Chapter II
**Structure Determination of Morpholine Biguanide Hydrochloride**

**Preparation of Compound**

Morpholine biguanide hydrochloride was prepared in our laboratory by one of our colleagues. Morpholine hydrochloride was fused with dicyandiamide to give morpholine biguanide hydrochloride. The scheme is as follows:

\[
\text{NH}_2\text{HCN} + \text{N} = \text{C} = \text{NH} - \text{C} = \text{NH}_2 \rightarrow \text{N} - \text{C} - \text{NH} - \text{C} - \text{NH}_2 \cdot \text{HCl}
\]

The product was dissolved in water and filtered. From the filtrate morpholine biguanide hydrochloride precipitated with acetone. It was purified by dissolving in water and reprecipitating with acetone. Morpholine biguanide hydrochloride was crystallised by slow evaporation at room temperature (28°C). Thin, transparent plate shaped crystals were obtained. These were examined under polarizing microscope. Single crystals were selected for structural analysis.
Cell Dimensions and Space Group

Rotation and Weissenberg photographs were taken about the three edges of the plate shaped crystals and it was found that the crystallographic axes coincided with the external edges of the plate. The repeat distances along the three axes were obtained from rotation photographs. From pinacoids in the zero layer Weissenberg photographs, $a^*$, $b^*$ and $c^*$ values were calculated. Values of three angles $\alpha^*$, $\beta^*$ and $\gamma^*$ were obtained from the separation of pinacoids in the Weissenberg photographs. The crystals were found to belong to orthorhombic system with unit cell dimensions

\[
\begin{align*}
\alpha &= 19.02 \, \text{Å} \\
b &= 9.92 \, \text{Å} \\
c &= 10.34 \, \text{Å} \\
\alpha &= \beta = \gamma = 90^\circ
\end{align*}
\]

The space group of the crystal was determined by noting the systematic absences of reflections in the Weissenberg photographs. Depending upon the nature of the internal symmetry, sets of systematically absent reflections can be derived from the expression for the structure factor ($F$).

While looking for the nature of systematically absent reflections, if any, in the X-ray photographs, the usual procedure followed is given below. First of all, the reflections of the general type $hkl$ are examined. If no systematic absence
is found, the lattice is primitive. Next, reflections with one index zero, i.e., reflections of type $hko$, $h0l$ and $0kl$ are considered, which provide a clue to the presence of glide planes. Lastly, the reflections with two indices zero are examined which give an idea of the presence of screw axis. The systematic absences observed in the Weissenberg photographs of morpholine biguanide hydrochloride are

<table>
<thead>
<tr>
<th>Type of Reflections</th>
<th>Indices</th>
<th>Systematic Extinctions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. General</td>
<td>$hkl$</td>
<td>none</td>
</tr>
<tr>
<td>2. With one index zero</td>
<td>$0kl$</td>
<td>$k$ odd</td>
</tr>
<tr>
<td></td>
<td>$h0l$</td>
<td>$l$ odd</td>
</tr>
<tr>
<td></td>
<td>$hko$</td>
<td>$h$ odd</td>
</tr>
<tr>
<td>3. With two indices zero</td>
<td>$h00$</td>
<td>$h$ odd</td>
</tr>
<tr>
<td></td>
<td>$0ko$</td>
<td>$k$ odd</td>
</tr>
<tr>
<td></td>
<td>$00l$</td>
<td>$l$ odd</td>
</tr>
</tbody>
</table>

The systematic absences indicate a primitive lattice with three glide planes and hence the space group Pbca. The density of the crystals measured by the method of flotation in a mixture of bromoform and benzene was 1.389 gm/c.c. while that calculated for eight formula units per unit cell is 1.400 cm³/c.c. This shows that one formula unit is present per asymmetric unit of the unit cell. The formula used for
the density calculation was, $NPV = \gamma M$ where, $V$ is the volume of the unit cell, $N$: the Avogadro number, $M$ the molecular weight, $\gamma$: the number of molecules in unit cell and $\rho$: the density of the crystal.

Two Dimensional Intensity Data

In order to derive maximum information from a projection, selection of a crystallographic axis with the smallest period of repetition is desirable because this is expected to give maximum resolution of peaks in the projection. In this case $b$ and $c$ axes being short and nearly equal were used for collecting intensity data. The plate shaped single crystals were cut into needles of almost square cross sections with $b$ and $c$ axes along the lengths of the crystals. Intensity data were recorded by multiple film Weissenberg technique using radiation. The intensities of these reflections were estimated visually by comparison with a standard set of intensity wedges specially prepared for the purpose. While making the standard wedges, special care was taken to ensure that even the strongest reflection recorded was not a saturated one. The reflections with intensities less than the minimum intensity of the measuring wedge were given a value half of the smallest measurable intensity and marked as unobserved reflections.

The intensities of reflections are largely affected due to absorption of X-rays by the crystal itself during their
passage through the crystal. If $I_o$ be the intensity of the incident beam and $I$ that of the transmitted beam then,

$$I = I_o e^{-\mu l}$$

(51)

where $\mu$ is the linear absorption coefficient of the crystal and $l$ is the path length transversed by X-rays inside the crystal. Calculation of the transmission factor 'A' for a crystal of irregular shape is indeed very involved but the task becomes much simpler if the crystal is in the form of a sphere or a cylinder. A general program for computation of absorption correction for application to two dimensional intensity data collected from a crystal of regular shape was written and used on IBM 1620.

For a cylindrical crystal of radius $R$ the path traversed by a beam entering at an angle $\theta$ to the $y$ axis (27), being reflected from an element of volume about the point of coordinates $R_x, R_y$ and leaving at a deflection angle $2\theta$ is

$$P = R \sqrt{\{1 - (x \cos \theta - y \sin \theta)^2\} + R \sqrt{1 - (x \cos \theta + y \sin \theta)^2} - 2 R \sin \theta}$$

(52)

This equation can be solved for different values of $\theta$ and integrated to give the area $\Delta S$ of the regions. Each area can be attributed a path length $L$. Hence we get values of $\Delta S$ and $L$ for successive values of $\theta$ and the
transmission factor for a particular $\theta$

$$A = \sum e^{-\mu R l}$$

$A^x = \frac{1}{A}$ is defined as the absorption factor.

Now the crystals with nearly square cross sections can be approximately treated as cylindrical without introducing much error. This approximation, however, becomes less valid as the equiinclination angle increases.

Linear absorption coefficient, $\mu$, for morpholine biguanide hydrochloride is 31.9 cm$^{-1}$ and the values of $\mu R$ for the crystals assumed to be cylindrical with their axes parallel to 'b' and 'c' were 0.35 and 0.48 respectively.

The intensity data were corrected for Lorentz and polarization factors. The expression for Lorentz and polarization factors for equiinclination recording is,

$$L P^{-1} = \frac{2 \xi \cos \theta}{1 + \cos^2 \theta}$$

where

$$\xi^2 + \xi^2 = 4 \sin^2 \theta$$

$\xi$ and $\xi$ being cylindrical coordinates of the reciprocal lattice points perpendicular and parallel to the rotation axis respectively.
For zero layer Weissenberg photographs, the expression (54) becomes

\[ Lp^{-1} = \frac{4 \sin \theta \cos \theta}{1 + \cos^2 \theta} \quad (56) \]

\[ = \frac{2 \sin 2\theta}{1 + \cos^2 \theta} \quad (57) \]

The intensities of the \( hko \) reflections were brought to the absolute scale by Wilson's (28) statistical plot. The observed and absolute structure factors are related by a scale factor \( K \). We have

\[ |F_0|^2 K = |F|^2 \quad (58) \]

According to A. J. C. Wilson if the reciprocal space be divided into equal \( \sin^2 \theta \) zones then for each zone

\[ K \langle |F_0|^2 \rangle = \sum f_j^2 \exp \left(-2B \frac{\langle \sin^2 \theta \rangle}{\lambda^2}\right) \quad (59) \]

or

\[ \ell_n \frac{\sum f_j^2}{\langle |F_0|^2 \rangle} = \ell_n K + \frac{2B}{\lambda^2} \langle \sin^2 \theta \rangle \quad (60) \]

where

\[ \langle |F_0|^2 \rangle \]

is the average of the observed intensities, the scattering factor of the \( j \) atom in the unit cell, \( B \) the temperature factor.
The reciprocal lattice net was divided into a number of equal \( \sin^2 \Theta \) zones, the number of reflections within this range being large enough for the application of statistical methods. The plot of \( \ln \left( \sum |f|^2 / \langle |F_0|^2 \rangle \right) \) against \( \sin^2 \Theta \) for \( hko \) data, shown in Fig. 2, for different \( \sin^2 \Theta \) zones gave the overall temperature factor \( B \) of the crystal and the scale factor \( K \). The \( hko \) intensities were multiplied by this factor to bring them to absolute scale.

Intensity values of the \( k0l \) data were corrected for absorption, Lorentz and polarization factors and brought to same scale as \( hko \) data by cross-layer correlation method, using the \( h00 \) reflections which are common in both the photographs. Unit weight was assigned to these common reflections and the sums of \( h00 \) intensities from the two layers were linearly correlated. The \( k0l \) reflections were then multiplied by the resulting scale factor.

**Two Dimensional Analysis**

**Location of Heavy Atom**

In order to determine the positional coordinates of heavy (chlorine) atom, Patterson syntheses projected along 'b' and 'c' were calculated. Maximum resolution was expected in these two Patterson maps since 'b' and 'c' axes are much smaller than 'a' axis and are nearly equal. Patterson function for
The Patterson syntheses were calculated with the help of Beever-Lipson strips. The suitable interval of division for calculation was ascertained by considering the limit of resolution. The expression for limit of resolution in this case is

\[ 0.6 \lambda / 2 \sin \Theta_m \] where, \( \lambda \) is the wavelength of X-rays and \( \Theta_m \) is the maximum Bragg angle observed. For \( Cu K_\alpha \) radiation (\( \lambda = 1.5418 \text{ Å} \)) with \( \Theta_m = 90^\circ \), this value becomes 0.46 Å. For the peaks to be well defined, it is necessary to choose an interval of division which is considerably less than this limiting value 0.46 Å. The value chosen in this case was about 0.16 Å, nearly one third the above value. Thus the intervals along the three crystallographic axes, a, b and c were 3°, 6° and 6° respectively. The orthorhombic system is given by

\[ P(uvw) = \frac{2}{V_c} \sum_{h,k,l} F(hkl)^2 \cos 2\pi h U \cos 2\pi k V \cos 2\pi l W \] (61)

For the (010) and (001) projections the expression (61) becomes

\[ P(UW) = \frac{4}{A_c} \sum_{h,k,l} F(hkl)^2 \cos 2\pi h U \cos 2\pi l W \] (62)

and

\[ P(UV) = \frac{4}{A_c} \sum_{h,k,l} F(hkl)^2 \cos 2\pi k U \cos 2\pi l V \] (63)
calculated h0l and hko Patterson syntheses showed symmetry corresponding to plane group $\text{Pmm}$.

Now, if $x, y, z$ be the positional coordinates of an atom in a unit cell with space group $\text{Pbc}a$, its equivalent positions are: $x, y, z; \bar{x}, \bar{y}, \bar{z}; \frac{1}{2} - x, \frac{1}{2} + y, z; \bar{x}, \frac{1}{2} + y, \frac{1}{2} - z; x, \frac{1}{2} - y, \frac{1}{2} + z; \frac{1}{2} - x, \bar{y}, \frac{1}{2} + z$ and $\frac{1}{2} + x, y, \frac{1}{2} - z$.

The interactions between eight chlorine atoms give rise to sixty-four Patterson peaks, eight of which lie at the origin. Strengths and positions of non-origin peaks are given below:

<table>
<thead>
<tr>
<th>Strength</th>
<th>Positional Coordinates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td></td>
</tr>
<tr>
<td>&quot;</td>
<td>$2x, 2y, 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$2\bar{x}, 2y, 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$2x, 2\bar{y}, 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$2x, 2y, 2\bar{z}$</td>
</tr>
<tr>
<td>Double</td>
<td></td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2}, \frac{1}{2} + 2y, 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$2x, \frac{1}{2}, \frac{1}{2} + 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2} + 2x, 2y, \frac{1}{2}$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2}, \frac{1}{2} - 2y, 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$2x, \frac{1}{2}, \frac{1}{2} - 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2} - 2x, 2y, \frac{1}{2}$</td>
</tr>
<tr>
<td>Four times</td>
<td></td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2} - 0, \frac{1}{2} + 2z$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$\frac{1}{2} + 2x, \frac{1}{2}, 0$</td>
</tr>
<tr>
<td>&quot;</td>
<td>$0, \frac{1}{2} + 2y, \frac{1}{2}$</td>
</tr>
</tbody>
</table>
Peaks related centrosymmetrically to all the above mentioned peaks are also present. Strength 'single' signifies the presence of one peak due to Cl-Cl interaction, 'double' means superposition of two such peaks and 'Four times' indicates the superposition of four Cl-Cl vectors.

Thus in the (001) projection we expect the following distribution of peaks:

<table>
<thead>
<tr>
<th>Position</th>
<th>Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>2x, 2y, 0</td>
<td>double</td>
</tr>
<tr>
<td>2x, 2y, 0</td>
<td>double</td>
</tr>
<tr>
<td>1/2 + 2x, 2y, 0</td>
<td>double</td>
</tr>
<tr>
<td>1/2 - 2x, 2y, 0</td>
<td>double</td>
</tr>
<tr>
<td>1/2, 1/2 + 2y, 0</td>
<td>Four times</td>
</tr>
<tr>
<td>2x, 1/4, 0</td>
<td>Four times</td>
</tr>
<tr>
<td>1/2 + 2x, 1/4, 0</td>
<td>Four times</td>
</tr>
<tr>
<td>0, 1/4, 2y, 0</td>
<td>Four times</td>
</tr>
<tr>
<td>1/4, 0, 0</td>
<td>Four times</td>
</tr>
</tbody>
</table>

with their centrosymmetrically placed peaks.

In $\text{hOCl}$ Patterson map the Cl-Cl interaction peaks are distributed as follows:
Peaks related centrosymmetrically to all the above mentioned peaks are also present.

Due to heavy superposition of peaks in both the projections, it was not possible to distinguish the Cl-Cl interaction peaks. So it became necessary to proceed with the three dimensional analysis of the structure.

3-D Data

The three dimensional data were collected along \( b \) and \( c \) axes. The crystals were cut parallel to these two axes with nearly square cross-sections and were taken to be cylinders for all practical purposes.

Equiinclination Weissenberg photographs were taken along \( C \)-axis using filtered CuK\(_\alpha\) radiation upto eighth layer.
Weissenberg photographs were also taken up to first layer 'b' axis. Out of 2235 possible reflections in the effective limiting sphere of CuKα radiation, nearly 1750 independent reflections were recorded. The intensity estimation was done visually as before using a standard intensity wedge. The unobserved reflections were given a statistically significant value i.e., one half the minimum observable intensity.

The intensities were corrected for absorption, Lorentz and polarization factors using R. Ahmed's programs written for IBM 1620 computer.

Due to divergence of the X-ray beam, spots on one half of the film get extended while those on the other half are contracted (29). A higher layer Weissenberg photograph is shown in Fig. 1. The visually estimated intensities need correction for this spot size variation. According to Phillip, the expression for the multiplier to the intensity value of an extended spot is

\[ A = 1 + \Delta A \]  
\[ \Delta A = \frac{180}{4\pi} \frac{\frac{1}{5} \left\{ \frac{(4 - s^2)}{s^2} - 1 \right\}^{\frac{1}{2}}}{2R_1/(4 - s^2)^{\frac{1}{2}} + R_2} \]  

where,

- \( R_1 \) = radius of the camera = 28.7 mm
- \( R_2 \) = distance between the first pinhole of the collimator and the crystal = 75.0 mm
Fig. 1. Morpholine biguanide HCl: Fifth layer Weissenberg photograph along c axis. odd reflections for odd values of k and the ho5 pinacoids are missing.

The extra marks on the lower half of the film are due to the ink marks.
For correcting the intensities of contracted spots the expression used was (30)

\[ A = 1 - 0.4 \Delta A \]  \hspace{1cm} (66)

Some of the spots recorded at very low values of \( \Theta \) on the contracted side get extended. Expression (64) was used for the intensity correction of such spots.

The 3-D data from different layers were correlated with the help of cross layer (h1l) data. The -h0l data were insufficient for the purpose of cross-correlation because alternative festoons on the Weissenberg photographs (for \( l = 2n \)) were missing. The intensities of -h10, -h11, -h12, ..., -h18 reflections from the cross layer data were compared with the intensity values of corresponding reflections from \( h0l, hkl, h1k, h1k2, ..., h1k8 \) layers, and the main data multiplied by scale factors for the respective layer. The 3-D data were brought to absolute scale by Wilson's statistical plot shown in Fig. 3.

3-D Patterson Synthesis

A three dimensional Patterson synthesis for \( x = 0^\circ \) to \( 180^\circ \), \( y = 0^\circ \) to \( 180^\circ \) and \( z = 0^\circ \) to \( 180^\circ \) at intervals of \( 3^\circ \), \( 6^\circ \) and \( 6^\circ \) respectively was calculated on IBM 1620 computer using F. R. Ahmed's program. From the distribution of non-origin peaks due to Cl-Cl interactions, we find that the sections through 3-D Patterson function at \( x = \frac{1}{2}, y = \frac{1}{2} \) and \( z = \frac{1}{2} \) are Harker
**Fig. 2.** Morpholine biguanide HCl : Wilson's statistical plot for scaling the hko intensity data to absolute scale.

**Fig. 3.** Morpholine biguanide HCl : Wilson's statistical plot for scaling the three dimensional intensity data to absolute scale.
sections. The positions of Cl-Cl vectors in the Harker and the coordinates of chlorine atoms as deduced from these are shown below:

<table>
<thead>
<tr>
<th>Harker Section at</th>
<th>Strength of Peak</th>
<th>Coordinates</th>
</tr>
</thead>
</table>
| $x = 180^\circ$    | 782.9            | $2y = 0$  
|                    |                  | $180^\circ - 2z = 34.5^\circ$, $2x = 145.5^\circ$ |
|                    | 463.4            | $2z = 145.5^\circ$  
|                    |                  | $180^\circ - 2y = 73.5^\circ$, $2y = 106.5^\circ$ |
| $y = 180^\circ$    | 695.8            | $2x = 0$  
|                    |                  | $180^\circ - 2x = 24^\circ$, $2x = 156^\circ$ |
|                    | 377.8            | $2x = 156^\circ$  
|                    |                  | $180^\circ - 2z = 36^\circ$, $2z = 144^\circ$ |
| $z = 180^\circ$    | 1155.0           | $2x = 0$  
|                    |                  | $180^\circ - 2y = 68.4^\circ$, $2y = 111.6^\circ$ |
|                    | 310.0            | $2y = 111.6^\circ$  
|                    |                  | $180^\circ - 2x = 22.5^\circ$, $2x = 157.5^\circ$ |

...Mean $2x = 156.7^\circ$, $x = 73.3^\circ$  
$2y = 109.0^\circ$, $y = 54.5^\circ$  
$2z = 145.7^\circ$, $z = 72.3^\circ$

The three Harker sections are shown in Figs. 4a and 4b.
Fig. 4a. Morpholine biguanide HCl: Harker sections at $y = 180^\circ$ and $z = 180^\circ$. Cross marks show the Cl-Cl interaction peaks.
Fig. 4b. Morpholine biguanide HCl: Harker section at $x=180^\circ$.

Fig. 5. Plot of $N$ (percentage of reflections having the phase of heavy atom) against $r = (\Sigma f_h^2/\Sigma f_l^2)^{1/2}$, where $f_h$ and $f_l$ are the scattering factors for heavy and light atoms respectively.
Before calculating the three dimensional Fourier, it was thought necessary to consider the possibility of applicability of heavy atom technique in this case. The structure factor equation can be written as

\[ F = F_H + F_L \]  \hspace{1cm} (67)

where

\[ F_H = \text{structure factor due to heavy atom only} \]
\[ F_L = \text{structure factor due to light atoms} \]

In a suitable heavy atom derivative, for a fairly large fraction of reflections, \( F \) and \( F_H \) have same signs. The actual number of structure factors having the same sign as \( F_H \) can be calculated according to Sim (31) in terms of a parameter defined by

\[ \gamma = \left( \frac{\sum f_H^2}{\sum f_L^2} \right)^{1/2} \]  \hspace{1cm} (68)

where

\[ f_H = \text{scattering factor of heavy atom} \]
\[ f_L = \text{scattering factor of light atom} \]

The variation of fraction of structure factors with signs determined by the heavy atom contributions with \( \gamma \) for the case of a molecule containing one heavy atom situated in a general position is shown in figure 5. It can be seen that for \( \gamma = 1 \), 80% of reflections have same sign for \( F \) and \( F_H \), and for \( \gamma = 2 \), the proportion increases to 90%. In case of morpholine biguanide hydrochloride, \( \gamma = 0.775 \) which indicates
that nearly 76% of the structure factors have the phase of the heavy atom. But the inclusion of the rest 24% of reflections with wrong signs will cause errors in the electron density calculations. According to Woolfson, for minimization of these errors, a weight must be assigned to each term \( P \). With \( \gamma = 0.775 \), a Fourier series calculated by assigning weight to each term according to the probable weight to each term according to the probable magnitude of the phase angle error gives a distinctly better resolution of the light atom peaks in the Fourier map than does the unweighted one.

The probability that the sign of \( P \) is same as that of \( F_H \) is given by

\[
P_+ = \frac{1}{2} + \frac{1}{2} \tanh \left( \frac{|1| F_H | F_L |}{\sum F_L^2} \right) \quad (69)
\]

and the weight to be assigned to the term is given by (32)

\[
W = 2 P_+ - 1 \quad (70)
\]

3-D Analysis of Structure

The structure factors of all the reflections were calculated using the coordinates of Chlorine atom only

\[
F_H = \sum_{j=1}^{N} f_j e^{2\pi i (h x + k y + l z)}
\]

\[
= \sum_{j=1}^{N} f_j \cos \chi n \ (h x + k y + l z) \quad (71)
\]
where \( f_H \) is the scattering factor of heavy atom (chlorine) at that angle. The disagreement factor \( R \), which is a useful guide in determining whether a structure analysis is proceeding in right direction or not was calculated at this stage, using all the reflections.

\[ R = \frac{\sum (|F_o| - |F_c|)^2}{\sum |F_o|} \]

where

- \( F_o \) = observed structure factor
- \( F_c \) = calculated structure factor

The value of \( R \) in this case was 0.60 which indicated that the findings were substantially right and it was worthwhile to proceed with the structure determination.

The observed structure factor for each reflection was multiplied by the above weighting function, the phase of heavy atom was assigned to it and a 3-D Fourier summation was calculated using all the reflections. The resulting Fourier map calculated for \( x = 0^\circ \) to \( 180^\circ \), \( y = 0^\circ \) to \( 90^\circ \) and \( z = 0^\circ \) to \( 360^\circ \) at the intervals of \( 3^\circ \), \( 6^\circ \), and \( 6^\circ \) respectively is shown in figure 6. The contours are drawn at the interval of \( 1 \text{ e} / \text{\AA}^3 \) starting from \( 1 \text{ e} / \text{\AA}^3 \).

The electron density peaks in the weighted fourier were used to construct a spoke and bead model. The peaks with heights between \( 4 \text{ e} / \text{\AA}^3 \) and \( 9 \text{ e} / \text{\AA}^3 \) were taken as atomic sites.
The expression used for electron density calculation was:

\[ p(xyz) = \frac{\theta}{V_c} \sum_{a=a} ^{h+k=2n} \sum_{l=2n+1} ^{k+l=2n+1} F(hk\ell) \cos 2\pi kx \cos 2\pi ky \cos 2\pi \ell z \]

\[ - \sum_{a=a} ^{h+k=2n+1} \sum_{l=2n+1} ^{k+l=2n+1} F(hk\ell) \cos 2\pi kx \sin 2\pi ky \sin 2\pi \ell z \]

\[ - \sum_{a=a} ^{h+k=2n+1} \sum_{l=2n+1} ^{k+l=2n+1} F(hk\ell) \sin 2\pi kx \cos 2\pi ky \sin 2\pi \ell z \]

\[ - \sum_{a=a} ^{h+k=2n+1} \sum_{l=2n+1} ^{k+l=2n+1} F(hk\ell) \sin 2\pi kx \sin 2\pi ky \cos 2\pi \ell z \]
Fig. 6a. Morpholine biguanide HCl. Chlorine phased weighted three dimensional Fourier sections at $\gamma = 0^\circ$, $6^\circ$, $12^\circ$, and $18^\circ$. The contours are drawn at the intervals of $1 \text{ e/Å}^3$ starting from $1 \text{ e/Å}^3$ in case of C, N and O and at the intervals of $2 \text{ e/Å}^3$ starting from $2 \text{ e/Å}^3$ in case of chlorine. One eighth of the unit cell is shown.
Fig. 6b. Morpholine biguanide $\text{HCl} :$ Chlorine phased three dimensional Fourier sections at $\gamma = 24^\circ, 30^\circ, 36^\circ$ and $42^\circ$. 
Fig. 6c. Morpholine biguanide HCl : Chlorine phased three
dimensional Fourier sections at $y = 48^\circ, 54^\circ, 60^\circ$, and $66^\circ$. 
Fig. 64. Morpholine biguanide HCl : Chlorine phased three dimensional Fourier sections at \( \gamma = 72^\circ, 78^\circ, 84^\circ \) and \( 90^\circ \).
corresponding to carbon, nitrogen and oxygen. The \( x \) and \( z \) coordinates of electron density maxima were plotted on a tracing paper in a scale of 2 cms to 1 A. Spokes were inserted at the \( x, z \) coordinates of the different peaks and the heights of the peaks along the \( y \)-direction were marked by coloured beads. Different colours were used to indicate variations in strengths of the maxima. Since no crystallographic data were available on any similar structure, the model had to be constructed on the basis of stereochemical considerations only. The peaks corresponding to one molecule were located and the proposed model satisfied nearly all the intermolecular and intramolecular characteristics of morpholine biguanide hydrochloride.

To test the proposed model, structure factors were calculated for \( hko \) data only as there was least superposition of peaks in this projection. The R-value with chlorine only was 0.54. On calculation of structure factors with all the atomic sites of the proposed model, the R-value came down to 0.30. This indicated that there was no gross error in the proposed model and it was ready for 3-D refinement.