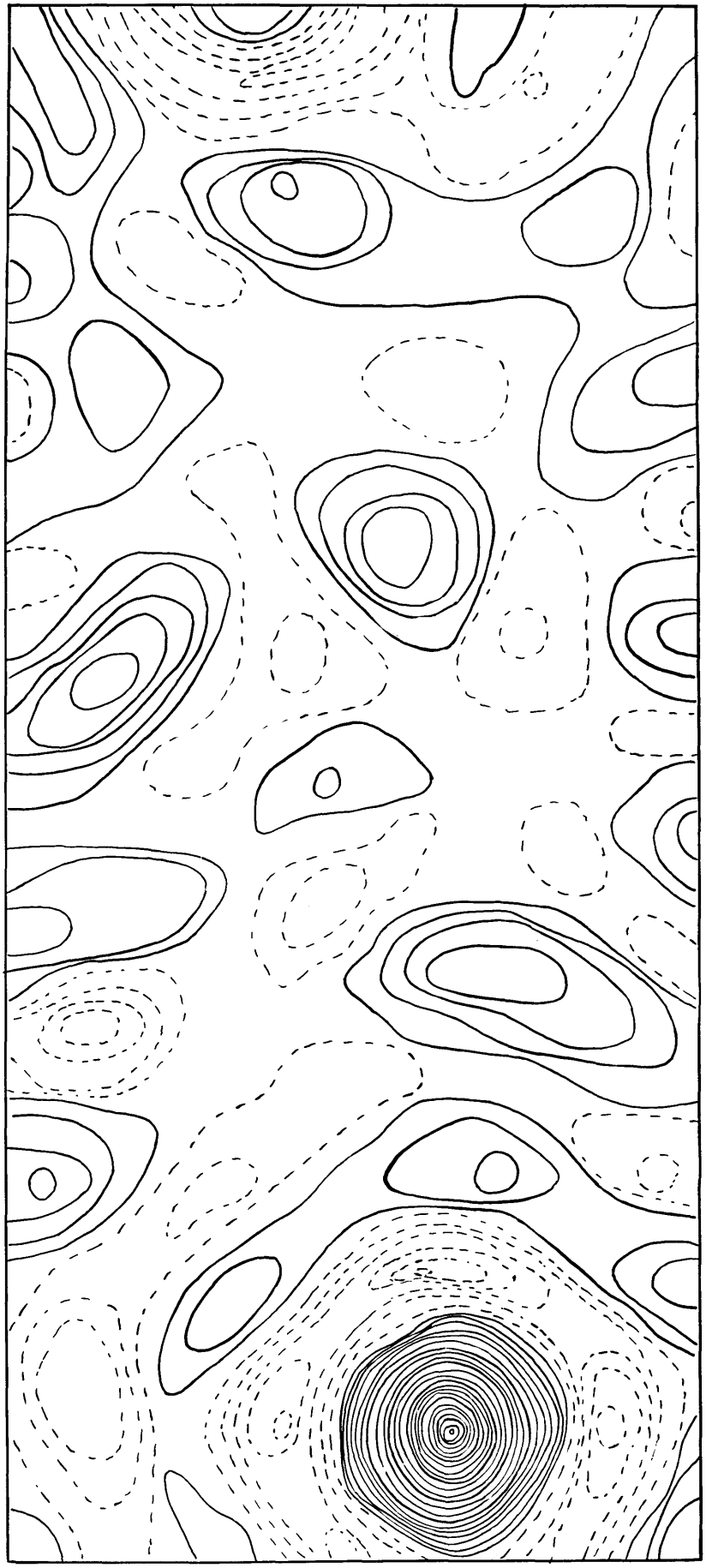
$B = 2\sin 2\pi\tfrac{1}{2}[h+k]\cos 2\pi\tfrac{1}{2}[2hx-2ky].$

if $h+k = 2n \qquad \sin\pi(h+k) = 0$

$B = 0$

if $h+k = 2n+1 \qquad \sin\pi(h+k) = 0$

$B = 0$



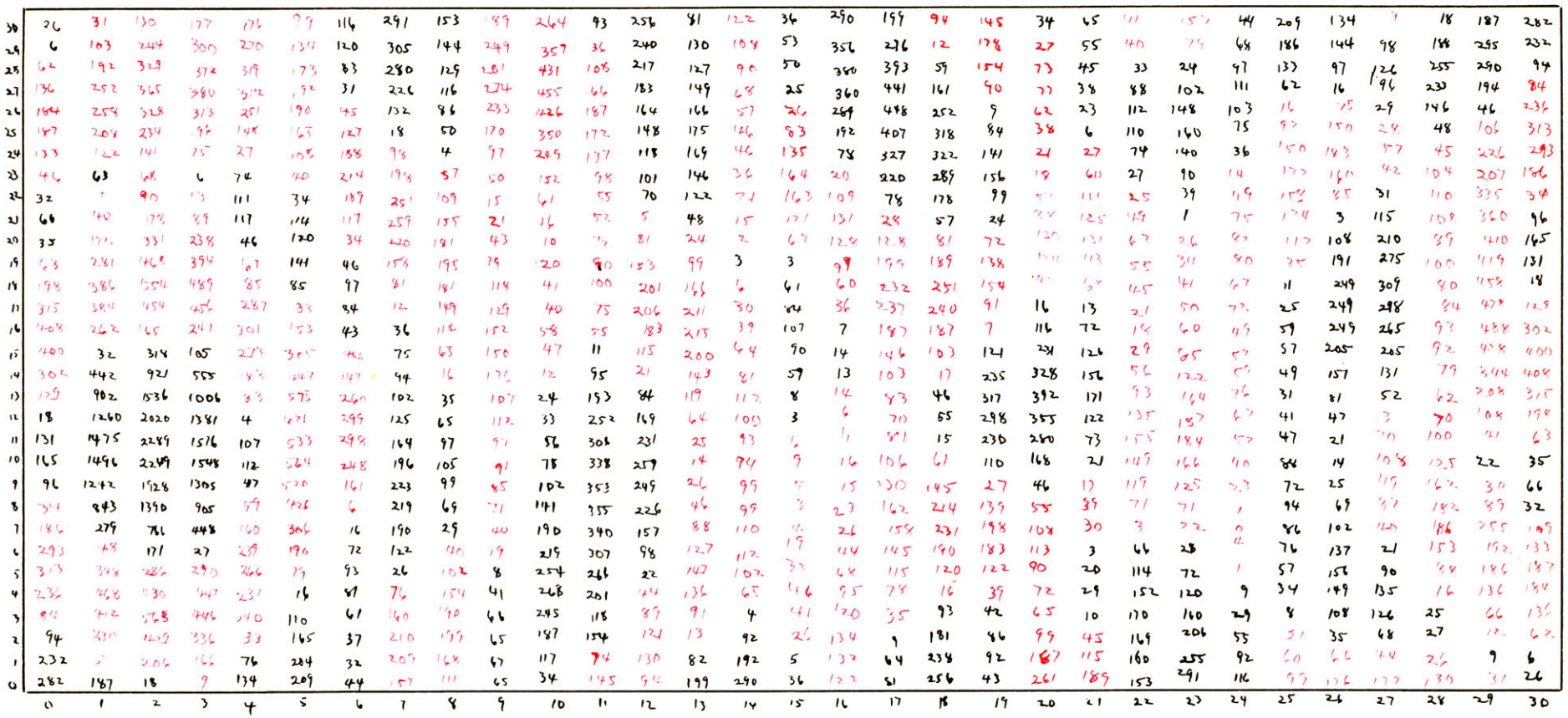


Figure 5. Electron Density ρ(xyo)

A sample calculation of the determination of the phases is given below.

Reflection	Parameters x	y	Structure Factor /m cos2πhx	cos2πky	Phase
220	.178	.034	-.618	.910	--
130	.178	.034	.437	.802	+

With the phase constants determined in the above manner and using the F values of Table I as obtained from the De Jong photographs an electron density ρ (xyo) was made. The electron density map ρ (xyo) of $\frac{1}{8}$ of the cell, the motif, is given in Figure 5. With 4 molecules per cell the position of 4 carbons, I bromine, 2 nitrogens and 4 oxygens (i.e. II positions) should appear in the electron density map of $\frac{1}{4}$ of the cell. In the electron density map obtained the bromine appeared where it was put in the structure. However, many more than I0 other peaks are seen to occur indicating quite some error.

Second Approximation-

Due to the error just indicated it was necessary to obtain more x-ray data. This was done by means of the Weissenberg method for the hko and hol reflections using CuKα radiation and the precession method for okl reflections using MoKα radiation. The intensities were determined by the Dawton method and corrections to obtain $|F|^2$ are given below. Since intensities are obtained experimentally and F's or their squares are used as coefficients of a Fourier series for electron density maps and Patterson projections it is necessary to compute F's. As $\frac{1}{Lp}$ is a function of sin θ

$(\frac{1}{Lp} = \sin 2\theta \frac{2}{1+\cos^2 2\theta})$, sin must be calculated for all reflections. This was done by means of the formula

$$\sin \theta_{hkl} = \frac{1}{2} \sqrt{a^{*2}h^2 + b^{*2}k^2 + c^{*2}l^2}$$

$a^* = \frac{\lambda}{a} = \frac{1.539}{8.21} = .18745$

$b^* = \frac{\lambda}{b} = \frac{1.539}{18.42} = .08355$

$c^* = \frac{\lambda}{c} = \frac{1.539}{5.40} = .2850$

$I \sim LpF^2$

Reflection hkO	$\sin\theta$	$1/Lp$ (9)	I Average	$F^2 = I(\frac{1}{Lp})$	$4F^2$ = F^2 used in series	$20F$ = Fused in series signs based on Br
020	.0835	.1688	14.95	2.52	10	32
040	.1671	.3482	15.6	5.43	22	47
060	.2506	.5500	.55	.30	1	11
080	.3342	.7861	5.83	4.58	18	−43
0100	.4177	1.0661	.33	.35	1	−12
0120	.5013	1.3910	5.63	7.83	31	−56
0140	.5848	1.7252	.93	1.60	6	−25
0160	.6684	1.9664	.85	1.67	7	−26
0180	.7519	1.9495	.10	.19	1	−19
0200	.8355	1.5873	.28	.44	2	−13
0220	.9190	.9823	.10	.098	0	6
110	.1026	.2085	4.21	.88	4	19
130	.1565	.3246	5.75	1.87	7	27
140	.1915	.4046	9.43	3.82	15	−39
150	.2290	.4950	29.59	14.65	59	77
160	.2675	.5945	25.73	15.30	61	−78
170	.3071	.7050	.21	.15	1	− 8
180	.3470	.8258	14.35	11.85	47	−69
190	.3875	.9593	.90	.86	3	−19
1100	.4280	1.1041	6.58	7.26	29	−54
1110	.4690	1.2612	.49	.62	2	−16
1120	.5100	1.4265	1.71	2.44	10	−31
1130	.5500	1.5893	.05	.08	0	− 6
1150	.6325	1.8843	.38	.72	3	−17
1160	.6799	1.9832	.11	.22	1	9
1170	.7163	1.9979	.13	.26	1	−10
1180	.7576	1.9356	.51	.99	4	20
1190	.7987	1.7865	.08	.14	1	− 7
1200	.8407	1.5552	.81	1.26	5	22
1220	.9238	0.9432	.32	.30	1	11

(9) M.J. Buerger and G.E. Klein. Correction of X-ray Diffraction Intensities for Lorentz and Polarization Factors. J. App. Phys. Vol. 16 No. 7 July 1945 408-418.

Reflection hKO	$\sin\theta$	$\frac{1}{Lp}$	I Average	$F^2 = I(\frac{1}{Lp})$	$4F^2$	20 F
200	.1874	.3950	1.33	.53	2	-15
210	.1920	.4057	6.19	2.51	10	-32
220	.2050	.4365	44.12	19.26	77	-88
230	.2255	.4864	26.13	12.71	51	-71
240	.2510	.5510	2.20	1.21	5	-22
250	.2805	.6299	2.67	1.68	7	-26
260	.3130	.7220	1.62	1.17	5	-22
270	.3474	.8268	20.91	17.29	69	-83
280	.3838	.9443	1.60	1.51	6	25
290	.420	1.0746	3.37	3.62	14	-38
2100	.4579	1.2178	2.94	3.58	14	38
2110	.4964	1.3711	.24	.33	1	-11
2130	.5745	1.6862	.41	.69	3	-17
2140	.614	1.8282	.47	.86	3	19
2150	.6540	1.9387	.11	.21	1	9
2160	.6942	1.9962	.14	.28	1	11
2180	.7799	1.8653	.29	.54	2	15
2190	.8155	1.7025	.22	.37	1	12
2210	.8981	1.1559	.44	.51	2	14
2230	.9789	.4348	.92	.40	2	13
310	.2840	.6394	37.86	24.21	97	-98
320	.2933	.6654	.36	.24	1	10
330	.3078	.7071	10.00	7.07	28	-53
340	.3270	.7639	1.21	.92	4	19
350	.3503	.8362	2.65	2.22	9	-30
360	.3766	.9220	.29	.27	1	10
370	.4057	1.0230	.30	.31	1	-11
3100	.5037	1.4007	.76	1.06	4	21
3110	.5386	1.5433	1.10	1.70	7	26
3130	.6115	1.8199	3.00	5.46	22	47
3150	.6868	1.9906	.43	.86	3	19
3170	.7638	1.9189	.73	1.40	6	24
400	.3749	.9163	.25	.23	1	-10
410	.3772	.9241	5.23	4.83	19	44
420	.3840	.9470	.36	.34	1	-12
430	.3953	.9864	.16	.16	1	8
440	.4105	1.0402	.05	.26	1	-10
450	.4292	1.1085	4.40	4.88	19	44
460	.4509	1.1906	.14	.17	1	- 8
470	.4755	1.2870	4.66	6.00	24	49
480	.5015	1.3918	.06	.08	0	6
490	.5305	1.5103	1.85	2.79	11	33
4110	.5930	1.7556	1.59	2.79	11	33
4120	.6260	1.8657	.18	.34	1	12
4170	.8030	1.7662	.36	.64	3	-16
4180	.8402	1.5583	.10	.16	1	8
4190	.8778	1.3011	.48	.64	3	-16
4210	.9540	.6840	.58	.40	2	-13

Reflection hkO	sin θ / λ	y(θ) LP	I Average	F^2	$4F^2$	20F
5I0	.4705	I.2672	4.07	5.I2	20	45
520	.4760	I.2890	.I4	.I8	I	8
530	.4852	I.3258	4.87	6.46	26	5I
540	.4975	I.3755	.7I	.98	4	20
550	.5I30	I.4386	0	0	0	0
560	.53I5	I.5I44	I.88	2.85	II	34
580	.5755	I.6900	.64	I.08	4	2I
590	.6008	I.7809	.I9	.34	I	-I2
5I00	.6277	I.8707	.28	.52	2	I4
5II0	.6560	I.9429	.49	.95	4	-I9
5I30	.7I73	I.9975	.24	.48	2	-I4
5I50	.7852	I.8449	.I0	.I8	I	- 8
5I90	.92I7	.9604	.39	.37	I	-I2
5200	.9579	.648I	.75	.49	2	-I4
600	.5624	I.639I	I.22	2.00	8	28
6I0	.5639	I.6449	.08	.I3	I	- 7
620	.5685	I.6590	I.20	I.99	8	28
650	.5867	I.7323	.64	I.II	4	2I
660	.6I56	I.8332	.I0	.I8	I	8
670	.6338	I.8880	.55	I.04	4	-20
690	.6765	I.9786	.I9	.38	2	-I2
6I00	.7006	I.9990	.I8	.36	I	-I2
6I20	.7536	I.9457	.48	.93	4	-I9
6I30	.78I8	I.8582	.05	.09	0	- 6
6I40	..8II4	I.724I	.74	I.28	5	-23
6I60	.8735	I.3323	.45	.60	2	-I5
6I70	.9058	I.0882	.I8	.20	I	9
6I80	.9390	.8I63	.45	.37	I	-I2
6I90	.9728	.50I6	.I3	.07	0	5
720	.66I4	I.9539	.66	I.29	5	-23
740	.6770	I.9794	.64	I.27	5	-23
760v	.7023	I.9996	.79	I.58	5	-25
780	.7364	I.9788	.40	.79	3	-I8
7I00	.7778	I.8730	.53	.99	4	-20
7I20	.8257	I.6457	.2I	.35	I	- I2
7I40	.8789	I.2930	.0I	.0I	0	2
800	.7498	I.9544	.45	.88	4	-I9
820	.7544	I.9439	.26	.5I	2	-I4
840	.7682	I.9053	.I6	.30	I	-II
850	.7780	I.8723	.20	.37	I	-I2
860	.7906	I.8226	.I0	.I8	I	- 8
870	.806I	I.75II	.30	.53	2	-I5
890	.8388	I.5669	.0I	.02	0	- 3
8I00	.8582	I.5070	.I6	.24	I	I0
8I20	.8794	I.2894	.02	.03	0	- 3
8I20	.90I9	I.II87	.23	.26	I	I0
8I40	.9509	0.7I2I	.92	.66	3	I6

Reflection hkO	sin θ / λ	1/Lp	I Average	F^2	$4F^2$	20F
9I0	.8445	I.5307	.50	.76	3	-I8
930	.8528	I.4764	.I3	.I9	I	-9
940	.8599	I.4283	.I5	.2I	I	9
950	.8690	I.3646	.06	.08	0	-6
960	.8800	I.2850	.I2	.I5	I	8
980	.9073	I.0764	.23	.25	I	I0
990	.9235	.9456	.I0	.09	0	6
9II0	.9606	.6227	.20	.I2	I	7
I000	----	~~.8228~~	0	0	0	0
I0I0	.9382	.8228	.05	.04	0	4
I030	.945I	.7630	.24	.I8	I	8
I050	.9602	.6266	.65	.4I	2	I3
I070	.98I8	.4008	.65	.26	I	I0

hol Reflections

Reflection hol	sin θ / λ	(1/Lp) (9)	I Average	F^2	$4F^2$	20F
002	.2850	.6423	28.97	I8.6I	74.44	86
004	.5700	I.6686	.97	I.62	6.48	-25
006	.8550	I.46I8	.83	I.2I	4.84	-22
I0I	.I706	.3564	I3.I5	4.69	I8.76	43
20I	.2355	.5II3	40.67	20.79	83.I6	-9I
30I	.3I52	.7288	2.52	I.84	7.36	27
40I	.40II	I.0067	.3I	.3I	I.24	-II
50I	.4899	I.3448	I.7I	2.30	9.20	-30
70I	.67I4	I.97I2	.90	I.77	7.08	-27
90I	.855I	I.46II	.20	.29	I.I6	-II
I02	.3000	.6845	4.29	2.94	II.76	-34
202	.34II	.8074	I.58	I.28	5.I2	23
302	.4004	I.0042	2.93	2.94	II.76	-34
502	.5485	I.5833	I.54	2.44	9.76	-3I
602	.6305	I.8787	.I0	.I9	.76	-9
802	.8022	I.7700	.I3	.23	.92	-I0
902	.8904	I.207I	.33	.40	I.60	I3
203	.4668	I.2587	6.II	7.65	30.60	-55
403	.5686	I.6634	I.76	2.93	II.72	-34
503	.6343	I.8894	.I0	.I9	.76	-9
803	.863I	I.4062	.025	.035	.I4	4

Reflection hOl	sin θ	1/Lp	I Average	F^2	$4F^2$	$20F$
104	.5776	1.6980	.14	.24	.96	−10
204	.6004	1.7820	.18	.32	1.28	−11
304	.6549	1.9404	1.36	2.64	10.56	−32
404	.6822	1.9859	.09	.18	.72	8
504	.7379	1.9764	.33	.65	2.60	−16
604	.8007	1.7773	0	0	0	0
804	.9680	.5510	00	0	0	0
105	.7186	1.9970	.43	.86	3.44	−19
205	.7367	1.9783	.05	.10	.40	−6
405	.8051	1.7560	.05	.09	.36	−6
505	.8523	1.4797	.04	.06	.24	5
605	.9078	1.0726	.03	.03	.12	−3
705	.9685	.5459	.71	.39	1.56	12
200	.1875	.3952	.53	.21	.84	9
400	.3749	.9163	.15	.14	.56	−7
600	.5624	1.6391	1.05	1.72	6.88	−26
800	.7498	1.9544	.53	1.04	4.16	−20
106	.8601	1.4269	0	0	0	0
206	.8753	1.3193	.28	.37	1.48	−12
306	.9005	1.1296	0	0	0	0

okl Reflections

Reflection okl	sin θ Mo	1/P (4)	1/graphically (10)	I I Average	F^2	$15F^2$	$16F$
020	.0385	1.0030			1.32	2	−18
040	.0770	1.0119	.74	3.36	2.52	4	24
060	.1154	1.0270	1.10	.67	.76	1	−14
080	.1540	1.0485	1.40	1.88	2.76	4	−26
0100	.1924	1.0767	1.72	.88	1.63	2	20
0120	.2309	1.1122	1.97	4.58	10.04	15	−50
0140	.2694	1.1555	2.18	2.07	5.21	8	36
0160	.3079	1.2072	2.31	1.30	3.63	5	−28
0180	.3464	1.2677	2.38	.50	1.51	2	20
0200	.3849	1.3376	2.32	.81	2.51	4	−26
031	.0875	1.0154	.85	12.28	10.55	16	−52
041	.1124	1.0256	.95	17.43	16.98	25	64
051	.1165	1.0275	1.08	2.43	2.70	4	26
061	.1328	1.0360	1.24	27.79	35.70	54	−96
071	.1498	1.0459	1.37	4.32	6.78	10	−47
081	.1674	1.0584	1.51	.97	1.55	2	20

Reflection hkl	sin θ/λ	I/p	I/L (10)	I	F²	σF²	(σF
091	.1852	1.0710	1.65	.55	.97	1	16
0101	.2033	1.0861	1.78	2.43	4.70	7	−35
0136	.2587	1.1427	2.11	.06	.15	.22	6
0186	.3524	1.2780	2.37	.72	2.18	3	24
0191	.3714	1.3125	2.35	.67	2.07	3	23
0201	.3904	1.3483	2.28	.76	2.34	4	−24
0211	.4093	1.3868	2.19	.40	1.22	2	−16
0221	.4284	1.4278	2.06	.70	2.06	3	23
002	.1313	1.0350	1.22	7.18	9.07	14	−48
012	.1327	1.0359	1.22	7.17	9.06	14	−48
022	.1368	1.0381	1.23	2.30	2.94	4	27
032	.1535	1.0420	1.31	12.42	16.95	25	64
092	.2173	1.0989	1.88	2.15	4.42	7	32
0112	.2488	1.1314	2.06	.70	1.63	2	−20
0128	.2656	1.1509	2.16	.80	1.99	3	23
0132	.2824	1.1721	2.22	.48	1.25	2	18
0142	.2997	1.1955	2.29	.61	1.67	3	−21
0152	.3170	1.2207	2.34	1.20	3.43	5	−30
0162	.3346	1.2482	2.37	.22	.65	1	13
0172	.3524	1.2780	2.38	.13	.40	1	10
0192	.3884	1.3444	2.29	.25	.77	1	−14
013	.1979	1.0813	1.73	.62	1.16	2	−17
033	.2052	1.0877	1.79	2.68	5.22	8	37
053	.2192	1.1007	1.89	3.33	6.93	10	−42
073	.2384	1.1201	2.02	3.20	7.24	11	43
093	.2621	1.1468	2.13	2.23	5.45	8	−37
0113	.2891	1.1809	2.25	.13	.35	1	9
0193	.4153	1.3995	2.17	.47	1.43	2	−19
0213	.4494	1.4751	1.83	.53	1.43	2	19
004	.2626	1.1474	2.13	1.43	3.50	5	−39
014	.2631	1.1479	2.13	1.85	4.52	7	34
034	.2688	1.1548	2.17	1.69	4.24	6	−33
064	.2868	1.1778	2.24	.18	.48	1	11
084	.3042	1.2018	2.30	.20	.55	1	12
094	.3144	1.2167	2.33	.03	.09	0	−5
0114	.3372	1.2524	2.36	.19	.56	1	12
0124	.3497	1.2734	2.38	.03	.09	0	5
0134	.3626	1.2959	2.35	.48	1.46	2	−19
0154	.3902	1.3479	2.30	.03	.09	0	5
0174	.4192	1.4079	2.12	.10	.30	0	−9
045	.3370	1.2520	2.37	.57	1.69	3	−21
065	.3478	1.2701	2.38	.45	1.36	2	19
075	.3547	1.2820	2.37	.40	1.22	2	−16
085	.3623	1.2954	2.36	.33	1.01	2	−16
095	.3710	1.3113	2.35	.10	.31	0	9
0105	.3804	1.3290	2.33	.38	1.18	2	17

Reflection (0kℓ)	sin θ (sin θ)	I/p	I/L (10)	I (Average)	F^2	15 F^2	16 F
006	.3939	I.3554	2.26	.95	2.9I	4	28
026	.3958	I.3590	2.24	.20	.6I	I	I2
0462	.40I3	I.3703	2,22	.I3	.40	I	I0
0I06	.4384	I.4504	I.90	.08	.22	0	8
0I26	.5466	I.4927	I.62	.20	.48	I	II
0I46	.4772	I.5406	I.08	.80	.59	I	I2

(10) M.J. Buerger. The Photography of the Reciprocal Lattice. ASXRED Monograph No.I

Patterson Synthesis-

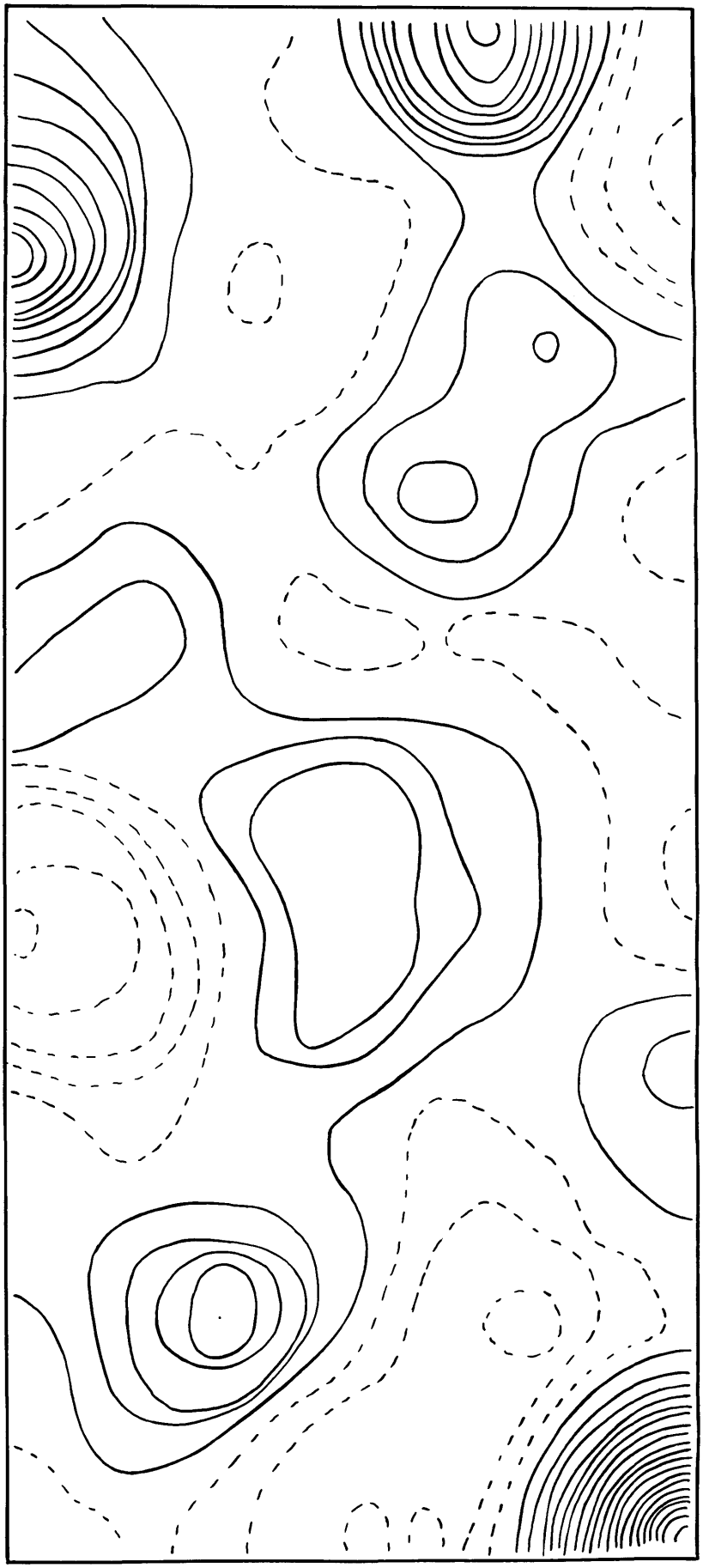
Patterson projections were obtained using the new above $|F|^2$'s. The three projections $\varrho(xyo)$, $\varrho(oyz)$, and $\varrho(xoz)$ for $\frac{1}{4}$ of the cell are given in Figures 6, 7 and8. The Br-Br vectors ~~are~~ of the ~~same~~ (xyo) projection are the same as obtained in the initial Patterson using De Jong values although the rest of the plot is different. The complete Pattersons for all three projections are given in Figure 9. The originsof the three projections are seen displaced as in each case the origin is on the symmetry element, the two fold screw axis, and the relation to one another may be seen from the space group representation of Figure 2.

Second Electron Density-

Using the new F values for hko reflections, a second electron density $\varrho(xyo)$ was prepared. This second electron density resulted in the correct number of atomic position peaks. The first electron density $\varrho(xyo)$ seemed to be much in error due to an insufficient number of terms having been used in the series. In the second electron density $\varrho(xyo)$ 137 terms were used, whereas in the first one only 75 terms were used. See Figure 10 a

Preliminary Comparison of Intensities-

A preliminary check of intensities for hko reflections was made using the atomic positions as indicated in the second electron density $\varrho(xyo)$ and assuming an average value of the atomic scattering factor (f) for the nitrogen, oxygen and carbon as it was impossible to determine which of these positions represented which atoms. A sample calculation of intensity follows.



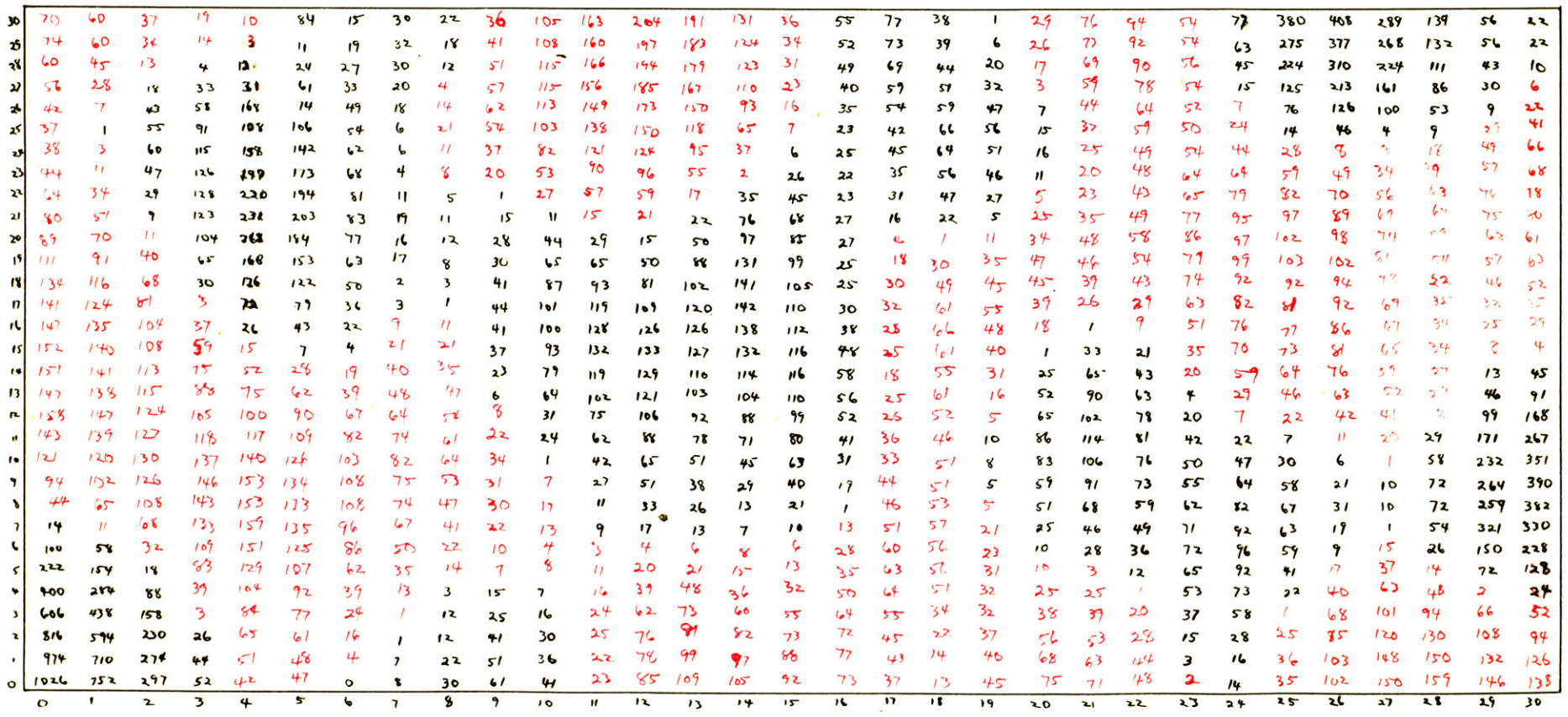
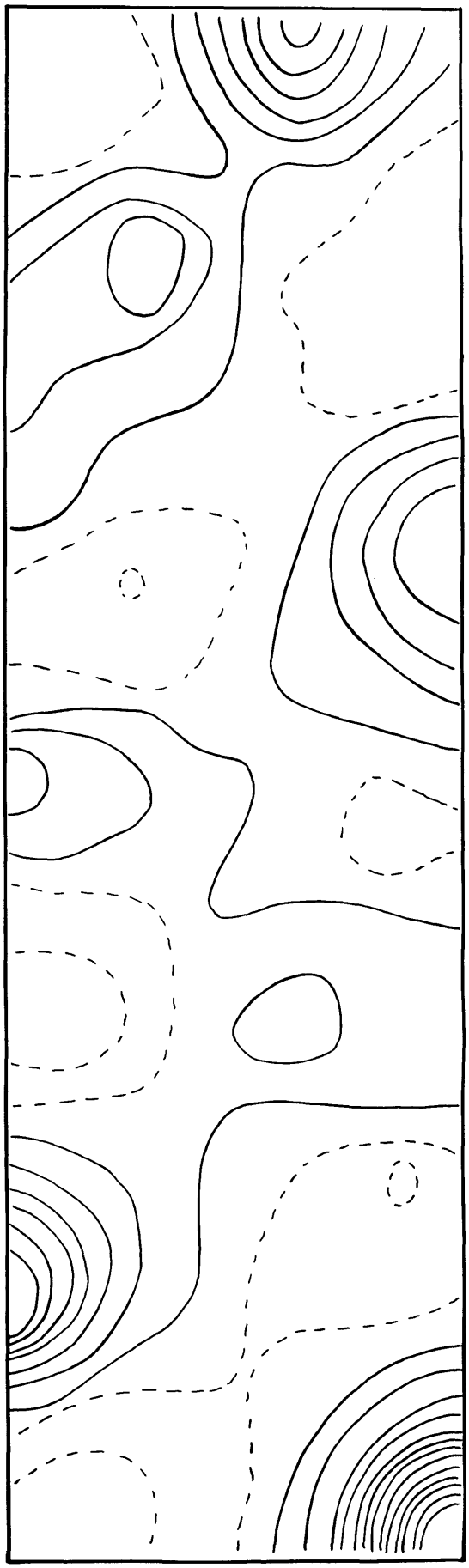
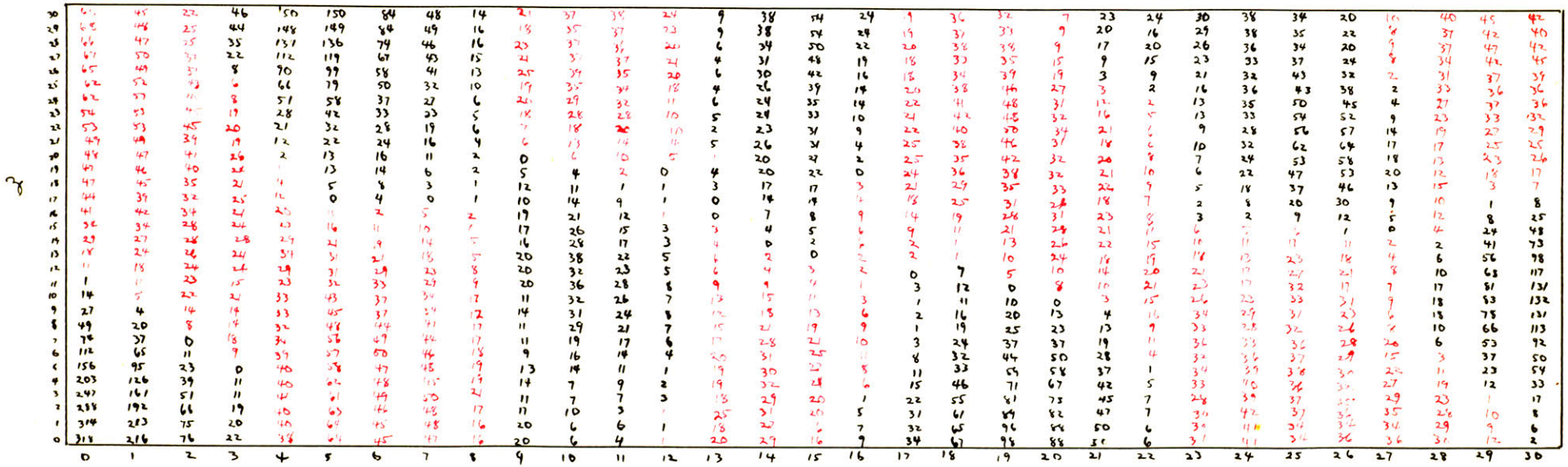


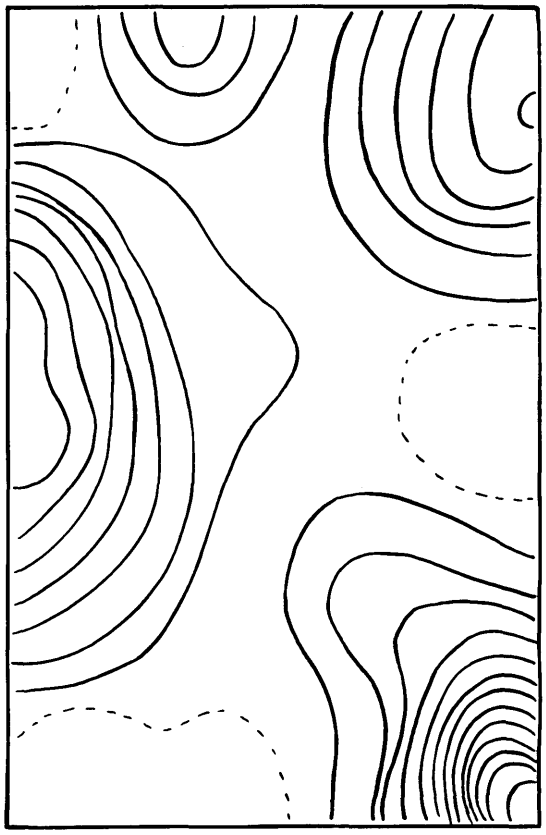
Figure 6. Patterson Projection (xy0)





1" = 1 Å

Figure 7 Patterson Projection (0kl)



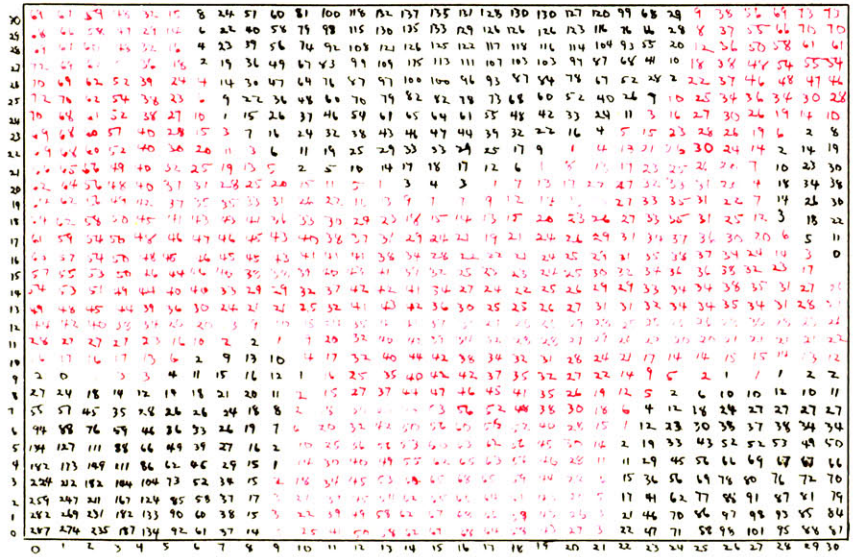


Figure 8. Patterson Projection (xoz)

1" = 1 Å

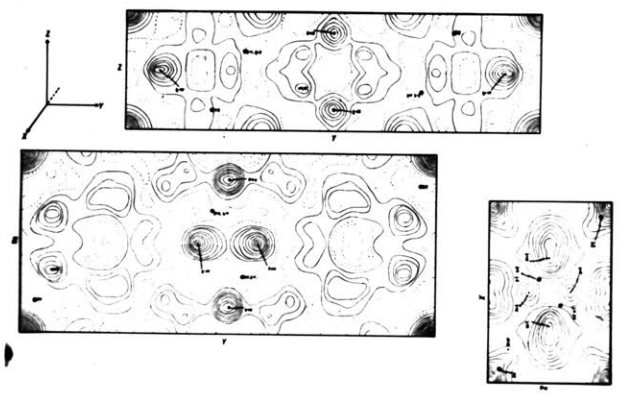


Figure 9. Patterson Projections

Reflection m	atom	Parameters x	y	$\sqrt{Lp}$ [11]	mf [12]	Structure Factor/m $\cos 2\pi hx$ [13]	$\cos 2\pi ky$ [13]	$\sqrt{I}$ +	−
I50									
	Br	.I78	.036	4I.5I		.437	.426	7.75	
	I	.093	.380	7.03		.834	.809	4.74	
	2	.I08	.430	7.03		.779	.588	3.22	
	3	.4I7	.283	7.03		-.867	-.86I	5.25	
	4	.067	.I80	7.03		.9I3	.809	5.I9	
	5	.I50	.I9I	7.03		.588	.960	3.97	
	6	.470	.II3	7.03		-.982	-.9I8	6.34	
	7	.470	.2I3	7.03		-.982	.9I8		6.34
	8	.4I7	.283	7.03		-.867	-.86I	5.25	
	9	.200	.333	7.03		.309	-.509		I.II
	I0	.26I	.430	7.03		-.I07	.588		.44

$\frac{\sqrt{I}}{4}$ =+ 33.80

The atomic scattering factor, f, is determined for each atom for each reflection as a function of $\sin\theta$. The rough comparison of observed and calculated intensities is given in Table 2.

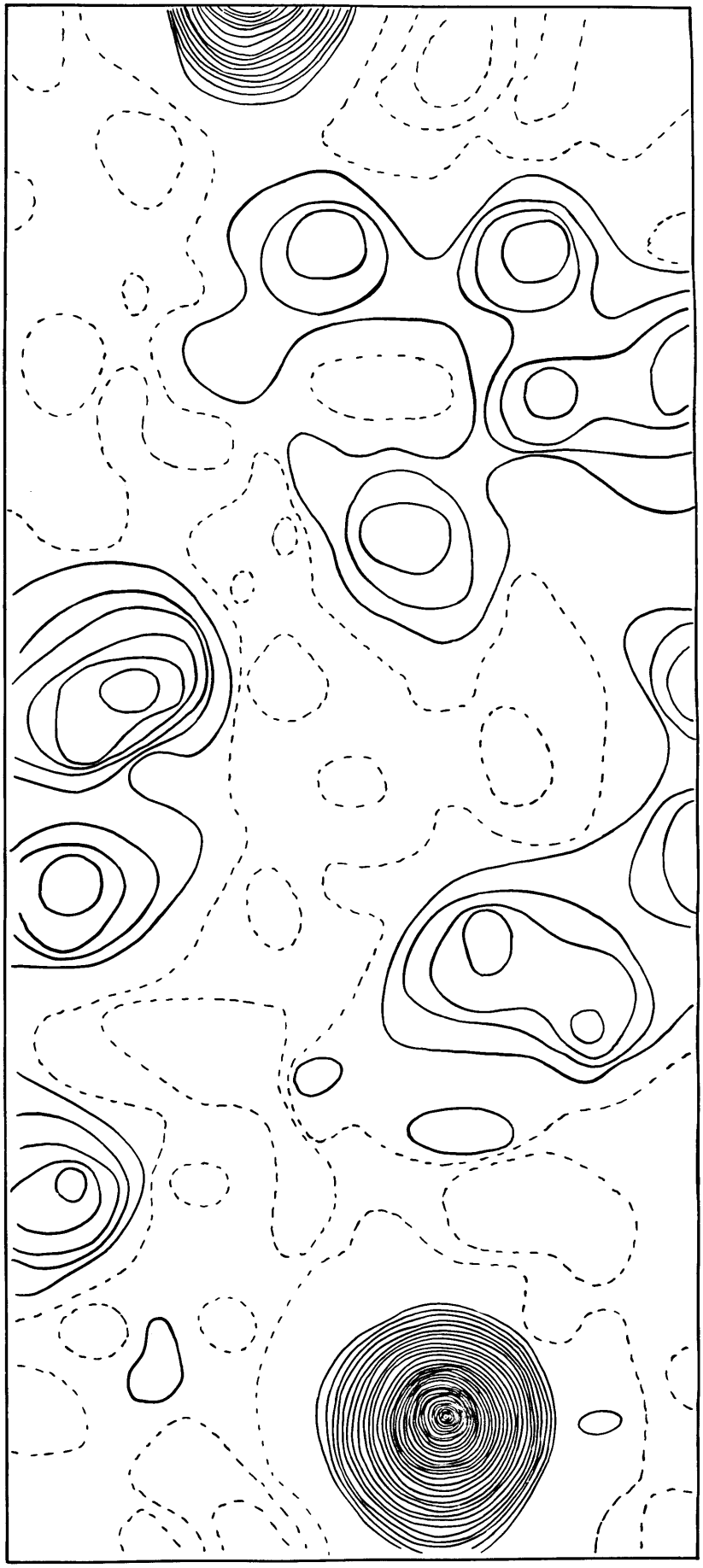
Electron Density maps $\varrho(xoz)$ and $\varrho(oyz)$-

Electron density maps $\varrho(xoz)$ and $\varrho(oyz)$ using the same proceedure as was used for $\varrho(xyo)$ were prepared and are given in Figure I0 and Figure II. The three complete electron density maps all drawn to the same origin are given in Figure I2.

(II) M.J. Buerger and G.E. Klein. Correction of Diffraction Amplitudes for Lorentz and Polarization Factors. J. App. Phys. Vol. I7 No. 4 April I946 285-306.

(I2) Internationale Tabellen zur bestimmung Kristallstrukturen. I. (Gebruder Borntraeger, Berlin (I935).

(I3) M.J. Buerger. Numerical Structure Factor Tables. Geol. Soc. America Special Paper 33.



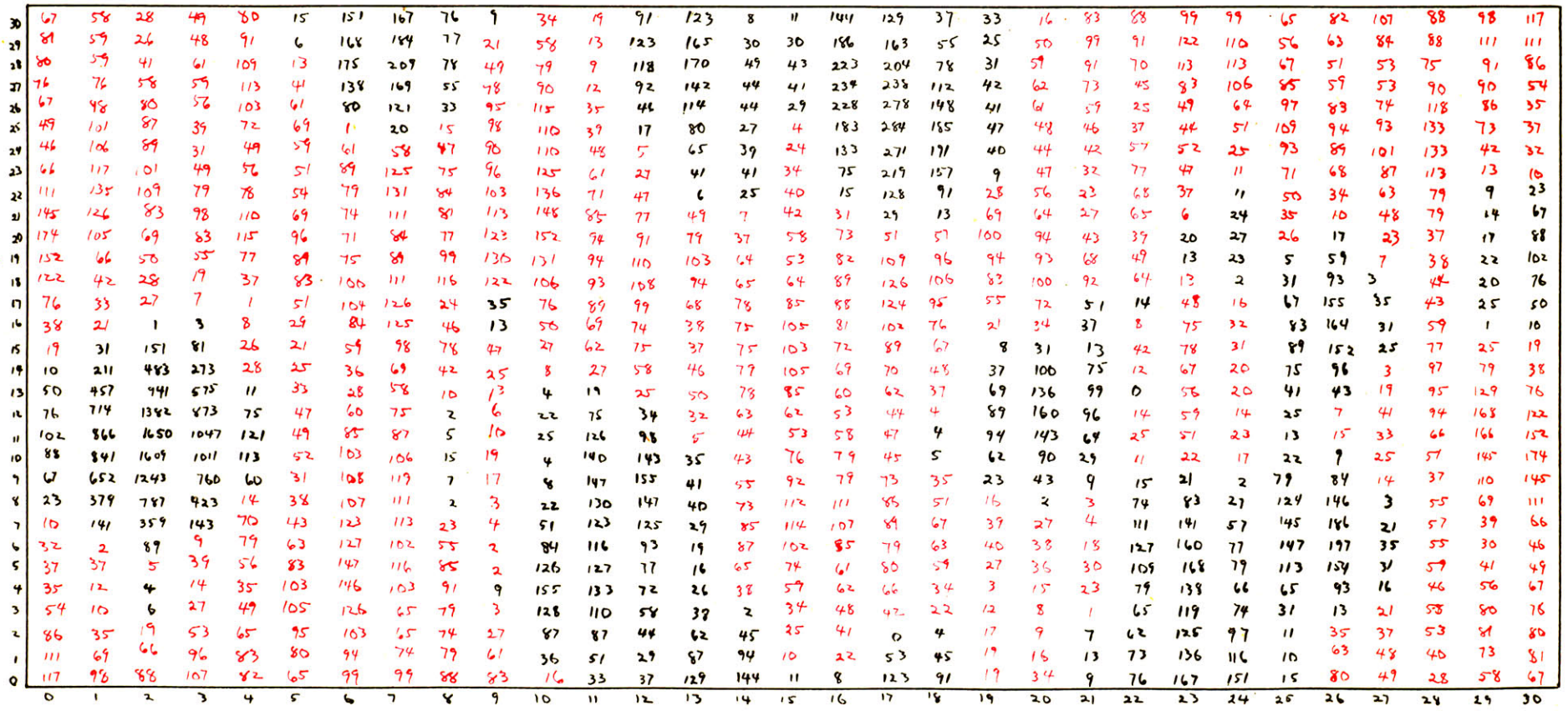
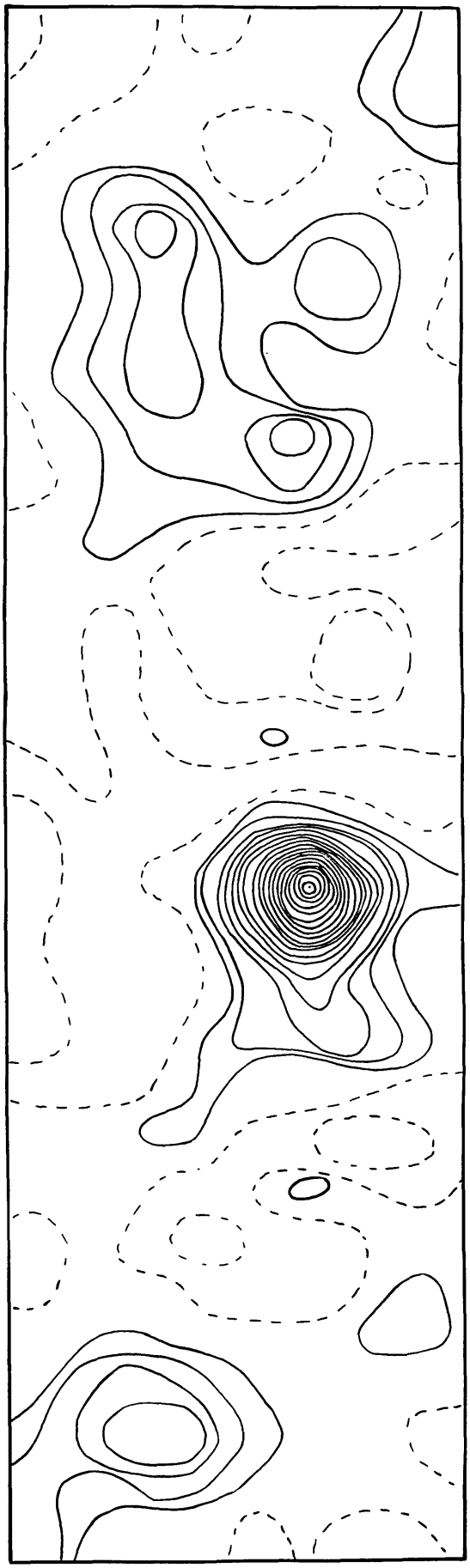


Figure 10a Electron Density ρ(xyo



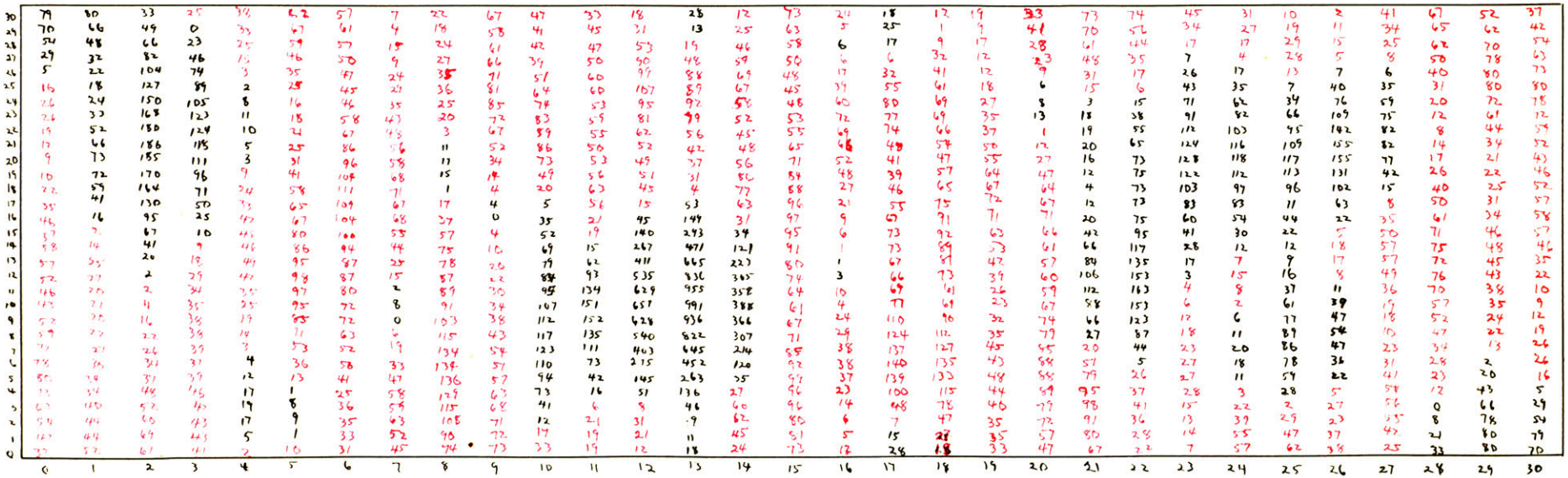
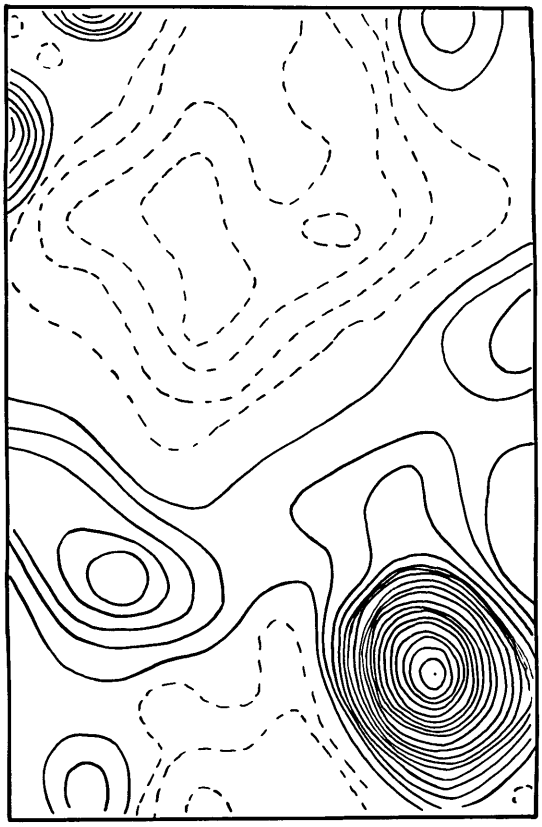


Figure 10 Electron Density $\rho(xyz)$

1" = 1 Å



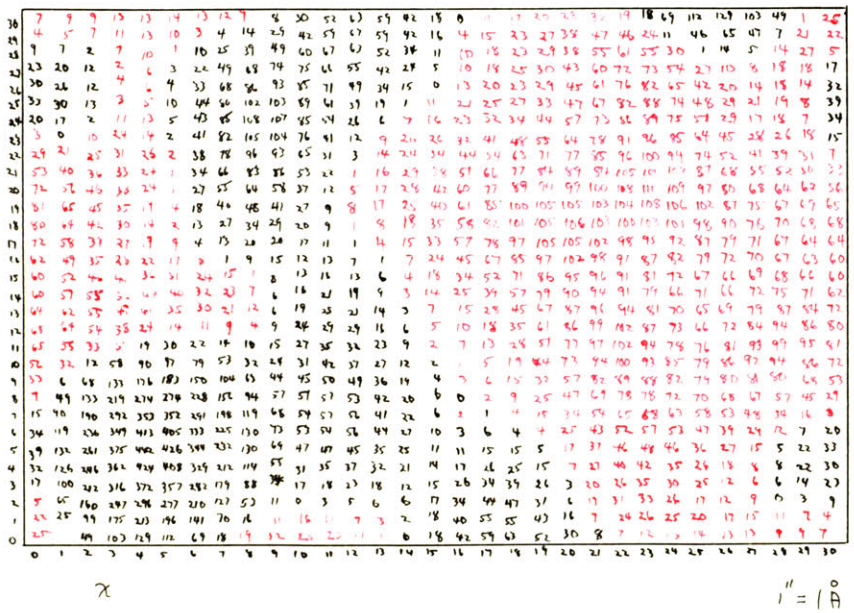


Figure 11 Electron Density $\rho(x0z)$

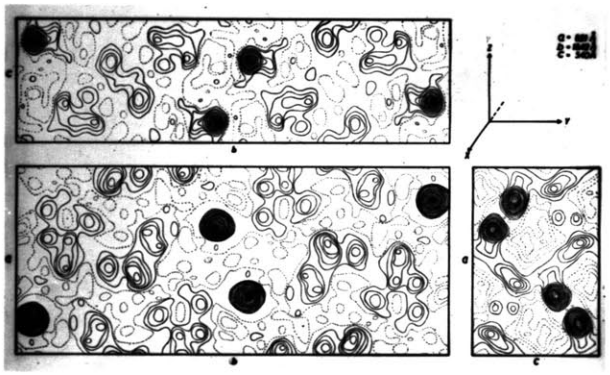


Figure 12 Electron Density Maps

Atomic Positions-

By comparing the three electron density maps and assuming the diglycine structure to be made up of glycine molecules of known structure[14] which of the peaks represented the oxygens, which the nitrogens and which the carbons were decided upon. The atomic positions so obtained are:

Atomic positions as determined from the electron density maps							Atomic positions referred to the same origin			
	x	y	y	z	x	z		x	y	z
Br	.178	.035	.215	.167	.072	.083	Br	.178	.035	.833
C	.089	.380	.370	.333	.161	.083	C	.089	.380	.833
C	.106	.430	.180	.120	.144	.370	C	.106	.430	.120
N	.268	.430	.180	.158	.482	.092	N	.268	.430	.158
O	.470	.112	.363	.183	.220	.433	O	.470	.112	.317
O	.196	.333	.417	.842	.054	.092	O	.196	.333	.842
C	.423	.283	.484	.017	.173	.233	C	.423	.283	.517
C	.065	.172	.422	.127	.315	.123	C	.065	.172	.373
N	.145	.193	.443	.342	.105	.408	N	.145	.193	.158
O	.475	.213	.037	.350	.225	.100	O	.475	.213	.650
O	.423	.283	.037	.350	.173	.400	O	.423	.283	.350

Second Intensity Comparison-

Intensity calculations were made using the parameters as determined from the electron density maps. A comparison of the observed and calculated intensities using a specific atomic scattering factor, f, for each of the atoms for each reflection is given in Table 2.

---

(14) G. Albrecht and R. Corey. J. A. C. S. 61, 5 (1939).

Final Electron Density $\rho(xyo)$-

Only ~~four~~ five sign changes were found from those determined by Br alone. The changes were for the 320, 0 22 0, 6I0, 370 and 940 reflections. An electron density map $\rho(xyo)$ made with tthese 4 sign changes showed a slight shift of parameters as indicated in Figure I3. The new set of parameters from the final electron density (xyo) are:

	x	y
Br	.I78	.035
C	.095	.380
C	.I04	.428
N	.278	.430
O	.462	.II0
O	.200	.338
C	.450	.267
C	.072	.I70
N	.I49	.I94
O	.472	.2I3
O	.4I8	.283

The agreement between observed and calculated intensities is somewhat improved with these new parameters as is shown for a few reflections in Table 2.

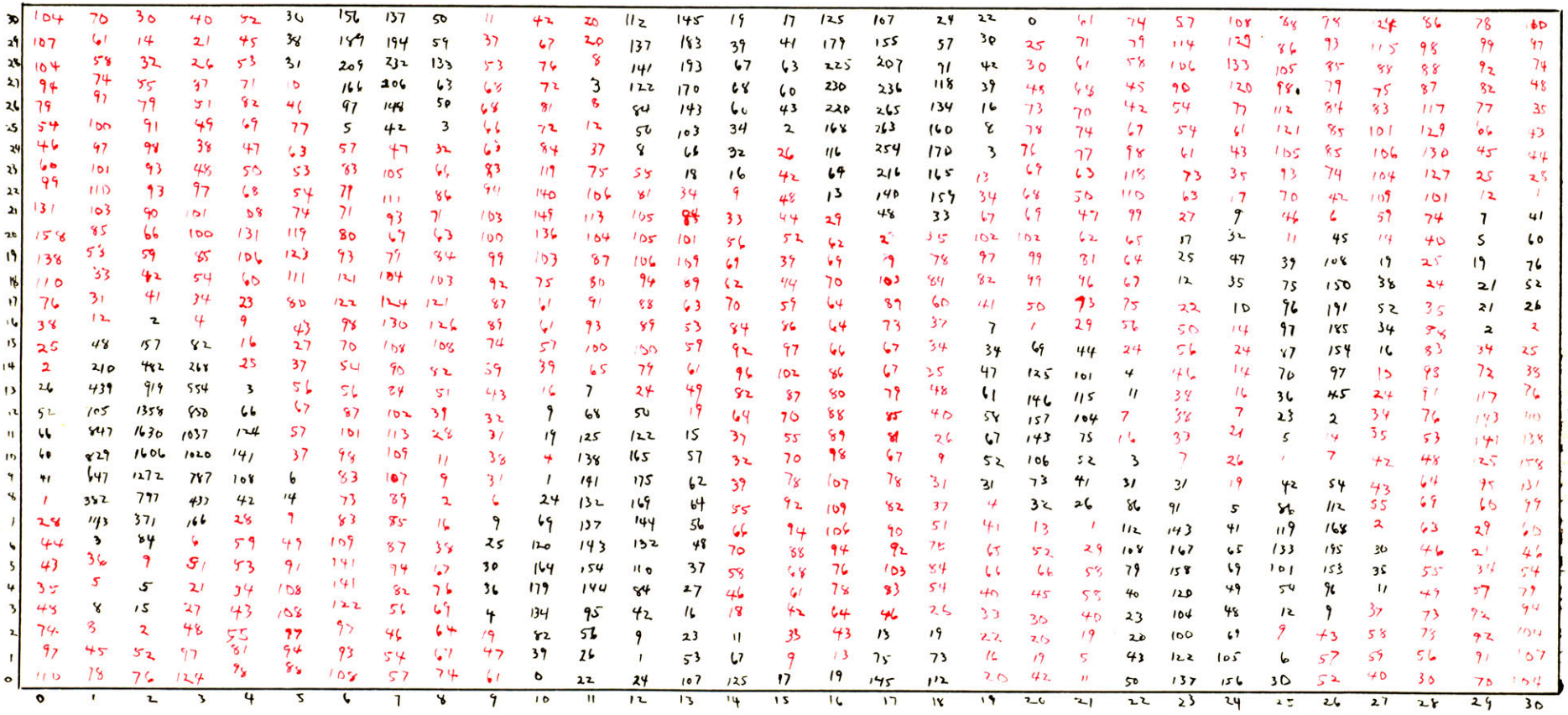


Figure 13 : Electron Density $\rho(x,y,0)$

Table 2

Comparison of Observed and Calculated Intensities Under Three Conditions

Condition I. Rough comparison using an average scattering factor f, for oxygen, nitrogen and carbon in intensity calculations.

Condition 2 Intensity comparison using correct f for oxygen, nitrogen and carbon as well as for bromine.

Condition 3 Intensity comparison using final x and y atomic positions and condition 2.

Reflection hko	Observed I	Observed $\sqrt{40I}$	Calc. $\sqrt{I}/4$ Condition I	Calc. $\sqrt{I}/4$ Condition 2	Calc. $\sqrt{I}/4$ Condition 3
220	44.I	42	40	4I	42
3I0	37.9	39	34.9	36	37
I50	29.6	34	33.8	3I	29
230	26.I	32		33	32
I60	25.7	32	23.3	29	3I
270	20.9	29	30	29	3I
040	I5.6	25	23.3	25	25
020	I5.0	24	22	2I	2I
I80	I4.4	24	27.I	25	24
330	I0.0	20	20.9	22	22
I40	9.34	I9	2I.I	25	23
II00	6.6	I6	I6.6	I9	I7.5
2I0	6.2	I6	I4.7	I2	I2
080	5.8	I5	20.I	I7	I3
I30	5.75	I5	9.6	I7	I8.7
0I20	5.6	I5	20.6	20	I8
4I0	5.2	I4	I4.9	I8	I8
530	4.9	I4	I8.7	20	I9
470	4.7	I4	I5.8	I9	I7
450	4.4	I3	20.I	I9.8	I9
II0	4.2	I3	II	II	I2
5I0	4.I	I3	I7	I4	I5
290	3.4	I2	I2.9	I4.6	I4.6
3I30	3.0	II	I6.3	I7	I7.8
2I00	2.9	II	I4.7	I5	I4.3

Reflection hkO	Observed I	Observed $\sqrt{40I}$	Calc. $\sqrt{I}/4$ Condition I	Calc. $\sqrt{I}/4$ Condition 2	Calc. $\sqrt{I}/4$ Condition 3
250	2.7	I0	I3.8	I2	II
350	2.7	I0	I3.5	I0.6	I4
240	2.2	9	I2.4	I2.0	9.5
560	I.9	9	I3.4	I4.8	I4.9
490	I.9	9	I3.7	I3	I5.0
II20	I.7	8	I3.4	I3	I5
260	I.6	8	7.6	I0	I2.4
280	I.6	8	9.6	I0	I0.6
4II0	I.6	8	I2.8	I3	I4
200	I.3	7	I2.5	I0	I0.5
600	I.2	7	7.98	I0	I0
340	I.2	7	7.8	6	7
620	I.2	7	I3.2	I3	I2
3II0	I.I	7	I3.5	I3	II
0I40	.93	6	II.8	II	II.6
8I40	.92	6		I7	I7.7
2230	.92	6		I9	
I90	.90	6	8.2	8	
0I60	.85	6	9.8	I2.5	I3
I200	.8I	6	I5.9	I5	
760	.79	6		I3	
3I00	.76	6	II.3	II	
5200	.75	5	I2.5	I3	
6I40	.74	5		I4	
3I70	.73	5	I0.05	I0	
540	.7I	5	6.96	I2	
720	.66	5		I0	
I050	.65	5			
I070	.65	5			
740	.64	5		I2	
580	.64	5	8.36	7	
640	.64	5	7.88	8	
42I0	.58	5		I4	
060	.55	5	3.6	6	
670	.55	5	8.7	9	
7I00	.53	5		I2	
II80	.5I	5	9.7	I0	
9I0	.50	4		II	
III0	.49	4	5.0	6	
5II0	.49	4		7	
4I90	.49	4		I0	
6I20	.48	4		9	
2I40	.47	4		I0	

Reflection hkO	Observed I	Observed $\sqrt{40I}$	Calc. $\sqrt{I}/4$ Condition I	Calc. $\sqrt{I}/4$ Condition 2	Calc. $\sqrt{I}/4$ Condition 3
6I80	.45			I2	
800	.45			I3	
6I60	.45			II	
22I0	.44			II	
3I50	.43			9	
2I30	.4I			8	
780	.40			7	
5I90	.39			I2	
II50	.38			7	
320	.36			3	
4I70	.36			I2	
420	.36			3	
0I00	.33			5	
I220	.32			8.5	
870	.30			8	
370	.30			6	
360	.29			7	
2I80	.29			9	
5I00	.28			4.5	
0200	.28			I0	
820	.26			6	
400	.25			4.5	
440	.25			5	
5I30	.24			9	
2II0	.24			4.5	
980	.25			I3.9	
8I20	.23			I0	
2I90	.22			7.9	
7I20	.2I			5	
I70	.2I			5	
9II0	.20			II	
850	.20			3	
690	.I9			8	
590	.I9			7	
6I00	.I8			6	
4I20	.I8			3	
6I70	.I8			6	
8I00	.I6			7	
840	.I6			7	
430	.I6			5	
940	.I5			II	
460	.I4			3.5	
520	.I4			5	
2I60	.I4			5	
6I90	.I3			8.6	
930	.I3			6	
II70	.I3			6	
960	.I2			5	
II60	.II			5	

Reflection	Observed I	Observed $\sqrt{40I}$	Calc. $\sqrt{I}/4$ Condition I	Calc. $\sqrt{I}/4$ Condition 2	Calc. $\sqrt{I}/4$ Condition 3
4K0					
2I50	.II			6.5	
990	.I0			6.8	
4I80	.I0			5	
5I50	.I0			5	
860	.I0			6	
660	.I0			6	
0I80	.I0			2	
0220	.I0			4	
II90	.08			3.6	
6I0	.08			3	
480	.06			2	
950	.06			7	
6I30	.05			6	
II30	.05			4	
I0I0	.05				
8II0	.02			5	
890	.0I			4	
7I40	.0I			2	
4I00	0				
3I20	0				
390	0				
380	0				
2I20	0				
4I40	0				
4200	0				
550	0				
570	0				
5I20	0				
5I40	0				
6II0	0				
6I50	0				
I000	0				

Comparison of Observed and Calculated Intensities under Condition(2)

Reflection (hkl)	Calc. $\sqrt{I}/4$ (Calc)	Calc. $I/I0^3$	Observed I
06I	4I	27	28
04I	34	I8.5	I7.4
032	33	I7.4	I2.4
03I	3I	I5.4	I2
002	27	II.6	7
0I2	28	I2.5	7
07I	I9	5.7	4.7
0I20	I8	5.2	4.6
053	2I	7.0	3.3
040	I7	4.6	3.4
073	20	6.4	3.2
033	I7	4.6	2.7
05I	II	I.9	2.4
0I0I	I5	3.6	2.4
022	I2	2.3	2.3
093	I4	3.I	2.2
092	I3	2.7	2.I
0I40	I2	2.3	2.0
080	I4	3.I	I.9
0I4	I3	2.7	I.85
034	I5	3.6	I.7
004	I8.7	5.6	I.4
0I60	II	I.9	I.3
0I52	II	I.9	I.20
08I	II	I.9	.97
006	20	6.4	895
0I22	I0	I.6	.8
0I46	I2	2.3	.8
0200	8	I.0	.8
0020			
0I8I	I0	I.6	.7
022I	I0	I.6	.7
0II2	I2	2.3	.7
020I	8	I.0	.76
0I9I	5	.4	.7
060	4	.25	.67
0I3	5	.4	.6
0I42	7	.8	.6
045	I0	I.6	.65
09I	7	.8	.55
02I3	II	I.9	.5
0I80	3	.I4	.5
0I32	7	.8	.5
0I93	8	I.0	.5
0I34	9	I.3	.5
065	I5	3.6	.5
02II	4	.25	.4
075	9	I.3	.4
0I05	9	I.3	.4
085	5	.4	.39
0I92	7	.8	.25
0I62	5	.4	.2
084	5	.4	.2

Reflection hkl	Calc. $\sqrt{I}/4$	Calc. I/I_0^3	Observed I
0II4	7	.8	.2
0I26	9	I.3	.2
064	3	.I4	.2
0I72	4	.25	.I3
0II3	2	.I6	.I3
046	6	.57	.I3
0I74	7	.8	.I
095	5	.4	.I
0I3I	3	.I4	.06
094	5	.4	.03
0I24	7	.8	.03
0I54	9	I.3	.03
0I06	4	.25	.08
024	4	.25	0

Comparison of Observed and Calculated Intensities Under Condition (2)

Reflection h0ℓ	Observed I	Observed $\sqrt{I}$	Observed $6\sqrt{I}$	Calc. $\sqrt{I}$
20I	40.67	6.38	38	36
002	28.97	5.38	32	3I
I0I	I3.I5	3.63	22	29
203	6.II	2.47	I5	25
I02	4.29	2.07	I2	I3.5
302	2.93	I.7I	I0	I9.5
30I	2.52	I.59	9.5	8.3
403	I.76	I.33	8	I3
50I	I.7I	I.3I	8	I2.9
202	I.58	I.26	7.5	7.8
502	I.54	I.24	7.4	I6
304	I.36	I.I7	7.0	I3
600	I.05	I.02	6.I	I0
004	.97	.98	5.9	I8.5
70I	.90	.95	5.7	I3
006	.83	.9I	5.5	23
705	.7I	.84	5.0	2I
200	.53	.73	4.4	I0
800	.53	.73	4.4	I3
I05	.43	.66	3.9	7
902	.33	.57	3.4	9
504	.33	.57	3.4	9
40I	.3I	.56	3.3	6
206	.28	.53	3.2	6
90I	.20	.44	2.7	9
204	.I8	.43	2.5	3
400	.I5	.39	2.3	4
I04	.I4	.37	2.3	4
802	.I3	.36	2.2	5
602	.I0	.32	I.9	7
503	.I0	.32	I.9	.22
404	.09	.30	I.8	2
205	.05	.22	I.3	7
405	.05	.22	I.3	5
505	.04	.20	I.2	8
605	.03	.I7	I.I5	6
803	.025	.I6	.95	8.5
60I	0			
402	0			
702	0			

Structure

Unit cell:

a	8.2I A
b	I8.42 A
c	5.40 A

Space group:

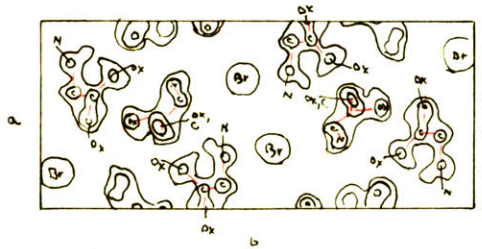
P $2_1 2_1 2_1$

Atomic positions:

	x	y	z
Br	.I78	.035	.833
C	.095	.380	.833
C	.I04	.428	.I20
N	.278	.430	.I58
O	.462	.II0	.3I7
O	.200	.338	.842
C	.450	.267	.5I7
C	.072	.I70	.373
N	.I49	.I94	.I58
O	.472	.2I3	.650
O	.4I8	.283	.350

Discussion

The above structure can only be regarded as an approximate structure. The atomic positions are in some error due principally to the overlapping of the atoms in projection. The atoms could not be further resolved as only zero level photographs were taken so that only projections of the cell could be obtained. No account has been taken so far of the position of the hydrogen atoms in the structure or of their contribution to calculated intensities. A projection of the determined structure on xyo is given in Figure 14. Here the shape of the glycine molecule may easily be seen.



N = Nitrogen
Ox = Oxygen
C = Carbon
Br = Bromine

Figure 14 Projection of structure on (xyo)

Acknowledgment

The writer is indebted to Prof.M. J. Buerger for the suggestion of diglycine hydrobromide for crystal structure determination. Suggestions during the work and reading of the manuscript were most helpful.