A Thesis

THE X-RAY CRYSTAL ANALYSES OF SOME ORGANIC AND CHARGE-TRANSFER COMPOUNDS, AND THE DEVELOPMENT OF RELATED COMPUTER PROGRAMS

Submitted to the University of Glasgow
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SUMMARY

The thesis is divided into three main parts; the first is devoted to a brief review of the theory and methods of X-ray Crystallography, the second describes a number of computer programs devised wholly or in part by the author, and the third presents four structural analyses.

The computer programs found in Part II are written in KDF 9 - ALGOL, a subset of ALGOL 60. The ASS system of crystallographic computer programs is described and two programs from its structure solution aspect are reported. These are a Fourier-coefficient weighting program which weights a structure factor, for use in a Fourier summation, depending on the probability that its phase is correct, and a program which will provide a complete analysis of structure-factor data. Two other programs, more connected with structure refinement, are then discussed, an isotropic structure-factor least-squares program and a general anisotropic structure-factor program.

Part III consists of the application of the X-ray method to organic structural problems. The crystal-structure analyses of the molecular complexes of anthracene and 1,12-dimethylbenzophenanthrene with 4-bromo-2,5,7-trinitrofluorenone are described and an account of the charge-transfer bonding involved in the compounds is given. Two further structural analyses, those of a derivative of a degradation product of a pigment obtained from the bacterium *Pseudomonas lemmonieri*, and of the derivative of a tertiary alcohol, obtained from a reaction designed to yield a doubly bridged tricyclic molecule, are then presented and the results discussed.

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David R. Pollard

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PART I

SOME METHODS OF X-RAY ANALYSIS.

1.1 Introduction

By the beginning of this century classical crystallography had developed so that it was possible to understand the external morphology and symmetry of crystals. In 1912 it was shown that X-rays were diffracted by matter in an analogous manner to that of light by a diffraction grating. This discovery, due to Max von Laue, led to deeper understanding both of the structure of crystals and of the nature of X-radiation itself.

X-Ray Crystallography is basically concerned with the interpretation of the diffraction patterns formed when matter and X-rays interact. It was soon deduced that, as X-rays are scattered by electrons, the diffraction pattern must be related to the electron distribution in the crystal. Initially simple substances were investigated but with increasing knowledge it soon became possible to study more complex inorganic compounds. Molecular crystals were a greater problem owing to the complex electron distribution and the scale of the calculations involved. The major breakthrough in this field came from the discovery that the interpretation of the diffraction pattern was simplified if a small number of atoms, whose scattering dominated in the diffraction, was included in the molecule under study. From this start structures of complex molecules have now been elucidated in the steroid, alkaloid, terpenoid, protein and many other fields.

The use of the electronic computer has made possible most recent advances in crystallography. With suitable programming the complete data collection and structure solution of some of the simpler compounds can be done automatically. It is difficult to estimate the value of crystallography to modern chemistry, it is sufficient to say that X-ray diffraction has provided not only important information on the structure of molecules but also the basis on which many different fields of study have developed.

1.2 The Geometry of X-ray Diffraction

Let a beam of X-rays, of wavelength λ, be incident on a crystal in a direction defined by the unit vector $\lambda \underline{s}_o$ (Fig. 1.1). At a point B, the path difference between a wave scattered in a direction defined by the unit vector $\lambda \underline{s}$ relative to one scattered at the origin A is

$$AC - BD = \lambda\, \underline{r}.\underline{s} - \lambda\, \underline{r}.\underline{s}_o = \lambda\, \underline{r}.\underline{S} \qquad (1)$$

and corresponding phase difference $2\pi\, \underline{r}.\underline{S}$, $\underline{r}$ is the vector position of B relative to A and $\underline{S} = \underline{s} - \underline{s}_o$. The reciprocal vector, $\underline{S}$, is defined in terms of the incident and diffracted rays and, from Fig. 1.2, it can be seen that

$$|\underline{S}| = 2 \sin \theta / \lambda \qquad (2)$$

where 2θ is the scattering angle.

If the volume around B has an electron content $\rho(r)\, dV$, the expression for the scattered wave relative to one scattered by a single electron at the origin is then

$$G_B(\underline{S}) = \rho(r)\, dV \exp 2\pi i\, \underline{r}.\underline{S}\ . \qquad (3)$$

The wave scattered by the unit cell is then

$$G(\underline{S}) = \int \rho(r) \exp(2\pi i\, \underline{r}.\underline{S})\, dV \qquad (4)$$

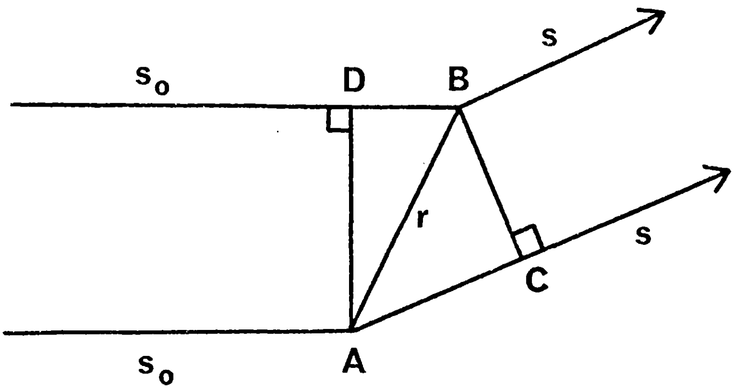
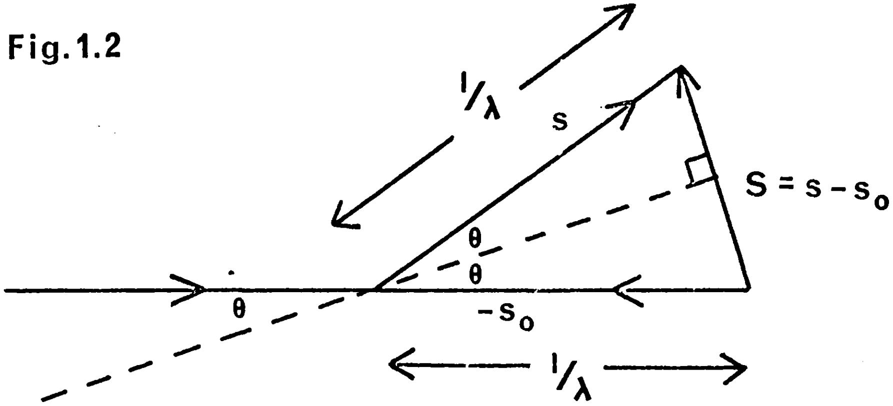


Fig.1.1

Fig.1.2



where the integral is over the whole unit cell and the value, $G(\underline{S})$, expresses the fact that the scattered amplitude is the Fourier transform of the scattering distribution.

In a crystal with lattice dimensions $\underline{a}$, $\underline{b}$ and $\underline{c}$ the electron density function $\rho(r)$ is periodic and thus points defined by

$$R = \underline{r} + u\,\underline{a} + v\,\underline{b} + w\,\underline{c} \tag{5}$$

where u, v and w are integers, have similar electron densities. Scattering from any of these points, C , is given by substituting (5) in (3),

$$\begin{aligned} G_C(\underline{S}) &= \rho(r)\, dV \exp 2\pi i\, R.\underline{S} \\ &= G_B(\underline{S}) \exp 2\pi i\, (u\,\underline{a} + v\,\underline{b} + w\,\underline{c}).\underline{S}\,. \end{aligned} \tag{6}$$

The resultant wave from the crystal will be large only if the waves scattered from these points are in phase, that is, the path difference between $G_B(\underline{S})$ and $G_C(\underline{S})$ must be a whole number of wavelengths. Thus the relation

$$\lambda(u\,\underline{a} + v\,\underline{b} + w\,\underline{c}).\underline{S} = n\lambda \tag{7}$$

with integers u, v, w and n must be valid, and so each of the equations

$$\begin{aligned} \underline{a}\cdot\underline{S} &= h \\ \underline{b}\cdot\underline{S} &= k \\ \underline{c}\cdot\underline{S} &= l \end{aligned} \qquad (8)$$

where h, k and l are integers must hold individually. The relations (8) are termed the Laue equations. The equation $\underline{a}\cdot\underline{S} = h$ defines, in reciprocal space, a family of planes normal to $\underline{a}$. The spacing between two adjacent planes is $1 / |\underline{a}|$. Since $\underline{a}$, $\underline{b}$ and $\underline{c}$ are never parallel the Laue equations define three sets of equally spaced planes whose intersections form a net called the Reciprocal Lattice. The equation defining such a lattice is

$$\underline{S} = h\,\underline{a}^* + k\,\underline{b}^* + l\,\underline{c}^* . \qquad (9)$$

From (8) and (9) it follows that $\underline{a}^*$ is normal to $\underline{b}$ and $\underline{c}$ and that the projection of $\underline{a}^*$ upon $\underline{a}$ is $1 / |\underline{a}|$. Similar results hold for $\underline{b}^*$ and $\underline{c}^*$. These reciprocal lattice constants are related to the primitive translations of the real space lattice by

$$\begin{aligned} \underline{a}^* &= \underline{b} \times \underline{c} / V \\ \underline{b}^* &= \underline{c} \times \underline{a} / V \\ \underline{c}^* &= \underline{a} \times \underline{b} / V \end{aligned} \qquad (10)$$

where V is the volume of the unit cell.

Values of $\underline{S}$, corresponding to diffracted beams, are those whose ends lie on the reciprocal lattice points and are determined by the coordinates h, k and l. From Fig 1.2, if AO is a fixed direction, the free end of $\underline{S}$ must lie on the surface of a sphere with its centre at A and radius $1 / \lambda$. This sphere is known as the *Sphere of Reflection* and only those points of the reciprocal lattice that lie on the surface of the sphere can be observed for a given incident-beam direction.

By rewriting the Laue equations (8) in the form

$$(\underline{a} / h).\underline{S} = (\underline{b} / k).\underline{S} = (\underline{c} / l).\underline{S} = 1 \qquad (11)$$

it can be seen that, by definition, $\underline{a}/h$, $\underline{b}/k$ and $\underline{c}/l$ are the intercepts which the plane with Miller indices hkl makes on the crystal axes. From (11) the projections of three points on this plane on to $\underline{S}$ are equal, $\underline{S}$ is therefore normal to the plane and $|\underline{S}| = 1 / d$ where d is the origin to plane distance. Substituting for $|\underline{S}|$ in equation (2) gives

$$2 d \sin \theta = \lambda \qquad (12)$$

From Fig 1.2, $\underline{s}$ and $\underline{s}_o$ have the same relation as the incident and reflected beams with respect to a mirror. When the Laue equations hold for a diffracted beam the plane normal to $\underline{S}$ is now identified as the lattice plane with Miller indices hkl .

In this way Bragg (1913) related the integers h, k and l of the Laue equations with corresponding Miller indices and allowed diffraction by a crystal to be considered in the simpler concept of reflection by a crystal plane.

1.3 The Structure Factor

The vector distance, from the origin, of the n^{th} atom of a group of N atoms with atomic coordinates x_n, y_n, z_n, is given by

$$\underline{r}_n = x_n + y_n + z_n \, . \tag{13}$$

Thus the wave scattered from the n^{th} atom, relative to the origin of the unit cell, is, by analogy to (4)

$$\begin{aligned} &\int \rho(\underline{r}) \exp 2\pi i \, (\underline{r} + \underline{r}_n).\underline{S} \, dV \\ &= \left\{ \int \rho(\underline{r}) \exp 2\pi i \, \underline{r}.\underline{S} \, dV \right\} \exp 2\pi i \, \underline{r}_n.\underline{S} \\ &= f_n(\underline{S}) \exp 2\pi i \, \underline{r}_n.\underline{S} \, . \end{aligned} \tag{14}$$

The function

$$f_n(\underline{S}) = \int \rho_n(\underline{r}) \exp 2\pi i \, \underline{r}.\underline{S} \, dV \tag{15}$$

is defined as the <u>atomic scattering factor</u> and is the Fourier transform of the atomic electron density. If the atom has spherical symmetry then $\rho(r)$ is a function of $|\underline{r}|$ only and $f_n(\underline{S})$ is then a function of $|\underline{S}|$, or, from (2), of $2 \sin \theta / \lambda$.

The atomic scattering factor is then the expression for the total wave scattered by an atom relative to a single electron. In atoms, owing to the varying electron distribution, there are phase differences between waves scattered at different points. For small angles of diffraction these differences are small and the total scattering is the sum of the individual electron scattering, that is, at $\sin\theta/\lambda = 0$ the scattering is Z the atomic number. Increasing $\sin\theta/\lambda$ gives larger phase differences and so decreases $f_n(\underline{S})$. Scattering factors for different atoms are difficult to calculate as $\rho(\underline{r})$ is known exactly only for hydrogen, values for spherically symmetric atoms have been tabulated, however, and can be found in International Tables, Vol. III, (1962).

The total wave scattered by the contents of the unit cell can now be considered as the sum of the waves scattered by N spherically symmetric atoms and the Fourier transform expression (4) can be written as

$$G(\underline{S}) = \sum_{n=1}^{N} f_n \exp 2\pi i\, \underline{r}_n \cdot \underline{S}\ . \tag{16}$$

We now require values of (16) at which the Laue equations are satisfied, these values, known as Structure Factors of the diffracted beams, are obtained by expressing (13) as

$$\underline{r}_n = x_n\underline{a} + y_n\underline{b} + z_n\underline{c} \tag{17}$$

where x_n, y_n and z_n are fractional coordinates. Designating values of G, which are satisfied, by F we have from (16)

$$F = \sum_{n=1}^{N} f_n \exp 2\pi i (x_n \underline{a}.\underline{S} + y_n \underline{b}.\underline{S} + z_n \underline{c}.\underline{S})$$

$$= \sum_{n=1}^{N} f_n \exp 2\pi i (hx_n + ky_n + lz_n)$$

$$= F\ (hkl)\ . \tag{18}$$

The structure factor, F(hkl), is therefore a function of h, k and l and is a particular value of the Fourier transform. The complex expression, F(hkl), can be written as

$$A + i B \tag{19}$$

where $$A = \sum_{n=1}^{N} f_n \cos 2\pi (hx_n + ky_n + lz_n) \tag{20}$$

and $$B = \sum_{n=1}^{N} f_n \sin 2\pi (hx_n + ky_n + lz_n)\ . \tag{21}$$

The <u>structure amplitude</u> $|F\ (hkl)|$ and phase $\alpha\ (hkl)$ are given by

$$|F\ (hkl)| = (A^2 + B^2)^{1/2} \tag{22}$$

$$\alpha\ (hkl) = \tan^{-1} B / A\ . \tag{23}$$

If, as in a centrosymmetric space group, the origin can be taken as a centre of symmetry, the B components vanish and the phase angle is limited to 0 or 180°.

The atomic scattering factor is normally calculated from the electron density of an atom at rest. Atoms at room temperature, however, have a considerable motion caused by thermal vibration, the electron density then appears more diffuse and increases the rate at which the amplitude of the scattered wave falls off with $\sin\theta/\lambda$. Waller (1927) showed that if f_o is the scattering factor for the atom at rest the corrected scattering factor, f, for an atom with isotropic motion is given by

$$f = f_o \exp(-B\sin^2\theta/\lambda^2) . \qquad (24)$$

B is the <u>temperature coefficient</u> and can be obtained from fundamental constants and heat capacity data (Debye, 1914), its value is given by

$$B = 8\pi^2 U \qquad (25)$$

where $U = \bar{u}^2$ and u is the root mean square amplitude of vibration of the atoms from their mean position.

In the general case each atom undergoes a motion such that the electron density is smeared over a small anisotropic volume denoted as a triaxial ellipsoid. Each atom has a different ellipsoid and all these ellipsoids are differently oriented. To describe this anisotropic motion U may now be replaced by the symmetrical second order tensor $\underline{U}$. The mean square amplitude of vibration in the direction of the unit vector $\underline{l}$ is then

$$\overline{u}^{2} = \sum_{i=1}^{3} \sum_{j=1}^{3} U_{ij}\, l_{i}\, l_{j} \qquad (26)$$

where both $\underline{U}$ and $\underline{l}$ are referred to the reciprocal lattice axes. The correction now to be applied to f_o is given by Cruickshank (1965) as

$$f = f_{o} \exp -2\pi^{2} (U_{11} h^{2} a^{*2} + U_{22} k^{2} b^{*2} + U_{33} l^{2} c^{*2}$$

$$+ 2U_{23}\, klb^{*}c^{*} + 2U_{31}\, lhc^{*}a^{*} + 2U_{12}\, hka^{*}b^{*}) \; . \qquad (27)$$

1.4 Application of Fourier Series

The electron density function, $\rho(r)$, is finite, single valued and periodic in three dimensions, consequently it may be represented by an appropriate Fourier series. If u, v and w are integers and x, y and z fractional coordinates then

$$\rho(r) = \rho(xyz)$$

$$= \sum_{u}\sum_{v=-\infty}^{+\infty}\sum_{w} A(uvw) \exp[-2\pi i (ux + vy + wz)] . \qquad (28)$$

As the structure factor is the Fourier transform of $\rho(xyz)$ then

$$F(hkl) = V\int_0^1\int_0^1\int_0^1 \rho(xyz) \exp[2\pi i(hx+ky+lz)]dxdydz . \qquad (29)$$

Substituting $\rho(xyz)$ in (29) by the Fourier series (28) gives, after rearrangement,

$$F(hkl) = V\sum_{u}\sum_{v=-\infty}^{+\infty}\sum_{w} [\int_0^1\int_0^1\int_0^1 \Big\{A(uvw) \exp 2\pi i (h-u)x. \exp 2\pi i (k-v)y. \exp 2\pi i (l-w)z. dxdydz\Big\}] . \qquad (30)$$

The triple integral in (30) is zero unless $h = u$, $k = v$ and $l = w$, therefore

$$F (hkl) = V.A(uvw) \qquad (31)$$

which inserting in (28) gives

$$\rho(xyz) = \frac{1}{V} \sum_{u} \sum_{v=-\infty}^{+\infty} \sum_{w} F(hkl) \exp[-2\pi i \, (hx+ky+lz)] \; . \qquad (32)$$

The structure factors, F (hkl), scaled by the reciprocal of the cell volume, are then the corresponding coefficients of the Fourier series which gives the electron density function. The zero term of the series (29) is

$$\begin{aligned} F\ (000) &= V \int_0^1 \int_0^1 \int_0^1 \rho(xyz)\ dx\ dy\ dz \\ &= Q \end{aligned}$$

where Q is the total number of electrons per unit cell and is a constant for a given compound.

1.5 Factors Affecting Intensity

The total energy, $E(hkl)$, of the X-ray beam reflected from a set of planes with Miller indices h, k and l, of an ideally imperfect small crystal rotating with uniform velocity can be represented as

$$E(hkl) = K \cdot L(hkl) \cdot p(hkl) \cdot |F(hkl)|^2 \qquad (34)$$

where K is a constant for the experiment and is given by

$$K = \frac{I_o \lambda^3 N^2 dV}{\omega} \cdot \frac{e^4}{m^2 c^4}$$

where

I_o is the intensity of the incident beam,

λ is the wavelength of the X-rays,

N is the number of unit cells per unit volume,

dV is the volume of crystal irradiated,

ω is the angular velocity of the crystal,

e is the electronic charge,

m is the mass of an electron,

c is the velocity of light.

The Lorentz factor, $L(hkl)$, allows for the varying time taken for reciprocal lattice points to pass through the surface of the sphere of reflection and is dependent on the experimental method employed. As the angular velocity of the upper layer planes depends on the angle of inclination of the planes to the axis of rotation, a rotation correction is normally included in the calculation of the Lorentz factor. Tunell (1939) gives, for equi-inclination Weissenberg photographs,

$$L(hkl) = \sin\theta\,(\cos^2\mu - \cos^2\theta)^{-1/2}(\sin 2\theta)^{-1} \qquad (36)$$

where μ is the angle between the incident beam and the plane normal to the rotation axis.

The polarisation factor, $p(hkl)$, takes into account the reduction in intensity caused by the polarisation of the X-ray beam after reflection by the crystal. In all cases it takes the form

$$p(hkl) = (1 + \cos^2 2\theta) / 2 . \qquad (37)$$

The energy of the diffracted beam is proportional to its intensity, $I(hkl)$, which may be measured by either the blackening produced on a photographic film or by counter techniques. Hence from (34)

$$I(hkl) \propto L(hkl) \,.\, p(hkl) \,.\, |F(hkl)|^2 . \qquad (38)$$

Thus a structure amplitude may be measured experimentally on an arbitrary scale. The absolute scale can be obtained by several methods: by comparison with a known standard (Robertson, 1934), from the decrease in average structure amplitude with scattering angle (Wilson, 1942), or, in the latter stages of the analysis by least-squares methods or by comparison with calculated structure factors.

Certain other corrections to the intensities are also required on account of the physical structure of the crystal itself. The most important of these is absorption. Since X-rays are absorbed by matter, the further a beam travels through a crystal the more it is attenuated. The intensity, I , of a transmitted beam can be represented as

$$I = I_o \exp (- \mu t) \tag{39}$$

where I_o is the intensity of the incident beam, t is the thickness of the crystal through which the beam passes and μ is the linear absorption coefficient for the crystal. Corrections are possible for variously shaped crystals and methods have been proposed by Albrecht (1939), Rogers and Moffett (1956) and Busing and Levy (1957).

If a beam of X-rays is incident on a plane in a crystal, the reflected beam is in an orientation such that it can be re-reflected from the same plane. Such a doubly reflected beam is parallel to the incident beam and exactly out of phase with it, so decreasing the intensity of the diffracted beam. The effect is known as <u>primary extinction</u> and corrections for it are difficult to make.

<u>Secondary extinction</u> is caused by layers of a crystal, nearest to the incident beam, shielding lower layers from the same beam. Thus when in a reflecting condition the incident beam received by a plane is that of the original beam less that which has been reflected by any previous plane. Several methods of correcting for the effect have been proposed, for example those of Chandrasekhar (1956) and Hamilton (1957).

Friedel's Law states that the intensities of the reflections (hkl) and $(\bar{h}\bar{k}\bar{l})$ are the same, although, for a non-centrosymmetric crystal the phase angles have opposite signs. This situation is altered, however, if an atom in the structure is excited by the incident radiation and scatters anomalously. The resulting diffracted wave has then to be corrected by both a real and imaginary component, the imaginary component advancing the phase by $\pi / 2$. This has an effect on the intensity scattered by the crystal and in general for non-centrosymmetric crystals the reflection (hkl) will not equal $(\bar{h}\bar{k}\bar{l})$. In Fig 1.3 , F^{R} and F^{R-} are the components of the diffracted wave from the normal atoms, F'^{A} and F'^{A-} are the real components from the anomalous scatterer, F''^{A} and F''^{A-} are the imaginary components, and F and $\bar{F}$ are the structure factor vectors. On account of the imaginary components, it can be seen that the structure amplitudes and magnitudes of the phase angles are not identical. Anomalous scattering can be used as a test of centrosymmetry, as a means of distinguishing enantiomorphic structures and to some extent for direct structure analysis.

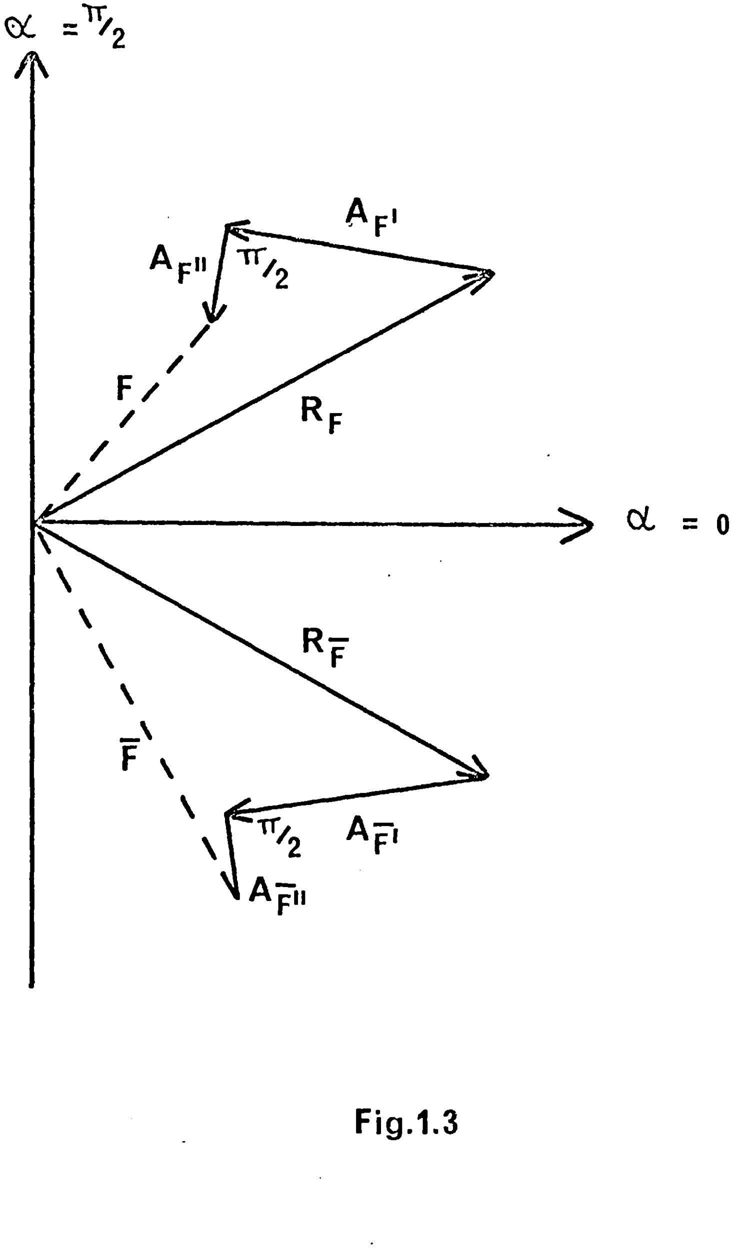


Fig.1.3

1.6 The Phase Problem

The determination of a crystal structure by X-ray diffraction is not straightforward. Structure factors, F(hkl), which have to be employed in the appropriate Fourier series (32) to give a representation of the electron density throughout the unit cell, are complex quantities and are characterised by both magnitude and phase. The magnitude of a structure factor can be routinely determined but as yet no experimental method of deriving phase angles has been found. This difficulty of solving a crystal structure without the immediate knowledge of the relevant phase angles has become known as the "phase problem". Subsequent sections of this chapter list some of the methods used in surmounting it.

1.6.1 Trial and Error

The simplest method is to postulate a chemically sensible model, compare the agreement between observed and calculated structure amplitudes and to discard the model if the agreement is bad. The process, for obvious reasons, is called "trial and error". Some factors can be helpful in the initial trial structure, thus the atomic radii, packing in the unit cell, previously known structures of similar type and the spatial grouping of atoms, for example the oxygen atoms in a silicate group, all give information on probable molecular orientations.

The position of a molecule can sometimes be found if a few reflections are outstandingly large. In planar hydrocarbons, for example coronene (Robertson and White, 1945), several large intensities may be sufficient to reveal the complete structure.

1.6.2 The Patterson Function

The most direct method for displaying the information contained in the measured structure amplitudes is to use the function derived by Patterson (1935) from considerations of the self-convolution of the electron density. It takes the form of the following Fourier series,

$$P(UVW) = \frac{1}{V} \sum_h \sum_k \sum_l |F(hkl)|^2 \cos 2\pi (hU + kV + lW). \qquad (40)$$

The most important property of this function is that it gives a peak, at the vector distance between every pair of atoms in a crystal, whose height is proportional to the atomic number of the atoms involved. Thus in a unit cell of N atoms there are N^2 peaks, N of which are at the origin. For small molecules the number of vectors is not large and in some cases the main features of a crystal structure can be determined directly. The extraction of the real structure from the Patterson function was simplified by Harker (1936) who pointed out that owing to particular symmetry elements in the unit cell some vector peaks are constrained to lie on certain lines and sections.

1.6.3 Superposition Methods

These are extensions of the Patterson function method whereby light atom positions can be determined from vector coincidences. One of the first developments was the "Vector Convergence Method" of Beevers and Robertson (1950) which was carried out by placing the origin of the Patterson on each heavy atom in turn, light atom positions were then revealed in the sum function of the superimposing vectors. Buerger's minimum function (1951) is a similar image seeking function where the value recorded on the resulting map is the lower of the two values, at any point, of two displaced Patterson maps. Some moderately complicated molecules have been solved using superposition methods, for example Cellobiose (Jacobson, Wunderlich and Lipscombe, 1961).

1.6.4 The Heavy Atom Method

If in a molecular structure there is an atom which is considerably heavier than the remaining atoms, then the scattering from that atom will dominate in the diffraction pattern. Phases of structure factors, derived from the heavy atom only, will agree to a fair approximation to the true phases and may be used, with measured structure amplitudes, to give an electron density distribution from which further information may be derived. This procedure known as the "Heavy Atom Method" has been the most widely applied of all for surmounting the phase problem. Many large and complicated structures have been solved in this way by either employing a heavy atom already present or introducing one in the form of a chemical derivative or a solvate.

The major disadvantage of the method comes from its own inherent supposition, that is, the scattering from the heavy atom dominating that of the lighter atoms; the accuracy with which the positions of the lighter atoms can be found is thus lessened. This difficulty may be overcome by the method of isomorphous substitution whereby some heavy atom derivative is employed to solve the structure while the final refinement is carried out with an isomorphous derivative in which a lighter atom has replaced the heavy atom. Examples of both methods are given in the structural studies of the phthalocyanins by Robertson (1936) and Robertson and Woodward (1940). At present the method of multiple isomorphous substitution is widely used in the determination of protein structures.

1.6.5 Direct Methods

The adjective "Direct" is usually reserved for those methods which attempt to derive the phases of the structure factors by mathematical relations of the X-ray diffraction data. The method arose out of the work of Harker and Kasper (1948) who derived relations in the form of inequality expressions. More recently Karle and Hauptman (1954) have developed a statistical method in which phase information is deduced from the magnitudes of the structure amplitudes. Their "Symbolic Addition Method" is largely based on an earlier equation due to Sayre (1952). In general direct methods have only been applied to fairly small structures in centrosymmetric space groups, however, moderately sized structures, including some in non-centrosymmetric space groups, for example the alkaloid Panamine by Karle and Karle (1966), have been attempted and solved.

1.7 Methods of Refinement

In the initial stages of an analysis the object is to increase the agreement between observed and calculated structure factors. This agreement is normally expressed as a residual, R , defined by

$$R = \frac{\sum |F\ \mathrm{obs}| - \sum |F\ \mathrm{calc}|}{\sum |F\ \mathrm{obs}|} . \qquad (41)$$

In an analysis the structure solution normally merges into the Fourier refinement. This Fourier refinement is important as it can both proceed when the structure is incompletely known and lead to further information.

1.7.1 Fourier Refinement

If the position of an atom, say a heavy atom, has been found, phases and amplitudes of the structure factors, derived from this atom, will approximate to the true phases and amplitudes (see Section 1.6.4, The Heavy Atom Method). A Fourier series now summed with coefficients which are more likely to be correct (using calculated phases and observed amplitudes) should give an electron density distribution from which other atoms can be determined. This process, when repeated, leads to improved phases which can be used to increase the definition of subsequent electron density distributions. The decision whether or not to include a structure factor can be taken either by the crystallographer or, equally well, by a computer program set to some arbitrary limit. Woolfson (1956) and Sim (1959) have pointed out that it can be better to weight each structure factor according to the reliability of its phase.

The largest error involved in a Fourier refinement is that due to series termination whereby a Fourier series is normally truncated short of an infinite number of terms. The effect is apparent in the modification of atomic positions by diffraction ripples from neighbouring atoms. Booth (1946) suggested a correction by employing two Fourier syntheses, one using F_{obs} values and the other F_{calc} values for coefficients. As the series termination errors in each synthesis are substantially the same, improved atomic positions can be obtained by subtracting from the derived F_{obs} positions the differences in atomic positions given from the F_{obs} and F_{calc} syntheses; this modification is known as a back-shift correction.

An important Fourier synthesis is that derived from $(F_o - F_c)$ terms. It arose out of Bunn's (1949) error synthesis which is computed using limited data depending on the relative size of Fo and Fc . When used with full data the (Fo - Fc) or difference synthesis can give rise to important information on errors in positional or thermal parameters. The most common use for a difference synthesis now is to check the correctness of a structure and determine hydrogen atom positions. On a difference map from a refined structure the level of electron density should be consistently small and show no significant deviation from that level.

1.7.2 Least Squares Refinement

The use of the least-squares technique, as applied to a crystal structure analysis, was first accomplished by Hughes (1941). The object is to minimise some function of the difference between observed and calculated intensities with respect to the structural parameters. The most common function employed is

$$M = \sum_{hkl} w \left(|Fo| - |Fc| \right)^2 \qquad (42)$$

where the sum is over all independent structure amplitudes and w is the weight for each term. The weight should be chosen to reflect the accuracy of the observed data and values which give the lowest standard deviations in derived parameters can be shown to be

$$w(hkl) = 1 / \sigma^2(hkl) \tag{43}$$

where σ^2 is the variance for each Fo . As σ^2 is not normally known it is common for a weight, depending on some function of Fo , to be used.

If from the $|Fc|$ there are $p_1, p_2 \ldots, p_n$ parameters to be determined, then, for M to be a minimum we must have $\partial M / \partial p_j = 0$ for $j = 1, 2 \ldots n$, that is,

$$\sum_{hkl} w \Delta \frac{\partial |Fc|}{\partial p_j} = 0 \tag{44}$$

where $\Delta = |Fo| - |Fc|$. For a trial set of p_j, not too far from the correct value, Δ may be expanded as a function of the parameters by a Taylor series of the first order. Stopping the series after two terms we obtain

$$\Delta(\underline{p} + \underline{e}) = \Delta(\underline{p}) - \sum_{i=1}^{n} e_i \frac{\partial |Fc|}{\partial p_i} \tag{45}$$

where e_i is a small change in a parameter p_i , and $\underline{p}$ and $\underline{e}$ are the whole set of parameters and changes. Substituting (45) into (44) we obtain the Normal Equations

$$\sum_{i=1}^{n} \left\{ \sum_{hkl} w \frac{\partial|Fc|}{\partial p_i} \cdot \frac{\partial|Fc|}{\partial p_j} \right\} e_i = \sum_{hkl} w \Delta \frac{\partial|Fc|}{\partial p_j} . \tag{46}$$

There are thus n equations which have to be set up and solved to find the n unknown parameters. It is more convenient to express the normal equations in matrix form as

$$\sum_{i} a_{ij} e_i = b_j \tag{47}$$

where

$$a_{ij} = \sum_{hkl} w \frac{\partial |Fc|}{\partial p_i} \cdot \frac{\partial |Fc|}{\partial p_j} \tag{48}$$

and

$$b_j = \sum_{hkl} w \Delta \frac{\partial|Fc|}{\partial p_j} . \tag{49}$$

The solution of (47) is

$$e_j = \sum_{i=1}^{n} (a^{-1})_{ij} b_i \tag{50}$$

where (a^{-1}) is the matrix inverse to a .

As the matrices which have to be used are generally large it can be difficult to retain the whole matrix in a computer high speed store. It is then customary to make an approximation to the complete matrix by setting up blocks of normal equations involving only the differentials of parameters of individual atoms. In the program to be described in Part II of this thesis a chain of 4×4 blocks involving an isotropic thermal and three positional parameters can be employed in this way.

Owing to the omission of higher terms in the Taylor series for Δ it is necessary to calculate several cycles of refinement before a minimum of M is obtained. The course of a refinement may be followed by the change in M or by the function

$$R' = \sum w \Delta^2 / \sum w |Fo|^2 . \qquad (51)$$

The value $(R')^{1/2}$ has a value similar to that of the usual discrepancy factor (41) which is also a useful indication of the state of refinement as it is not affected by changes in the weighting scheme.

1.8 Accuracy of Results

At the completion of a structure refinement it is necessary to know the accuracy with which molecular parameters have been determined so that meaningful comparisons between different structures can be drawn. For the relative weights normally employed the variance of a parameter, p_i, can be obtained from the inverse matrix of the normal equations and is

$$\sigma^2(p_i) = (a^{-1})_{ii} \left(\sum w \Delta^2\right) / (m - n) \qquad (52)$$

where m is the number of observations and n is the number of parameters. The covariance of parameters p_i and p_j can also be estimated as

$$\begin{aligned} \mathrm{cov}(p_i, p_j) &= (a^{-1})_{ij} \left(\sum w \Delta^2\right) / (m - n) \\ &= \sigma^2(p_i)\, \sigma^2(p_j)\, r_{ij} \qquad (53) \end{aligned}$$

where r_{ij} is the correlation coefficient between the two parameters. The above formulae are valid only if the weights are appropriate to the data, a test for this is that $w\Delta^2$ has constant averages in groups, say of increasing $|Fo|$ or $\sin\theta/\lambda$. From statistical methods and using equations (52) and (53) the standard deviations of the required molecular parameters can be established.

When comparing an experimental value x, with standard deviation s, based on n degrees of freedom, to a theoretical value p we define

$$t = (x - p) / s . \tag{54}$$

This is a random variable having a Student distribution with n degrees of freedom. From tables of this distribution (Fisher and Yates, 1953) the probability that x differs from its theoretical or expected value, p, because of random experimental errors may be determined from the value of t. When n is large the distribution is normal and for the 1 % and 0.1 % significance points $t = 2.6$ and 3.3 respectively.

If the goodness of fit of a set of experimental results to their expected values is under consideration the χ^2 test may be used. If d_i is the deviation of the i^{th} of a set of n points from the least-squares plane through the points, then

$$\chi^2 = \sum_{i=1}^{n} (d_i / \sigma)^2 \tag{55}$$

where σ is the average positional standard deviation of a point. The probability that deviations from the plane are caused by random experimental errors can then be found by looking up tables of χ^2 for $n - 3$ degrees of freedom.

PART II

CRYSTALLOGRAPHIC COMPUTER PROGRAMS.

1.1 Introduction

Immediately after the work for this thesis started Glasgow University's DEUCE computer was replaced by an English Electric KDF 9. As no programs were available this created the opportunity for developing an integrated suite of programs for general crystallographic use on the KDF 9 computer.

The current set of programs, called the ASS System (for Automatic Structure Solution), is now in use in several other universities. It is capable, at present, of proceeding from the initial data reduction to a complete isotropic structure-factor least-squares refinement. The following sections give a short description of the system and list some of the author's contributions to it.

1.2 The ASS System

The ASS system is basically a data handling procedure whereby selected information can be obtained by the crystallographer to determine the course of an analysis. The work in setting up the system was carried out in collaboration with Dr. K. W. Muir and Dr. D. R. McGregor who also developed the automatic Fourier refinement aspect. Several other programs have been adapted, notably Dr. J. G. Sime's Fourier program, and various other people have contributed programs.

In the system crystallographic data is treated as belonging to one of two types,

(1) Permanent Information which does not change throughout the analysis, for example unit cell dimensions, space group symmetry, chemical elements present with their scattering factors, and experimental reflection data.

(2) Temporary Information relating to the proposed model at the time, for example atomic coordinates and thermal parameters, structure factors, electron density maps and peak coordinates.

All of this information is stored on a magnetic tape acting as a "file", the system allows only one set of type (2) data to be retained at any one time, the temporary data is thus overwritten when newer data becomes available. Data storage was accomplished by the simple approach of defining rigorously the arrangement of the arrays on magnetic tape. This method is not so general as that given in the "Index of Lists" compiled by Cruickshank, Freeman, Rollet, Sime, Smith, Truter and Wells (1964) but as the programmers were working together a loss in generality was no disadvantage and simplified the programs considerably.

The main function of the ASS is that it can carry out cycles of calculations consisting of structure factors, Fourier coefficient weighting, Fourier summation and peak searching, with little help from the crystallographer. Two programs, which are derived from earlier programs of Dr. K. W. Muir, will be described here as belonging to this aspect and a further two programs will be discussed later.

1.2.1 Coefficient Weighting Program

In the typical application of the heavy-atom method in a centrosymmetrical case, the fraction of correct phases determined by the heavy atom alone is dependent on the ratio of the sums of the squares of the scattering factors of the heavy and light atoms, a parameter r thus defined is

$$r = \left(\sum_{H} f^2 / \sum_{L} f^2\right)^{1/2}. \qquad (1)$$

In a Fourier synthesis derived from structure factors based on a heavy atom only, to minimise the errors caused by wrong phases, a weight should be assigned to each term, F, according to the probability that the sign of F is the same as the sign of F_H (Woolfson, 1956). This probability is given by

$$P = 1/2 + 1/2 \tanh (|F| |F_H| / \sum f_L^2) \qquad (2)$$

and the weight to be applied to the term is

$$w = 2P - 1 . \qquad (3)$$

The same argument applies for the non-centrosymmetrical case except that the probability is defined in terms of a parameter X where

$$X = 2 |F| |F_H| / \sum f_L^2 \qquad (4)$$

and the weight is given by

$$w = I_1(X) / I_0(X) \qquad (5)$$

where I_0 and I_1 are the zero-order and first-order modified Bessel functions of the first kind (Sim, 1961).

The weighting program was developed from an earlier version, written by Dr. K. W. Muir, which was only applicable for the non-centrosymmetrical case and used paper tape input and output. In the present program, structure factor data is accepted from a magnetic tape in the ASS format, contributions to $\sum f_L^2$, that is atoms omitted from the structure factor calculation, are determined and corrected for thermal vibration by applying an overall isotropic thermal parameter. A check is now made to decide which of the two weighting functions is to be used and if (3) is applicable the weight is calculated directly. If (5) has to be used a Chebyshev series is entered for values of X less than five and for values over five a simple equation is employed. The weighted F obs value is now transferred to a magnetic work tape and when all the structure factor data has been processed the intermediate values on the work tape are scaled and returned to the file tape for subsequent use by the Fourier program.

The program has been in general use for some years and at Glasgow it has been applied routinely in about thirty analyses.

1.2.2 Analysis of Structure Factors

This program gives a complete analysis of calculated structure factor data by magnitude of F obs, sin θ / λ, reciprocal lattice net or batch of reflections, and by various combinations of indices. A special analysis for small values of F obs or "unobserved" data is also incorporated. In each section totals of |F obs|, |F calc|, Δ, Δ^2 and number of reflections are collected and printed together with values of R, average Δ and average Δ^2. In the analysis by F obs and sin θ / λ variable limits can be set and for that by indices combinations of h, k, l, h + k, k + l, h + l, h + k + l either odd or even are determined.

In both the above programs the time per run is negligible and limited only by the peripheral transfer rate.

1.3 Isotropic Structure Factor Least Squares (ILS)

With the initial version of the ASS in operation it became apparent that a fast simplified structure-factor least-squares program was required. The Cruickshank Glasgow SFLS program then available, although completely general, proved inefficient in machine time for large structures, required lengthy paper tape input and if a subsequent Fourier summation was contemplated a large quantity of paper tape had to be output (with associated punching errors). As the ASS was designed as a data handling system an integrated least-squares program, which was magnetic tape oriented in data input and output, could easily be developed. By limiting the program to isotropic refinement only and simplifying the logic a useful increase in speed over the Cruickshank program was envisaged (a factor of three for comparable calculations is normally obtained).

The ILS program is designed to calculate structure factors and accumulate least-squares totals which are later solved for the parameter changes. The parameters which can be varied are, an overall scale for $|F\ obs|$ or batch scale factors, atomic coordinates, individual atomic isotropic vibration parameters U. The program is designed to work for most space groups by using the relevant expressions for

P1 or P$\bar{1}$, but as yet positions such as (x, 2 x, z) in a hexagonal space group can not be dealt with. The least-squares totals accumulated are for either

(1) a matrix involving scale factors and an overall vibration parameter U, 4 × 4 matrices for individual isotropic U and atomic coordinates.

(2) a full matrix calculation,

the choice depending on the store limit of the computer.

A number of options are available in the program, specified atoms can be left out of the least-squares calculations, any given shift factor can be applied, several cycles of calculations can be carried out with the new parameters and their estimated standard deviations being printed after every cycle, the printing of the calculated structure factors can be suppressed. Three weighting schemes are available,

i $w = 1$,
ii $w = (A + |Fo| + B|Fo|^2 + C|Fo|^3)^{-1}$,
iii $w = (1 + [(|Fo| - B) / A]^2)^{-1}$.

There is virtually no limit on the number of F obs which may be included as these are processed one at a time as the calculation proceeds. Symmetry related atoms are dealt with by rotation matrices and translation vectors; instead of transforming the coordinates, however, the equivalent transformations for the indices are made. With atoms in special positions an occupation number can be set to scale the formfactor to give the correct contribution to the structure factor.

ILS is a one segment program written in ALGOL for a KDF 9 computer. For a 16K machine a large number of atoms, of any type, may be accomodated for the block diagonal approximation. A limit of about 120 parameters is demanded in the full matrix case with the limit increased for a machine of larger store size. Input to the program consists of a small paper tape containing a series of keys, not normally altered in the analysis, and a list of atomic parameters and scale factors to be refined. Output consists of a new parameter tape and various printed results. The calculated structure factors and new parameters are written onto the magnetic file tape ready for Fourier or Molecular Geometry calculations. For ten atoms in a centrosymmetric monoclinic space group with 1000 F obs the time required for a block diagonal calculation is 3 minutes 37 seconds and for a full matrix calculation 8 minutes 24 seconds of which approximately 2 minutes are required for the matrix inversion and solution (the ALGOL procedure used is very inefficient). The program currently in use has been modified so that the setting up of the normal equations is now accomplished by a machine-code procedure written by Mr. W. Bowie. With this modification the times taken for the above calculations are 2 minutes 52 seconds and 5 minutes 10 seconds respectively.

ILS has now been in use since 1966 and most structures solved in Glasgow since then have been refined to typical R values of 0.12 - 0.15 before proceeding to a full anisotropic refinement. The program was used in all the analyses reported in this thesis.

1.4 Anisotropic Structure Factor Program (ASF)

ASF was developed to ease the final calculations of electron density and difference syntheses after an anisotropic refinement. The program is again magnetic tape oriented and, apart from a list of atomic parameters input on paper tape, works from a standard ASS file tape.

ASF is a structure factor program handling any combination of atoms with isotropic or anisotropic thermal parameters as for space groups $P1$ or $P\bar{1}$. The program processes one reflection at a time with transformations made to the indices and not the coordinates, symmetry and special positions are dealt with as in the ILS program. Printed output is designed to give the crystallographer as much information as possible, thus full unit cell data, symmetry information, rotation matrices and translation vectors, and all atomic coordinates and thermal parameters are listed at the start of the calculation. Structure factors can be either printed or omitted and at the conclusion an analysis of the structure factors by reciprocal lattice net is provided.

The program was not written to be particularly efficient and consequently does not attain the predicted speed of the computer. A typical run with ten atoms, all anisotropic, in a centrosymmetrical monoclinic space group with 1000 F obs terms takes 2 minutes 37 seconds. As an example of the KDF 9 ALGOL used in the programs described, the text of both ILS and ASF is given in Appendix I.

PART III

CRYSTAL STRUCTURE DETERMINATIONS.

CHAPTER 1

Anthracene, 4-Bromo-2,5,7-trinitrofluorenone complex.

1.1 Introduction

The term "polarisation bonding", first used by McKeown, Ubbelohde and Woodward (1951), is now employed to describe the intermolecular attraction found in certain molecular complexes. Consideration of the class of molecular complexes in which the colour of the compound is deeper than that of the components gives rise to two main groups. The first group includes compounds of benzoquinones with aromatic hydrocarbons, amines or phenols, the quinhydrones being representative, and the second group molecular complexes of nitro-compounds with unsaturated hydrocarbons or their derivatives, picrates being examples.

The bonding involved in these compounds was postulated, first by Moore, Shepherd and Goodall (1931) to be a form of covalent link formation, then by Briegleb (1937) to be the result of electrostatic interactions. Hammick and Yule (1940), and Gibson and Loeffler (1940) suggested a polarisation mechanisation similar to an oxidation-reduction reaction for formation of compounds of aromatic amines and nitro-compounds. Weiss (1942) extended this idea to cover the whole group of molecular compounds by assuming an essentially ionic complex was formed by a complete electron transfer from the unsaturated hydrocarbon or its derivative (donor A) to the quinone or polynitro compound (acceptor B) according to the reaction

$$A + B \rightleftharpoons [\overset{+}{A}][\overset{-}{B}] \quad .$$

Mulliken (1952), and Nakamoto (1952) interpreted complex formation as a charge transfer process involving π- orbital overlap, stabilisation depending on overlap of the highest filled orbital of the donor and lowest unfilled orbital of the acceptor, and thus on the symmetry and orientation of the molecules. Structural features derived from this were discussed by Mulliken (1952) and by Harding and Wallwork (1953). More recently the charge-transfer spectra of some complexes has been interpreted in terms of molecular orbital theories by Dewar and Lepley (1961) and Lepley (1962).

Complexes of 2,4,7-trinitrofluorenone (Orchin and Woolfolk, 1946) and 2,4,5,7-tetranitrofluorenone (Newman and Lutz, 1956) have been prepared and studied on account of their stability and good complexing properties, the steric effects of this complex formation being discussed by Orchin (1951). More recently 4-bromo-2,5,7-trinitrofluorenone (BTNF) was prepared by Newman and Blum (1964) and this also proved to be a good complexing agent. As several complexes of BTNF were available this present work was started with three objects in view: to see if BTNF was suitable for general use as a complexing agent in other X-ray studies, to determine the bonding involved in BTNF complex formation, and to study the molecular overcrowding in BTNF itself. To do this two molecular complexes have been investigated; in both cases the structure of the complexed entity was well established from previous work. The complexed molecules were anthracene and 1,12-dimethyl-3,4-benzophenanthrene.

1.2 Experimental

Crystal data

Anthracene : 4-Bromo-2,5,7-trinitrofluorenone.

$C_{14}H_{10}$: $C_{13}H_4N_3O_7Br$.

F.W. = 574.8 , M.Pt. = 208 - 209 °C .

Monoclinic, a = 6.95 ± 2 , b = 11.58 ± 2 , c = 29.26 ± 4 Å

$\beta = 98^\circ 40' \pm 20'$.

U = 2328 Å^3 , D_m = 1.62 gm.cm.$^{-3}$, Z = 4 , D_x = 1.64 gm.cm.$^{-3}$

Linear absorption coefficient μ = 31.8 cm.$^{-1}$ (Cu K_α).

F(000) = 1152.

Systematic absences,

0k0 when k is odd,

h0l when l is odd.

Space group $P2_1/c$ (C_{2h}^5 , No. 14).

The crystals were supplied by Professor M. S. Newman and consisted of small dark brown needles elongated along the a axis. Cell dimensions were determined from rotation and Weissenberg photographs, taken about the a axis with Cu K_α radiation (λ = 1.5418 Å), and from a precession photograph of the h0l zone taken with Mo K_α radiation (λ = 0.7107 Å).

Intensity data were estimated visually from multiple-film Weissenberg photographs of the reciprocal lattice nets 0kl to 5kl with four films per pack (Robertson, 1943) using the interfilm scale factors of Rossman (1956). Lorentz, polarisation and rotation factors (Tunell, 1939) were applied by the computer program written by A. A. Hock, M. R. Truter and M. Wells, absorption corrections were not considered necessary as care had been taken to select a small crystal. The resulting 1977 structure amplitudes, corresponding to some 37 % of the data accessible to Cu K_α radiation, were put on an approximately absolute scale by comparison with the first set of structure factors. The final scale factors were determined by the least-squares method and no unobserved reflections were used in the analysis.

1.3 Structure Solution and Refinement

In the space group $P2_1/c$ there are four equivalent positions, ± (x, y, z) , ± (x, 1/2 - y, 1/2 + z); as Z = 4 the complex molecule may be taken as the asymmetric unit. Owing to the presence of various symmetry elements, atomic vectors are concentrated on certain sections and lines of the three-dimensional Patterson function (Harker, 1936). Thus the heavy-atom vectors 2x, 2y, 2z ; 2x, 1/2, 1/2 + 2z and 0, 1/2 + 2y, 1/2 result from the centre of symmetry, two-fold screw axis and c glide respectively. From the (100) Patterson projection, Harker section at V = 1/2 , (Fig. 1.1), and line section U = 0, W = 1/2 , a consistent set of fractional coordinates for the bromine atom was found to be,

Br(1)	0.1651	0.0384	0.2452 .

The vector positions were determined by the interpolation formula of Booth (1948) and are marked on the Patterson projection.

Solution of the phase problem was attempted by use of the ASS crystallographic program system (Part II of this thesis). From the above bromine atom the system gave 24 out of the possible 37 non hydrogen light atoms in two successive cycles and lowered the residual R from 0.588 (bromine alone) to 0.469. One further cycle was sufficient to reveal the remaining atoms, and structure factors on all atoms gave $R = 0.367$. In each case a cycle of the system consisted of structure factor, electron-density and peak-searching calculations.

Inspection of the last electron-density distribution then showed that the electron density at N(2), (numbering in Fig. 1.2), was rising to over 16 e/Å^3 and at O(5) and O(4) falling to 5 and 3 e/Å^3 compared with values of 7 and 8 e/Å^3 at other nitrogen and oxygen positions. The abnormal values can be explained by considering the shape of the 4-bromo-2,5,7-trinitrofluorenone molecule (henceforth BTNF). Application of a two-fold rotation axis along the axis of the carbonyl group would give rise to a second molecule whose only distinguishing feature from the first is the interchange of the 4-bromo and 5-nitro groups. If the BTNF molecule was disordered in this manner, the electron density would rise near the Br(1) and N(2) positions, and fall at the O(5) and O(4) positions. Further inspection of the electron-density distribution showed the presence of two "lobes" of electron density near the bromine which were interpreted as the oxygen atoms of the disordered nitro group. No other atoms showed evidence of further disorder and a structure factor calculation, incorporating a 2:1 disorder ratio, gave R = 0.292 . A computer program written to calculate the degree of disorder by minimising R , gave a ratio of 0.65 : 0.35 for the interchange and a further structure factor calculation, using these values, gave R = 0.255 .

The structure was then refined by the least-squares method, using the program described in Part II of this thesis. Three positional and one thermal parameter per atom, together with individual layer scale factors, were employed. The disorder was accounted for by introducing two sets of atoms for the 4-bromo and 5-nitro positions: the first, Br(1),N(2),O(4) and O(5), was given an occupancy factor of 0.65 , and the second, Br(2),N(4),O(8) and O(9), one of 0.35 . The positions of the 0.35 nitro group were not varied in the refinement. Five cycles of the block diagonal approximation to the normal matrix, with indicated shifts multiplied by 0.75, reduced R to 0.134. The function minimised was $\sum_{i=1}^{s} w_i (|Fo| - |Fc|)_i^2$ where the sum was taken over the independent structure amplitudes and the weight for each observation, w, was given by the scheme suggested by Cruickshank et al. (1961), that is

$$w = (A + |Fo| + B |Fo|^2)^{-1}$$

with A = 2 F min and B = 2 / F max. During this initial refinement some errors in scaling and indexing were corrected, one reflection, the $21\bar{3}$, being omitted on account of the inaccuracy involved in determining its structure amplitude. One further cycle of refinement with 14 hydrogen atoms, at assumed positions, included in the structure factor calculations lowered R to 0.129.

As an anisotropic refinement was now contemplated, and as intensity data had only been collected around the a axis, degeneracy of the U_{11} thermal parameters was avoided by putting the structure amplitudes on a common scale, by applying the current refined layer scale factors, and subsequently refining an overall scale factor. Refinement was completed using the Glasgow Structure-Factor Least-Squares program (Cruickshank and Smith, 1965). A block diagonal approximation, involving 9 × 9 blocks of three positional and six anisotropic thermal parameters per atom together with a 2 × 2 block of overall scale and dummy thermal parameter, was used with indicated shifts multiplied by 0.75. Five cycles of refinement produced convergence, final values of R and R′ were 0.082 and 0.0115. The weighting scheme was

$$w = k \exp - (A + B|Fo| + C|Fo|^2 + D/s + E/s^2 + Fs),$$

where k is a constant, s is $\sin^2\theta / \lambda^2$ and A,B,C,D, E and F are fitted by a least-squares program written by

D. McGregor (1967) so $w = 1 / \langle \Delta^2 \rangle$ where $\langle \Delta^2 \rangle$ is the local average of $(k|Fo| - |Fc|)^2$. A bivariate analysis of $\sum w \Delta^2$ batched by magnitude of Fo and $\sin \theta / \lambda$ showed no significant variation over the ranges considered.

The standard deviation of the electron density $\sigma(\rho)$, estimated from the formula of Cruickshank (1949), was 0.11 e/Å^3. Deviations greater than $3\sigma(\rho)$ on a final (Fo - Fc), or difference, electron-density distribution were situated at the 4-bromo and 5-nitro positions, indicating, perhaps, that not sufficient account had been taken of the disorder. The hydrogen positions were confirmed from a low order difference map ($\sin \theta / \lambda$ less than 0.355 , Jellinek, 1958); all 14 were found with peak values ranging from 0.3 to 0.5 e/Å^3.

Atomic formfactors used were those of Freeman (1959) for carbon, Freeman and Watson (1962) for bromine, and Berghuis et al.(1955) for oxygen and nitrogen. The numbering scheme adopted with reference to the 0.65 component and the final electron-density distribution, shown by means of superimposed contour sections drawn parallel to (100), are given in Figs. 1.2 and 1.3 .

The packing of the molecules is shown in Fig. 1.4 and views of the "sandwich" of molecules formed in Figs. 1.5

and 1.6 . Atomic parameters of the model are given in Tables 1.1 to 1.4 and refer to one "sandwich" of the molecules; final observed and calculated structure factors together with an analysis by magnitude of Fo and reciprocal lattice net in Tables 1.5 and 1.6 ; bond lengths, interbond angles, interatomic distances and selected mean planes in Tables 1.7 to 1.10 . Estimated standard deviations when quoted are in units of the last decimal place and were determined from the inverse matrix of the normal equations.

DIAGRAMS AND TABLES.

COURSE OF ANALYSIS.

I Fourier refinement.

		R
1	Br alone.	0.588
2	Br + 15 other atoms.	0.510
3	Br + 24 other atoms.	0.469
4	Br, 3 N, 7 O, 27 C.	0.367
5	As above, 2:1 disorder.	0.292
6	As above, 0.65:0.35 disorder.	0.255

II Least-squares refinement.

Cycles of refinement	Final R	Final R dash	*
1 - 5	0.134	-	(a)
6	0.129	-	(b)
7 - 11	0.082	0.0115	(c)

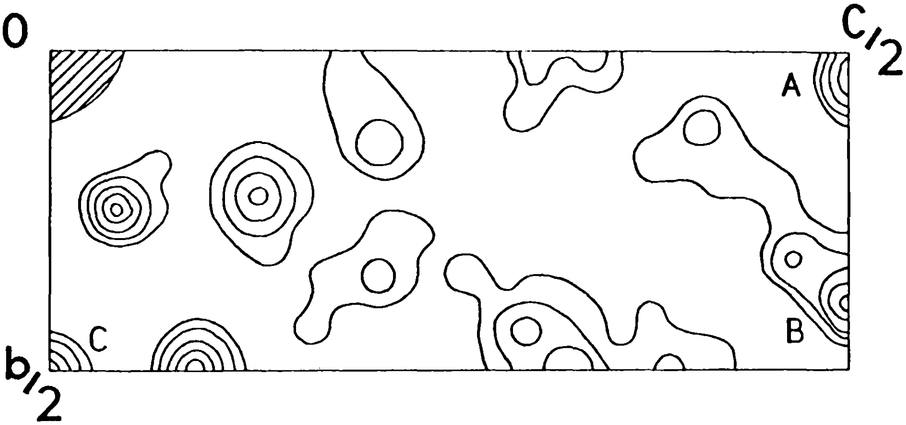
* (a) isotropic, individual layer scale factors.

(b) as above, 14 hydrogens included but not refined.

(c) anisotropic, one overall scale factor, weighting scheme changed.

FIG. 1.1

The Patterson projection on to (100) and Harker section at $V = 1/2$. Contours are at arbitrary intervals and peaks corresponding to vectors between bromine atoms are marked at A,B and C.


0
c/2
A
B
C
b/2

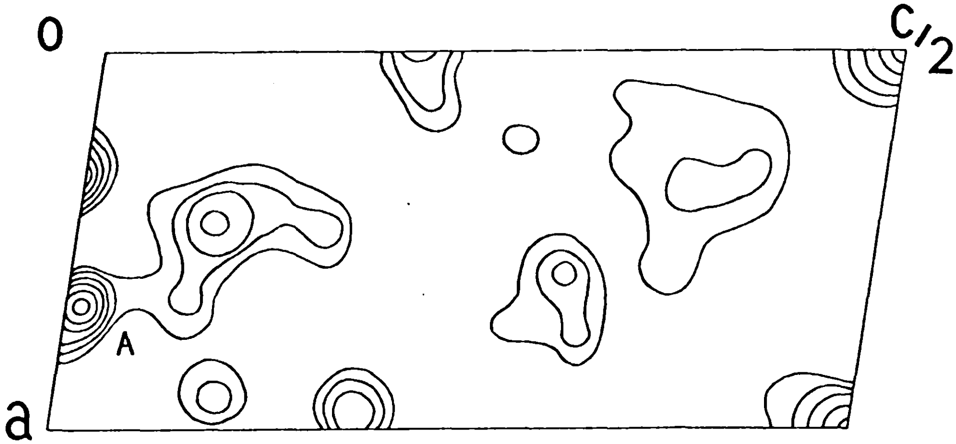

0
c/2
A
a

FIG. 1.2

Molecular drawing showing the numbering of the heavier atoms.

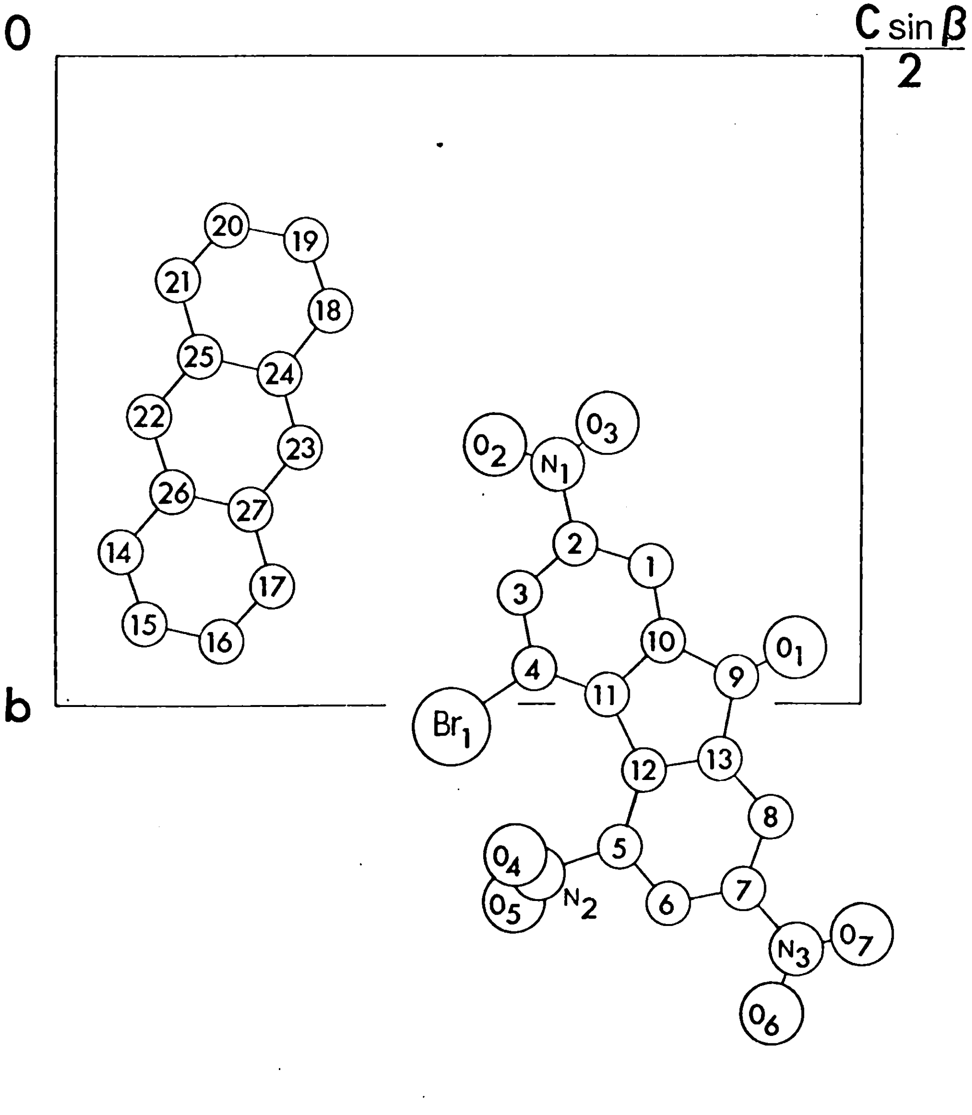

0
C sin β / 2
b
20
19
21
18
25
24
22
23
26
27
14
17
15
16
O2
O3
N1
2
1
3
10
O1
4
11
9
Br1
12
13
8
O4
5
O5
N2
6
7
N3
O7
O6

FIG. 1.3

Composite final electron-density synthesis projected on (100). Contours are at levels of 1 $e/\mathring{A}^3$ starting from 1 $e/\mathring{A}^3$ except around the bromine atom which is contoured at 4 $e/\mathring{A}^3$ intervals.

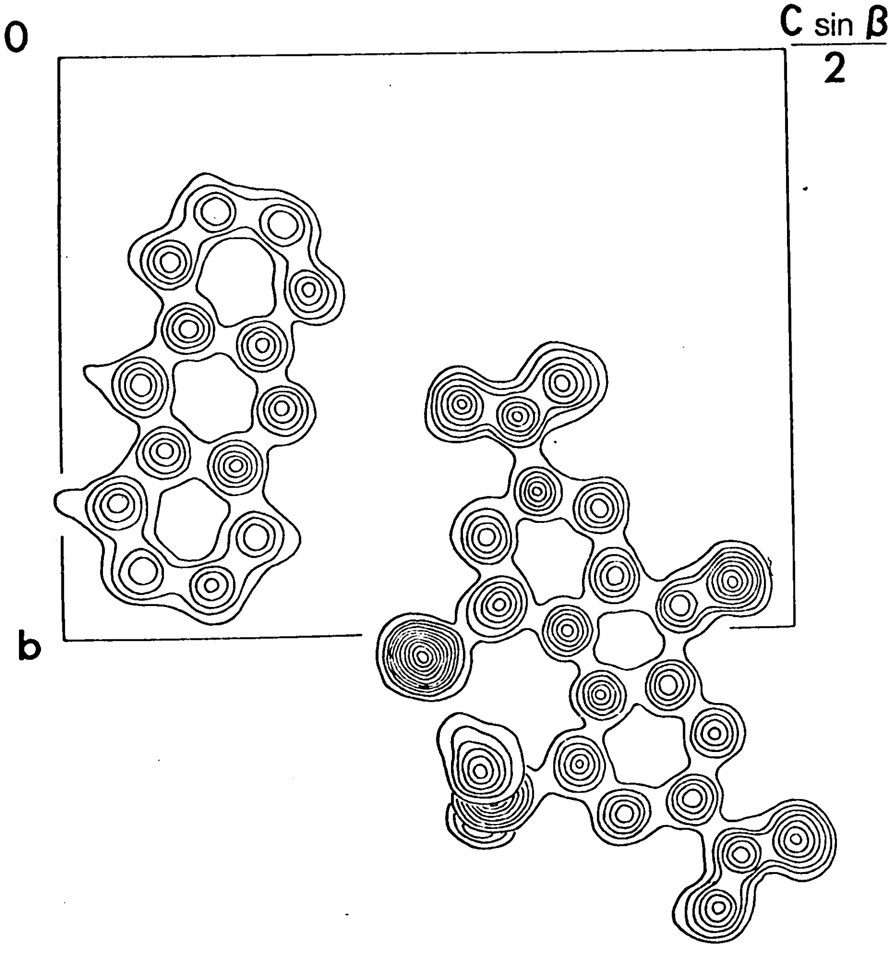

0
$\frac{C \sin \beta}{2}$
b

FIG. 1.4

The molecular packing viewed down the (100) projection. (For clarity only the contents of one half of the unit cell are shown.)

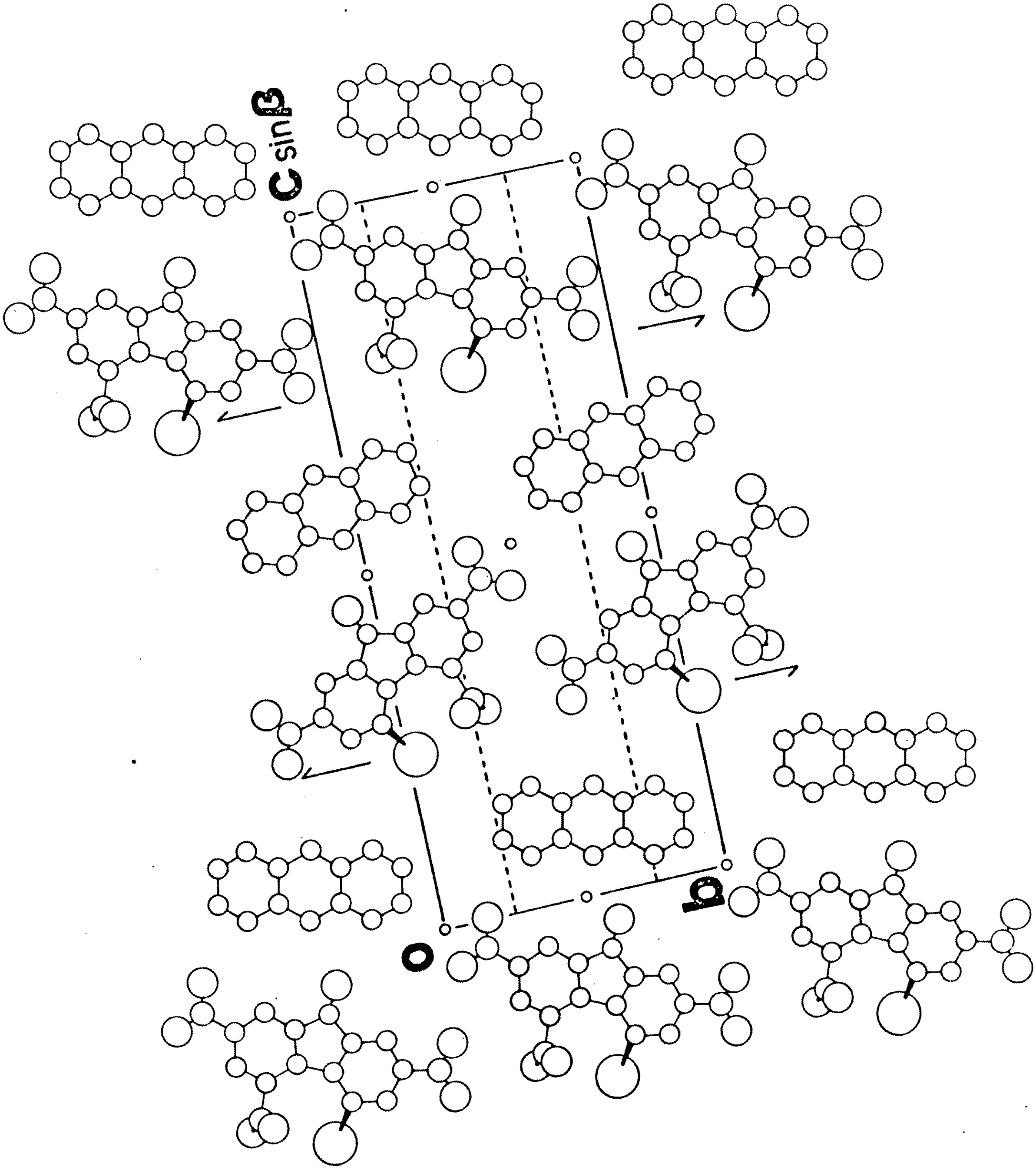

c sinβ
b
o

FIG. 1.5

Arrangement of the molecules showing overlap viewed down (100).

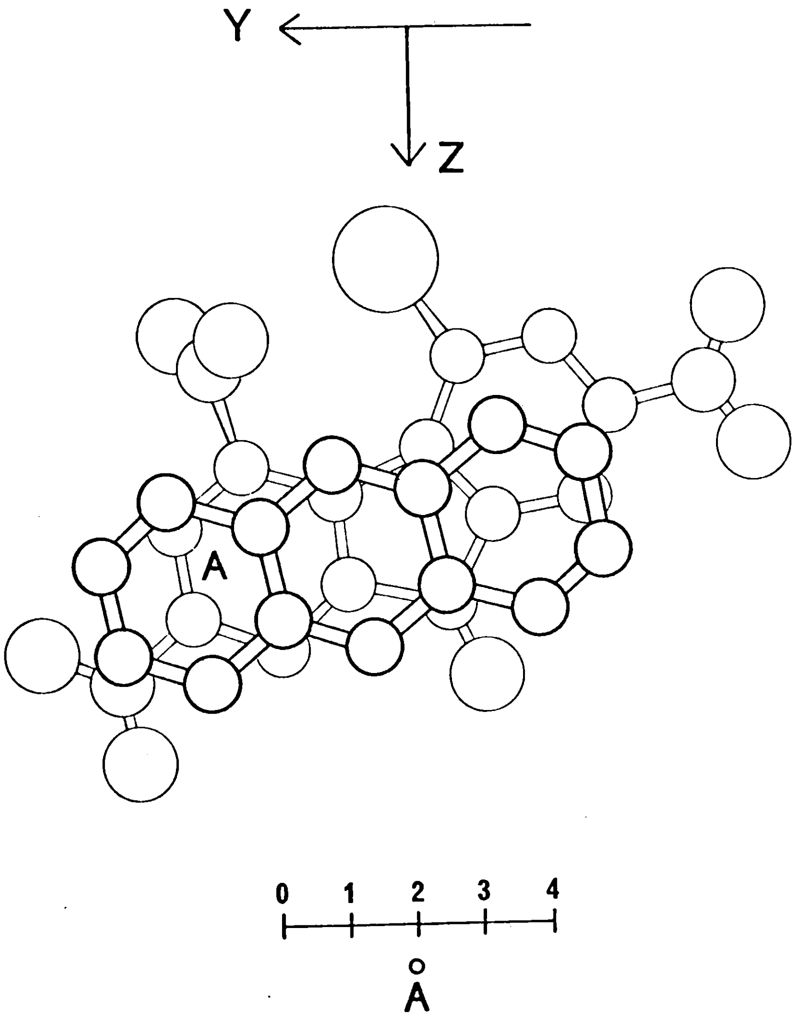

Y
Z
A
0
1
2
3
4
Å

FIG. 1.6

Arrangement of the molecules showing overlap viewed down (CC1).

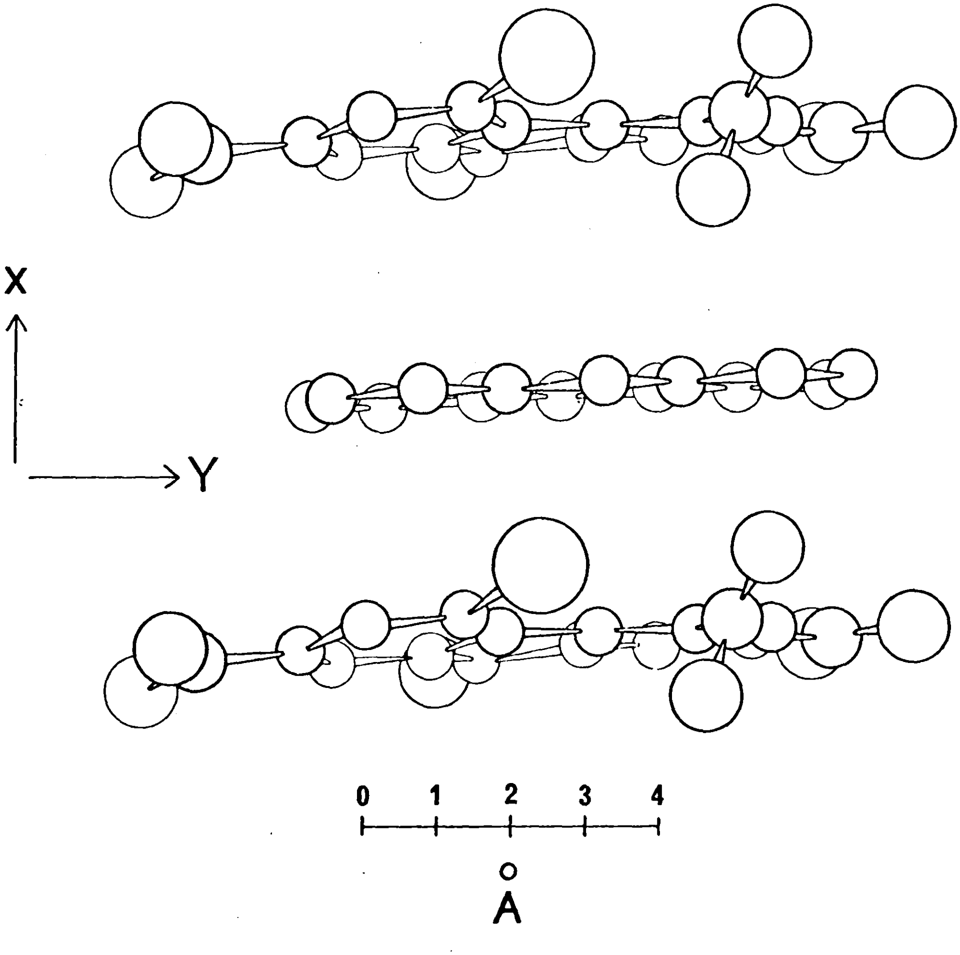

X
Y
0
1
2
3
4
Å

TABLE 1.1

FRACTIONAL COORDINATES AND E.S.D.S.

(A) 4 - BROMO 2,5,7 - TRINITROFLUORENONE.

ATOM	X/a	Y/b	Z/c
Br(1)	0.16225 ± 24	1.03383 ± 14	0.24501 ± 5
Br(2)	0.33418 ± 44	1.26565 ± 26	0.28833 ± 9
O(1)	0.3768 ± 9	0.9116 ± 6	0.4586 ± 2
O(2)	0.3346 ± 11	0.5978 ± 6	0.2714 ± 3
O(3)	0.4177 ± 15	0.5648 ± 7	0.3420 ± 3
O(4)	0.4243 ± 17	1.2301 ± 12	0.2853 ± 4
O(5)	0.1254 ± 16	1.3029 ± 11	0.2847 ± 4
O(6)	0.2789 ± 13	1.4789 ± 6	0.4440 ± 3
O(7)	0.3172 ± 12	1.3576 ± 7	0.5010 ± 2
N(1)	0.3597 ± 10	0.6286 ± 7	0.3107 ± 3
N(2)	0.2624 ± 17	1.2631 ± 10	0.3004 ± 4
N(3)	0.2999 ± 11	1.3800 ± 7	0.4601 ± 3
C(1)	0.3599 ± 11	0.7857 ± 7	0.3677 ± 3
C(2)	0.3358 ± 11	0.7518 ± 7	0.3218 ± 3
C(3)	0.2849 ± 10	0.8274 ± 8	0.2863 ± 3
C(4)	0.2630 ± 10	0.9447 ± 7	0.2956 ± 3
C(5)	0.2862 ± 11	1.2198 ± 7	0.3501 ± 3
C(6)	0.2850 ± 11	1.3081 ± 7	0.3804 ± 3
C(7)	0.3025 ± 11	1.2844 ± 8	0.4270 ± 3

C(8)	0.3272 ± 12	1.1727 ± 8	0.4438 ± 3
C(9)	0.3525 ± 11	0.9585 ± 8	0.4214 ± 3
C(10)	0.3446 ± 10	0.9025 ± 8	0.3757 ± 3
C(11)	0.3009 ± 11	0.9866 ± 7	0.3410 ± 3
C(12)	0.2958 ± 10	1.1033 ± 7	0.3644 ± 3
C(13)	0.3224 ± 11	1.0853 ± 7	0.4116 ± 3

(B) ANTHRACENE.

C(14)	-0.1905 ± 13	1.2647 ± 9	0.4613 ± 3
C(15)	-0.1979 ± 15	1.3749 ± 9	0.4461 ± 4
C(16)	-0.2158 ± 14	1.4015 ± 9	0.3992 ± 4
C(17)	-0.2149 ± 13	1.3157 ± 8	0.3678 ± 3
C(18)	-0.1798 ± 13	0.8947 ± 10	0.3318 ± 3
C(19)	-0.1593 ± 14	0.7841 ± 9	0.3465 ± 4
C(20)	-0.1401 ± 14	0.7604 ± 8	0.3951 ± 4
C(21)	-0.1425 ± 13	0.8454 ± 8	0.4258 ± 3
C(22)	-0.1674 ± 12	1.0549 ± 9	0.4437 ± 3
C(23)	-0.2013 ± 12	1.1076 ± 8	0.3499 ± 3
C(24)	-0.1819 ± 11	0.9895 ± 8	0.3638 ± 3
C(25)	-0.1655 ± 11	0.9623 ± 8	0.4124 ± 3
C(26)	-0.1853 ± 11	1.1714 ± 8	0.4291 ± 3
C(27)	-0.1995 ± 11	1.1977 ± 7	0.3809 ± 3

TABLE 1.2

ORTHOGONAL COORDINATES AND E.S.D.S.

(A) 4 - BROMO 2,5,7 - TRINITROFLUORENONE.

ATOM	X'	Y	Z'
Br(1)	1.115 ± 2	11.975 ± 2	6.998 ± 1
Br(2)	2.297 ± 3	14.660 ± 3	8.085 ± 3
O(1)	2.590 ± 6	10.559 ± 7	13.022 ± 5
O(2)	2.300 ± 7	6.925 ± 8	7.589 ± 7
O(3)	2.871 ± 10	6.542 ± 8	9.569 ± 8
O(4)	2.917 ± 11	14.248 ± 13	7.904 ± 11
O(5)	0.862 ± 11	15.091 ± 13	8.199 ± 11
O(6)	1.917 ± 9	17.130 ± 8	12.699 ± 8
O(7)	2.180 ± 8	15.725 ± 8	14.326 ± 7
N(1)	2.473 ± 7	7.282 ± 8	8.714 ± 8
N(2)	1.804 ± 11	14.630 ± 12	8.513 ± 12
N(3)	2.062 ± 8	15.985 ± 8	13.146 ± 8
C(1)	2.474 ± 8	9.101 ± 8	10.381 ± 8
C(2)	2.308 ± 7	8.708 ± 8	9.064 ± 8
C(3)	1.958 ± 7	9.584 ± 9	8.078 ± 7
C(4)	1.808 ± 7	10.942 ± 8	8.372 ± 7
C(5)	1.967 ± 7	14.128 ± 8	9.944 ± 8
C(6)	1.959 ± 8	15.151 ± 8	10.831 ± 8
C(7)	2.079 ± 8	14.877 ± 9	12.175 ± 9

C(8)	2.249 ± 8	13.583 ± 10	12.640 ± 8
C(9)	2.423 ± 8	11.102 ± 9	11.960 ± 7
C(10)	2.368 ± 7	10.453 ± 9	10.632 ± 7
C(11)	2.068 ± 7	11.428 ± 8	9.663 ± 7
C(12)	2.033 ± 7	12.780 ± 8	10.352 ± 7
C(13)	2.216 ± 7	12.571 ± 8	11.705 ± 7

(B) ANTHRACENE.

C(14)	-1.309 ± 9	14.649 ± 11	13.695 ± 9
C(15)	-1.360 ± 10	15.925 ± 10	13.258 ± 12
C(16)	-1.483 ± 10	16.233 ± 11	11.905 ± 12
C(17)	-1.477 ± 9	15.240 ± 10	10.986 ± 10
C(18)	-1.236 ± 9	10.363 ± 11	9.897 ± 9
C(19)	-1.095 ± 9	9.083 ± 10	10.303 ± 12
C(20)	-0.963 ± 9	8.808 ± 10	11.707 ± 13
C(21)	-0.980 ± 9	9.792 ± 10	12.608 ± 10
C(22)	-1.150 ± 8	12.219 ± 10	13.156 ± 8
C(23)	-1.383 ± 8	12.829 ± 9	10.448 ± 8
C(24)	-1.250 ± 8	11.461 ± 9	10.835 ± 8
C(25)	-1.138 ± 8	11.146 ± 9	12.238 ± 8
C(26)	-1.274 ± 8	13.568 ± 9	12.749 ± 8
C(27)	-1.371 ± 7	13.873 ± 9	11.353 ± 8

TABLE 1.3

ANISOTROPIC TEMPERATURE PARAMETERS AND E.S.D.S.

(A) 4 - BROMO 2,5,7 - TRINITROFLUORENONE.

ATOM	U11	U22	U33	2U23	2U31	2U12
Br(1)	0.0824	0.0805	0.0574	0.0138	-0.0010	0.0121
	11	10	7	16	13	17
Br(2)	0.0835	0.0700	0.0683	0.0155	0.0241	-0.0023
	21	17	15	28	26	29
O(1)	0.0578	0.0810	0.0569	0.0276	0.0236	0.0031
	41	42	33	64	55	64
O(2)	0.0955	0.0823	0.0841	-0.0056	0.0315	0.0097
	55	47	44	80	76	80
O(3)	0.1703	0.0806	0.0927	0.0103	0.0266	0.0291
	86	55	54	87	107	105
O(4)	0.0659	0.1309	0.0963	0.0273	0.0675	0.0082
	82	100	77	149	120	142
O(5)	0.0974	0.1108	0.0727	0.0320	0.0073	0.0773
	84	92	66	127	111	141
O(6)	0.1284	0.0693	0.1107	-0.0338	0.0772	-0.0232
	68	47	58	87	100	91
O(7)	0.1168	0.0944	0.0714	-0.0448	0.0471	-0.0057
	60	54	42	79	78	90
N(1)	0.0518	0.0806	0.0715	0.0129	0.0251	0.0005
	50	54	46	82	71	80
N(2)	0.0422	0.0676	0.1038	-0.0513	0.0754	0.0004
	80	73	84	132	124	115
N(3)	0.0601	0.0743	0.0874	-0.0281	0.0528	-0.0262
	54	52	54	87	81	82
C(1)	0.0293	0.0631	0.0625	0.0231	0.0220	0.0055
	51	53	47	84	70	78

C(2)	0.0174	0.0590	0.0740	-0.0121	0.0294	-0.0096
	48	51	51	86	71	73
C(3)	0.0169	0.0715	0.0614	0.0087	0.0298	-0.0124
	46	55	46	82	66	75
C(4)	0.0190	0.0730	0.0526	0.0189	0.0179	0.0059
	47	56	42	79	64	75
C(5)	0.0265	0.0607	0.0574	0.0088	0.0233	0.0041
	49	51	45	79	66	76
C(6)	0.0363	0.0499	0.0740	0.0031	0.0260	-0.0027
	53	48	53	83	77	76
C(7)	0.0331	0.0640	0.0743	-0.0056	0.0374	-0.0068
	55	55	54	90	79	81
C(8)	0.0402	0.0787	0.0620	-0.0117	0.0217	-0.0178
	58	63	50	93	78	91
C(9)	0.0290	0.0777	0.0502	0.0015	0.0292	0.0006
	50	58	41	84	65	82
C(10)	0.0216	0.0754	0.0486	0.0122	0.0143	-0.0053
	47	55	41	80	64	78
C(11)	0.0242	0.0634	0.0541	0.0150	0.0246	-0.0033
	47	51	41	79	64	75
C(12)	0.0161	0.0682	0.0523	0.0069	0.0156	-0.0106
	46	52	42	80	63	74
C(13)	0.0256	0.0621	0.0559	0.0158	0.0305	-0.0086
	49	51	43	79	68	74

(B) ANTHRACENE.

C(14)	0.0341	0.0936	0.0793	-0.0413	0.0250	-0.0125
	58	71	59	109	84	96
C(15)	0.0592	0.0648	0.1241	-0.0602	0.0263	-0.0051
	69	64	88	122	116	100

C(16)	0.0497	0.0709	0.1273	0.0320	0.0207	0.0216
	67	65	90	129	115	101
C(17)	0.0408	0.0718	0.0898	0.0244	0.0232	0.0007
	59	61	63	107	90	93
C(18)	0.0355	0.1105	0.0740	-0.0568	0.0407	-0.0236
	58	81	58	113	85	104
C(19)	0.0454	0.0706	0.1255	-0.0480	0.0427	-0.0196
	65	65	84	124	108	97
C(20)	0.0356	0.0612	0.1526	-0.0018	0.0416	-0.0157
	61	60	100	130	114	91
C(21)	0.0471	0.0690	0.0892	0.0070	0.0417	-0.0042
	60	60	64	106	93	92
C(22)	0.0330	0.0900	0.0614	0.0061	0.0230	-0.0188
	56	70	49	94	75	90
C(23)	0.0327	0.0753	0.0598	0.0063	0.0339	-0.0236
	53	58	48	87	72	87
C(24)	0.0195	0.0828	0.0635	-0.0194	0.0316	-0.0215
	49	60	47	89	69	81
C(25)	0.0319	0.0645	0.0685	0.0101	0.0407	-0.0106
	53	54	50	89	76	81
C(26)	0.0265	0.0755	0.0593	-0.0132	0.0323	0.0028
	51	59	47	87	69	81
C(27)	0.0208	0.0627	0.0695	0.0124	0.0182	-0.0040
	49	53	50	84	69	75

The thermal parameters are values of U_{ij} ($\mathring{A}^2$) in the expression

$$\exp\left[-2\pi^2 \left(U_{11} h^2 a^{*2} + U_{22} k^2 b^{*2} + U_{33} l^2 c^{*2} + 2U_{23}\, k\, l\, b^*c^* + 2U_{31}\, l\, h\, c^*a^* + 2U_{12}\, h\, k\, a^*b^* \right)\right]$$

TABLE 1.4

(A) ASSUMED TEMPERATURE FACTORS AND FRACTIONAL COORDINATES OF THE HYDROGEN ATOMS.

NUMBERS REFER TO THE CARBON ATOM TO WHICH THE HYDROGEN IS BONDED.

ATOM	X/a	Y/b	Z/c	U iso
H(1)	0.3941	0.7236	0.3962	0.0562
H(3)	0.2582	0.7945	0.2511	0.0514
H(6)	0.2673	1.3956	0.3673	0.0548
H(8)	0.3471	1.1546	0.4807	0.0626
H(14)	-0.1782	1.2454	0.4981	0.0690
H(15)	-0.1936	1.4433	0.4721	0.0840
H(16)	-0.2282	1.4918	0.3878	0.0845
H(17)	-0.2315	1.3376	0.3308	0.0758
H(18)	-0.2015	0.9115	0.2941	0.0701
H(19)	-0.1598	0.7125	0.3219	0.0842
H(20)	-0.1198	0.6715	0.4081	0.0890
H(21)	-0.1255	0.8234	0.4622	0.0678
H(22)	-0.1553	1.0356	0.4804	0.0641
H(23)	-0.2197	1.1253	0.3133	0.0597

(B) ASSUMED TEMPERATURE FACTORS AND FRACTIONAL COORDINATES OF THE 0.35 OCCUPATION NITRO GROUP.

ATOM	X/a	Y/b	Z/c	U iso
O(8)	0.0553	1.0840	0.2595	0.0600
O(9)	0.2362	1.0099	0.2248	0.0600
N(4)	0.1767	1.0175	0.2580	0.0700

TABLE 1.5

FINAL OBSERVED AND CALCULATED STRUCTURE FACTORS.

H	K	L	Fo	Fc
0	0	4	31.2	-27.1
0	0	6	95.4	-107.6
0	0	8	8.3	11.1
0	0	10	102.5	-110.3
0	0	12	26.9	-20.7
0	0	14	47.1	40.9
0	0	16	21.4	22.4
0	0	18	11.6	-12.6
0	0	20	36.4	33.6
0	0	22	20.0	-16.6
0	0	24	58.1	50.7
0	0	26	15.9	-14.7
0	0	28	18.4	16.8
0	0	30	15.1	-12.8
0	0	32	11.7	10.4
0	1	1	77.4	-78.1
0	1	2	78.3	75.2
0	1	3	57.7	67.3
0	1	4	74.4	79.2
0	1	5	90.0	89.1
0	1	6	60.4	60.1
0	1	7	21.4	-20.0
0	1	8	25.8	-26.8
0	1	9	53.7	52.3
0	1	10	60.0	58.6
0	1	11	14.7	-14.4
0	1	12	46.2	-44.9
0	1	14	48.7	-45.5
0	1	15	40.5	-39.5
0	1	16	32.0	32.5
0	1	17	61.9	57.4
0	1	18	31.8	-29.8
0	1	20	35.1	28.0
0	1	21	21.4	24.0
0	1	23	20.6	-23.8
0	2	0	10.0	-14.4
0	2	1	7.0	-6.8
0	2	2	15.2	-15.3
0	2	3	114.5	113.8
0	2	4	69.2	71.9
0	2	5	15.9	13.2
0	2	6	88.4	-94.3
0	2	7	53.8	49.8
0	2	8	47.4	50.4
0	2	9	29.4	-29.8
0	2	10	109.3	-122.6
0	2	11	82.9	79.5
0	2	12	107.4	110.6
0	2	13	47.0	-47.1
0	2	14	61.8	-70.2
0	2	16	27.7	31.7
0	2	17	33.3	-35.2
0	2	18	43.7	-39.9
0	2	19	10.1	9.9
0	2	20	35.6	34.4
0	2	21	22.5	-23.8
0	2	22	6.4	-5.6
0	2	23	20.3	-21.6
0	2	24	11.6	7.9
0	2	25	14.4	13.0
0	2	26	6.8	-4.5
0	2	28	11.6	-12.1
0	3	1	19.1	-19.9
0	3	2	53.9	-50.9
0	3	3	9.2	-7.5
0	3	4	113.1	101.9
0	3	5	29.7	-29.1
0	3	6	96.9	-104.5
0	3	7	21.8	-18.2
0	3	8	54.8	63.6
0	3	9	33.4	-31.8
0	3	10	40.7	-46.2
0	3	11	47.3	-46.5
0	3	12	29.0	27.0
0	3	13	27.7	28.2
0	3	14	5.0	-6.6
0	3	15	20.2	-19.5
0	3	16	11.5	-11.8
0	3	17	25.8	25.8
0	3	18	10.1	10.3
0	3	19	10.4	-10.8
0	3	20	12.4	-14.6
0	3	22	26.9	26.0
0	3	23	21.1	-23.8
0	3	24	23.4	23.3
0	3	26	12.7	-13.4
0	3	27	9.5	-8.7
0	4	0	76.1	66.4
0	4	1	106.4	-105.0
0	4	2	98.7	-105.3
0	4	3	91.4	100.0
0	4	4	19.7	19.1
0	4	5	16.8	-17.7
0	4	6	50.9	-47.4
0	4	7	65.2	66.6
0	4	8	19.8	19.7
0	4	9	60.9	-62.5
0	4	10	14.2	-14.6
0	4	11	37.3	38.3
0	4	12	7.0	7.8
0	4	13	7.3	-8.4
0	4	17	17.6	-17.3
0	4	18	16.0	-15.6
0	4	19	16.5	15.1
0	4	20	11.1	10.8
0	4	21	15.3	-13.6
0	4	22	23.5	-28.2
0	4	23	11.7	11.8
0	4	24	11.8	11.2
0	4	25	13.6	-13.8
0	4	26	20.2	-18.2
0	4	27	16.3	15.0
0	4	28	11.2	11.3
0	4	30	8.4	-8.8
0	5	2	42.9	-37.9
0	5	4	8.2	-11.7
0	5	5	6.3	5.9
0	5	6	63.6	53.9
0	5	7	26.8	-27.0
0	5	8	29.7	-30.3
0	5	9	15.5	13.6
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H	K	L	Fo	Fc
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4	5	-4	6.4	-4.5
4	5	-5	12.9	-10.8
4	5	-7	19.5	17.8
4	5	-8	19.8	20.1
4	5	-9	5.6	-4.3
4	5	-10	15.0	-13.8
4	5	-11	5.8	4.4
4	5	-12	28.1	27.5
4	5	-14	26.1	-27.2
4	5	-15	16.6	19.3
4	5	-16	27.1	31.1
4	5	-17	11.2	-8.3
4	5	-18	13.9	-13.6
4	5	-20	12.5	12.2
4	5	1	13.6	14.0
4	5	3	22.3	-24.1
4	5	4	14.8	12.8
4	5	6	20.5	-20.8
4	5	7	5.9	-6.8
4	5	8	23.4	24.8
4	5	10	16.0	-16.4
4	5	12	20.0	18.9
4	5	14	6.6	-6.0
4	5	15	6.7	6.3
4	5	16	13.3	14.2
4	5	17	11.5	9.8
4	5	18	6.6	-5.9
4	5	19	9.2	-10.3
4	6	0	15.4	15.2
4	6	-1	10.0	-11.3
4	6	-2	5.8	7.3
4	6	-3	36.1	36.5
4	6	-4	18.8	-17.7
4	6	-5	11.6	-12.6
4	6	-6	11.0	11.1
4	6	-7	11.1	12.2
4	6	-9	19.1	-17.6
4	6	-11	10.7	9.4
4	6	-13	11.9	-8.6
4	6	-16	9.3	-9.8
4	6	-17	16.9	15.7
4	6	-20	14.9	-14.3
4	6	2	8.4	-7.6
4	6	3	11.2	10.6
4	6	5	13.7	12.8
4	6	8	7.8	7.0
4	6	9	12.1	9.6
4	6	11	11.4	-12.1
4	6	12	18.8	-19.9
4	6	15	16.9	-21.0
4	6	17	20.1	21.6
4	6	18	12.8	13.1
4	6	21	13.0	13.4
4	6	23	10.4	-11.8
4	6	25	7.5	7.5
4	7	0	22.2	24.7
4	7	-1	6.3	-7.5
4	7	-2	8.8	-8.3
4	7	-4	8.9	10.1
4	7	-5	12.6	11.3
4	7	-6	8.9	-8.3
4	7	-7	9.0	-8.0
4	7	-8	16.9	18.2
4	7	-9	9.1	-7.6
4	7	-12	14.7	16.1
4	7	-15	13.3	11.6
4	7	-16	18.9	-19.7
4	7	-18	23.5	-28.0
4	7	-19	13.2	12.1
4	7	-20	14.5	17.7
4	7	1	6.3	-5.7
4	7	2	22.9	-26.7
4	7	3	6.4	7.9
4	7	4	23.2	26.4
4	7	6	21.6	-21.5
4	7	7	6.6	-8.9
4	7	8	6.6	5.7
4	7	9	8.1	7.6
4	7	10	9.4	-9.8
4	8	-1	17.4	-18.5
4	8	-3	11.4	9.6
4	8	-4	14.7	14.6
4	8	-5	9.3	7.4
4	8	-6	11.4	-10.5
4	8	-7	13.2	-12.8
4	8	-8	16.2	-18.7
4	8	-9	18.2	-20.3
4	8	-10	11.5	-12.0
4	8	-12	9.4	7.1
4	8	1	17.5	17.3
4	8	2	14.8	14.5
4	8	3	8.1	-7.7
4	8	4	9.4	-10.7
4	8	5	6.7	5.3
4	8	6	6.7	4.1
4	8	7	12.5	-12.6
4	8	8	11.6	9.8
4	8	9	17.6	18.4
4	8	10	6.6	-8.3
4	9	0	17.6	20.1
4	9	-2	9.4	-9.1
4	9	-4	11.6	-12.3
4	9	-5	24.0	-26.7
4	9	-6	18.3	-19.0
4	9	-8	16.3	16.2
4	9	-9	23.9	26.8
4	9	-11	9.3	-10.0
4	9	-12	13.1	14.1
4	9	2	9.4	-7.6
4	9	4	12.4	-11.4
4	9	5	23.8	26.6
4	9	6	6.6	-7.0
4	9	7	9.3	7.8
4	9	9	12.8	-14.9
4	9	10	12.7	-14.4
4	9	12	13.7	14.0
4	9	13	11.9	-11.8
4	10	0	11.2	11.5
4	10	-1	11.2	-11.0
4	10	-2	9.1	-9.4
4	10	1	12.9	-16.1
4	10	2	12.8	13.1
4	10	3	9.0	-9.7
4	10	4	8.9	-6.4
4	10	5	10.8	12.1
4	10	6	20.1	-20.6
4	10	7	10.6	10.5
4	10	8	7.4	5.6
4	10	9	8.4	7.5
5	0	-8	11.3	11.8
5	0	-10	20.5	-19.6
5	0	-12	22.7	19.2
5	0	-14	36.6	-36.5
5	0	-16	13.7	11.3
5	0	-20	6.0	-5.9
5	0	-22	15.0	-17.4
5	0	-24	10.5	12.7
5	0	6	10.7	-9.6
5	0	8	17.4	14.7
5	0	10	23.7	-24.1
5	0	12	5.8	-4.8
5	0	16	6.1	7.5
5	0	22	14.9	13.5
5	0	24	10.8	-10.5
5	1	0	10.5	7.7
5	1	-1	13.4	-9.8
5	1	-3	10.0	-10.8
5	1	-4	4.8	4.6
5	1	-5	20.6	-19.5
5	1	-6	14.3	-16.9
5	1	-7	20.6	20.4
5	1	-8	9.2	9.3
5	1	-9	23.6	-19.0
5	1	-11	28.5	26.5
5	1	-13	17.2	-14.5
5	1	-14	17.3	14.3
5	1	-15	18.1	18.1
5	1	-16	12.1	-13.1
5	1	-17	20.2	-19.0
5	1	-19	10.2	10.4
5	1	-20	7.3	5.5
5	1	-22	10.6	-8.6
5	1	1	7.3	-7.5
5	1	2	10.4	-7.5
5	1	3	20.2	-17.3
5	1	4	15.4	12.7
5	1	5	19.1	19.3
5	1	6	14.6	-13.5
5	1	7	23.8	-23.0
5	1	9	31.9	31.6
5	1	10	10.2	9.7
5	1	11	16.0	-14.6
5	1	12	10.0	10.5
5	1	13	22.9	21.7
5	1	14	7.4	5.9
5	1	15	19.7	-20.1
5	1	16	14.4	-16.1
5	1	17	10.6	9.6
5	1	18	6.1	6.2
5	1	19	8.4	-7.6
5	1	20	10.1	10.5
5	1	22	9.4	-8.3
5	1	23	11.5	-11.7
5	2	0	23.5	23.7
5	2	-1	12.5	10.4
5	2	-2	21.3	-19.4
5	2	-3	7.4	-5.4
5	2	-4	21.7	24.8
5	2	-6	23.1	-26.3
5	2	-7	15.4	16.3
5	2	-10	4.4	-5.7
5	2	-11	14.5	-13.3
5	2	-12	16.9	17.4
5	2	-13	9.3	-8.9
5	2	-14	18.0	15.1
5	2	-16	6.8	6.0
5	2	-17	12.7	11.7
5	2	-18	11.7	-12.5
5	2	-23	10.6	-13.3
5	2	-24	12.1	-12.9
5	2	-26	11.6	-12.4
5	2	-28	19.1	23.9
5	2	1	5.4	-5.7
5	2	2	11.3	-11.9
5	2	3	22.7	21.7
5	2	4	7.7	-6.0
5	2	5	18.4	-16.3
5	2	7	12.4	13.2
5	2	9	10.2	9.1
5	2	11	16.3	-14.1
5	3	0	5.2	2.9
5	3	-1	21.9	-22.2
5	3	-2	10.5	-9.3
5	3	-3	15.9	17.3
5	3	-4	13.9	11.5
5	3	-5	18.3	-15.9
5	3	-7	13.7	14.3
5	3	-8	16.1	14.5
5	3	-9	4.6	4.5
5	3	-11	32.8	31.2
5	3	-12	36.1	37.7
5	3	-13	9.1	-8.0
5	3	-14	10.9	-10.4
5	3	-15	12.5	-9.8
5	3	-16	8.1	-8.4
5	3	-18	11.9	-12.6
5	3	-19	6.0	6.4
5	3	-20	13.6	-12.5
5	3	-21	20.3	-19.7
5	3	-22	10.6	-13.0
5	3	-24	13.4	13.6
5	3	-25	19.0	20.2
5	3	-27	11.0	11.2
5	3	1	26.5	28.8
5	3	2	14.3	-14.3
5	3	3	24.5	-24.9
5	3	4	8.4	-6.4
5	3	5	22.9	20.8
5	3	7	8.4	-8.7
5	3	9	5.6	7.3
5	3	10	8.2	-6.2
5	3	11	11.8	-10.7
5	3	12	11.2	9.7
5	3	13	18.7	19.1
5	3	14	12.2	-10.1
5	3	15	8.7	-7.3
5	3	16	12.2	12.1
5	3	17	12.8	12.1
5	3	18	14.6	-14.4
5	3	20	17.4	18.0
5	3	23	14.5	-13.6
5	4	0	6.0	-4.8
5	4	-5	14.7	-13.2
5	4	-6	6.8	-6.7
5	4	-7	15.1	12.1
5	4	-8	24.5	25.7
5	4	-9	10.8	12.2
5	4	-10	11.1	-9.0
5	4	-11	27.2	-26.8
5	4	-12	15.9	-14.5
5	4	-15	18.4	-16.1
5	4	-16	16.2	13.4
5	4	-17	15.9	14.6
5	4	-18	12.1	-10.4
5	4	-19	12.2	-13.1
5	4	-20	8.7	6.8
5	4	-21	15.0	15.6
5	4	1	15.6	13.6
5	4	2	17.8	16.7
5	4	3	22.7	22.4
5	4	4	16.6	17.4
5	4	6	14.6	-14.6
5	4	7	11.2	-9.8
5	4	8	19.9	21.2
5	4	9	24.5	-26.9
5	4	10	7.3	6.5
5	4	11	9.5	9.8
5	4	12	6.1	-4.5
5	5	0	8.5	6.6
5	5	-1	9.2	-10.3
5	5	-2	17.2	-15.2
5	5	-3	18.7	19.9
5	5	-4	9.2	8.7
5	5	-5	14.1	15.8
5	5	-6	15.6	-15.8
5	5	-7	13.3	14.2
5	5	-8	7.7	8.2
5	5	-9	27.2	-26.1
5	5	-16	11.4	10.9
5	5	-17	12.2	-13.0
5	5	-18	9.7	-9.9
5	5	-19	15.0	14.3
5	5	-20	11.4	11.2
5	5	1	6.7	-6.0
5	5	2	31.6	-32.8
5	5	3	9.6	-8.8
5	5	4	9.8	9.4
5	5	5	29.2	32.1
5	5	6	14.8	-14.4
5	5	8	25.0	25.5
5	5	9	12.1	-11.6
5	5	11	13.0	-13.4
5	5	12	13.7	14.6
5	5	13	12.2	14.3
5	5	14	11.4	-10.3
5	5	15	21.2	-24.4
5	5	16	7.2	-4.3
5	5	18	11.2	-10.3
5	5	20	10.2	9.5
5	6	-1	13.7	19.0
5	6	-2	16.3	-16.3
5	6	-3	9.9	-8.6
5	6	-4	12.9	-11.8
5	6	-5	19.9	19.9
5	6	-6	10.0	-9.8
5	6	-7	15.8	-20.3
5	6	-8	17.0	16.4
5	6	-9	10.2	-10.7
5	6	1	13.7	-14.5
5	7	0	19.1	20.0
5	7	-2	21.3	-22.7
5	7	-4	19.1	19.6
5	7	-6	6.0	-6.4
5	7	-7	12.1	-11.2
5	7	-8	10.5	8.7
5	7	2	13.6	-12.6
5	7	3	13.6	-13.0

TABLE 1.6

(A) ANALYSIS OF STRUCTURE FACTOR DATA AS A FUNCTION OF LAYER LINE INDEX. ALL FIGURES ARE ON ABSOLUTE SCALE.

h	$\Sigma\|F_o\|$	$\Sigma\|F_c\|$	$\Sigma\|\Delta\|$	N	R	$\Sigma\|\Delta\|/N$
0	6321	6258	463	227	0.0732	2.04
1	12342	12193	1052	449	0.0852	2.34
2	9614	9413	821	451	0.0854	1.82
3	7175	7044	515	345	0.0717	1.49
4	5270	5146	498	307	0.0944	1.62
5	2858	2784	242	198	0.0847	1.22
ALL	43580	42835	3591	1977	0.0824	1.82

(B) ANALYSIS OF STRUCTURE FACTORS BY MAGNITUDE OF Fo.

RANGE	$\Sigma\|F_o\|$	$\Sigma\|F_c\|$	$\Sigma\|\Delta\|$	N	R	$\Sigma\|\Delta\|/N$
0 - 10	3403	3194	580	454	0.1704	1.28
10 - 20	11492	11108	1081	798	0.0941	1.36
20 - 40	13644	13563	930	495	0.0682	1.88
40 - 70	8237	8247	554	157	0.0673	3.53
70 - 100	4213	4144	241	51	0.0572	4.73
100 - 250	2591	2580	203	22	0.0783	9.23

TABLE 1.7

BOND LENGTHS AND E.S.D.S IN ANGSTROMS.

(A) 4 - BROMO 2,5,7 - TRINITROFLUORENONE.

Br(1) - C(4)	1.853 ± 8	C(2) - C(3)	1.364 ± 11
Br(2) - C(5)	1.961 ± 8	C(3) - C(4)	1.398 ± 12
O(1) - C(9)	1.205 ± 10	C(4) - C(11)	1.404 ± 11
O(2) - N(1)	1.193 ± 11	C(5) - C(6)	1.354 ± 11
O(3) - N(1)	1.199 ± 11	C(5) - C(12)	1.411 ± 12
O(4) - N(2)	1.325 ± 17	C(6) - C(7)	1.377 ± 12
O(5) - N(2)	1.094 ± 16	C(7) - C(8)	1.385 ± 13
O(6) - N(3)	1.239 ± 11	C(8) - C(13)	1.378 ± 12
O(7) - N(3)	1.214 ± 11	C(9) - C(10)	1.479 ± 11
N(1) - C(2)	1.478 ± 12	C(9) - C(13)	1.505 ± 12
N(2) - C(5)	1.526 ± 14	C(10) - C(11)	1.407 ± 11
N(3) - C(7)	1.474 ± 12	C(11) - C(12)	1.518 ± 12
C(1) - C(2)	1.384 ± 11	C(12) - C(13)	1.382 ± 10
C(1) - C(10)	1.380 ± 12		

(B) ANTHRACENE.

C(14) - C(15)	1.350 ± 15	C(20) - C(21)	1.335 ± 15
C(14) - C(26)	1.437 ± 13	C(21) - C(25)	1.413 ± 13
C(15) - C(16)	1.393 ± 17	C(22) - C(25)	1.412 ± 13
C(16) - C(17)	1.353 ± 15	C(22) - C(26)	1.415 ± 13
C(17) - C(27)	1.419 ± 13	C(23) - C(24)	1.428 ± 13
C(18) - C(19)	1.351 ± 15	C(23) - C(27)	1.382 ± 12
C(18) - C(24)	1.444 ± 14	C(24) - C(25)	1.442 ± 12
C(19) - C(20)	1.437 ± 17	C(26) - C(27)	1.432 ± 11

TABLE 1.8

BOND ANGLES AND E.S.D.S IN DEGREES.

(A) 4 - BROMO 2,5,7 - TRINITROFLUORENONE.

O(2) - N(1) - O(3)	122.4± 9	C(6) - C(5) - C(12)	122.2± 7
O(2) - N(1) - C(2)	119.8± 8	C(5) - C(6) - C(7)	119.2± 8
O(3) - N(1) - C(2)	117.6± 8	N(3) - C(7) - C(6)	119.5± 8
O(4) - N(2) - O(5)	135.4±13	N(3) - C(7) - C(8)	118.8± 8
O(4) - N(2) - C(5)	104.3±10	C(6) - C(7) - C(8)	121.7± 8
O(5) - N(2) - C(5)	120.0±11	C(7) - C(8) - C(13)	117.1± 8
O(6) - N(3) - O(7)	121.4± 8	O(1) - C(9) - C(10)	126.8± 8
O(6) - N(3) - C(7)	117.3± 8	O(1) - C(9) - C(13)	127.5± 7
O(7) - N(3) - C(7)	118.6± 8	C(10) - C(9) - C(13)	105.7± 6
C(2) - C(1) - C(10)	116.2± 7	C(1) - C(10) - C(9)	126.2± 7
N(1) - C(2) - C(1)	119.1± 7	C(1) - C(10) - C(11)	124.8± 7
N(1) - C(2) - C(3)	118.5± 7	C(9) - C(10) - C(11)	108.8± 7
C(1) - C(2) - C(3)	122.4± 8	C(4) - C(11) - C(10)	115.7± 7
C(2) - C(3) - C(4)	120.0± 7	C(4) - C(11) - C(12)	136.2± 7
Br(1) - C(4) - C(3)	115.2± 6	C(10) - C(11) - C(12)	108.0± 6
Br(1) - C(4) - C(11)	123.9± 6	C(5) - C(12) - C(11)	136.2± 7
C(3) - C(4) - C(11)	120.6± 7	C(5) - C(12) - C(13)	115.7± 7
Br(2) - C(5) - C(6)	114.7± 6	C(11) - C(12) - C(13)	107.9± 7
Br(2) - C(5) - C(12)	121.7± 6	C(8) - C(13) - C(9)	126.8± 7
N(2) - C(5) - C(6)	111.4± 8	C(8) - C(13) - C(12)	123.8± 8
N(2) - C(5) - C(12)	126.2± 8	C(9) - C(13) - C(12)	109.4± 7

(B) ANTHRACENE.

C(15)- C(14)- C(26)	119.9± 9	C(18)- C(24)- C(25)	117.7± 8
C(14)- C(15)- C(16)	121.8±10	C(23)- C(24)- C(25)	118.7± 8
C(15)- C(16)- C(17)	119.8±10	C(21)- C(25)- C(22)	124.0± 8
C(16)- C(17)- C(27)	122.1± 9	C(21)- C(25)- C(24)	118.2± 8
C(19)- C(18)- C(24)	121.8± 9	C(22)- C(25)- C(24)	117.8± 8
C(18)- C(19)- C(20)	119.0±10	C(14)- C(26)- C(22)	122.0± 8
C(19)- C(20)- C(21)	121.2± 9	C(14)- C(26)- C(27)	118.7± 8
C(20)- C(21)- C(25)	122.1± 9	C(22)- C(26)- C(27)	119.3± 8
C(25)- C(22)- C(26)	122.5± 8	C(17)- C(27)- C(23)	123.9± 8
C(24)- C(23)- C(27)	123.1± 8	C(17)- C(27)- C(26)	117.5± 8
C(18)- C(24)- C(23)	123.6± 8	C(23)- C(27)- C(26)	118.6± 8

TABLE 1.9

NON - BONDED DISTANCES.

(A) INTERPLANAR DISTANCES LESS THAN 3.6 Å.

C(14)....C(8)	ii	3.48 Å	C(22)....C(13)	ii	3.55 Å
C(14)....C(7)	ii	3.52	C(23)....O(4)	ii	3.30
C(15)....N(3)	i	3.42	C(23)....C(12)	i	3.42
C(15)....O(6)	i	3.54	C(23)....C(12)	ii	3.59
C(15)....N(3)	ii	3.58	C(24)....C(10)	ii	3.51
C(15)....C(7)	ii	3.59	C(24)....C(11)	i	3.52
C(16)....C(6)	ii	3.60	C(24)....C(11)	ii	3.56
C(17)....O(4)	ii	3.36	C(24)....C(12)	i	3.57
C(17)....C(6)	i	3.44	C(25)....C(9)	ii	3.40
C(17)....C(6)	ii	3.55	C(25)....C(10)	ii	3.48
C(18)....C(4)	i	3.45	C(25)....C(9)	i	3.57
C(18)....C(11)	i	3.48	C(26)....C(8)	ii	3.48
C(19)....C(1)	ii	3.49	C(26)....C(8)	i	3.52
C(19)....C(2)	ii	3.50	C(26)....C(13)	ii	3.53
C(19)....C(1)	i	3.57	C(27)....O(4)	ii	3.55
C(20)....C(1)	ii	3.46	C(27)....C(5)	ii	3.56
C(22)....C(9)	ii	3.49			

(B)) SOME INTRAMOLECULAR DISTANCES.

Br(1)....O(4)	i	3.04	Br(1)....N(2)	i	3.13
Br(1)....O(5)	i	3.35			

(C) INTERMOLECULAR DISTANCES, OTHER THAN INTERPLANAR, LESS THAN 3.6 Å.

O(2).....O(4)	iii	2.96 Å	O(3).....C(16)	viii	3.40 Å
O(5).....C(3)	iv	3.28	O(3).....O(6)	vi	3.42
O(4).....C(3)	v	3.32	Br(1)....O(2)	iv	3.50
O(3).....C(6)	vi	3.36	O(2).....C(23)	vii	3.53
Br(1)....O(5)	vii	3.38	O(4).....N(1)	v	3.58

The Roman numerals refer to the following transformations applied to the coordinates found in Table 1.1 , the transformation is applied to the second atom of each contact.

i	x,	y,	z.
ii	1 - x,	y,	z.
iii	1 - x,	-1/2 + y,	1/2 - z.
iv	- x,	1/2 + y,	1/2 - z.
v	1 - x,	1/2 + y,	1/2 - z.
vi	x,	1 - y,	z.
vii	- x,	-1/2 + y,	1/2 - z.
viii	1 + x,	1 - y,	z.

TABLE 1.10

MEAN MOLECULAR PLANES.

PLANE NO.	ATOMS DEFINING PLANE.
1	C(1) to C(13)
2	C(1),C(2),C(3),C(4),C(10) and C(11)
3	C(5),C(6),C(7),C(8),C(12) and C(13)
4	C(9),C(10),C(11),C(12),C(13) and O(1)
5	N(1),O(2),O(3) and C(2)
6	N(2),O(4),O(5) and C(5)
7	N(3),O(6),O(7) and C(7)
8	C(14) to C(27)

PLANE EQUATIONS.

PLANE NO.	P	Q	R	S	RMS D
1	0.9848	0.0944	-0.1459	1.7108	0.064
2	0.9709	0.1301	-0.2012	1.5230	0.022
3	0.9935	0.0638	-0.0942	1.8907	0.021
4	0.9860	0.1176	-0.1180	2.2771	0.021
5	0.9523	0.1914	-0.2378	1.7019	0.015
6	-0.3991	-0.8852	-0.2392	-15.6763	0.017
7	0.9925	0.0906	-0.0825	2.4072	0.001
8	0.9954	0.0703	-0.0659	-1.1396	0.021

In this Table P, Q and R are the direction cosines of the plane normal, S is the plane to origin distance and RMS D is the root mean square deviation in Å of the atoms in the plane from the plane.

The plane equation is then

$$PX' + QY + RZ' = S$$

where X', Y and Z' are in Å and are referred to orthogonal axes defined by X parallel to a*, Y parallel to b and Z parallel to c.

DEVIATIONS FROM MEAN PLANES (Å).

PLANE NO.	ATOM AND DISTANCE.					
1	C(1)	0.069	C(2)	0.061	C(3)	-0.057
	C(4)	-0.120	C(5)	0.108	C(6)	0.067
	C(7)	-0.036	C(8)	-0.059	C(9)	-0.023
	C(10)	0.057	C(11)	-0.006	C(12)	-0.013
	C(13)	-0.050	Br(1)	-0.504	N(1)	0.140
	N(2)	0.204	N(3)	-0.091	O(1)	-0.064
2	C(1)	-0.025	C(2)	0.027	C(3)	0.001
	C(4)	-0.028	C(10)	-0.002	C(11)	0.028
	Br(1)	-0.290	N(1)	0.072	C(9)	-0.132
	C(12)	0.032				
3	C(5)	0.027	C(6)	0.001	C(7)	-0.024
	C(8)	0.018	C(12)	-0.031	C(13)	0.010
	N(2)	0.032	N(3)	-0.062	C(9)	0.097
	C(11)	-0.018				

4	C(9)	0.006	C(10)	0.033	C(11)	-0.034
	C(12)	0.009	C(13)	0.005	O(1)	-0.019
	C(1)	0.007	C(4)	-0.196	C(5)	0.150
	C(8)	0.046				
5	N(1)	-0.026	O(2)	0.009	O(3)	0.009
	C(2)	0.007				
6	N(2)	-0.030	O(4)	0.010	O(5)	0.013
	C(5)	0.007				
7	N(3)	0.002	O(6)	-0.001	O(7)	-0.001
	C(7)	-0.001				
8	C(14)	-0.036	C(15)	0.032	C(16)	0.020
	C(17)	0.017	C(18)	-0.014	C(19)	0.009
	C(20)	0.029	C(21)	0.022	C(22)	-0.013
	C(23)	-0.024	C(24)	-0.013	C(25)	-0.016
	C(26)	-0.015	C(27)	0.002	C(1)	3.558
	C(2)	3.452	C(3)	3.230	C(4)	3.157
	C(5)	3.435	C(6)	3.441	C(7)	3.453
	C(8)	3.500	C(9)	3.544	C(10)	3.531
	C(11)	3.365	C(12)	3.380	C(13)	3.458

SOME DIHEDRAL ANGLES.

PLANE A	PLANE B	∠ AB	PLANE A	PLANE B	∠ AB
1	2	3.9°	2	8	8.6°
1	3	3.5	3	4	3.4
1	4	2.1	3	6	64.5
1	5	7.9	3	7	1.7
1	6	63.8	3	8	1.7
1	7	3.7	4	8	4.1
1	8	4.8	5	8	12.3
2	3	7.3	6	8	63.7
2	4	4.9	7	8	1.5
2	5	4.2			

1.4 Discussion

The results of this analysis are of sufficient accuracy for details of the molecules involved to be discussed and some conclusions drawn. The discussion on polarisation bonding in this complex will be withheld until after the analysis of the BTNF:dimethyl benzophenanthrene complex and the bonding involved in both compounds dealt with then.

The main structural feature to be expected in BTNF is a steric strain caused by the juxtaposition of the substituents at the 4- and 5- positions, Fig. 1.2 . This is shown by departure from normal valence angles, planarity and expected covalent bond lengths; but as disorder in this compound is present at the 4- and 5- positions some care must be taken in assessing the results. The C(11)-C(12) bond of 1.518 Å is three estimated standard deviations greater than the comparable bond of 1.48 Å in free fluorenone (Burns and Iball, 1954) and 1.47 Å in 9-dicyanomethylene-2,4,7-trinitrofluorene (Silverman, Krukonis and Yannoni, 1967). Thus the increase is just significant and is probably caused by steric repulsion between the bromine atom and the nitro group at C(5). The interbond angles Br(1)-C(4)-C(11) and N(2)-C(5)-C(12) have been increased from their usual value of 120° to 123.9°

and 126.2°, again angles C(4)-C(11)-C(12) and C(5)-C(12)-C(11) have both been increased to 136.2° from an undistorted value of 132°. Br(1) and N(2) are -0.50 Å and 0.20 Å respectively out of the plane derived from the carbon skeleton of the BTNF molecule (Table 1.10, equation 1). This would imply the benzene rings are twisted in opposite directions, the dihedral angle between the mean planes of the benzene rings is 7.3° (Table 1.10, equations 2 and 3). Relief from overcrowding, however, must also arise from bond angle deformation at C(4) and C(5).

The bond lengths and interbond angles in the aromatic rings do not differ significantly from accepted values. The mean bond length of 1.385 Å agrees well with the value of 1.392 Å found in crystalline benzene (Cox, Cruickshank and Smith, 1958), and the mean bond angle of 119.9° is almost exactly the expected value of 120°. The mean plane through the cyclopentanone system is

$$0.9860\,X' + 0.1176\,Y - 0.1180\,Z' = 2.2771$$

where X', Y and Z' (in Å) are orthogonal coordinates with respect to orthogonal axes defined in Table 1.10 and the root mean square deviation of the atoms from this plane is 0.021 Å. The C(9)-O(1), C(9)-C(10) and C(9)-C(13) bond lengths agree favourably with similar bonds in anthraquinone (Lonsdale, Milledge and Karimat, 1966) and 3,3 - dibromobenzophenone (Ramaseshan and Venkatesan, 1958). The Br(1)-C(4) bond of 1.853 Å compares with the expected value of 1.85 Å (Sutton, 1965); the Br(2)-C(5) bond of 1.961 Å is significantly greater than this value, however, it is doubtful whether this represents a true bond length as the increase is probably caused by effects of the disorder. Bond lengths and angles of the 2- and 7- nitro groups are similar to those found in other analyses, for example 9:10-dinitroanthracene (Trotter, 1959) and m-dinitrobenzene (Trotter, 1961).

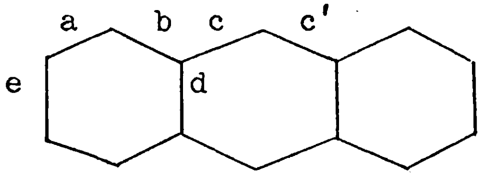
The structure of anthracene is well established and an assessment of various experimental results has been given by Cruickshank and Sparks (1960). For comparison, the chemically equivalent bond lengths and interbond angles from the experimental results have been averaged and set out in Table 1.A , together with results from other analyses. The average standard deviations of the means for the experimental results are 0.010 Å for a bond length and 0.7° for a bond angle.

The equation of the best plane through the anthracene molecule is

$$0.9954\,X' + 0.0703\,Y - 0.0659\,Z' = -1.1396$$

TABLE 1.A

COMPARISON OF DIMENSIONS FOUND FOR ANTHRACENE.
BOND LENGTHS IN ANGSTROMS, BOND ANGLES IN DEGREES.



a	b	c	d	e	Reference *
1.364	1.419	1.391	1.440	1.390	(1)
1.375	1.444	1.405	1.433	1.418	(2)
1.38	1.44	1.40	1.44	1.42	(3)
1.347	1.428	1.409	1.437	1.415	(4)
∠ae	∠ab	∠bd	∠cd	∠cc'	
120.9	120.7	118.5	118.9	122.2	(1)
121.3	119.3	119.3	119.5	121.0	(2)
120.0	121.0	119.0	119.5	121.0	(3)
120.4	121.5	118.0	118.6	122.8	(4)

* (1) Sinclair, Robertson and Mathieson (1950).

(2) Mason (1964), 290 °K results.

(3) Brown, Wallwork and Wilson (1964), -100 °C results.

(4) Present analysis.

and the root mean square distance of the fourteen atoms from this plane is 0.021 Å . The mean interbond angles do not differ significantly from those found by Sinclair, Robertson and Mathieson (1950), Brown, Wallwork and Wilson (1964) and Mason (1964). Average bond lengths agree well with other reported values, none being more than two standard deviations different from the "best available" values quoted by Cruickshank and Sparks (1960). It is interesting, however, to note that in the four a type bonds, that is the most chemically reactive, the individual values are consistently lower, by about 0.020 Å , than Cruickshank and Spark's value. This could imply localisation of the π- electrons at these sites and involvement in the charge transfer bonding.

Although the standard deviations are large, the anisotropic vibration tensors give a physically acceptable description of the atomic vibration. In general the component of the anisotropic vibration tensor normal to the molecular planes, in this case the U_{11} , is greater than the remaining components, this situation was also found by Hanson (1965) in the azulene, s-trinitrobenzene complex. Individual U_{ij} tend to increase towards the extremities of the molecules as is normally found in most structure analyses.

CHAPTER 2

1,12-Dimethyl-3,4-benzophenanthrene , 4-Bromo-2,5,7-trinitrofluorenone complex.

2.1 Experimental

Crystal data

1,12-Dimethyl-3,4-benzophenanthrene : 4-Bromo-2,5,7-trinitrofluorenone.

$C_{20}H_{16} : C_{13}H_4N_3O_7Br.$

F.W. = 650.5 , M.Pt. = 144 - 145 $^{\circ}$C .

Monoclinic, $a = 17.13 \pm 3$, $b = 7.12 \pm 2$, $c = 23.22 \pm 4$ $\overset{\circ}{A}$

$\beta = 106^{\circ}\ 33' \pm 20'$.

$U = 2713\ \overset{\circ}{A}{}^3$, $D_m = 1.58$ gm.cm.$^{-3}$, $Z = 4$, $D_x = 1.59$ gm.cm.$^{-3}$

Linear absorption coefficient $\mu = 16.7$ cm.$^{-1}$ (Mo K_{α}).

F(000) = 1320.

Systematic absences,

h0l when l is odd,

(0k0 when k is odd).

Space group $P2_1/c$ (No. 14), but later shown to be
Pc (C_s^2 , No. 7).

Crystals, consisting of reddish brown needles elongated along b, were supplied by Professor M. S. Newman. Rotation and equatorial layer Weissenberg photographs taken about the b axis with Cu K_{α} radiation (λ = 1.5418 Å) and precession photographs of the 0kl and hk0 zones taken with Mo K_{α} radiation (λ = 0.7107 Å) gave the cell dimensions and space group.

Intensity data were collected by means of a Hilger and Watts linear diffractometer (Arndt and Phillips, 1961) see Appendix II. The diffractometer has equi-inclination geometry and molybdenum radiation was used together with balanced strontium oxide and zirconium oxide filters (Ross, 1926). The reciprocal lattice nets h0l to h7l were surveyed systematically using a 3° scan through each reflection coupled with a one minute oscillation cycle; in all some 6500 reflections were measured. As the random background count was of the order of 20 counts per minute, only reflections of 30 counts per minute or over were used in the subsequent analysis. The intensities were corrected for Lorentz, polarisation and rotation factors (Tunell, 1939) by the computer program of A. A. Hock, M. R. Truter and M. Wells; no absorption corrections were made as the crystal was almost transparent to molybdenum radiation. In all 1646 structure amplitudes (approximately 27 %

of the copper sphere) were derived. The data, initially assumed to be on the same scale, were later found to show a decrease in average intensity with duration of exposure. This was due to crystal deterioration and was partially allowed for by having separate scale factors for each reciprocal lattice net in the least-squares refinement.

2.2 Structure Solution and Refinement

From the initial systematic absences the space group was taken to be $P2_1/c$; as $Z = 4$, giving one molecule of complex per asymmetric unit, heavy atom atomic vectors should occur on the Harker section $V = 1/2$ and line section $U = 0$, $W = 1/2$. The two- and three-dimensional Patterson functions (Section at $V = 1/2$, Fig. 2.1) could be interpreted for the bromine atom by assuming the z - coordinate to be almost exactly 0.25 ; the general 2x, 2y, 2z vector thus appears on the section at $V = 1/2$ together with the 2x, 1/2, 1/2 + 2z vector. The bromine fractional coordinates derived by the method of Booth (1948) were

Br(1)	0.3870	0.2500	0.0884 .

Recognisable molecules were obtained in an electron-density map, phased from structure factors for the bromine atom alone, even though a pseudo mirror plane was present. A further electron-density synthesis, computed from structure factors which were based on the BTNF molecule, gave a clear 1,12-dimethyl-3,4-benzophenanthrene (henceforth DMBP) molecule although the mirror symmetry was not completely broken. The two complexed molecules so found would not refine to any R value better than 0.35 and gave inconsistent molecular packing.

Re-examination of the basic data revealed that the 0k0 reflections were few in number. Thus, if the systematic halving of these data were accidental, the space group would not be $P2_1/c$ but Pc. In the space group Pc, with two molecules of complex per asymmetric unit, the heavy atom vectors in the Patterson function were consistent with one bromine at the origin and the other at $y = 0.5$. Coordinates derived were,

Br(1)	0.7739	0.5000	0.1793
Br(2)	0.0000	0.0000	0.0000 .

This approach proved fruitful and in four cycles of structure-factor and electron-density syntheses, employing the weighting function of Sim (1959), all 88 atoms were located and the residual R stood at 0.244 .

Refinement was continued by means of the isotropic least-squares program described in Part II of this Thesis, and, in view of the number of parameters and planes involved, an anisotropic analysis was not contemplated. Six cycles of minimisation of the function $\sum w\,(|Fo| - |Fc|)^2$ with weights, w, given by

$$w = (A + |Fo| + B\,|Fo|^2)^{-1}$$

where A = 2 F min. and B = 2 / F max. (Cruickshank et al., 1961), reduced the value of R from 0.244 to 0.157 . A block diagonal approximation, involving a chain of 4 × 4 blocks for thermal and positional parameters and a 9 × 9 block for scale factors and overall thermal parameter, was used with indicated shifts multiplied by 0.65 . To save computing time per run the two BTNF molecules were refined for the first three cycles, and the DMBP molecules for the second three cycles. Four further cycles, with all molecules included in the least-squares calculations and shifts multiplied by 0.5 , reduced R to 0.150 . The ratio of average coordinate shift to average standard deviation was then 0.25 ; the refinement was terminated as no significant improvement in the postulated model was considered likely with the data available.

Electron-density and difference syntheses were then computed from the last set of structure factor data ; $\sigma(\rho)$, derived from the formula of Cruickshank (1949), was 0.3 e/Å^3 . Features on the difference map greater than $3\sigma(\rho)$ were associated with the two bromine atoms and were probably due to anisotropic thermal vibration.

Atomic formfactors were those of Freeman (1959) for carbon, Berghuis et al. (1955) for nitrogen and oxygen, Freeman and Watson (1962) for bromine. The numbering scheme adopted for the analysis, the final electron-density synthesis, shown by superimposed contour sections parallel to (010), and molecular packing in the unit cell are in Figs. 2.2 , 2.3 and 2.4 respectively. Final atomic parameters are given in Tables 2.1 and 2.2 ; observed and calculated structure amplitudes with an analysis by magnitude of Fo and reciprocal lattice net in Tables 2.3 and 2.4 ; bond lengths, interbond angles, interatomic distances and mean molecular planes in Tables 2.5 to 2.8 . When quoted, standard deviations are in units of the last decimal place and were determined from the inverse matrix of the normal equations.

DIAGRAMS AND TABLES.

COURSE OF ANALYSIS.

I Fourier refinement.

		R	*
1	2 Br atoms.	0.807	(a)
2	2 Br, 3 N, 7 O, 13 C.	0.544	
3	All non hydrogen atoms.	0.357	
4	As above.	0.244	

II Least-squares refinement.

Cycles of refinement	Final R	
1 - 3	0.176	(b)
4 - 6	0.157	(c)
7 - 10	0.150	(d)

* (a) only half of the structure factor data phased.

(b) isotropic, individual layer scale factors, only BTNF molecules included.

(c) as above, only DMBP molecules included.

(d) as above, all molecules included.

FIG. 2.1

Section at V = 1/2 through the Patterson function.

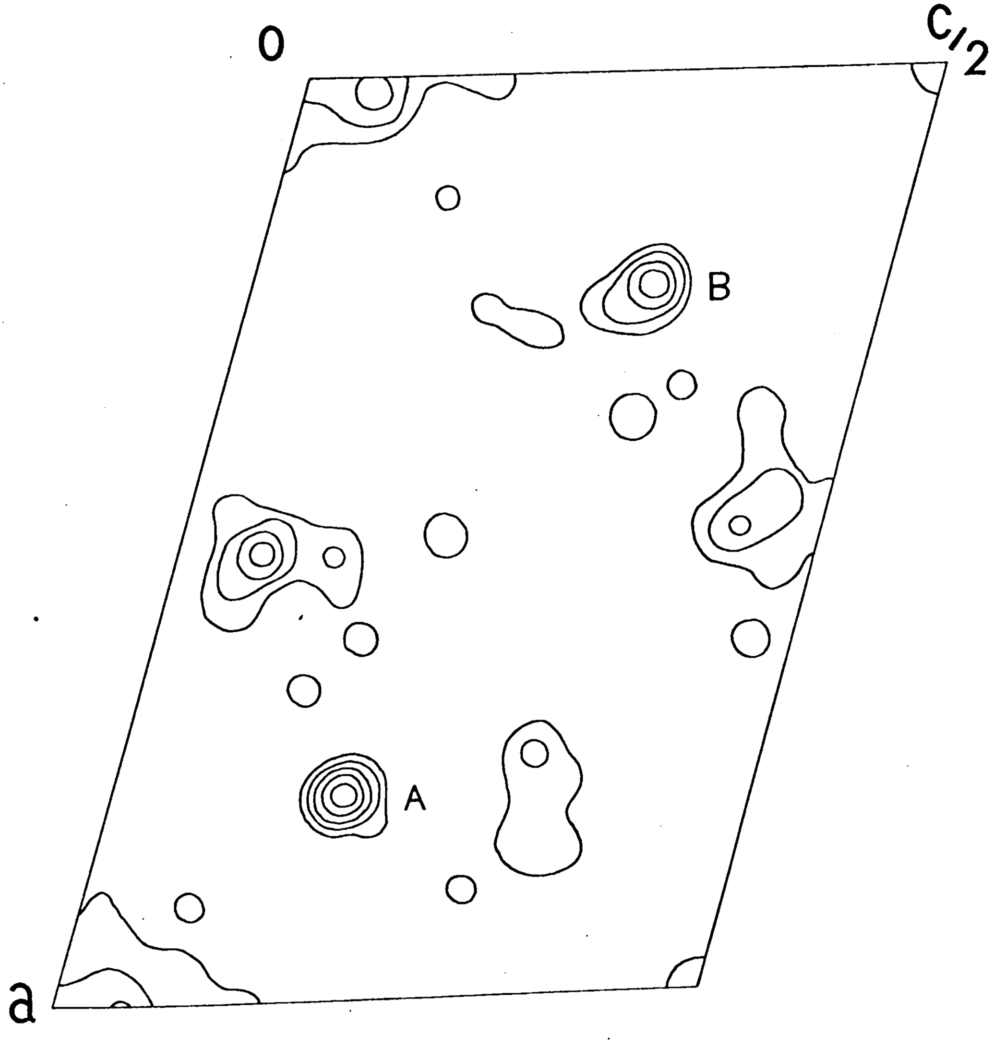

0
c/2
B
A
a

FIG. 2.2

Atomic arrangement showing numbering of the heavier atoms.

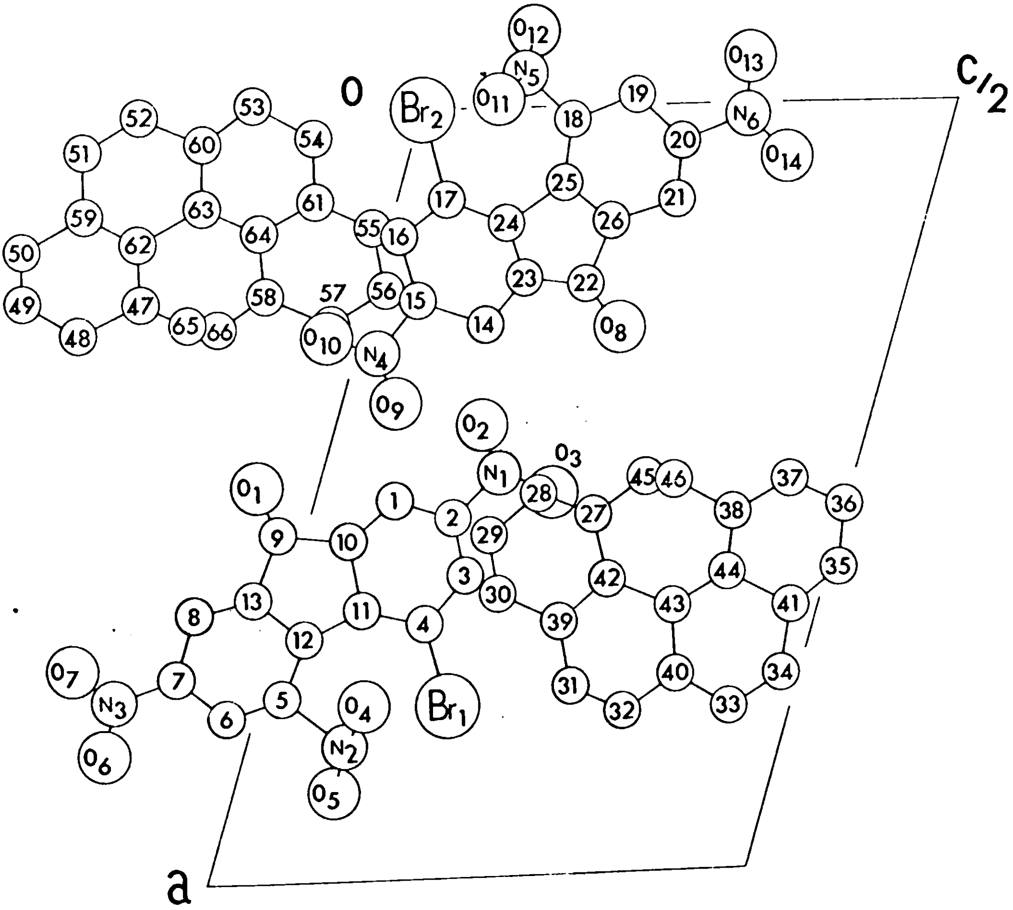

O_{12}
N_5
O_{13}
0
Br_2
O_{11}
18
19
N_6
c/2
53
52
54
60
20
O_{14}
51
25
61
17
21
63
59
55
16
24
26
50
62
64
23
22
57
56
15
49
47
58
65
66
14
O_8
48
O_{10}
N_4
O_9
O_2
O_3
N_1
45
46
37
28
36
O_1
1
2
27
38
29
9
10
3
42
44
35
30
13
43
41
8
11
4
39
12
40
34
O_7
7
N_3
5
O_4
31
Br_1
32
33
6
N_2
O_6
O_5
a

FIG. 2.3

Composite final electron-density synthesis viewed down the <u>b</u> axis. Contours are at intervals of 1 e/Å^3 starting from 1 e/Å^3 except at the bromine atom which is contoured at 5 e/Å^3 intervals.

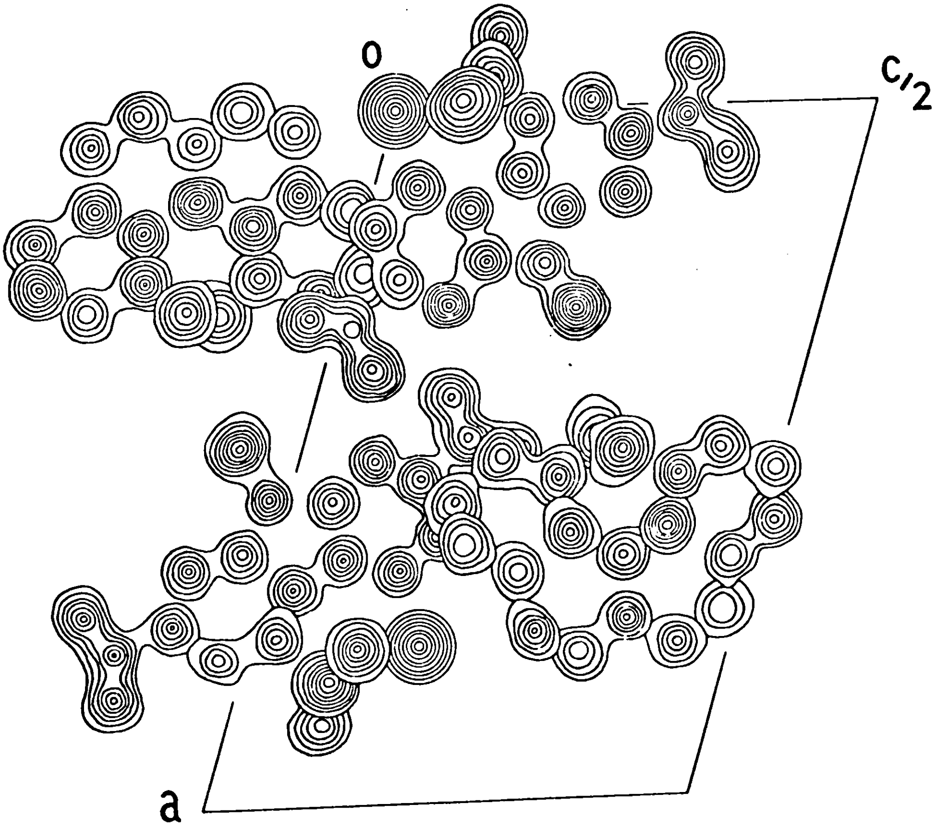

0
c/2
a

FIG. 2.4

The molecular packing viewed down the b axis.

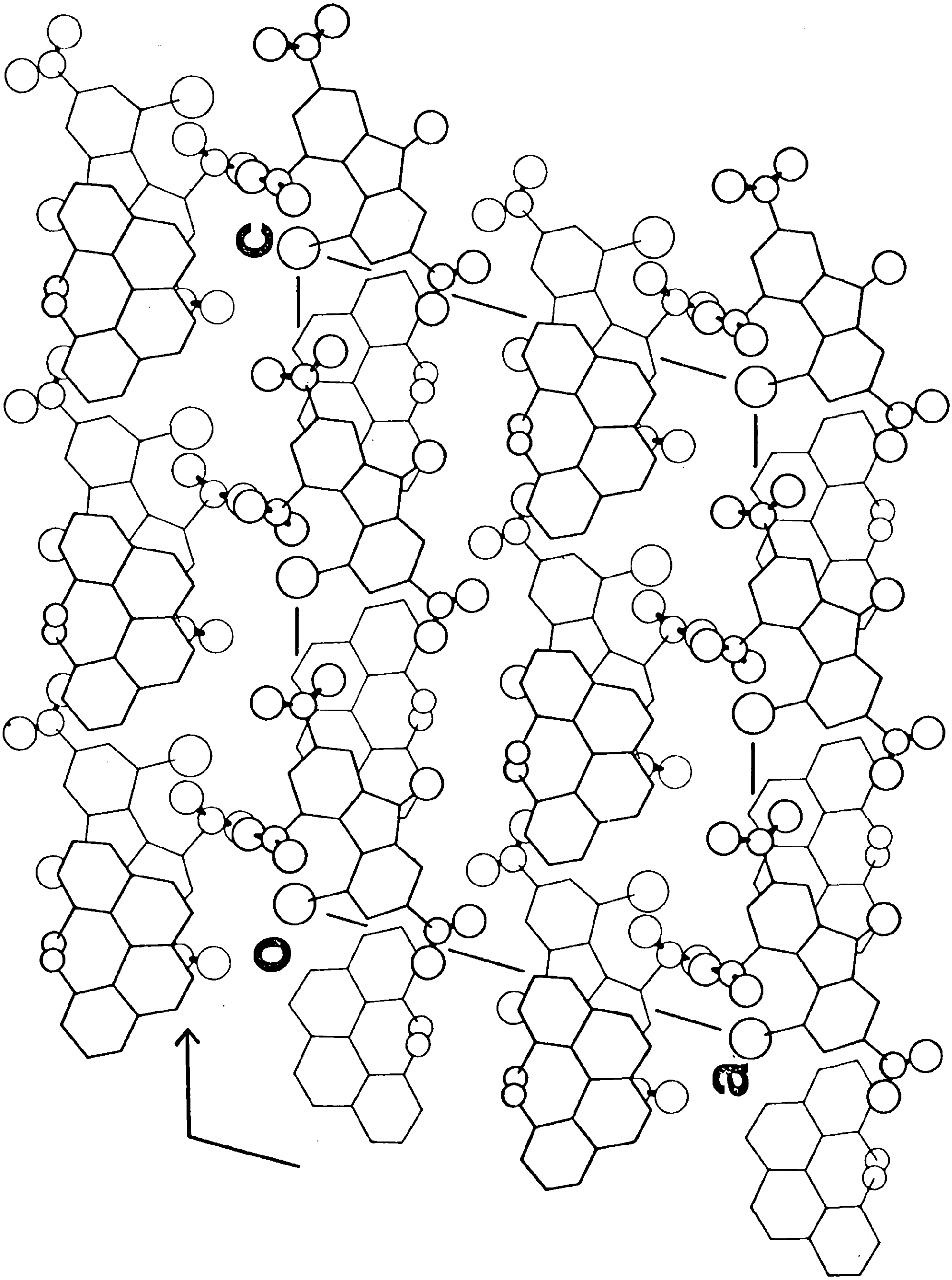

c
o
a

TABLE 2.1

FRACTIONAL COORDINATES AND ISOTROPIC TEMPERATURE FACTORS WITH E.S.D.S.

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE A

ATOM	X/a	Y/b	Z/c	U iso
Br(1)	0.7737(4)	0.5511(10)	0.1784(3)	0.052(2)
O(1)	0.4814(20)	0.5235(56)	-0.0608(15)	0.036(9)
O(2)	0.4140(32)	0.6743(86)	0.1378(24)	0.089(17)
O(3)	0.5020(25)	0.6554(64)	0.2214(18)	0.056(12)
O(4)	0.7792(25)	0.1483(66)	0.1147(18)	0.059(12)
O(5)	0.8840(29)	0.2390(72)	0.0940(20)	0.064(11)
O(6)	0.8289(23)	0.3700(60)	-0.1266(16)	0.042(10)
O(7)	0.7113(24)	0.4164(61)	-0.1780(17)	0.009(11)
N(1)	0.4449(24)	0.6325(63)	0.1655(17)	0.025(11)
N(2)	0.8221(20)	0.2459(55)	0.0899(14)	0.007(8)
N(3)	0.7576(31)	0.3812(79)	-0.1332(22)	0.051(14)
C(1)	0.5063(31)	0.5662(83)	0.0758(23)	0.008(13)
C(2)	0.5306(23)	0.5553(61)	0.1344(16)	0.006(9)
C(3)	0.6035(23)	0.5637(65)	0.1611(17)	0.006(10)
C(4)	0.6639(30)	0.5124(85)	0.1355(23)	0.034(14)
C(5)	0.7585(30)	0.3412(80)	0.0222(21)	0.027(13)
C(6)	0.7811(39)	0.3366(99)	-0.0221(28)	0.052(18)
C(7)	0.7281(25)	0.3929(67)	-0.0811(17)	0.009(10)
C(8)	0.6486(32)	0.4432(91)	-0.0825(23)	0.037(15)

C(9)	0.5477(23)	0.4698(66)	-0.0242(17)	0.005(9)
C(10)	0.5577(32)	0.4888(87)	0.0432(23)	0.036(14)
C(11)	0.6456(23)	0.4507(69)	0.0723(17)	0.005(9)
C(12)	0.6833(33)	0.4113(86)	0.0283(24)	0.035(14)
C(13)	0.6308(26)	0.4487(73)	-0.0310(20)	0.019(11)

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE B

Br(2)	0.0000(0)	0.0000(0)	0.0000(0)	0.054(0)
O(8)	0.2872(16)	0.0293(48)	0.2436(12)	0.014(7)
O(9)	0.3826(24)	0.1805(61)	0.0511(17)	0.048(11)
O(10)	0.2922(22)	0.1555(58)	-0.0296(16)	0.039(10)
O(11)	-0.0114(25)	-0.3225(65)	0.0706(18)	0.056(12)
O(12)	-0.1009(26)	-0.2199(68)	0.0842(19)	0.057(12)
O(13)	-0.0669(34)	-0.1173(90)	0.2944(24)	0.097(19)
O(14)	0.0717(26)	-0.0981(68)	0.3568(19)	0.061(13)
N(4)	0.3131(32)	0.1540(88)	0.0211(23)	0.058(16)
N(5)	-0.0432(19)	-0.2533(52)	0.0890(14)	0.003(8)
N(6)	0.0156(24)	-0.1054(62)	0.3066(17)	0.025(10)
C(14)	0.2790(23)	0.0446(66)	0.1144(17)	0.008(9)
C(15)	0.2458(45)	0.0734(118)	0.0474(33)	0.077(23)
C(16)	0.1617(30)	0.0620(83)	0.0115(22)	0.030(13)
C(17)	0.1154(26)	0.0070(72)	0.0452(19)	0.017(11)
C(18)	0.0173(26)	-0.1441(68)	0.1435(18)	0.012(10)
C(19)	-0.0117(27)	-0.1697(68)	0.1988(19)	0.012(10)
C(20)	0.0460(28)	-0.1113(73)	0.2510(20)	0.022(12)

C(21)	0.1220(23)	-0.0614(62)	0.2611(16)	0.006(9)
C(22)	0.2336(30)	0.0242(83)	0.2003(22)	0.029(13)
C(23)	0.2200(25)	-0.0000(69)	0.1383(18)	0.012(10)
C(24)	0.1481(28)	0.0003(78)	0.1081(20)	0.024(12)
C(25)	0.0972(29)	-0.0735(78)	0.1515(21)	0.027(13)
C(26)	0.1482(26)	-0.0424(74)	0.2044(19)	0.020(11)

DIMETHYL BENZOPHENANTHRENE MOLECULE A

C(27)	0.5314(27)	0.1739(72)	0.2666(20)	0.020(12)
C(28)	0.5028(36)	0.1710(94)	0.2094(26)	0.047(17)
C(29)	0.5562(30)	0.1011(77)	0.1733(21)	0.024(12)
C(30)	0.6304(46)	0.0496(120)	0.1989(35)	0.074(23)
C(31)	0.7505(32)	0.0476(89)	0.2883(24)	0.042(14)
C(32)	0.7854(34)	0.0811(89)	0.3448(25)	0.039(15)
C(33)	0.7833(38)	0.1121(109)	0.4435(28)	0.054(18)
C(34)	0.7400(39)	0.1429(102)	0.4822(28)	0.058(18)
C(35)	0.6066(27)	0.0664(75)	0.5124(19)	0.020(11)
C(36)	0.5262(46)	-0.0092(121)	0.4993(35)	0.082(23)
C(37)	0.4911(28)	-0.0768(73)	0.4408(20)	0.021(12)
C(38)	0.5302(31)	-0.0486(86)	0.3982(23)	0.034(14)
C(39)	0.6666(42)	0.0736(109)	0.2614(30)	0.078(20)
C(40)	0.7385(27)	0.1024(70)	0.3852(19)	0.017(11)
C(41)	0.6532(34)	0.0762(93)	0.4744(25)	0.044(16)
C(42)	0.6132(30)	0.1041(78)	0.2972(21)	0.022(12)
C(43)	0.6498(40)	0.0769(107)	0.3645(29)	0.058(19)

C(44)	0.6076(23)	0.0411(66)	0.4066(17)	0.006(9)
C(45)	0.4846(25)	0.2709(69)	0.3053(19)	0.011(11)
C(46)	0.4873(24)	-0.1533(64)	0.3322(17)	0.005(10)

DIMETHYL BENZOPHENANTHRENE MOLECULE B

C(47)	0.2411(25)	0.5412(72)	0.2887(19)	0.014(10)
C(48)	0.2830(36)	0.5737(98)	0.2411(26)	0.047(16)
C(49)	0.2397(26)	0.5055(71)	0.1811(18)	0.018(10)
C(50)	0.1715(35)	0.4455(95)	0.1684(26)	0.006(16)
C(51)	0.0455(31)	0.3681(81)	0.1975(22)	0.033(13)
C(52)	0.0027(34)	0.3498(81)	0.2398(24)	0.038(14)
C(53)	-0.0104(36)	0.4175(94)	0.3435(25)	0.045(16)
C(54)	0.0350(30)	0.4535(82)	0.4066(22)	0.036(13)
C(55)	0.1492(39)	0.4435(107)	0.4830(28)	0.056(18)
C(56)	0.2324(44)	0.3927(106)	0.5139(30)	0.063(20)
C(57)	0.2743(38)	0.3410(100)	0.4704(28)	0.053(18)
C(58)	0.2358(33)	0.3352(88)	0.4038(23)	0.035(14)
C(59)	0.1290(25)	0.4101(66)	0.2172(18)	0.012(10)
C(60)	0.0399(46)	0.4053(119)	0.3050(34)	0.073(22)
C(61)	0.1157(21)	0.4397(60)	0.4244(15)	0.005(9)
C(62)	0.1657(30)	0.4611(82)	0.2711(22)	0.030(13)
C(63)	0.1199(23)	0.4078(60)	0.3202(17)	0.004(9)
C(64)	0.1568(34)	0.4042(91)	0.3810(25)	0.039(15)
C(65)	0.2706(28)	0.6735(73)	0.3388(20)	0.017(11)
C(66)	0.2775(31)	0.2058(78)	0.3680(22)	0.038(13)

TABLE 2.2

ORTHOGONAL COORDINATES AND E.S.D.S

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE A

ATOM	X'	Y	Z'
Br(1)	12.700 ± 7	3.922 ± 7	0.368 ± 7
O(1)	7.902 ± 33	3.725 ± 40	-3.760 ± 33
O(2)	6.797 ± 53	4.799 ± 61	1.181 ± 54
O(3)	8.241 ± 41	4.664 ± 46	2.692 ± 40
O(4)	12.627 ± 42	1.056 ± 47	-1.088 ± 41
O(5)	14.511 ± 48	1.701 ± 51	-2.128 ± 45
O(6)	13.607 ± 37	2.639 ± 43	-6.984 ± 36
O(7)	11.758 ± 39	2.963 ± 43	-7.627 ± 38
N(1)	7.796 ± 39	4.502 ± 45	1.526 ± 38
N(2)	13.495 ± 32	1.750 ± 39	-1.924 ± 32
N(3)	12.436 ± 51	2.713 ± 56	-6.788 ± 49
C(1)	8.312 ± 51	4.029 ± 59	-0.708 ± 50
C(2)	8.711 ± 38	4.095 ± 43	0.528 ± 37
C(3)	9.907 ± 38	4.012 ± 46	0.814 ± 38
C(4)	10.897 ± 49	3.647 ± 60	-0.095 ± 51
C(5)	12.451 ± 49	2.428 ± 57	-3.179 ± 49
C(6)	12.823 ± 64	2.391 ± 71	-4.416 ± 62
C(7)	11.952 ± 41	2.796 ± 47	-5.435 ± 39
C(8)	10.647 ± 53	3.154 ± 64	-5.079 ± 52
C(9)	8.990 ± 38	3.344 ± 47	-3.233 ± 38

C(10)	9.154 ± 52	3.436 ± 62	-1.717 ± 51
C(11)	10.597 ± 38	3.207 ± 49	-1.469 ± 38
C(12)	11.212 ± 55	2.927 ± 61	-2.674 ± 54
C(13)	10.355 ± 43	3.194 ± 52	-3.798 ± 44

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE B

Br(2)	0.000 ± 0	0.000 ± 0	0.000 ± 0
O(8)	4.714 ± 27	0.209 ± 34	4.258 ± 27
O(9)	6.281 ± 39	1.285 ± 43	-0.672 ± 38
O(10)	4.797 ± 37	1.107 ± 41	-2.113 ± 36
O(11)	-0.188 ± 41	-2.296 ± 47	1.695 ± 40
O(12)	-1.656 ± 43	-1.565 ± 48	2.448 ± 43
O(13)	-1.000 ± 57	-0.835 ± 64	7.134 ± 55
O(14)	1.177 ± 42	-0.698 ± 48	7.937 ± 41
N(4)	5.139 ± 52	1.096 ± 63	-1.031 ± 51
N(5)	-0.709 ± 32	-1.803 ± 37	2.278 ± 32
N(6)	0.256 ± 39	-0.750 ± 44	7.045 ± 37
C(14)	4.580 ± 38	0.325 ± 47	1.300 ± 39
C(15)	4.035 ± 74	0.522 ± 84	-0.097 ± 75
C(16)	2.655 ± 50	0.441 ± 59	-0.521 ± 49
C(17)	1.895 ± 42	0.050 ± 52	0.487 ± 43
C(18)	0.284 ± 43	-1.026 ± 48	3.248 ± 41
C(19)	-0.193 ± 44	-1.208 ± 48	4.674 ± 43
C(20)	0.756 ± 46	-0.828 ± 52	5.605 ± 45

C(21)	2.003 ± 37	-0.437 ± 44	5.469 ± 36
C(22)	3.786 ± 49	0.172 ± 59	3.528 ± 50
C(23)	3.621 ± 41	-0.014 ± 49	2.135 ± 40
C(24)	2.431 ± 46	0.002 ± 56	1.787 ± 45
C(25)	1.596 ± 48	-0.523 ± 56	3.044 ± 48
C(26)	2.432 ± 43	-0.301 ± 52	4.033 ± 43

DIMETHYL BENZOPHENANTHRENE MOLECULE A

C(27)	8.723 ± 45	1.238 ± 51	3.600 ± 45
C(28)	8.254 ± 60	1.217 ± 67	2.410 ± 59
C(29)	9.131 ± 49	0.725 ± 55	1.325 ± 47
C(30)	10.348 ± 76	0.353 ± 85	1.544 ± 77
C(31)	12.320 ± 53	0.339 ± 63	3.034 ± 53
C(32)	12.893 ± 56	0.580 ± 63	4.177 ± 55
C(33)	12.858 ± 63	0.798 ± 78	6.478 ± 62
C(34)	12.148 ± 65	1.017 ± 72	7.598 ± 63
C(35)	9.957 ± 44	0.451 ± 53	8.941 ± 44
C(36)	8.638 ± 75	-0.066 ± 86	9.029 ± 77
C(37)	8.071 ± 45	-0.547 ± 52	7.840 ± 44
C(38)	8.704 ± 52	-0.346 ± 61	6.661 ± 52
C(39)	10.942 ± 68	0.524 ± 78	2.819 ± 66
C(40)	12.124 ± 44	0.729 ± 50	5.344 ± 43
C(41)	10.722 ± 56	0.542 ± 66	7.833 ± 57
C(42)	10.066 ± 49	0.741 ± 56	3.910 ± 48
C(43)	10.668 ± 66	0.548 ± 76	5.294 ± 64

C(44)	9.975 ± 37	0.292 ± 47	6.480 ± 38
C(45)	7.954 ± 42	1.928 ± 49	4.727 ± 42
C(46)	7.999 ± 40	-1.091 ± 46	5.354 ± 39

DIMETHYL BENZOPHENANTHRENE MOLECULE B

C(47)	3.957 ± 42	3.852 ± 51	5.528 ± 42
C(48)	4.645 ± 59	4.062 ± 70	4.219 ± 58
C(49)	3.935 ± 42	3.597 ± 51	3.052 ± 41
C(50)	2.815 ± 58	3.171 ± 67	3.075 ± 59
C(51)	0.743 ± 51	2.620 ± 58	4.367 ± 50
C(52)	0.044 ± 56	2.490 ± 58	5.557 ± 52
C(53)	-0.171 ± 59	2.971 ± 67	8.027 ± 57
C(54)	0.575 ± 49	3.228 ± 59	9.258 ± 49
C(55)	2.449 ± 63	3.157 ± 76	10.489 ± 63
C(56)	3.815 ± 72	2.795 ± 76	10.801 ± 68
C(57)	4.502 ± 63	2.427 ± 71	9.586 ± 62
C(58)	3.871 ± 54	2.386 ± 62	8.228 ± 52
C(59)	2.117 ± 41	2.919 ± 47	4.414 ± 40
C(60)	0.642 ± 76	2.884 ± 85	6.893 ± 75
C(61)	1.899 ± 35	3.129 ± 43	9.293 ± 35
C(62)	2.720 ± 49	3.281 ± 59	5.488 ± 48
C(63)	1.966 ± 38	2.902 ± 43	6.851 ± 38
C(64)	2.574 ± 56	2.877 ± 64	8.083 ± 56
C(65)	4.442 ± 46	4.793 ± 52	6.539 ± 45
C(66)	4.555 ± 50	1.464 ± 56	7.194 ± 49

TABLE 2.3

FINAL OBSERVED AND CALCULATED STRUCTURE AMPLITUDES.

H	K	L	Fo	Fc
20	0	-8	31.3	26.5
19	0	-4	24.9	24.3
18	0	0	45.3	50.5
18	0	-24	23.2	20.3
17	0	4	26.7	19.5
17	0	0	43.7	43.4
16	0	-14	34.5	35.4
16	0	-8	24.9	34.4
16	0	-4	64.2	73.8
16	0	-2	51.9	46.5
16	0	4	22.6	19.9
16	0	10	25.5	21.7
15	0	8	42.3	45.0
15	0	-4	22.4	29.3
15	0	-8	38.1	47.3
15	0	-10	22.7	19.7
15	0	-14	58.2	65.7
14	0	-16	41.9	55.4
14	0	-14	42.9	49.5
14	0	-8	52.2	54.4
14	0	-6	32.9	34.6
14	0	-4	44.3	45.1
14	0	0	32.8	29.2
14	0	4	26.9	24.0
14	0	6	32.5	30.9
13	0	10	40.0	36.6
13	0	6	77.2	85.4
13	0	4	43.6	45.2
13	0	2	21.5	31.1
13	0	0	47.8	45.0
13	0	-4	32.3	30.9
13	0	-6	51.4	59.3
13	0	-8	91.4	108.9
13	0	-18	24.0	36.9
12	0	-18	51.4	54.2
12	0	-14	77.4	80.9
12	0	-10	29.7	30.2
12	0	-8	73.8	76.2
12	0	-2	45.4	47.8
12	0	2	46.9	30.6
12	0	4	33.2	21.2
12	0	6	59.7	50.6
12	0	14	27.9	26.4
11	0	-26	24.5	28.7
11	0	-24	23.4	20.9
11	0	-22	25.8	25.9
11	0	-18	20.7	16.7
11	0	-16	24.9	26.0
11	0	-14	45.0	47.5
11	0	-12	23.3	27.1
11	0	-10	23.2	28.2
11	0	-8	74.5	79.5
11	0	-4	33.6	34.3
11	0	-2	54.0	57.5
11	0	2	78.3	75.9
11	0	4	35.5	37.5
11	0	6	19.2	23.8
11	0	8	45.4	43.2
11	0	10	29.9	39.0
11	0	12	47.9	41.4
10	0	14	56.2	48.1
10	0	12	72.9	72.0
10	0	10	50.3	68.1
10	0	8	20.7	17.2
10	0	6	46.9	52.2
10	0	2	66.5	60.7
10	0	0	21.3	30.4
10	0	-4	43.7	56.0
10	0	-6	28.1	33.0
10	0	-8	53.0	56.4
10	0	-16	58.5	67.5
9	0	-22	24.8	32.1
9	0	-16	22.8	31.6
9	0	-14	20.2	26.8
9	0	-12	37.2	52.1
9	0	-8	92.0	87.6
9	0	-6	72.1	74.8
9	0	-4	67.8	55.0
9	0	-2	99.0	98.2
9	0	0	34.4	40.2
9	0	2	51.3	60.6
9	0	6	68.0	66.2
9	0	16	26.0	22.6
9	0	22	26.3	24.1
8	0	16	28.2	33.2
8	0	14	52.9	59.7
8	0	10	77.9	72.4
8	0	8	17.3	25.0
8	0	6	41.1	40.5
8	0	4	58.1	67.4
8	0	0	224.5	217.3
8	0	-2	81.6	73.7
8	0	-4	52.2	40.0
8	0	-8	86.5	74.6
8	0	-10	22.1	19.9
8	0	-12	30.4	41.5
8	0	-14	21.6	32.2
8	0	-16	26.8	25.3
8	0	-18	67.3	69.3
8	0	-20	33.3	32.7
8	0	-24	26.5	26.4
7	0	-22	44.9	44.0
7	0	-18	59.9	64.4
7	0	-14	44.2	57.5
7	0	-12	90.6	90.2
7	0	-8	141.6	130.9
7	0	-6	52.0	47.4
7	0	-4	47.2	56.5
7	0	2	94.2	76.2
7	0	4	124.3	111.0
7	0	6	31.0	36.5
7	0	8	64.4	71.9
7	0	10	69.9	62.6
7	0	14	25.4	29.2
7	0	16	24.9	22.8
7	0	20	29.6	13.9
6	0	20	32.1	27.8
6	0	18	40.1	32.3
6	0	16	28.2	33.8
6	0	14	81.8	70.5
6	0	12	54.8	58.5
6	0	10	30.7	29.3
6	0	4	15.5	24.0
6	0	2	51.4	53.2
6	0	0	171.4	135.6
6	0	-2	71.5	60.5
6	0	-4	135.8	145.7
6	0	-6	25.0	30.9
6	0	-8	172.5	137.9
6	0	-10	73.2	69.1
6	0	-14	31.2	43.2
6	0	-16	60.2	79.2
6	0	-18	28.4	19.6
6	0	-22	30.8	28.5
5	0	-22	38.8	45.2
5	0	-20	44.7	48.9
5	0	-18	77.0	86.8

H	K	L	Fo	Fc
5	0	-16	48.1	56.8
5	0	-14	49.3	53.1
5	0	-10	97.9	105.3
5	0	-8	61.9	45.1
5	0	-6	130.8	120.7
5	0	-4	13.9	17.6
5	0	-2	90.0	75.2
5	0	0	37.4	53.0
5	0	2	99.9	90.4
5	0	4	60.7	54.1
5	0	6	127.2	112.9
5	0	8	71.3	66.7
5	0	10	89.5	78.3
5	0	12	47.8	47.3
5	0	14	28.3	18.8
5	0	16	23.2	17.7
5	0	20	40.1	30.4
4	0	18	27.2	30.5
4	0	16	78.0	70.3
4	0	14	39.9	34.4
4	0	12	41.2	36.4
4	0	10	81.6	69.0
4	0	8	31.1	16.5
4	0	6	36.8	78.9
4	0	4	128.5	107.1
4	0	2	32.9	32.5
4	0	0	285.6	271.3
4	0	-2	66.8	38.6
4	0	-4	65.7	73.5
4	0	-6	173.7	161.3
4	0	-8	64.2	36.7
4	0	-10	40.2	28.6
4	0	-12	56.5	60.8
4	0	-14	55.2	48.7
4	0	-16	22.9	31.5
4	0	-18	71.0	76.7
3	0	-22	21.6	21.1
3	0	-20	38.5	47.7
3	0	-18	50.4	54.4
3	0	-16	27.9	40.1
3	0	-12	85.9	79.9
3	0	-10	71.6	65.8
3	0	-8	124.2	108.6
3	0	-6	92.3	89.6
3	0	-4	147.3	139.2
3	0	-2	14.3	44.9
3	0	2	141.0	134.6
3	0	4	144.9	133.7
3	0	6	78.4	54.4
3	0	8	53.3	48.4
3	0	12	42.2	36.3
3	0	14	34.9	41.6
3	0	16	54.3	44.9
3	0	20	26.0	27.1
3	0	22	36.5	27.4
3	0	26	27.5	29.2
2	0	-20	82.3	94.5
2	0	-18	19.1	21.3
2	0	-16	51.0	51.9
2	0	-14	59.5	61.2
2	0	-12	53.7	62.2
2	0	-10	74.1	70.1
2	0	-8	64.8	71.2
2	0	-6	92.7	74.1
2	0	-4	9.8	6.9
2	0	4	164.2	156.9
2	0	6	25.4	28.6
2	0	8	84.0	55.1
2	0	10	62.8	54.0
2	0	12	87.9	87.2
2	0	14	135.2	122.2
2	0	18	51.7	47.9
1	0	20	37.8	34.5
1	0	14	71.2	61.7
1	0	12	95.9	84.8
1	0	10	76.1	66.3
1	0	8	43.3	45.1
1	0	6	14.4	15.2
1	0	4	178.4	158.5
1	0	-6	54.8	57.3
1	0	-8	132.5	109.9
1	0	-10	161.8	160.5
1	0	-12	64.0	61.6
1	0	-16	39.9	47.4
1	0	-18	26.1	32.2
1	0	-20	53.6	62.9
1	0	-22	46.8	46.7
0	0	4	52.8	62.8
0	0	6	146.1	121.6
0	0	10	101.0	87.9
0	0	12	44.2	61.0
0	0	16	30.6	30.9
18	1	2	28.3	15.4
16	1	-14	23.3	19.0
17	1	-9	24.9	20.3
17	1	-8	23.5	29.1
17	1	-4	48.2	53.6
17	1	-2	23.1	22.0
17	1	0	23.8	16.8
17	1	2	39.0	42.6
16	1	12	28.2	12.2
16	1	-10	25.6	33.1
16	1	-13	22.7	19.6
15	1	-18	29.7	40.1
15	1	-12	22.3	18.5
15	1	-11	20.5	23.4
15	1	-10	77.2	77.3
15	1	-8	36.3	43.7
15	1	-2	33.1	29.0
15	1	0	40.8	47.4
15	1	4	30.5	28.0
15	1	6	25.3	22.5
14	1	8	32.7	31.5
14	1	4	23.3	18.8
14	1	0	37.7	38.0
14	1	-5	26.9	23.8
14	1	-6	62.1	61.8
14	1	-8	25.9	24.1
14	1	-9	34.7	25.5
14	1	-11	22.2	22.4
14	1	-14	25.9	33.3
13	1	14	25.1	21.5
13	1	10	22.4	27.0
13	1	3	19.8	16.8
13	1	2	36.3	31.5
13	1	1	19.9	12.0
13	1	0	28.1	30.4
13	1	-2	29.3	37.7
13	1	-3	30.6	29.9
13	1	-4	40.9	44.9
13	1	-6	26.0	21.5
13	1	-10	82.5	91.3
13	1	-12	21.1	14.3
13	1	-13	24.7	19.6
13	1	-14	62.6	77.9
13	1	-20	24.3	31.3
12	1	-14	25.5	31.1

H	K	L	Fo	Fc
12	1	-13	49.7	44.5
12	1	-10	51.2	66.0
12	1	-6	26.4	35.9
12	1	-4	60.5	62.8
12	1	-2	21.6	32.1
12	1	2	54.6	62.6
12	1	5	20.5	20.6
12	1	6	71.5	63.9
12	1	14	24.3	16.1
11	1	16	27.8	20.4
11	1	11	23.0	21.2
11	1	10	62.7	67.6
11	1	6	76.9	73.9
11	1	1	22.9	23.3
11	1	0	47.8	48.7
11	1	-2	56.4	56.5
11	1	-4	49.3	53.7
11	1	-6	35.8	38.2
11	1	-3	19.8	19.8
11	1	-11	34.1	34.3
11	1	-12	19.9	23.5
11	1	-14	33.8	41.0
11	1	-16	29.8	31.8
11	1	-20	21.2	14.4
11	1	-22	28.9	28.2
10	1	-14	23.1	30.5
10	1	-13	19.3	11.2
10	1	-12	35.2	42.9
10	1	-8	76.7	83.5
10	1	-6	63.0	67.7
10	1	-4	16.8	19.9
10	1	-3	16.5	11.6
10	1	-2	77.8	88.0
10	1	0	65.3	64.7
10	1	2	22.3	26.6
10	1	3	35.3	31.5
10	1	4	60.0	50.4
10	1	6	27.0	26.8
10	1	7	34.0	32.9
10	1	9	19.2	27.4
10	1	10	45.1	46.4
10	1	14	30.0	25.7
9	1	19	30.2	13.3
9	1	14	41.5	39.3
9	1	9	42.4	43.1
9	1	8	45.5	45.5
9	1	7	18.7	21.9
9	1	6	24.6	26.2
9	1	0	25.9	18.2
9	1	-2	58.0	67.6
9	1	-3	54.6	47.1
9	1	-4	38.6	49.9
9	1	-8	30.9	35.2
9	1	-9	29.5	32.1
9	1	-10	17.0	22.6
9	1	-12	32.5	32.2
9	1	-13	18.0	20.2
9	1	-14	25.5	35.9
9	1	-16	18.8	22.7
9	1	-18	33.7	37.4
9	1	-20	27.5	35.6
9	1	-24	32.4	24.6
9	1	-30	27.6	30.5
8	1	-20	68.9	73.3
8	1	-19	21.0	34.8
8	1	-18	45.1	47.2
8	1	-16	20.9	24.3
8	1	-14	39.4	50.3
8	1	-10	76.5	85.7
8	1	-6	38.0	37.4
8	1	-4	83.3	84.0
8	1	-3	27.6	26.8
8	1	0	35.9	31.4
8	1	1	32.6	35.9
8	1	2	99.2	89.8
8	1	3	15.1	27.3
8	1	4	37.2	32.3
8	1	5	24.7	24.5
8	1	6	61.2	51.6
8	1	8	74.6	68.9
8	1	16	21.9	22.0
8	1	18	24.8	25.0
7	1	-22	27.7	32.4
7	1	-19	30.0	33.6
7	1	-18	67.8	62.6
7	1	-16	55.6	63.9
7	1	-15	19.2	17.9
7	1	-12	51.6	53.1
7	1	-11	77.2	67.8
7	1	-10	156.5	141.0
7	1	-8	17.2	13.1
7	1	-7	33.6	35.1
7	1	-4	114.0	111.7
7	1	-3	18.8	18.2
7	1	-2	46.4	51.0
7	1	0	50.8	56.4
7	1	2	113.4	112.5
7	1	4	37.8	37.0
7	1	5	34.3	25.1
7	1	6	59.7	56.5
7	1	8	47.8	49.1
7	1	10	20.4	15.7
7	1	12	60.8	46.8
7	1	13	25.2	24.0
7	1	16	26.1	22.8
6	1	22	25.2	21.9
6	1	20	22.8	20.7
6	1	17	24.1	16.7
6	1	16	28.7	24.1
6	1	12	63.8	62.4
6	1	11	36.3	27.5
6	1	10	89.3	86.5
6	1	9	22.0	23.3
6	1	8	27.5	26.1
6	1	7	21.6	17.4
6	1	6	80.1	73.9
6	1	4	141.5	127.0
6	1	3	43.9	51.3
6	1	1	41.4	32.8
6	1	0	29.7	33.4
6	1	-1	33.2	26.5
6	1	-2	61.6	63.2
6	1	-3	32.4	18.7
6	1	-4	17.7	20.8
6	1	-5	21.1	18.2
6	1	-6	138.3	124.2
6	1	-7	47.0	41.4
6	1	-8	35.2	42.3
6	1	-9	61.0	52.9
6	1	-10	22.0	13.4
6	1	-11	44.5	46.7
6	1	-12	45.3	50.9
6	1	-13	30.6	35.1
6	1	-15	18.6	16.3
6	1	-17	20.2	17.7
6	1	-20	23.5	31.1
6	1	-23	25.7	9.9

H	K	L	Fo	Fc
5	1	16	44.8	43.1
5	1	14	63.2	64.0
5	1	10	76.1	73.6
5	1	9	76.0	62.5
5	1	8	41.8	38.6
5	1	7	23.6	32.4
5	1	5	17.1	23.9
5	1	4	71.7	66.6
5	1	3	13.6	10.6
5	1	2	73.9	72.5
5	1	1	17.9	25.3
5	1	0	13.6	16.7
5	1	-2	84.6	69.7
5	1	-3	51.6	42.5
5	1	-4	48.5	39.6
5	1	-5	46.0	29.5
5	1	-6	95.1	86.9
5	1	-7	31.5	30.0
5	1	-8	28.7	12.1
5	1	-9	42.0	42.4
5	1	-10	150.1	131.3
5	1	-11	50.8	53.3
5	1	-12	38.2	40.9
5	1	-14	58.2	67.4
5	1	-17	35.7	35.8
5	1	-19	27.2	38.1
5	1	-20	23.2	31.5
4	1	-22	21.1	15.6
4	1	-20	33.8	30.7
4	1	-18	42.8	50.3
4	1	-16	43.3	44.8
4	1	-14	59.9	66.3
4	1	-12	76.2	69.5
4	1	-11	18.2	20.3
4	1	-10	82.3	91.7
4	1	-9	55.2	50.1
4	1	-8	119.0	123.1
4	1	-7	50.8	59.6
4	1	-6	38.5	36.5
4	1	-5	44.5	33.2
4	1	-4	71.2	70.5
4	1	-3	35.9	27.9
4	1	-2	84.1	77.6
4	1	-1	90.6	68.8
4	1	0	29.3	28.5
4	1	1	44.4	39.2
4	1	2	101.6	101.9
4	1	3	27.2	11.9
4	1	5	21.3	24.5
4	1	6	86.2	75.3
4	1	7	54.9	42.2
4	1	8	63.5	65.5
4	1	9	71.6	63.8
4	1	11	35.6	22.8
4	1	12	18.4	19.8
4	1	14	31.5	34.7
4	1	18	22.2	28.6
4	1	19	21.1	10.9
3	1	22	28.9	24.2
3	1	18	38.4	25.2
3	1	17	20.1	21.8
3	1	16	54.2	53.0
3	1	14	42.1	43.8
3	1	12	43.6	43.0
3	1	10	104.4	102.4
3	1	9	74.9	62.8
3	1	8	89.8	84.1
3	1	7	13.3	19.7
3	1	6	114.0	104.2
3	1	5	44.4	41.7
3	1	4	22.5	23.0
3	1	3	20.3	20.4
3	1	2	64.4	54.2
3	1	1	12.9	13.2
3	1	0	122.5	130.0
3	1	-1	56.3	48.5
3	1	-2	38.7	41.1
3	1	-3	56.4	50.2
3	1	-4	177.8	164.3
3	1	-5	134.2	111.2
3	1	-6	66.7	62.9
3	1	-7	32.6	25.3
3	1	-8	42.7	43.5
3	1	-9	56.6	41.0
3	1	-10	27.0	37.4
3	1	-11	19.0	18.6
3	1	-16	46.6	53.6
3	1	-19	28.9	25.6
2	1	-22	33.4	37.4
2	1	-20	27.1	25.9
2	1	-18	28.3	34.9
2	1	-17	23.3	31.3
2	1	-16	50.0	43.4
2	1	-15	18.3	27.1
2	1	-13	28.1	20.3
2	1	-12	131.7	123.0
2	1	-11	18.1	24.3
2	1	-10	93.3	74.9
2	1	-9	17.2	20.6
2	1	-8	32.0	30.7
2	1	-7	53.6	42.2
2	1	-6	205.9	173.4
2	1	-5	33.8	28.1
2	1	-4	57.9	53.3
2	1	-3	74.0	72.4
2	1	-2	19.7	27.1
2	1	-1	8.4	15.9
2	1	0	55.2	102.3
2	1	1	97.9	117.0
2	1	2	69.8	63.1
2	1	3	126.4	96.5
2	1	4	84.6	84.3
2	1	5	57.0	39.3
2	1	6	120.3	107.6
2	1	7	36.1	24.2
2	1	8	93.6	85.7
2	1	9	13.9	32.1
2	1	10	103.1	94.6
2	1	12	61.9	49.8
2	1	16	33.9	30.5
2	1	18	21.9	21.4
2	1	22	32.0	34.7
1	1	20	24.1	18.4
1	1	19	32.4	34.3
1	1	18	30.4	33.3
1	1	16	37.1	40.1
1	1	15	19.5	17.1
1	1	14	52.0	54.0
1	1	13	22.1	21.6
1	1	11	36.6	25.9
1	1	10	31.2	24.7
1	1	9	79.0	64.6
1	1	8	55.8	42.9
1	1	5	65.4	45.8
1	1	4	78.2	71.0
1	1	3	44.6	53.4
1	1	-4	75.0	60.1

H	K	L	Fo	Fc
1	1	-5	101.9	92.0
1	1	-6	139.5	144.6
1	1	-7	32.3	34.7
1	1	-8	55.7	62.7
1	1	-9	76.8	57.9
1	1	-10	62.0	50.3
1	1	-11	27.5	26.4
1	1	-12	31.1	37.2
1	1	-14	64.1	63.6
1	1	-16	22.2	22.6
1	1	-17	23.9	35.8
1	1	-20	34.7	33.5
0	1	21	21.7	15.5
0	1	20	77.7	85.2
0	1	18	21.7	28.6
0	1	16	35.4	34.9
0	1	14	42.9	46.7
0	1	13	24.4	24.9
0	1	12	18.6	13.8
0	1	11	88.2	49.6
0	1	10	66.0	66.3
0	1	8	151.9	143.2
0	1	7	13.5	22.7
0	1	6	29.6	22.5
0	1	5	82.3	65.1
0	1	4	154.5	141.3
19	2	-10	24.7	19.7
18	2	-4	32.6	32.0
17	2	-6	25.5	25.2
17	2	4	34.6	28.8
16	2	4	29.7	26.6
16	2	-6	23.0	19.6
15	2	-11	25.3	23.6
15	2	-10	30.9	44.3
15	2	-8	41.5	45.5
15	2	2	31.2	25.9
15	2	7	25.2	31.6
15	2	8	29.7	24.3
14	2	0	34.6	23.0
14	2	-2	23.2	33.2
14	2	-5	37.5	41.3
14	2	-8	43.9	49.3
14	2	-10	22.2	30.4
14	2	-12	27.5	28.0
14	2	-13	38.6	32.9
14	2	-16	27.6	37.4
13	2	-15	29.3	25.5
13	2	-13	35.4	28.0
13	2	-12	37.4	44.7
13	2	-11	28.0	35.7
13	2	-9	20.8	27.9
13	2	-8	23.5	24.2
13	2	-7	62.8	58.4
13	2	-6	53.2	60.6
13	2	-4	24.4	21.6
13	2	0	39.8	49.2
13	2	1	25.0	23.5
13	2	2	25.3	31.8
13	2	4	21.1	17.5
13	2	6	30.6	35.5
13	2	10	49.8	50.6
12	2	11	26.9	20.3
12	2	10	26.4	19.2
12	2	3	19.6	20.3
12	2	2	19.9	11.0
12	2	0	20.3	16.3
12	2	-1	25.7	13.9
12	2	-2	49.7	53.4
12	2	-4	31.2	28.2
12	2	-6	23.9	35.4
12	2	-8	39.3	49.3
12	2	-10	30.2	38.4
12	2	-12	19.8	19.4
12	2	-14	51.4	57.0
12	2	-18	34.8	40.5
11	2	-19	26.9	18.0
11	2	-14	25.2	23.8
11	2	-12	35.5	33.1
11	2	-11	16.8	19.2
11	2	-10	40.9	50.9
11	2	-9	35.3	38.2
11	2	-8	48.8	47.8
11	2	-7	31.5	28.9
11	2	-6	18.9	22.4
11	2	-5	18.0	22.6
11	2	-4	31.2	40.4
11	2	-3	46.0	37.6
11	2	-2	54.0	51.4
11	2	2	37.4	41.1
11	2	4	34.3	35.1
11	2	5	36.6	37.1
11	2	6	31.4	42.9
11	2	7	26.5	40.1
11	2	8	48.8	40.8
10	2	12	58.0	55.5
10	2	8	38.4	32.1
10	2	2	51.0	49.2
10	2	1	49.4	44.1
10	2	-2	31.2	33.1
10	2	-3	31.2	40.1
10	2	-4	49.0	51.6
10	2	-6	15.5	22.5
10	2	-9	23.3	34.6
10	2	-12	24.6	29.5
10	2	-15	26.5	26.6
10	2	-16	36.1	39.8
10	2	-20	35.0	36.0
10	2	-22	25.8	24.8
9	2	-24	30.8	36.8
9	2	-23	24.4	19.0
9	2	-22	24.8	32.1
9	2	-16	23.5	30.7
9	2	-12	37.8	42.8
9	2	-11	40.9	39.0
9	2	-10	46.0	43.8
9	2	-9	19.9	18.8
9	2	-7	56.2	51.4
9	2	-6	69.8	81.5
9	2	-4	32.1	34.0
9	2	-2	32.5	23.3
9	2	-1	43.3	45.9
9	2	0	64.6	63.5
9	2	1	42.3	34.4
9	2	2	24.4	24.1
9	2	3	18.4	30.9
9	2	6	61.2	53.3
9	2	7	26.0	20.1
9	2	11	47.0	50.6
9	2	14	22.3	14.2
9	2	17	25.4	23.6
9	2	24	30.4	22.1
8	2	16	35.9	24.5
8	2	14	25.9	24.7
8	2	13	23.4	31.5
8	2	12	27.1	34.6
8	2	11	40.8	36.1
8	2	10	54.8	52.0

H	K	L	Fo	Fc
8	2	9	39.9	43.9
8	2	4	40.4	43.1
8	2	2	21.3	16.2
8	2	1	24.2	27.3
8	2	0	96.8	87.2
8	2	-1	21.0	26.6
8	2	-2	60.9	67.6
8	2	-3	25.1	29.8
8	2	-4	27.9	31.1
8	2	-6	38.4	46.3
8	2	-8	20.9	7.5
8	2	-9	36.6	36.5
8	2	-10	54.7	46.7
8	2	-12	40.7	40.0
8	2	-14	38.1	40.2
8	2	-17	36.0	36.0
8	2	-18	21.7	41.5
7	2	-21	25.3	21.5
7	2	-20	33.3	35.6
7	2	-18	54.2	57.3
7	2	-14	29.1	26.0
7	2	-13	25.6	25.3
7	2	-12	59.0	57.8
7	2	-11	30.0	31.3
7	2	-10	29.8	40.4
7	2	-8	71.9	78.7
7	2	-7	50.0	55.8
7	2	-5	41.4	51.9
7	2	-4	85.8	78.4
7	2	-3	87.3	75.0
7	2	-2	19.4	23.0
7	2	-1	14.4	19.2
7	2	1	60.9	49.2
7	2	2	46.5	47.7
7	2	3	55.3	49.5
7	2	4	95.1	90.8
7	2	5	23.3	10.0
7	2	8	62.5	55.4
7	2	9	18.9	23.7
7	2	10	66.8	67.2
7	2	11	19.2	26.0
7	2	14	21.0	8.4
6	2	15	31.1	38.1
6	2	14	39.1	30.6
6	2	12	46.4	41.9
6	2	10	25.1	26.5
6	2	9	38.4	27.1
6	2	8	64.0	65.5
6	2	6	23.3	22.7
6	2	3	31.9	25.8
6	2	2	114.1	117.4
6	2	1	75.2	71.2
6	2	0	55.3	57.8
6	2	-1	42.7	39.5
6	2	-2	83.9	85.4
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7	4	0	26.5	28.0
7	4	1	23.2	31.6
7	4	2	42.6	40.3
7	4	4	30.2	39.4
7	4	5	32.3	33.7
7	4	8	37.1	36.2
7	4	9	35.2	26.5
6	4	15	26.3	16.8
6	4	14	33.6	21.0
6	4	13	26.5	20.8
6	4	12	32.1	28.3
6	4	9	63.0	50.8
6	4	8	39.2	37.0
6	4	7	30.3	18.9
6	4	5	23.0	35.0
6	4	2	63.0	56.8
6	4	1	32.6	37.5
6	4	-1	42.0	39.9
6	4	-2	46.0	47.4
6	4	-3	41.3	30.1
6	4	-6	58.3	42.6
6	4	-8	23.0	26.6
6	4	-9	35.0	30.2
6	4	-10	33.6	29.1
6	4	-12	26.1	23.1
6	4	-14	21.8	22.8
6	4	-15	44.0	39.2
6	4	-17	51.2	47.3
6	4	-19	32.9	35.3
5	4	-20	27.0	16.8
5	4	-14	25.9	29.6
5	4	-12	25.4	33.2
5	4	-11	43.9	41.4
5	4	-10	41.8	42.9
5	4	-9	18.4	18.8
5	4	-7	33.2	33.8
5	4	-6	36.8	44.8
5	4	-5	114.8	94.5
5	4	-1	54.8	51.3
5	4	0	21.1	29.5
5	4	1	14.9	16.6
5	4	2	21.9	30.3
5	4	3	22.6	26.5
5	4	4	49.2	45.1
5	4	5	41.2	36.2
5	4	6	47.7	45.4
5	4	7	52.4	39.1
4	4	16	45.5	38.0
4	4	14	22.8	20.3
4	4	12	25.1	22.9
4	4	10	28.4	25.5
4	4	6	15.9	24.9
4	4	5	19.0	17.9
4	4	4	24.9	33.9
4	4	3	42.5	65.0
4	4	-1	71.7	62.1
4	4	-2	12.9	15.9
4	4	-3	27.6	42.5
4	4	-4	39.7	44.6
4	4	-6	63.9	56.9
4	4	-7	47.6	39.1
4	4	-8	26.9	38.5
4	4	-10	42.8	37.7
4	4	-12	20.2	9.5
4	4	-13	25.6	13.9
4	4	-14	20.3	20.8
3	4	-19	56.5	52.4
3	4	-18	43.1	46.6
3	4	-14	22.4	25.1
3	4	-12	29.1	31.0
3	4	-9	25.1	26.5
3	4	-8	70.8	68.7
3	4	-7	43.3	39.2
3	4	-4	40.4	40.2
3	4	-3	87.6	84.4
3	4	-2	24.8	27.2
3	4	-1	73.8	69.0
3	4	0	53.2	59.0
3	4	1	69.0	61.4
3	4	2	25.9	28.9
3	4	3	36.2	22.3
3	4	5	76.0	64.3
3	4	6	22.9	19.9
3	4	7	29.2	36.9
3	4	8	34.5	31.1
3	4	9	34.2	29.5
3	4	11	50.0	39.5
3	4	21	23.6	10.0
2	4	17	24.4	21.0
2	4	14	30.3	36.3
2	4	13	20.1	16.9
2	4	12	29.3	23.7
2	4	9	21.7	18.7
2	4	8	33.8	27.4
2	4	4	47.3	50.2
2	4	0	14.6	19.9
2	4	-1	51.3	61.0
2	4	-2	162.3	148.3
2	4	-3	30.4	31.2
2	4	-4	23.1	25.3
2	4	-5	29.2	39.8
2	4	-6	20.3	16.9
2	4	-8	71.4	65.3
2	4	-9	38.3	31.4
2	4	-10	38.8	42.9
2	4	-11	24.3	17.0
2	4	-12	13.3	20.5
2	4	-13	25.7	26.2
1	4	-13	23.0	38.5
1	4	-12	57.2	63.2
1	4	-11	50.6	39.0
1	4	-10	43.9	41.0
1	4	-9	48.7	40.5
1	4	-8	41.2	40.5
1	4	-5	112.5	93.0
1	4	-4	12.2	18.0
1	4	-3	33.2	30.2
1	4	-2	50.9	61.2
1	4	-1	56.3	58.3
1	4	1	107.5	103.9
1	4	2	73.3	74.0
1	4	3	11.1	14.2
1	4	6	16.0	20.0
1	4	7	55.8	41.7
1	4	9	35.5	33.3
1	4	10	24.0	26.8
1	4	12	30.9	29.3
1	4	18	37.4	30.4
0	4	17	51.7	42.6
0	4	16	23.5	14.1
0	4	13	33.2	36.1
0	4	12	39.3	38.9
0	4	11	25.5	25.8
0	4	10	52.8	47.4
0	4	7	42.3	34.1
0	4	6	22.9	17.1
0	4	5	32.7	30.3
0	4	2	83.2	87.2
0	4	1	21.6	30.6
17	5	0	36.0	20.7
14	5	-8	34.5	19.5
14	5	1	33.3	12.3
14	5	11	34.1	15.4
13	5	3	27.3	7.4
13	5	-8	32.8	31.8
12	5	-22	32.5	17.6
12	5	-11	33.7	37.8
12	5	-2	28.9	17.4
11	5	11	37.8	27.5
11	5	1	25.7	21.8
11	5	-1	66.6	57.8
11	5	-11	39.1	31.1
11	5	-12	29.3	22.9
11	5	-19	33.9	14.2
11	5	-22	34.9	19.0
10	5	-24	34.1	15.8
10	5	-14	26.9	24.6
10	5	-13	32.3	14.4
10	5	-12	26.9	22.9
10	5	-10	24.9	24.2
10	5	-3	22.5	25.6
10	5	0	27.4	33.9
10	5	5	27.0	17.3
9	5	-14	30.5	27.1
9	5	-13	37.7	30.7
9	5	-4	21.9	32.3
9	5	-3	39.7	30.9
9	5	-1	25.5	29.1
9	5	15	37.3	10.2
8	5	12	29.1	12.3
8	5	10	26.4	18.6
8	5	5	36.3	35.1
8	5	3	36.1	24.0
8	5	1	21.6	8.1
8	5	0	21.6	30.3
8	5	-1	20.1	20.3
8	5	-3	24.7	19.0
8	5	-5	28.8	19.0
8	5	-8	32.2	21.1
8	5	-9	47.8	51.2
8	5	-13	33.0	31.6
8	5	-14	29.7	33.3
7	5	16	31.5	26.0
7	5	10	28.0	27.2
7	5	9	24.9	21.4
7	5	7	50.5	39.4
7	5	5	30.3	26.6
7	5	4	30.5	33.7
7	5	3	21.5	32.2
7	5	0	39.0	42.9
7	5	-1	51.5	47.2
7	5	-3	23.3	29.6
7	5	-5	22.1	19.4
7	5	-8	23.3	20.3
7	5	-11	21.8	17.2
6	5	-18	27.1	26.6
6	5	-13	35.5	33.5
6	5	-11	23.7	31.5
6	5	-10	29.3	31.3
6	5	-8	27.4	33.9
6	5	-7	21.0	15.8
6	5	-6	19.1	18.7
6	5	-4	17.3	26.0
6	5	0	30.5	29.3
6	5	1	68.0	59.0
6	5	6	41.3	47.5
6	5	7	36.0	35.2
6	5	11	30.8	29.0
5	5	15	34.3	34.5
5	5	7	34.2	37.1
5	5	1	17.3	37.2
5	5	0	17.3	19.0
5	5	-1	55.1	49.6
5	5	-2	27.3	34.4
5	5	-3	44.8	42.7
5	5	-4	32.8	27.1
5	5	-6	29.7	35.0
5	5	-8	34.9	31.6
5	5	-13	25.3	32.6
5	5	-14	23.9	30.2
5	5	-15	27.6	21.3
5	5	-17	25.3	23.3
4	5	-14	41.4	37.8
4	5	-13	24.1	23.5
4	5	-9	25.1	15.1
4	5	-8	46.6	51.9
4	5	-7	29.6	36.5
4	5	-5	19.3	16.6
4	5	-4	24.7	27.1
4	5	-3	52.5	47.3
4	5	1	28.1	25.4
4	5	5	34.5	30.4
4	5	6	40.7	39.6
4	5	7	50.5	34.1
4	5	8	42.3	41.1
4	5	9	51.8	41.4
4	5	14	25.7	24.6
4	5	21	36.1	24.7
4	5	23	56.5	12.8
3	5	12	26.0	24.4
3	5	6	29.4	19.1
3	5	3	22.0	36.3
3	5	2	23.5	37.7
3	5	1	22.4	24.4
3	5	-1	25.6	36.4
3	5	-2	26.5	33.1
3	5	-3	17.5	28.9
3	5	-4	49.3	54.5
3	5	-5	40.0	25.5
3	5	-6	22.1	33.0
3	5	-7	24.3	31.6
3	5	-8	36.0	39.6
3	5	-12	24.5	28.7
3	5	-13	29.5	37.0
3	5	-15	61.6	55.7
2	5	-14	26.7	19.3
2	5	-10	57.0	52.4
2	5	-7	57.4	49.9
2	5	-6	24.5	29.6
2	5	-5	55.6	55.4
2	5	-3	13.2	21.8
2	5	4	24.9	28.8
2	5	5	26.6	19.2
2	5	6	20.9	33.3
2	5	7	44.5	35.8
2	5	8	20.3	26.3
2	5	9	21.1	30.4
2	5	14	26.6	24.7
1	5	15	35.9	27.4
1	5	11	22.2	16.5
1	5	10	31.6	25.0
1	5	7	23.7	24.7
1	5	5	38.1	28.3
1	5	4	18.8	23.2
1	5	3	15.8	21.7
1	5	0	18.5	40.7
1	5	-2	25.2	38.9
1	5	-3	49.1	58.0
1	5	-6	29.9	30.1
1	5	-7	59.6	47.9
1	5	-13	31.6	31.0
1	5	-16	26.5	19.9
0	5	10	36.5	33.9
0	5	9	22.6	22.0
0	5	8	30.7	29.3
0	5	3	43.0	38.9
12	6	5	35.9	8.9
12	6	-1	28.4	26.7
11	6	-13	29.1	7.6
11	6	-9	39.9	29.2
11	6	-4	29.3	33.9
11	6	3	31.7	16.2
11	6	9	34.2	27.9
10	6	-8	34.0	31.3
9	6	-13	31.3	7.3
9	6	-7	23.2	24.7
9	6	-2	25.6	29.5
9	6	0	26.4	19.6
9	6	4	34.9	23.2
8	6	13	30.6	6.6
8	6	6	26.8	25.2
8	6	2	23.6	25.4
8	6	-2	21.7	14.8
8	6	-17	39.8	37.0
7	6	-13	31.0	32.8
7	6	-9	29.5	25.1
7	6	-4	23.8	21.8
7	6	-3	22.6	16.6
7	6	-2	38.2	31.6
7	6	5	27.1	24.0
7	6	8	25.5	18.5
6	6	3	32.3	27.0
6	6	0	18.7	22.4
6	6	-5	27.9	21.9
6	6	-9	25.0	21.6
6	6	-14	25.1	20.1
5	6	-9	24.7	29.9
5	6	-5	22.4	24.0
5	6	-4	28.7	25.6
5	6	-2	22.7	23.9
5	6	-1	33.4	30.4
5	6	0	17.0	23.3
5	6	4	39.1	35.8
5	6	7	24.7	29.5
5	6	9	24.1	24.3
5	6	19	32.7	12.4
4	6	11	24.6	24.2
4	6	4	18.9	21.7
4	6	3	49.3	43.4
4	6	2	29.7	30.3
4	6	1	28.3	29.2
4	6	-4	60.8	57.1
4	6	-6	35.4	28.7
4	6	-22	32.5	13.8
3	6	-14	24.9	28.8
3	6	-7	41.6	32.9
3	6	-5	15.2	12.7
3	6	-3	20.1	21.6
3	6	-2	28.9	41.8
3	6	1	50.8	52.5
3	6	2	25.2	33.6
3	6	5	22.7	18.9
2	6	15	29.6	18.4
2	6	5	17.2	14.0
2	6	3	17.6	14.2
2	6	1	18.2	29.1
2	6	0	24.6	33.4
2	6	-9	27.7	19.6
2	6	-18	30.7	24.0
1	6	-9	23.4	28.6
1	6	-7	19.6	32.2
1	6	-6	15.9	21.7
1	6	-5	16.4	13.9
1	6	-3	37.9	42.5
1	6	2	17.0	21.6
1	6	7	19.0	28.7
1	6	13	37.6	27.4
0	6	14	31.0	33.5
0	6	6	19.1	20.0
0	6	4	37.0	38.0
0	6	3	19.0	26.0
0	6	2	13.4	30.3
16	7	-11	36.5	10.3
14	7	-7	30.2	13.4
13	7	-12	32.8	3.2
11	7	1	28.7	13.5
8	7	8	27.1	11.6
8	7	-5	27.0	23.0
7	7	-20	30.9	13.2
7	7	-7	21.3	14.0
7	7	11	27.9	6.7
6	7	-16	27.7	16.6
5	7	-8	20.5	16.6
5	7	-4	20.3	34.4
5	7	-3	20.9	22.7
4	7	6	19.9	26.9
4	7	-8	24.7	27.3
4	7	-9	36.7	34.9
4	7	-11	24.7	23.0
2	7	11	30.5	24.5
1	7	-13	27.9	27.7
1	7	-3	10.9	23.7
0	7	7	21.3	31.9
0	7	5	15.5	21.9

TABLE 2.4

(A) ANALYSIS OF STRUCTURE FACTOR DATA AS A FUNCTION OF LAYER LINE INDEX. ALL FIGURES ARE ON ABSOLUTE SCALE.

k	$\Sigma\|F_o\|$	$\Sigma\|F_c\|$	$\Sigma\|\Delta\|$	N	R	$\Sigma\|\Delta\|/N$
0	13315	12990	1732	229	0.1301	7.56
1	15950	15469	2128	345	0.1334	6.17
2	14191	13797	1964	337	0.1384	5.83
3	11299	10969	1636	282	0.1448	5.80
4	7616	7339	1243	208	0.1632	5.98
5	4694	4363	1064	147	0.2266	7.24
6	2123	1951	502	76	0.2366	6.61
7	564	451	225	22	0.3985	10.23
ALL	69752	67329	10494	1646	0.1504	6.38

(B) ANALYSIS OF STRUCTURE FACTORS BY MAGNITUDE OF Fo.

RANGE	$\Sigma\|F_o\|$	$\Sigma\|F_c\|$	$\Sigma\|\Delta\|$	N	R	$\Sigma\|\Delta\|/N$
0 - 20	2289	2852	790	135	0.3451	5.85
20 - 40	25905	25266	5118	897	0.1976	5.71
40 - 80	26425	25355	3014	479	0.1141	6.29
80 - 100	6543	6044	723	73	0.1105	9.90
100 - 150	5571	5111	538	45	0.0966	11.96
150 - 300	3020	2709	311	17	0.1029	18.28

TABLE 2.5

BOND LENGTHS AND E.S.D.S IN ANGSTROMS.

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE A.

Br(1)- C(4)	1.88 ± 5	C(2) - C(3)	1.23 ± 5
O(1) - C(9)	1.27 ± 5	C(3) - C(4)	1.39 ± 6
O(2) - N(1)	1.10 ± 7	C(4) - C(11)	1.47 ± 6
O(3) - N(1)	1.26 ± 6	C(5) - C(6)	1.29 ± 8
O(4) - N(2)	1.39 ± 6	C(5) - C(12)	1.43 ± 7
O(5) - N(2)	1.04 ± 6	C(6) - C(7)	1.40 ± 7
O(6) - N(3)	1.19 ± 6	C(7) - C(8)	1.40 ± 7
O(7) - N(3)	1.11 ± 6	C(8) - C(13)	1.32 ± 7
N(1) - C(2)	1.41 ± 5	C(9) - C(10)	1.53 ± 6
N(2) - C(5)	1.77 ± 6	C(9) - C(13)	1.48 ± 6
N(3) - C(7)	1.44 ± 6	C(10)- C(11)	1.48 ± 7
C(1) - C(2)	1.30 ± 6	C(11)- C(12)	1.38 ± 7
C(1) - C(10)	1.44 ± 7	C(12)- C(13)	1.44 ± 7

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE B.

Br(2)- C(17)	1.96 ± 4	O(11)- N(5)	0.92 ± 6
O(8) - C(22)	1.18 ± 6	O(12)- N(5)	0.99 ± 6
O(9) - N(4)	1.21 ± 7	O(13)- N(6)	1.26 ± 7
O(10)- N(4)	1.13 ± 6	O(14)- N(6)	1.28 ± 6

N(4) - C(15)	1.56 ± 9	C(18)- C(25)	1.42 ± 7
N(5) - C(18)	1.59 ± 5	C(19)- C(20)	1.38 ± 6
N(6) - C(20)	1.53 ± 6	C(20)- C(21)	1.31 ± 6
C(14)- C(15)	1.51 ± 8	C(21)- C(26)	1.50 ± 6
C(14)- C(23)	1.32 ± 6	C(22)- C(23)	1.41 ± 6
C(15)- C(16)	1.45 ± 9	C(22)- C(26)	1.52 ± 7
C(16)- C(17)	1.32 ± 7	C(23)- C(24)	1.24 ± 6
C(17)- C(24)	1.41 ± 6	C(24)- C(25)	1.60 ± 7
C(18)- C(19)	1.51 ± 6	C(25)- C(26)	1.31 ± 6

DIMETHYL BENZOPHENANTHRENE MOLECULE A.

C(27)- C(28)	1.28 ± 7	C(35)- C(36)	1.42 ± 9
C(27)- C(42)	1.47 ± 7	C(35)- C(41)	1.35 ± 7
C(27)- C(45)	1.53 ± 6	C(36)- C(37)	1.40 ± 9
C(28)- C(29)	1.48 ± 8	C(37)- C(38)	1.35 ± 7
C(29)- C(30)	1.29 ± 9	C(38)- C(44)	1.43 ± 7
C(30)- C(39)	1.42 ± 10	C(38)- C(46)	1.66 ± 7
C(31)- C(32)	1.30 ± 8	C(39)- C(42)	1.42 ± 8
C(31)- C(39)	1.41 ± 9	C(40)- C(43)	1.47 ± 8
C(32)- C(40)	1.41 ± 7	C(41)- C(44)	1.57 ± 7
C(33)- C(34)	1.34 ± 9	C(42)- C(43)	1.52 ± 8
C(33)- C(40)	1.35 ± 8	C(43)- C(44)	1.40 ± 8
C(34)- C(41)	1.52 ± 9		

DIMETHYL BENZOPHENANTHRENE MOLECULE B.

C(47) - C(48)	1.49 ± 7	C(55) - C(56)	1.45 ± 10
C(47) - C(62)	1.36 ± 7	C(55) - C(61)	1.32 ± 7
C(47) - C(65)	1.46 ± 7	C(56) - C(57)	1.44 ± 9
C(48) - C(49)	1.44 ± 7	C(57) - C(58)	1.50 ± 8
C(49) - C(50)	1.20 ± 7	C(58) - C(64)	1.39 ± 8
C(50) - C(59)	1.53 ± 7	C(58) - C(66)	1.54 ± 8
C(51) - C(52)	1.39 ± 7	C(59) - C(62)	1.28 ± 6
C(51) - C(59)	1.41 ± 7	C(60) - C(63)	1.32 ± 9
C(52) - C(60)	1.52 ± 9	C(61) - C(64)	1.41 ± 7
C(53) - C(54)	1.46 ± 8	C(62) - C(63)	1.60 ± 7
C(53) - C(60)	1.40 ± 9	C(63) - C(64)	1.37 ± 7
C(54) - C(61)	1.33 ± 6		

TABLE 2.6

BOND ANGLES AND E.S.D.S IN DEGREES.

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE A.

O(2) - N(1) - O(3)	125 ± 5	C(5) - C(6) - C(7)	121 ± 6
O(2) - N(1) - C(2)	116 ± 4	N(3) - C(7) - C(6)	117 ± 4
O(3) - N(1) - C(2)	118 ± 4	N(3) - C(7) - C(8)	125 ± 4
O(4) - N(2) - O(5)	135 ± 5	C(6) - C(7) - C(8)	118 ± 4
O(4) - N(2) - C(5)	104 ± 3	C(7) - C(8) - C(13)	118 ± 5
O(5) - N(2) - C(5)	117 ± 4	O(1) - C(9) - C(10)	119 ± 4
O(6) - N(3) - O(7)	119 ± 5	O(1) - C(9) - C(13)	131 ± 4
O(6) - N(3) - C(7)	119 ± 4	C(10) - C(9) - C(13)	107 ± 3
O(7) - N(3) - C(7)	120 ± 5	C(1) - C(10) - C(9)	131 ± 5
C(2) - C(1) - C(10)	120 ± 5	C(1) - C(10) - C(11)	121 ± 4
N(1) - C(2) - C(1)	119 ± 4	C(9) - C(10) - C(11)	105 ± 4
N(1) - C(2) - C(3)	119 ± 4	C(4) - C(11) - C(10)	108 ± 4
C(1) - C(2) - C(3)	121 ± 4	C(4) - C(11) - C(12)	142 ± 4
C(2) - C(3) - C(4)	124 ± 4	C(10) - C(11) - C(12)	109 ± 4
Br(1) - C(4) - C(3)	119 ± 4	C(5) - C(12) - C(11)	140 ± 5
Br(1) - C(4) - C(11)	118 ± 3	C(5) - C(12) - C(13)	108 ± 4
C(3) - C(4) - C(11)	123 ± 4	C(11) - C(12) - C(13)	112 ± 4
N(2) - C(5) - C(6)	120 ± 5	C(8) - C(13) - C(9)	125 ± 4
N(2) - C(5) - C(12)	113 ± 4	C(8) - C(13) - C(12)	129 ± 5
C(6) - C(5) - C(12)	127 ± 5	C(9) - C(13) - C(12)	106 ± 4

4 BROMO - 2,5,7 TRINITROFLUORENONE MOLECULE B.

O(9) - N(4) - O(10)	124 ± 5	C(18)- C(19)- C(20)	113 ± 4
O(9) - N(4) - C(15)	123 ± 5	N(6) - C(20)- C(19)	115 ± 4
O(10)- N(4) - C(15)	111 ± 5	N(6) - C(20)- C(21)	113 ± 4
O(11)- N(5) - O(12)	141 ± 5	C(19)- C(20)- C(21)	132 ± 4
O(11)- N(5) - C(18)	107 ± 4	C(20)- C(21)- C(26)	113 ± 4
O(12)- N(5) - C(18)	112 ± 4	O(8) - C(22)- C(23)	135 ± 5
O(13)- N(6) - O(14)	132 ± 4	O(8) - C(22)- C(26)	120 ± 4
O(13)- N(6) - C(20)	113 ± 4	C(23)- C(22)- C(26)	101 ± 4
O(14)- N(6) - C(20)	115 ± 4	C(14)- C(23)- C(22)	120 ± 4
C(15)- C(14)- C(23)	111 ± 4	C(14)- C(23)- C(24)	121 ± 4
N(4) - C(15)- C(14)	110 ± 5	C(22)- C(23)- C(24)	113 ± 4
N(4) - C(15)- C(16)	121 ± 6	C(17)- C(24)- C(23)	129 ± 4
C(14)- C(15)- C(16)	127 ± 6	C(17)- C(24)- C(25)	123 ± 4
C(15)- C(16)- C(17)	110 ± 5	C(23)- C(24)- C(25)	106 ± 4
Br(2)- C(17)- C(16)	112 ± 3	C(18)- C(25)- C(24)	135 ± 4
Br(2)- C(17)- C(24)	127 ± 3	C(18)- C(25)- C(26)	123 ± 4
C(16)- C(17)- C(24)	120 ± 4	C(24)- C(25)- C(26)	102 ± 4
N(5) - C(18)- C(19)	109 ± 3	C(21)- C(26)- C(22)	127 ± 4
N(5) - C(18)- C(25)	131 ± 4	C(21)- C(26)- C(25)	121 ± 4
C(19)- C(18)- C(25)	118 ± 4	C(22)- C(26)- C(25)	112 ± 4

DIMETHYL BENZOPHENANTHRENE MOLECULE A.

C(28)- C(27)- C(42)	122 ± 5
C(28)- C(27)- C(45)	121 ± 5
C(42)- C(27)- C(45)	117 ± 4
C(27)- C(28)- C(29)	118 ± 5
C(28)- C(29)- C(30)	122 ± 5
C(29)- C(30)- C(39)	121 ± 7
C(32)- C(31)- C(39)	123 ± 5
C(31)- C(32)- C(40)	121 ± 5
C(34)- C(33)- C(40)	115 ± 6
C(33)- C(34)- C(41)	125 ± 6
C(36)- C(35)- C(41)	127 ± 5
C(35)- C(36)- C(37)	117 ± 6
C(36)- C(37)- C(38)	120 ± 5
C(37)- C(38)- C(44)	126 ± 5
C(37)- C(38)- C(46)	115 ± 4
C(44)- C(38)- C(46)	118 ± 4
C(30)- C(39)- C(31)	122 ± 6
C(30)- C(39)- C(42)	117 ± 6
C(31)- C(39)- C(42)	121 ± 6
C(32)- C(40)- C(33)	114 ± 5
C(32)- C(40)- C(43)	120 ± 5
C(33)- C(40)- C(43)	125 ± 5
C(34)- C(41)- C(35)	133 ± 5
C(34)- C(41)- C(44)	111 ± 4
C(35)- C(41)- C(44)	115 ± 4
C(27)- C(42)- C(39)	117 ± 5
C(27)- C(42)- C(43)	127 ± 5
C(39)- C(42)- C(43)	116 ± 5
C(40)- C(43)- C(42)	114 ± 5
C(40)- C(43)- C(44)	119 ± 5
C(42)- C(43)- C(44)	127 ± 5
C(38)- C(44)- C(41)	113 ± 4
C(38)- C(44)- C(43)	129 ± 4
C(41)- C(44)- C(43)	118 ± 4

DIMETHYL BENZOPHENANTHRENE MOLECULE B.

C(48)- C(47)- C(62)	117 ± 4	C(50)- C(59)- C(62)	118 ± 4
C(48)- C(47)- C(65)	111 ± 4	C(51)- C(59)- C(62)	123 ± 4
C(62)- C(47)- C(65)	126 ± 4	C(52)- C(60)- C(53)	120 ± 6
C(47)- C(48)- C(49)	116 ± 5	C(52)- C(60)- C(63)	112 ± 6
C(48)- C(49)- C(50)	124 ± 5	C(53)- C(60)- C(63)	127 ± 6
C(49)- C(50)- C(59)	120 ± 5	C(54)- C(61)- C(55)	116 ± 4
C(52)- C(51)- C(59)	119 ± 5	C(54)- C(61)- C(64)	118 ± 4
C(51)- C(52)- C(60)	122 ± 5	C(55)- C(61)- C(64)	126 ± 4
C(54)- C(53)- C(60)	113 ± 5	C(47)- C(62)- C(59)	125 ± 5
C(53)- C(54)- C(61)	121 ± 5	C(47)- C(62)- C(63)	120 ± 4
C(56)- C(55)- C(61)	126 ± 6	C(59)- C(62)- C(63)	115 ± 4
C(55)- C(56)- C(57)	109 ± 6	C(60)- C(63)- C(62)	120 ± 5
C(56)- C(57)- C(58)	125 ± 6	C(60)- C(63)- C(64)	114 ± 5
C(57)- C(58)- C(64)	118 ± 5	C(62)- C(63)- C(64)	124 ± 4
C(57)- C(58)- C(66)	116 ± 5	C(58)- C(64)- C(61)	115 ± 5
C(64)- C(58)- C(66)	124 ± 5	C(58)- C(64)- C(63)	121 ± 5
C(50)- C(59)- C(51)	117 ± 4	C(61)- C(64)- C(63)	124 ± 5

TABLE 2.7

NON - BONDED DISTANCES.

(A) INTERPLANAR DISTANCES LESS THAN 3.5 Å BETWEEN 4 - BROMO 2,5,7 - TRINITROFLUORENONE MOLECULE A AND DIMETHYL BENZOPHENANTHRENE MOLECULE A.

C(28)....N(1)	i	3.43 Å	C(36)....C(9)	iii	3.36 Å
C(28)....O(3)	i	3.46	C(37)....C(9)	iii	2.99
C(28)....C(2)	i	3.47	C(37)....O(1)	iii	3.18
C(29)....C(3)	i	3.42	C(37)....C(13)	iii	3.50
C(29)....C(2)	i	3.49	C(38)....C(8)	iii	3.42
C(31)....O(7)	iii	3.48	C(38)....C(9)	iii	3.47
C(32)....O(6)	iii	3.33	C(38)....C(13)	iii	3.49
C(32)....N(3)	iii	3.39	C(40)....N(3)	iii	3.50
C(33)....C(6)	iii	3.27	C(41)....C(13)	iv	3.40
C(34)....C(13)	iv	3.42	C(41)....C(5)	iii	3.49
C(34)....C(8)	iv	3.47	C(45)....O(3)	i	3.42
C(34)....C(6)	iii	3.50	C(45)....O(1)	iv	3.45
C(35)....O(4)	iii	3.45	C(46)....O(3)	v	3.00
C(35)....C(10)	iv	3.46			

(B) INTERPLANAR DISTANCES LESS THAN 3.5 Å BETWEEN 4 - BROMO 2,5,7 - TRINITROFLUORENONE MOLECULE B AND DIMETHYL BENZOPHENANTHRENE MOLECULE B.

C(47)....C(21)	ii	3.44 Å	C(56)....C(15)	iii	3.40 Å
C(48)....O(8)	ii	3.27	C(56)....C(16)	iii	3.45
C(48)....C(22)	ii	3.41	C(56)....N(4)	iv	3.49
C(49)....C(22)	i	3.46	C(59)....C(26)	i	3.26
C(50)....C(22)	i	3.18	C(59)....C(22)	i	3.33
C(50)....C(23)	i	3.42	C(62)....C(21)	ii	3.47
C(50)....C(24)	i	3.44	C(65)....O(10)	iv	3.22
C(51)....C(26)	i	3.39	C(65)....C(21)	ii	3.26
C(51)....C(19)	ii	3.43	C(65)....O(8)	ii	3.42
C(51)....C(21)	i	3.48	C(66)....O(8)	i	3.20
C(52)....C(20)	i	3.39	C(66)....O(10)	iii	3.46

(C) SOME INTRAMOLECULAR DISTANCES.

Br(1)....O(4)	i	3.22	Br(2)....O(11)	i	2.86
Br(1)....O(5)	i	3.80	Br(2)....O(12)	i	3.34
Br(1)....N(2)	i	3.26	Br(2)....N(5)	i	2.99
C(45)....C(46)	i	3.08	C(65)....C(66)	i	3.40

(D) INTERMOLECULAR DISTANCES, OTHER THAN INTERPLANAR, LESS THAN 3.5 Å.

O(12)....C(34)	iv	3.10 Å	O(13)....C(31)	x	3.40 Å
O(5).....C(51)	vii	3.24	O(9).....C(1)	i	3.41
O(9).....C(36)	viii	3.27	O(2).....C(14)	ii	3.45
O(5).....O(12)	vii	3.29	O(13)....C(32)	x	3.47
O(6).....C(53)	ix	3.38	O(6).....O(13)	xi	3.49
O(12)....C(33)	vi	3.39	O(14)....C(16)	iii	3.49

The Roman numerals refer to the following transformations applied to the coordinates found in Table 2.1 , the transformation is applied to the second atom of each contact.

i	x,	y,	z.
ii	x,	1 + y,	z.
iii	x,	- y,	1/2 + z.
iv	x,	1 - y,	1/2 + z.
v	x,	-1 + y,	z.
vi	-1 + x,	- y,	-1/2 + z.
vii	1 + x,	y,	z.
viii	x,	- y,	-1/2 + z.
ix	1 + x,	1 - y,	-1/2 + z.
x	-1 + x,	y,	z.
xi	1 + x,	- y,	-1/2 + z.

TABLE 2.8

MEAN MOLECULAR PLANES.

PLANE NO.	ATOMS.	PLANE NO.	ATOMS.
1	C(1) -C(13),O(1)	7	C(27)-C(44)
2	C(1) -C(4),C(10),C(11)	8	C(27)-C(30),C(39),C(42)
3	C(5) -C(8),C(12),C(13)	9	C(35)-C(38),C(41),C(44)
4	C(14)-C(26),O(8)	10	C(47)-C(64)
5	C(14)-C(17),C(23),C(24)	11	C(47)-C(50),C(59),C(62)
6	C(18)-C(21),C(25),C(26)	12	C(55)-C(58),C(61),C(64)

PLANE EQUATIONS.

PLANE NO.	P	Q	R	S	RMS D *
1	0.2352	0.9647	-0.1181	5.9029	0.168
2	0.1899	0.9463	-0.2615	5.4749	0.065
3	-0.3242	-0.9449	-0.0452	-6.2274	0.036
4	0.2489	-0.9623	-0.1095	0.4902	0.135
5	0.1339	-0.9724	-0.1911	0.0520	0.042
6	0.3275	-0.9449	-0.0041	1.0383	0.024
7	0.0877	-0.9930	-0.0794	-0.0110	0.394
8	0.2928	0.9458	-0.1402	3.1962	0.065
9	-0.3893	0.9169	-0.0876	-4.2707	0.063
10	0.1014	-0.9925	-0.0682	-3.2426	0.384
11	-0.4079	0.9101	-0.0738	1.4779	0.021
12	0.2889	0.9497	-0.1211	2.4344	0.036

* These symbols are defined in Table 1.10.

The plane equation is given by

$$PX' + QY + RZ' = S$$

where X', Y and Z' are in Å and are referred to orthogonal axes defined in Table 1.10.

DISTANCES FROM MEAN PLANES (Å).

INDIVIDUAL DISTANCES FROM A PLANE, OF ATOMS DEFINING THE PLANE, ARE NOT QUOTED.

PLANE NO.	ATOM AND DISTANCE.			
1	N(1) 0.09	N(2) -0.81	N(3) 0.44	Br(1) 0.83
2	N(1) -0.13	Br(1) 0.55	C(9) 0.24	C(12) 0.12
3	N(2) 0.29	N(3) -0.06	C(9) 0.30	C(11)-0.17
4	N(4) -0.15	N(5) 0.82	N(6) -0.48	Br(2) -0.49
5	N(4) -0.23	Br(2) -0.05	C(22)-0.39	C(25) 0.09
6	N(5) 0.42	N(6) -0.28	C(22) 0.02	C(24)-0.25
7	C(45)-1.58	C(46) 1.37		
8	C(31) 0.31	C(43)-0.30	C(45) 0.29	
9	C(34)-0.19	C(43) 0.16	C(46)-0.31	
10	C(65)-1.51	C(66) 1.76		
11	C(51) 0.28	C(63)-0.14	C(65) 0.59	
12	C(54)-0.33	C(63) 0.06	C(66)-0.60	

SOME DIHEDRAL ANGLES.

PLANE A	PLANE B	∠ AB °	PLANE A	PLANE B	∠ AB °
1	4	31.0	4	5	8.1
1	7	21.9	4	6	7.6
1	10	22.2	5	6	15.5
4	7	9.6	7	8	25.5
4	10	9.0	7	9	20.3
7	10	1.0	8	9	40.0
1	2	8.7	10	11	20.0
1	3	10.7	10	12	25.2
2	3	19.3	11	12	40.9

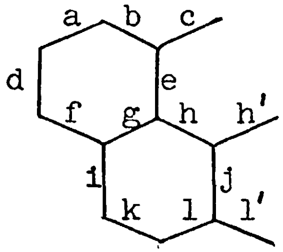
2.3 Discussion

Owing to the limited accuracy of the results it is impossible to achieve detailed information about the molecular geometry of the complex. However the structure can be considered as essentially correct and gross structural features to be established. The crystal and molecular structure of DMBP itself was discussed by Harnik, Herbstein and Schmidt (1954), by Hirshfield and Schmidt (1956) and later determined at 80°K by Hirshfield, Sandler and Schmidt (1963). They found that the molecule had a crystallographic twofold axis and the outer benzene rings were twisted in opposite directions, giving a methyl - methyl separation of 3.25 Å .

Assuming the two DMBP molecules in this analysis to be identical, the values of chemically equivalent bonds and interbond angles have been averaged and set out, with those of Hirshfield, Sandler and Schmidt for comparison, in Table 2.A . A fair agreement is shown in the two sets of results. The standard deviation of the means, for the experimental values quoted, are approximately 0.045 Å for a bond length and 2.2° for a bond angle. Both the DMBP molecules are highly strained with a mean separation of the methyl groups of 3.24 Å . In each case the outer benzene rings are twisted in opposite directions out of a

TABLE 2.A

MEAN VALUES OF BOND LENGTHS, IN ANGSTROMS, AND BOND ANGLES, IN DEGREES, IN 1,12-DIMETHYL BENZOPHENANTHRENE.



bond	A	B *	bond	A	B *
a	1.44	1.39	g	1.42	1.41
b	1.41	1.40	h	1.47	1.45
c	1.55	1.51	i	1.42	1.44
d	1.34	1.38	j	1.40	1.41
e	1.41	1.44	k	1.37	1.37
f	1.45	1.41	l	1.42	1.43

angle	A	B *	angle	A	B *	angle	A	B *
ad	118	119	fg	119	121	hh'	126	126
ab	120	123	fi	122	119	hj	117	117
bc	116	118	gi	118	120	ik	122	119
be	121	118	eg	118	118	kl	118	121
ce	121	123	eh	124	123	jl	121	120
df	124	120	gh	118	118	ll'	117	120

* (A) This analysis.

(B) Hirshfield, Sandler and Schmidt (1963).

TABLE 2.B

BOND LENGTHS IN THE BTNF MOLECULES.

bond	mean (Å)	S. dev. of mean (Å)	No.	Literature *
C - C aromatic	1.39	0.02	24	1.394
C sp^2 - C sp^2	1.49	0.03	6	1.47
C = O	1.23	0.05	2	1.215
C - N	1.55	0.05	6	1.472
N - O	1.16	0.04	12	1.22
C - Br	1.92	0.02	2	1.85

* Sutton (1965).

reference plane through the molecules (Table 2.8 , equations 7 and 10). The dihedral angle between the planes of these benzene rings is 40.0° in molecule A and 40.9° in molecule B (Table 2.8, equations 8,9,11 and 12).

Average bond distances found in the BTNF molecules are listed in Table 2.B and agree reasonably well with both the dimensions found in the preceding analysis and expected values (Sutton, 1965). The mean interbond angle in the benzene rings is $119.8(1.4)^{\circ}$ and in the five membered ring $107.3(1.3)^{\circ}$. Each of the 2-nitro groups is almost coplanar with its respective fluorenone grouping but the 5- and 7-nitro groups are not; for the 2- , 5- and 7-nitro groups the mean angles of rotation out of the fluorenone skeleton are 4.5° , 45.5° and 13.2° . Molecular overcrowding at the 4- and 5- positions is again minimised by deformation of the fluorenone skeleton. The best planes through the benzene rings and the pentanone systems can be found in Table 2.8 (equations 1 to 6). Each of the benzene rings is twisted out of the plane of the fluorenone group, the dihedral angle between the aromatic rings is 19.3° in molecule A and 15.5° in molecule B.

2.4 Bonding in Bromotrinitrofluorenone Complexes

Both compounds studied show a plane to plane stacking which is common to other molecular complexes. With strong charge transfer forces involved, molecules stacking this way should show a preferred orientation to allow maximum overlap of the respective π - molecular orbitals (Mulliken, 1952). This effect was discussed by Wallwork (1961) with reference to the acceptors s-trinitrobenzene, phenoquinone and chloranil. The relative orientations of the two complexes under discussion are given in Fig. 1.5 and Fig. 2.4 .

In the anthracene complex, the anthracene and BTNF molecules lie almost parallel with a dihedral angle between the two best molecular planes of 4.8° (Table 1.10 , equations 1 and 8). The two benzene rings (marked A in Fig. 1.5) would be almost directly above one another but are shifted laterally through approximately 1.2 Å , a comparable movement was found in the complex of s-trinitrobenzene with anthracene (Brown, Wallwork and Wilson, 1964). This movement, however, brings the 7-nitro group closer to the anthracene molecule and probably involves it in the charge transfer process. Sinomiya (1940) predicts maximum stability of complexes in which nitro groups are coplanar with the benzene ring to which they are bonded. The 7-nitro group is only 1.7° out of the plane of the benzene ring to which it is attached, while the 2- and 5-nitro groups are 4.0° and 84.8° out of the planes of their respective benzene rings and do not appear to be directly involved in the charge transfer bonding. The perpendicular separation of the molecular planes is approximately 3.42 Å and shows no decrease in the normal van der Waals distance of about 3.40 Å .

The DMBP complex shows features which indicate that strong local polarising forces are involved. Each BTNF : DMBP complex pair shows only a small degree of overlap and the mean planes through the component molecules do not lie parallel. The dihedral angle between the planes for the complexed A molecules is 21.9° and for the B molecules 9.0° . In each of the sets of molecules the carbonyl and 2- and 7-nitro groups make close contact with positions on the DMBP molecules. The carbonyl and 2-nitro group of each BTNF molecule approach the 2- and 12- positions of the DMBP molecule and the 7-nitro group is adjacent to the 6- and 7- positions. Levy, Newman and Szwark (1955) showed that the deformation in DMBP increased electron localisation and gave rise to large methyl affinities compared to the parent or mono methyl substituted benzophenanthrene. Berthier, Coulson, Greenwood and Pullman (1948) predicted, for benzophenanthrene, maximum electron density at the 5- and 8- positions, and appreciable values at the 2- and 12- positions. Assuming similar charge locations in DMBP there are interactions between these polarisable positions and the polarising regions in the BTNF molecules, that is the carbonyl and nitro groups. Carbonyl group bonding participation has been discussed by Prout and Wallwork (1966) and found in the perylene : fluoranil complex (Hanson, 1963) and in the bis-8-hydroxyquinolinatapalladium (II) : chloranil complex (Kamenar, Prout and Wright, 1966).

BTNF appears to form complexes suitable for study by X-ray methods, providing one accepts the increase in the number of atoms in the analysis and the possibility of disorder. The bonding in the anthracene complex is that of overlapping π molecular orbitals and in the DMBP complex that of electrostatic interactions of the polarising and polarisable groups. This difference is probably due to the planarity of the anthracene and large deviation from planarity of the DMBP molecule, in one case allowing overlap of the relevant π molecular orbitals and in the other case precluding it.

CHAPTER 3

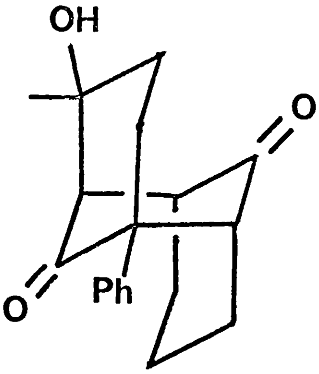
2-Phenyl-5-methyl-11-bromoacetoxy tricyclo [$5,3,1,0^{4,11}$] undecan-5-ol-2-carboxylic acid lactone.

3.1 Introduction

Some recent reports of doubly bridged tricyclic molecules (Davies, Erdtman and Nilsson, 1966 ; Birch, Butler and Siddall, 1964 ; Severin, 1959) prompted interest in the conformation of molecules similar to type I . The synthesis of I was attempted by Lawson (1966) via the precursors II , III and IV . On hydroboration 2-phenyl bicyclo-[3,3,1]-non-2-ene-9-one , II , gave one major product which was then oxidised to the dione III . The dione was alkylated with 1,3-dichloro-but-2-ene and the product IV treated with 80 % H_2SO_4 to give the alcohol I .

The resulting compound [$C_{19}H_{22}O_3$] gave an infra-red carbonyl absorption band at 1774 reciprocal centimetres and showed mono-substituted benzene absorption patterns in both infra-red and ultra-violet spectra. Nuclear magnetic resonance measurements indicated the presence of a tertiary alcohol, and a methyl group adjacent to an oxygen atom. The alcohol, which dissolved slowly in alkali, gave mono-formate and mono-acetate derivatives but could not be

I



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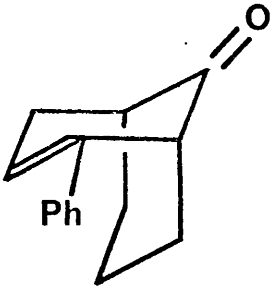

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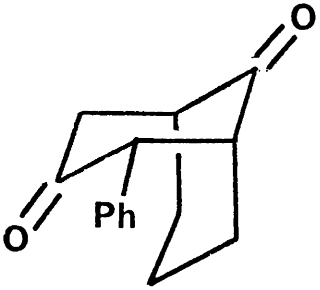
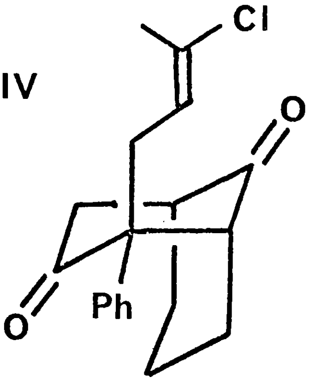
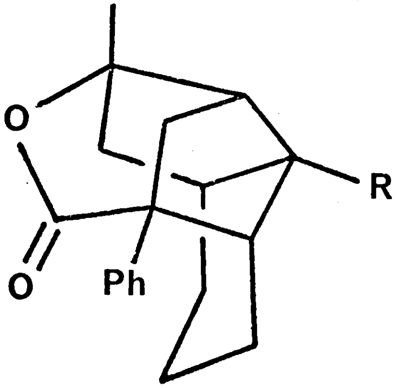


IV



V



dehydrated. Further chemical and physical investigations by Buchanan and Lawson (1966) did not lead to a definite assignment of the molecular structure.

The alcohol formed a bromoacetate derivative and the X-ray analysis of this compound is described in this chapter. The results establish the structure of the derivative as (V ; R = $-O(CO)CH_2Br$) , from which it may be concluded that the alcohol is (V ; R = -OH). Lawson has shown structure (V ; R = -OH) to be in agreement with the known properties of the alcohol and Buchanan, Ferguson, Lawson and Pollard (1966) rationalised its production from IV by a fragmentation reaction which was caused by strain in the molecular system and had been previously observed only in bicyclo-[3,2,1]-octenones.

3.2 Experimental

Crystal data

2-Phenyl-5-methyl-11-bromoacetoxy tricyclo [$5,3,1,0^{4,11}$] undecan-5-ol-2-carboxylic acid lactone.

$C_{21}H_{23}O_4Br$

F.W. = 419.3 , M.Pt. = 153°C .

Monoclinic, $a = 7.16 \pm 2$, $b = 15.30 \pm 3$, $c = 17.71 \pm 3$ Å

$\beta = 108^{\circ}\ 40' \pm 20'$

$U = 1838$ Å^3 , $D_m = 1.50$ gm.cm.$^{-3}$, $Z = 4$, $D_x = 1.52$ gm.cm.$^{-3}$

Linear absorption coefficient $\mu = 37.5$ cm.$^{-1}$ (Cu K_{α}).

F(000) = 864.

Systematic absences,

0k0 when k is odd,

h0l when l is odd.

Space group $P2_1/c$ (C^5_{2h} , No. 14).

Cell dimensions were determined from a small, colourless, needle shaped crystal by means of Weissenberg and rotation photographs, taken about the <u>a</u> axis with Cu K_{α} radiation, and from precession photographs of the h0l zone taken with Mo K_{α} radiation.

The intensities of 2234 independent X-ray reflections were estimated visually from multiple film Weissenberg photographs of the 0kl to 6kl reciprocal lattice nets. Lorentz, polarisation and rotation factors, appropriate to a small mosaic crystal, were applied; absorption corrections were not considered necessary. The resulting data, corresponding to some 53 % of that accessible to Cu K_α radiation, were initially put on an absolute scale by ensuring $k \sum |Fo| = \sum |Fc|$ for each reciprocal lattice net. The final scale factors were determined by least-squares methods. No unobserved reflections were used in the analysis.

3.3 Structure Solution and Refinement

From consideration of the vectors on the three-dimensional Patterson function, the Harker section at V = 1/2 is shown in Fig. 3.1 , fractional coordinates for the bromine atom were determined as

Br(1) 0.0769 0.2033 0.0213 .

Structure factors for all the reflections, calculated for this bromine position, gave an R factor of 0.506 . An electron density distribution was then computed using the observed structure amplitudes with the phases of the structure factors, and revealed a molecule consisting of four fused rings. Three oxygen atoms could be definitely assigned, those of the bromoacetate and carbonyl groups ; these, together with 17 other carbon atoms, were included in the subsequent structure factor calculation. After three rounds of structure factor and electron-density calculations the positions of all the atoms were known and the value of R was 0.232 .

The structure was now refined by the least-squares method using the author's program, three coordinates and a single thermal parameter per atom were employed. Five cycles of the block diagonal approximation, with all observations given unit weight, and indicated shifts multiplied by 0.85 , reduced the R factor to 0.148 .

Intensity data were only collected by rotating the crystal around the a axis and as Lingafelter and Donohue (1966) have pointed out this leads to degeneracy in the U_{11} thermal parameters when an anisotropic refinement including layer scale factors is carried out. To avoid this the data were now put on a common scale and subsequent refinement only involved one overall scale parameter. Each atom's isotropic thermal parameter was then replaced by six anisotropic temperature parameters and refinement continued using Cruickshank and Smith's (1965) least squares program. The block diagonal approximation, with weights given by

$$w = 1 / (A + |Fo| + B |Fo|^2)$$

where $A = 2$ F min. and $B = 2 /$ F max., and shifts multiplied by 0.75 , reduced R to 0.096 in five cycles. During this stage 20 hydrogen atoms, that is all except the methyl hydrogens, were included in the structure factor calculations at positions determined from geometrical considerations and with isotropic thermal parameters corresponding to that of the atom to which each was bonded. The hydrogen atoms were not refined. Convergence was produced by three further cycles of anisotropic refinement employing a weighting scheme such that

$$w = k \exp - (A + B|Fo| + C|Fo|^2 + D/s + E/s^2 + Fs),$$

where k is a constant, $A = -0.60$, $B = 0.079$, $C = -0.00053$, $D = 0.051$, $E = -0.00021$, $F = 3.2$ and $s = \sin^2 \theta / \lambda^2$. These parameters were determined by the least-squares method so that $w = 1 / \langle \Delta^2 \rangle$ where $\langle \Delta^2 \rangle$ is the local average of $(|Fo| - |Fc|)^2$, (McGregor, 1967). Parameter shifts in all cases were negligible compared to the corresponding standard deviations.

The electron-density standard deviation, $\sigma(\rho)$, was 0.15 e/Å^3. Features greater than $3\sigma(\rho)$, on a final (Fo - Fc) electron-density distribution, consisted of a region, 0.6 e/Å^3, at the heavy atom site, and two peaks close to the methyl group. The hydrogen positions were confirmed from a low order ($\sin \theta / \lambda < 0.355$) difference synthesis and three small peaks around the methyl group could be assigned as hydrogens.

Throughout the analysis the atomic formfactors used were those of Freeman (1959) for carbon, Berghuis et al. (1955) for oxygen, and Freeman and Watson (1962) for bromine. Atomic fractional and orthogonal coordinates are given in Tables 3.1 and 3.2 , anisotropic thermal parameters in Table 3.3 and assumed fractional coordinates and isotropic thermal parameters for the hydrogen atoms in Table 3.4 . Final observed and calculated structure factors together with an analysis by magnitude of Fo and reciprocal lattice net are in Tables 3.5 and 3.6 ; bond lengths, interbond angles, the more important non-bonded interatomic distances and finally mean planes are given in Tables 3.7 to 3.10 respectively. When quoted, standard deviations are in units of the last decimal place and were determined from the inverse matrix of the normal equations.

DIAGRAMS AND TABLES.

COURSE OF ANALYSIS.

I Fourier refinement.

		R
1	Br alone.	0.506
2	Br, 3 O, 17 C.	0.319
3	Br, 4 O, 21 C.	0.232

II Least-squares refinement.

Cycles of refinement	Final R	Final R dash	*
1 - 5	0.148	-	(a)
6 - 7	0.119	0.026	(b)
8	0.102	0.019	(c)
9 - 10	0.096	0.018	(d)
11 - 13	0.093	0.016	(e)

* (a) isotropic, unit weights, individual layer scale factors.

(b) anisotropic, weighting scheme applied, one overall scale parameter.

(c) as above, 20 hydrogens included but not refined.

(d) as above.

(e) as above, weighting scheme changed.

FIG. 3.1

The Harker section at V = 1/2. Contours are at arbitrary intervals.

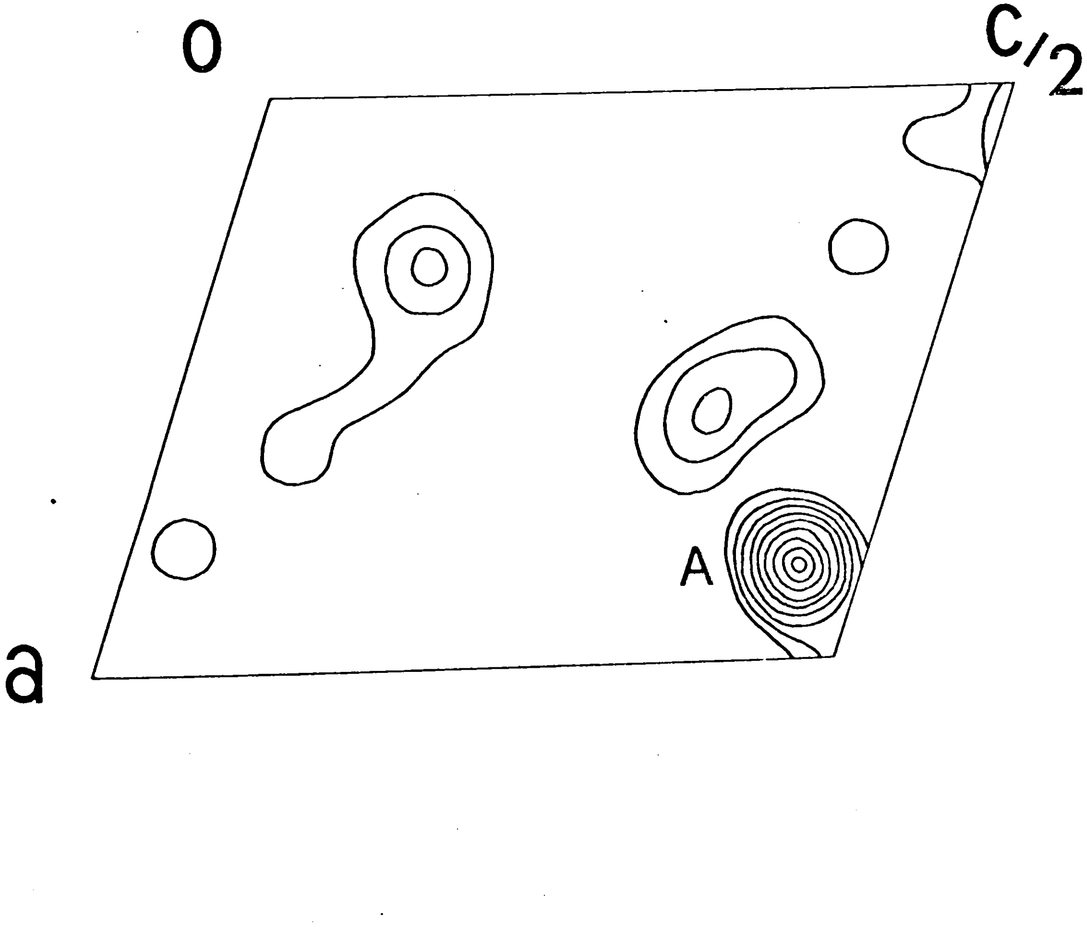

0
c/2
A
a

FIG. 3.2

Composite final electron-density synthesis viewed down (100) and corresponding atomic arrangement. Contours are at 1 e/$\mathring{A}^3$ intervals starting at 1 e/$\mathring{A}^3$ except at the bromine which is contoured at 5 e/$\mathring{A}^3$ intervals.

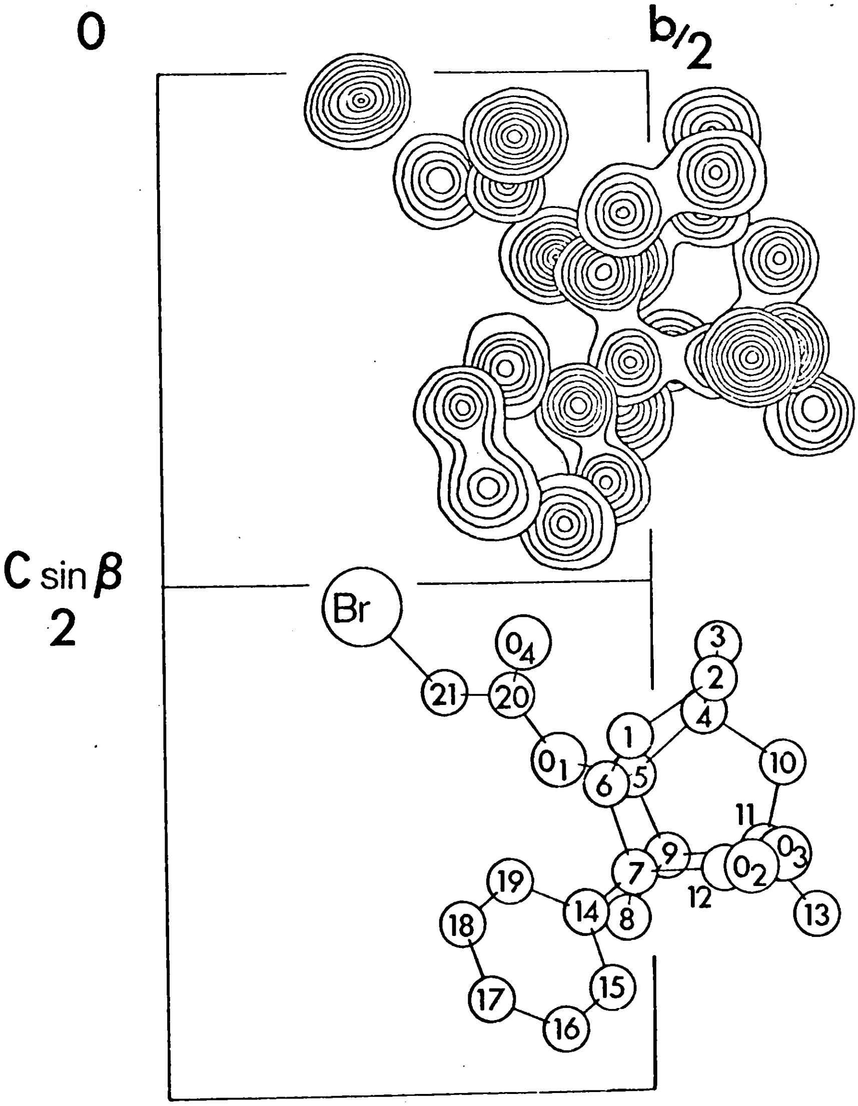
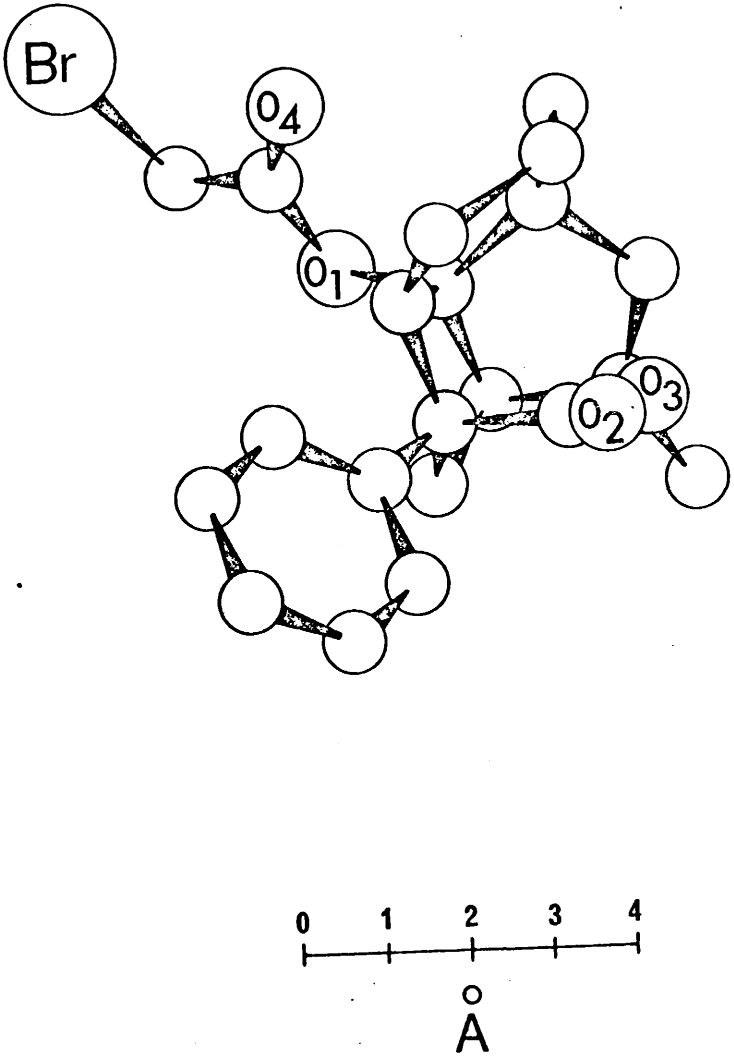

0
b/2
C sin β
2
Br
O4
3
2
21
20
4
1
10
O1
6
5
11
9
O2
O3
7
19
12
14
8
13
18
15
17
16

FIG. 3.3

Molecular diagram.



Br
O4
O1
O2
O3
0
1
2
3
4
Å

FIG. 3.4

The molecular packing viewed down the (100) projection.

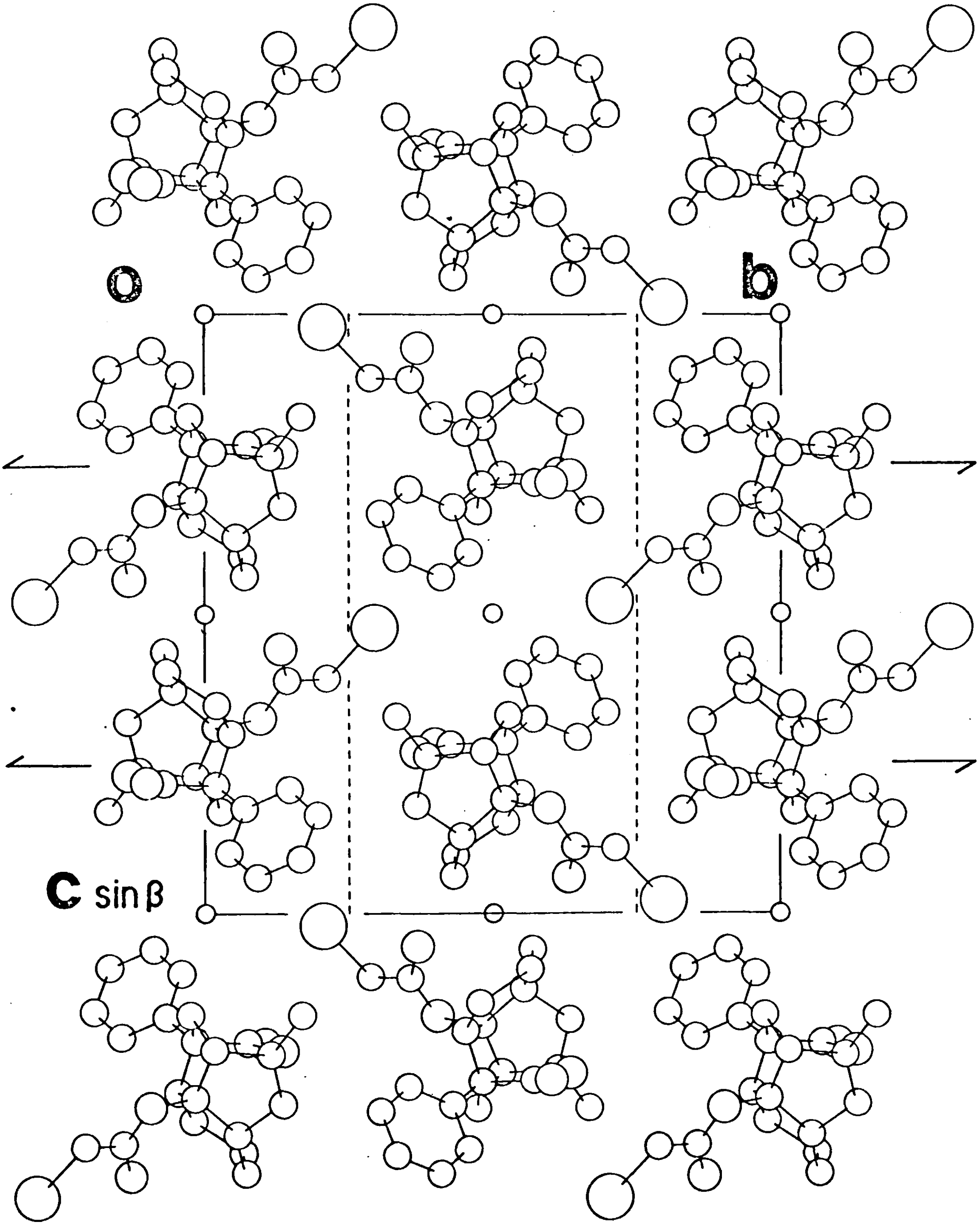

o
b
c sin β

TABLE 3.1

FRACTIONAL COORDINATES AND E.S.D.S.

ATOM	X/a	Y/b	Z/c
Br(1)	0.08263 ± 18	0.20349 ± 7	0.02268 ± 7
O(1)	0.1205 ± 7	0.4027 ± 4	0.1754 ± 3
O(2)	-0.3863 ± 8	0.6022 ± 4	0.2824 ± 3
O(3)	-0.1010 ± 7	0.6344 ± 3	0.2705 ± 3
O(4)	-0.1134 ± 9	0.3679 ± 5	0.0589 ± 4
C(1)	-0.3829 ± 12	0.4763 ± 6	0.1348 ± 5
C(2)	-0.3716 ± 13	0.5647 ± 6	0.0971 ± 5
C(3)	-0.1971 ± 13	0.5678 ± 6	0.0646 ± 5
C(4)	0.0045 ± 13	0.5531 ± 6	0.1287 ± 5
C(5)	0.0116 ± 11	0.4796 ± 5	0.1903 ± 5
C(6)	-0.1872 ± 11	0.4499 ± 5	0.2002 ± 5
C(7)	-0.1699 ± 10	0.4793 ± 5	0.2870 ± 4
C(8)	0.0515 ± 11	0.4721 ± 5	0.3306 ± 5
C(9)	0.1338 ± 10	0.5142 ± 5	0.2708 ± 5
C(10)	0.0894 ± 13	0.6348 ± 6	0.1798 ± 5
C(11)	0.0952 ± 10	0.6138 ± 5	0.2640 ± 5
C(12)	-0.2269 ± 10	0.5742 ± 5	0.2820 ± 4
C(13)	0.2386 ± 12	0.6688 ± 6	0.3309 ± 5
C(14)	-0.2945 ± 10	0.4286 ± 5	0.3266 ± 5
C(15)	-0.3011 ± 13	0.4557 ± 6	0.4003 ± 5

C(16)	-0.4056 ± 14	0.4070 ± 7	0.4401 ± 5
C(17)	-0.5005 ± 14	0.3316 ± 7	0.4090 ± 6
C(18)	-0.4995 ± 15	0.3046 ± 6	0.3360 ± 6
C(19)	-0.3989 ± 12	0.3521 ± 6	0.2938 ± 5
C(20)	0.0417 ± 14	0.3551 ± 5	0.1091 ± 5
C(21)	0.1872 ± 13	0.2847 ± 5	0.1079 ± 5

TABLE 3.2

ORTHOGONAL COORDINATES AND E.S.D.S.

ATOM	X'	Y	Z'
Br(1)	0.560 ± 1	3.114 ± 1	0.212 ± 1
O(1)	0.817 ± 5	6.162 ± 6	2.830 ± 5
O(2)	-2.619 ± 5	9.215 ± 6	5.886 ± 6
O(3)	-0.685 ± 5	9.707 ± 5	5.022 ± 5
O(4)	-0.768 ± 6	5.630 ± 7	1.303 ± 6
C(1)	-2.596 ± 8	7.288 ± 9	3.264 ± 9
C(2)	-2.519 ± 9	8.640 ± 9	2.570 ± 8
C(3)	-1.336 ± 9	8.689 ± 9	1.596 ± 8
C(4)	0.030 ± 9	8.464 ± 9	2.269 ± 8
C(5)	0.079 ± 7	7.338 ± 7	3.343 ± 8
C(6)	-1.269 ± 7	6.885 ± 8	3.974 ± 8
C(7)	-1.152 ± 7	7.333 ± 7	5.470 ± 7
C(8)	0.349 ± 8	7.223 ± 8	5.736 ± 8
C(9)	0.907 ± 7	7.868 ± 8	4.488 ± 8
C(10)	0.606 ± 9	9.714 ± 9	2.978 ± 8
C(11)	0.645 ± 7	9.392 ± 8	4.457 ± 8
C(12)	-1.538 ± 7	8.785 ± 8	5.512 ± 7
C(13)	1.618 ± 8	10.233 ± 10	5.313 ± 9
C(14)	-1.997 ± 7	6.558 ± 8	6.458 ± 8
C(15)	-2.041 ± 9	6.973 ± 9	7.779 ± 8

C(16)	-2.750 ± 10	6.228 ± 11	8.721 ± 9
C(17)	-3.393 ± 10	5.073 ± 11	8.388 ± 10
C(18)	-3.386 ± 10	4.661 ± 9	7.094 ± 11
C(19)	-2.705 ± 8	5.387 ± 9	6.117 ± 8
C(20)	0.283 ± 9	5.433 ± 8	1.837 ± 8
C(21)	1.269 ± 9	4.356 ± 8	1.482 ± 8

TABLE 3.3

ANISOTROPIC TEMPERATURE PARAMETERS AND E.S.D.S.

ATOM	U11	U22	U33	2U23	2U31	2U12
Br(1)	0.0892	0.0580	0.0775	-0.0237	0.0688	-0.0085
	8	6	7	10	12	10
O(1)	0.0264	0.0483	0.0568	-0.0103	0.0387	0.0115
	30	31	31	47	47	45
O(2)	0.0283	0.0550	0.0621	0.0030	0.0378	0.0055
	31	35	34	53	51	48
O(3)	0.0259	0.0393	0.0652	0.0010	0.0406	0.0027
	30	28	34	47	51	43
O(4)	0.0387	0.0733	0.0622	-0.0217	0.0144	0.0230
	36	44	37	63	57	59
C(1)	0.0308	0.0579	0.0502	0.0016	0.0119	0.0038
	48	51	45	72	74	72
C(2)	0.0395	0.0587	0.0509	0.0097	0.0224	0.0102
	50	52	46	76	77	76
C(3)	0.0465	0.0632	0.0502	0.0184	0.0271	0.0063
	54	54	47	79	80	81
C(4)	0.0451	0.0524	0.0481	0.0035	0.0397	-0.0038
	53	47	43	69	77	74
C(5)	0.0189	0.0424	0.0560	-0.0190	0.0320	-0.0036
	40	40	44	65	68	60
C(6)	0.0141	0.0482	0.0501	-0.0089	0.0186	0.0026
	38	41	41	65	63	60
C(7)	0.0174	0.0470	0.0423	-0.0014	0.0184	0.0032
	39	41	38	59	62	58
C(8)	0.0267	0.0518	0.0506	-0.0056	0.0265	-0.0004
	46	45	43	67	73	63

C(9)	0.0126	0.0465	0.0577	-0.0081	0.0242	0.0156
	38	41	45	66	66	58
C(10)	0.0413	0.0488	0.0585	-0.0001	0.0498	-0.0184
	49	46	47	71	78	72
C(11)	0.0115	0.0468	0.0665	-0.0147	0.0302	-0.0006
	38	40	49	72	66	60
C(12)	0.0181	0.0471	0.0476	-0.0054	0.0299	0.0017
	39	40	40	63	62	61
C(13)	0.0238	0.0677	0.0693	-0.0350	0.0428	-0.0236
	46	55	54	86	80	76
C(14)	0.0154	0.0452	0.0532	0.0121	0.0239	0.0036
	39	40	42	64	64	59
C(15)	0.0402	0.0606	0.0502	0.0128	0.0341	-0.0010
	52	51	45	76	78	77
C(16)	0.0523	0.0791	0.0612	0.0336	0.0718	0.0318
	60	68	53	94	92	99
C(17)	0.0473	0.0702	0.0732	0.0435	0.0521	0.0011
	58	62	61	100	93	94
C(18)	0.0513	0.0519	0.0875	0.0149	0.0661	-0.0182
	60	52	70	91	104	86
C(19)	0.0343	0.0461	0.0643	0.0016	0.0401	-0.0067
	49	45	50	74	79	70
C(20)	0.0530	0.0433	0.0606	-0.0070	0.0568	-0.0016
	55	42	49	72	85	75
C(21)	0.0457	0.0446	0.0502	-0.0057	0.0288	0.0008
	52	44	44	66	77	70

The thermal parameters, in $\mathring{A}^2$, are from the expression defined in Table 1.3 .

TABLE 3.4

(A) ASSUMED TEMPERATURE FACTORS AND FRACTIONAL COORDINATES OF HYDROGEN ATOMS.

NUMBERS REFER TO THE ATOM TO WHICH EACH HYDROGEN IS BONDED.

ATOM	X/a	Y/b	Z/c	U iso
H(1)i	-0.414	0.431	0.087	0.0492
H(1)ii	-0.497	0.478	0.163	0.0492
H(2)i	-0.508	0.576	0.050	0.0517
H(2)ii	-0.353	0.611	0.143	0.0517
H(3)i	-0.218	0.520	0.020	0.0558
H(3)ii	-0.193	0.629	0.040	0.0558
H(4)	0.091	0.535	0.091	0.0471
H(6)	-0.200	0.383	0.191	0.0385
H(8)i	0.100	0.498	0.390	0.0428
H(8)ii	0.102	0.408	0.345	0.0428
H(9)	0.290	0.500	0.289	0.0394
H(10)i	0.234	0.652	0.177	0.0507
H(10)ii	0.000	0.689	0.157	0.0507
H(15)	-0.230	0.514	0.426	0.0503
H(16)	-0.412	0.428	0.497	0.0602
H(17)	-0.579	0.297	0.442	0.0633
H(18)	-0.570	0.246	0.312	0.0619
H(19)	-0.400	0.331	0.235	0.0493

H(21)i	0.220	0.252	0.165	0.0487
H(21)ii	0.316	0.313	0.100	0.0487

(B) FRACTIONAL COORDINATES OF THE METHYL GROUP HYDROGEN ATOMS.

ATOM	X/a	Y/b	Z/c
H(13)i	0.199	0.737	0.320
H(13)ii	0.252	0.648	0.381
H(13)iii	0.351	0.670	0.326

TABLE 3.5

FINAL OBSERVED AND CALCULATED STRUCTURE FACTORS.

H	K	L	Fo	Fc
0	0	2	60.5	-60.3
0	0	4	37.7	50.5
0	0	6	36.2	39.1
0	0	8	27.4	-25.1
0	0	10	34.5	33.3
0	0	12	3.8	-5.7
0	0	14	17.0	-15.0
0	0	16	16.4	-15.3
0	0	18	6.7	6.2
0	0	20	15.8	-11.0
0	1	2	38.5	-34.4
0	1	3	11.0	15.8
0	1	4	30.2	-38.6
0	1	5	83.1	56.4
0	1	6	69.7	-83.8
0	1	7	39.9	-39.6
0	1	8	32.7	-35.3
0	1	9	41.9	44.4
0	1	10	74.5	-81.9
0	1	11	25.3	-22.3
0	1	12	31.8	-29.9
0	1	13	28.6	26.5
0	1	14	28.8	-27.2
0	1	15	24.5	-25.3
0	1	16	4.3	-4.7
0	1	17	2.6	-4.5
0	1	18	11.0	-8.4
0	1	21	9.4	-7.0
0	2	0	63.0	66.9
0	2	1	14.9	-8.9
0	2	2	152.3	-165.7
0	2	3	71.0	78.1
0	2	4	58.8	-66.6
0	2	5	65.5	-75.9
0	2	6	47.7	-53.2
0	2	7	2.7	-1.2
0	2	8	37.7	-40.3
0	2	9	62.1	-66.1
0	2	11	26.0	-26.0
0	2	12	10.3	-7.8
0	2	13	11.7	-10.5
0	2	14	19.4	18.5
0	2	15	21.8	-21.0
0	2	16	11.0	10.0
0	2	17	2.6	-3.9
0	2	18	11.0	8.3
0	2	20	3.5	2.9
0	2	21	8.3	5.9
0	3	1	83.4	-93.5
0	3	2	54.0	53.4
0	3	3	36.0	-51.7
0	3	4	118.3	117.4
0	3	5	69.6	-76.8
0	3	6	20.1	-17.9
0	3	7	35.4	-33.5
0	3	8	56.0	55.4
0	3	9	14.6	-15.4
0	3	10	6.4	10.1
0	3	11	7.6	9.4
0	3	12	43.3	39.9
0	3	14	13.2	12.1
0	3	16	10.1	7.7
0	3	17	17.6	15.8
0	3	18	2.4	4.2
0	3	19	19.2	12.6
0	4	0	44.5	-35.9
0	4	1	19.0	14.7
0	4	2	56.5	62.2
0	4	3	5.4	-1.7
0	4	4	10.6	-6.7
0	4	6	49.3	48.7
0	4	7	114.8	121.7
0	4	8	36.4	39.5
0	4	9	39.7	42.9
0	4	10	16.2	18.7
0	4	11	28.5	28.1
0	4	13	37.8	38.8
0	4	15	7.9	7.4
0	4	16	5.0	-5.2
0	4	17	12.5	11.0
0	4	18	10.7	-9.0
0	5	1	109.9	123.6
0	5	2	46.0	42.8
0	5	3	63.7	74.1
0	5	4	49.4	45.6
0	5	5	54.2	55.7
0	5	6	20.0	-16.0
0	5	7	40.4	44.1
0	5	8	3.9	-4.2
0	5	9	19.7	-19.3
0	5	10	6.0	-5.6
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6	3	-3	26.6	22.1
6	3	-4	5.2	2.8
6	3	-5	26.9	30.2
6	3	-6	24.0	25.4
6	3	-7	13.0	-13.0
6	3	-8	31.6	30.7
6	3	-9	32.4	29.4
6	3	-10	8.4	-8.3
6	3	-11	15.7	-16.8
6	3	-12	18.8	20.4
6	3	-15	11.7	-10.7
6	3	-16	16.5	17.0
6	3	-17	8.1	-8.9
6	4	0	30.8	28.5
6	4	1	11.1	-11.4
6	4	2	19.3	-22.7
6	4	4	11.0	13.0
6	4	5	9.5	-11.7
6	4	-1	10.0	-7.0
6	4	-2	47.0	-39.9
6	4	-3	62.3	57.2
6	4	-5	15.1	-14.9
6	4	-6	8.9	-8.2
6	4	-7	26.6	21.3
6	4	-8	7.9	-9.0
6	4	-9	25.3	21.6
6	4	-10	3.6	-3.4
6	4	-11	5.2	1.3
6	4	-13	24.3	24.8
6	4	-14	8.7	8.5
6	4	-15	13.7	13.0
6	4	-18	13.8	14.8
6	5	0	15.8	-14.0
6	5	3	14.4	-13.8
6	5	5	3.9	-5.9
6	5	-1	31.2	-27.1
6	5	-2	14.4	11.7
6	5	-3	22.4	-17.6
6	5	-5	9.4	-7.4
6	5	-7	36.8	-34.4
6	5	-9	23.0	21.0
6	5	-10	13.1	-11.9
6	5	-11	19.1	-18.6
6	5	-12	11.6	-13.2
6	5	-13	12.0	14.1
6	5	-14	8.7	9.3
6	5	-17	12.9	10.1
6	6	2	15.9	-13.3
6	6	3	11.3	15.0
6	6	-1	13.9	-10.7
6	6	-3	8.7	-7.8
6	6	-5	16.0	-11.0
6	6	-6	7.9	-9.2
6	6	-7	28.7	-27.3
6	6	-8	3.7	-3.0
6	6	-9	5.3	-3.2
6	6	-10	9.3	-8.8
6	6	-12	8.7	9.7
6	6	-13	13.8	-12.3
6	6	-14	7.7	7.2
6	6	-15	11.4	-10.6
6	7	1	23.7	21.2
6	7	3	11.6	13.7
6	7	4	10.4	11.6
6	7	5	5.2	9.9
6	7	-1	17.9	15.7
6	7	-2	12.2	-9.3
6	7	-3	11.2	9.7
6	7	-4	7.4	-6.5
6	7	-8	12.4	-10.6
6	7	-9	15.6	13.9
6	7	-12	18.3	-19.3
6	8	3	3.8	-4.1
6	8	4	3.7	5.4
6	8	-2	26.2	26.9
6	8	-4	15.8	12.6
6	8	-5	24.1	22.2
6	8	-6	18.1	16.9
6	8	-7	6.7	-7.8
6	8	-9	20.8	22.5
6	8	-11	14.1	14.7
6	8	-13	6.5	6.7
6	8	-15	6.8	6.6
6	8	-16	9.0	-11.0
6	9	1	8.6	-11.2
6	9	2	9.2	-11.9
6	9	4	5.0	6.8
6	9	-3	18.7	-18.2
6	9	-6	16.0	11.8
6	9	-8	20.0	21.5
6	9	-9	5.5	-4.2
6	9	-10	5.5	6.3
6	9	-11	5.4	-6.0
6	9	-12	11.7	11.8
6	9	-14	17.0	17.4
6	10	0	10.9	-10.2
6	10	2	11.0	-13.5
6	10	-1	5.4	-7.9
6	10	-2	11.5	-13.3
6	10	-4	13.2	-12.0
6	10	-6	18.4	-18.3
6	11	1	7.7	-9.1
6	11	-1	8.1	9.0
6	11	-2	5.2	6.1
6	11	-3	3.7	-3.3
6	11	-4	11.5	-11.6
6	11	-6	11.5	-12.4
6	11	-8	7.3	-9.0
6	11	-9	9.5	10.2
6	11	-10	17.2	-16.2
6	12	0	5.7	6.7
6	12	-1	8.2	-10.1
6	12	-2	6.9	8.8
6	12	-3	6.0	5.6
6	12	-4	5.0	7.9
6	12	-9	8.1	-7.5

TABLE 3.6

(A) ANALYSIS OF STRUCTURE FACTOR DATA AS A FUNCTION OF LAYER LINE INDEX. ALL FIGURES ARE ON ABSOLUTE SCALE.

h	$\sum \lvert F_o \rvert$	$\sum \lvert F_c \rvert$	$\sum \lvert \Delta \rvert$	N	R	$\sum \lvert \Delta \rvert / N$
0	6300	6198	676	256	0.1072	2.64
1	11383	11362	973	424	0.0855	2.20
2	9982	9851	720	433	0.0722	1.66
3	7912	7720	671	356	0.0848	1.89
4	6419	6233	612	341	0.0953	1.79
5	3993	3830	554	235	0.1388	2.36
6	2558	2552	308	171	0.1204	1.80
ALL	48547	47746	4514	2234	0.0930	2.02

(B) ANALYSIS OF STRUCTURE FACTORS BY MAGNITUDE OF Fo.

RANGE	$\sum \lvert F_o \rvert$	$\sum \lvert F_c \rvert$	$\sum \lvert \Delta \rvert$	N	R	$\sum \lvert \Delta \rvert / N$
0 - 5	535	640	201	144	0.3760	1.40
5 - 10	3710	3710	592	484	0.1595	1.22
10 - 20	10903	10453	1109	760	0.1017	1.46
20 - 40	15877	15528	1273	559	0.0802	2.28
40 - 60	8318	8163	625	173	0.0752	3.61
60 - 100	6934	6922	557	95	0.0804	5.87
100 - 200	2269	2330	156	19	0.0688	8.21

TABLE 3.7

BOND LENGTHS AND E.S.D.S IN ANGSTROMS.

Br(1)- C(21)	1.913 ± 8	C(6) - C(7)	1.566 ± 11
O(1) - C(5)	1.480 ± 9	C(7) - C(8)	1.528 ± 10
O(1) - C(20)	1.343 ± 10	C(7) - C(12)	1.503 ± 11
O(2) - C(12)	1.221 ± 9	C(7) - C(14)	1.514 ± 10
O(3) - C(11)	1.479 ± 9	C(8) - C(9)	1.511 ± 11
O(3) - C(12)	1.349 ± 9	C(9) - C(11)	1.547 ± 11
O(4) - C(20)	1.195 ± 11	C(10) - C(11)	1.514 ± 12
C(1) - C(2)	1.522 ± 13	C(11) - C(13)	1.545 ± 12
C(1) - C(6)	1.558 ± 11	C(14) - C(15)	1.385 ± 11
C(2) - C(3)	1.533 ± 12	C(14) - C(19)	1.410 ± 11
C(3) - C(4)	1.540 ± 12	C(15) - C(16)	1.395 ± 13
C(4) - C(5)	1.556 ± 11	C(16) - C(17)	1.363 ± 15
C(4) - C(10)	1.548 ± 12	C(17) - C(18)	1.359 ± 15
C(5) - C(6)	1.556 ± 10	C(18) - C(19)	1.396 ± 13
C(5) - C(9)	1.510 ± 11	C(20) - C(21)	1.504 ± 12

TABLE 3.8

BOND ANGLES AND E.S.D.S IN DEGREES.

C(5) - O(1) - C(20)	119.3±6	C(6) - C(7) - C(12)	106.5±6
C(11) - O(3) - C(12)	124.2±6	C(6) - C(7) - C(14)	115.8±6
C(2) - C(1) - C(6)	113.3±7	C(8) - C(7) - C(12)	108.5±6
C(1) - C(2) - C(3)	110.9±7	C(8) - C(7) - C(14)	113.5±6
C(2) - C(3) - C(4)	113.7±7	C(12) - C(7) - C(14)	109.4±6
C(3) - C(4) - C(5)	115.8±7	C(7) - C(8) - C(9)	100.9±6
C(3) - C(4) - C(10)	114.4±7	C(5) - C(9) - C(8)	105.9±6
C(5) - C(4) - C(10)	104.9±6	C(5) - C(9) - C(11)	103.8±6
O(1) - C(5) - C(4)	110.6±6	C(8) - C(9) - C(11)	112.0±6
O(1) - C(5) - C(6)	110.0±6	C(4) - C(10) - C(11)	106.6±7
O(1) - C(5) - C(9)	105.6±6	O(3) - C(11) - C(9)	110.8±6
C(4) - C(5) - C(6)	117.6±6	O(3) - C(11) - C(10)	107.7±6
C(4) - C(5) - C(9)	106.7±6	O(3) - C(11) - C(13)	103.8±6
C(6) - C(5) - C(9)	105.7±6	C(9) - C(11) - C(10)	103.5±7
C(1) - C(6) - C(5)	118.5±6	C(9) - C(11) - C(13)	114.8±7
C(1) - C(6) - C(7)	115.2±6	C(10) - C(11) - C(13)	116.2±7
C(5) - C(6) - C(7)	103.8±6	O(2) - C(12) - O(3)	115.5±7
C(6) - C(7) - C(8)	102.7±6	O(2) - C(12) - C(7)	125.2±7

O(3) - C(12) - C(7)	119.2±6	C(17) - C(18) - C(19)	120.8±9
C(7) - C(14) - C(15)	119.1±7	C(14) - C(19) - C(18)	120.5±8
C(7) - C(14) - C(19)	123.2±7	O(1) - C(20) - O(4)	126.3±8
C(15) - C(14) - C(19)	117.6±7	O(1) - C(20) - C(21)	107.6±7
C(14) - C(15) - C(16)	120.1±8	O(4) - C(20) - C(21)	126.1±8
C(15) - C(16) - C(17)	121.8±9	Br(1) - C(21) - C(20)	112.3±6
C(16) - C(17) - C(18)	119.2±9		

TABLE 3.9

NON - BONDED DISTANCES.

(A) INTERMOLECULAR DISTANCES LESS THAN 3.8 Å

O(2).....C(13)	i	3.23 Å	O(4).....C(3)	v	3.71 Å
O(3).....C(21)	ii	3.34	C(12)....C(21)	ii	3.73
O(2).....C(21)	ii	3.44	Br(1)....C(16)	vi	3.73
O(1).....C(19)	iii	3.49	C(8).....C(17)	iii	3.74
O(1).....C(18)	iii	3.58	Br(1)....O(2)	vii	3.77
O(2).....C(11)	i	3.62	Br(1)....O(3)	vii	3.77
O(2).....C(9)	i	3.63	C(3).....C(20)	v	3.78
O(2).....C(10)	i	3.64	Br(1)....C(15)	vi	3.79
O(2).....C(18)	iv	3.68	C(19)....C(21)	i	3.80

The Roman numerals refer to the following transformations applied to the coordinates found in Table 3.1 . The transformation is applied to the second atom of each contact.

i	-1 + x,	y,	z.
ii	- x,	1/2 + y,	1/2 - z.
iii	1 + x,	y,	z.
iv	-1 - x,	1/2 + y,	1/2 - z.
v	- x,	1 - y,	- z.
vi	x,	1/2 - y,	-1/2 + z.
vii	- x,	-1/2 + y,	1/2 - z.

(B) SOME INTRAMOLECULAR DISTANCES.

Br(1)....O(4)	3.05 Å	C(1).....C(19)	3.43 Å
O(1).....C(8)	3.13	C(2).....C(5)	3.01
O(1).....C(7)	3.50	C(2).....C(12)	3.11
O(2).....C(14)	2.79	C(2).....C(10)	3.33
O(2).....C(15)	2.99	C(2).....C(7)	3.46
O(2).....C(1)	3.25	C(3).....C(6)	2.99
O(2).....C(6)	3.30	C(4).....C(20)	3.07
O(2).....C(2)	3.37	C(5).....C(12)	3.07
O(3).....C(8)	2.78	C(6).....C(19)	2.98
O(3).....C(5)	3.00	C(6).....C(20)	3.01
O(3).....C(6)	3.07	C(6).....C(11)	3.19
O(3).....C(4)	3.10	C(7).....C(11)	2.91
O(3).....C(2)	3.24	C(8).....C(15)	3.15
O(4).....C(5)	2.79	C(8).....C(13)	3.29
O(4).....C(6)	2.99	C(9).....C(12)	2.81
O(4).....C(4)	3.10	C(10)....C(12)	3.45
O(4).....C(3)	3.12	C(12)....C(15)	2.95
O(4).....C(1)	3.15	C(12)....C(13)	3.48
C(1).....C(12)	2.90	C(14)....C(17)	2.81
C(1).....C(4)	3.04	C(15)....C(18)	2.76
C(1).....C(14)	3.33	C(16)....C(19)	2.74

TABLE 3.10

MEAN MOLECULAR PLANES.

PLANE NO.	ATOMS DEFINING PLANE.
1	O(3),C(7),C(9),C(11) and C(12)
2	C(5),C(6),C(7) and C(9)
3	C(4),C(5),C(9) and C(10)
4	C(1),C(3),C(4) and C(6)
5	C(14) to C(19)
6	O(1),O(4),C(20) and C(21)

PLANE EQUATIONS.

PLANE NO.	P	Q	R	S	RMS D *
1	-0.4115	-0.0896	-0.9070	-5.1477	0.004
2	-0.3571	0.9102	-0.2099	5.9165	0.027
3	0.8725	-0.2478	-0.4212	-3.0913	0.055
4	0.1252	-0.7884	-0.6023	-8.0077	0.029
5	-0.8246	0.5342	-0.1864	3.9577	0.009
6	0.4365	0.6057	-0.6652	2.2036	0.006

* These symbols are defined in Table 1.10. The plane equation is

$$PX' + QY + RZ' = S$$

where X', Y and Z' are in Å and are referred to orthogonal axes also defined in Table 1.10.

DEVIATIONS FROM MEAN PLANES (Å).

PLANE NO.	ATOM AND DISTANCE.					
1	O(3)	0.005	C(7)	0.003	C(9)	-0.001
	C(11)	-0.002	C(12)	-0.006	O(2)	0.062
	C(8)	-0.845				
2	C(5)	0.032	C(6)	-0.032	C(7)	0.021
	C(9)	-0.022	C(8)	-0.671		
3	C(4)	0.065	C(5)	-0.066	C(9)	0.043
	C(10)	-0.042	C(11)	-0.550		
4	C(1)	-0.029	C(3)	0.030	C(4)	-0.028
	C(6)	0.028	C(2)	-0.667	C(5)	0.219
5	C(14)	-0.012	C(15)	0.001	C(16)	0.012
	C(17)	-0.013	C(18)	0.002	C(19)	0.010
	C(7)	-0.110				
6	O(1)	0.003	O(4)	0.005	C(20)	-0.011
	C(21)	0.003	Br(1)	-0.214	C(5)	0.052

3.4 Discussion

The X-ray analysis of this tertiary alcohol derivative both determines the structure of the rearrangement product as (V ; R = $-O(CO)CH_2Br$), and gives information on the conformation of δ- lactones and on the tricyclo [$5,3,1,0^{4,11}$] undecane system. The numbering scheme adopted for the analysis with a final three-dimensional electron density distribution, shown by superimposed contour sections drawn parallel to (100), can be found in Fig. 3.2 and an overall view of the molecule in Fig. 3.3 .

Mean values for interatomic bond distances, apart from carbon to oxygen single bonds, are given in Table 3.A and agree well with literature values. The figures in parentheses are the respective root mean square deviations from the means and are similar to the estimated standard deviations of individual measurements determined from the least-squares totals, the latter thus being a reasonable estimate of the random errors. Carbon to oxygen single bonds in the structure are of two types, O(1)-C(20) and O(3)-C(12) , as shown in Fig. 3.2 , are O - C sp^2 bonds and are similar to the "longer" O - C bonds found in esters and carboxylic acids, Sutton (1965) quotes a value of 1.358 ± 0.005 Å for O - C sp^2 and this compares with the mean value of 1.346 Å found in the analysis.

TABLE 3.A

MEAN BOND LENGTHS IN ANGSTROMS.

Bond	Type	Mean	Number	Literature *
C - C	$sp^3 - sp^3$	1.538(18)	14	1.537
	$sp^2 - sp^3$	1.507 (5)	3	1.510
	benzene	1.385(18)	6	1.394
C - Br	aliphatic	1.913	1	1.938
C = O		1.208(13)	2	1.215

* Sutton (1965).

O(1)-C(5) and O(3)-C(11) are O - C sp^3 bonds and a value of 1.426 ± 0.005 Å is normally expected. In the present analysis a mean value for the two bond lengths of 1.480 Å is significantly greater (six e.s.d's) than the quoted value. Bond lengthening of this type has been observed, however, in other lactones and esters, Kim, Jeffrey, Rosenstein and Corfield (1967) found a value of 1.475 Å in β-D-Glucurono-γ-lactone and Tichy (1966) reports 1.465 Å in pentane-2,4-diol diacetate.

The unusual carbon skeleton of the molecule results in considerable steric strain. Examination of a Dreiding model of the structure reveals that the δ - lactone can adopt a boat conformation with O(3) and C(8) at the prow and stern but that there is a close contact of about 2.6 Å between the carbonyl group oxygen, O(2), and the CH$_2$ group at C(2) . In the crystal strain is relieved by rotation of the lactone ring carbonyl oxygen, O(2), about the C(7)-C(12) bond so that the O(2)...C(2) separation is increased to 3.37 Å . This mechanism also has the effect of rotating the lactone ether oxygen, O(3), into the plane of four of the other atoms in the δ - lactone ring, that is atoms C(7),C(9),C(11) and C(12) . The remaining atom in the ring, C(8), and carbonyl oxygen, O(2), are then -0.845 and 0.062 Å respectively from this plane (Table 3.10

equation 1). The δ - lactone ring is thus in a similar conformation to that found by Ferguson and Islam (1967) in a p-bromophenacyl ester palmarin derivative. Bond angles in the lactone ring, with the exception of angle C(7)-C(8)-C(9) do not differ significantly from trigonal or tetrahedral values. Angle C(7)-C(8)-C(9) is 100.9° and the contraction from the tetrahedral value is caused by C(8) acting as the bridgehead atom between the δ - lactone and a cyclopentane ring. A value of 96.7° has been reported by Macdonald and Trotter (1965) for the more strained bridgehead angle in anti-8-tricyclo [3,2,1,$0^{2,4}$] octyl-p-bromobenzenesulphonate.

Both cyclopentane rings adopt envelope conformations. The ring C(5),C(6),C(7),C(8) and C(9) has a "flap" from C(7) to C(9) with C(8) -0.671 Å out of the best plane through the remaining atoms in the ring (Table 3.10, equation 2) , this large deviation is predictable as C(8) is the bridging atom between the cyclopentane and lactone rings. The second cyclopentane ring C(4),C(5),C(9),C(10) and C(11) folds at C(9) to C(10) with C(11) -0.550 Å out of the best plane through the other four atoms (Table 3.10 equation 3) . Again, this deviation is due to the O(2)...C(2) interaction whose effect is to rotate C(10) about the C(9)-C(11) bond away from O(2) leaving C(4), C(5),C(9) and C(10) coplanar.

The cyclohexane ring is a flattened chair with C(1), C(3),C(4) and C(6) forming the seat, C(2) and C(5) are -0.667 and 0.219 Å respectively out of the best plane through the above four atoms (Table 3.10 , equation 4). For cyclohexane rings, in the undistorted chair form, a value of 0.73 Å has been determined by Sim (1965) for the perpendicular distance of the two other atoms from the plane of the "seat". In the present analysis C(2) is forced back into the plane defined by C(1),C(3),C(4) and C(6) on account of the O(2)...C(2) interaction, while C(5) is held close to the plane as it is at the junction of the cyclohexane and cyclopentane rings. Bucourt and Hainaut (1965) quote a value of 112.4° for the bond angle in undistorted cyclohexane derivatives and values of 113.3, 110.9 and 113.7° for angles C(2)-C(1)-C(6), C(1)-C(2)-C(3) and C(2)-C(3)-C(4) do not differ significantly from this. Angles C(3)-C(4)-C(5), C(4)-C(5)-C(6) and C(1)-C(6)-C(5) are 115.8, 117.6 and 118.5° respectively and are significantly greater (five e.s.d's) than 112.4° showing the strain imposed at the junction of the cyclohexane and two cyclopentane rings.

Bond distances and interbond angles in the phenyl ring do not deviate from expected values. The six atoms are coplanar with a root mean square deviation of the atoms from the plane (Table 3.10, equation 5) of 0.009 Å . The bond lengths and interbond angles in the bromoacetate group are characteristic of carboxylic acids. As is common angles O(1)-C(20)-O(4) and O(4)-C(20)-C(21) of 126.3 and 126.1° are greater than 120° while angle O(1)-C(20)-C(21), of 107.6° , is less than 120° . Angle O(4)-C(20)-C(21) is greater than is normally found (van Bommel and Bijvoet (1958) quote a value of 123.0° in Ammonium Hydrogen D-Tartrate) and this expansion must be caused by the O(4)...Br(1) interaction (the separation of these atoms is 3.05 Å whereas the sum of their van der Waals radii is 3.35 Å). As expected, atoms O(1),O(4),C(20) and C(21) are coplanar (Table 3.10, equation 6) with the bromine atom -0.214 Å out of this plane in the direction specified by atoms C(1) and C(6) as opposed to C(3) and C(4).

The intramolecular distances in the molecule (Table 3.9) show some interesting contacts. The lactone carbonyl oxygen, O(2), is 2.99 Å distant from one of the phenyl group carbon atoms, C(15), and the other carbonyl oxygen, O(4), is situated almost equally from atoms C(1), C(3),C(4) and C(6) . This position for O(4) is probably a minimum in energy for the rotation of the acetate group about the O(1)-C(5) bond. This aspect of ester conformation has been discussed by Mathieson (1965) who concludes that contacts of this type are likely to be caused by interactions between the partial negative charge of the oxygen and partial positive charge of the hydrogen atoms, in the present case O(4) and the axial hydrogens on C(1) and C(3) , that is, H(1)i and H(3)i .

The packing of the molecules in the unit cell is shown in Fig. 3.4 . The shortest intermolecular distance is a carbonyl oxygen to methyl group contact of 3.23 Å (Table 3.9) . From the coordinates of the methyl group hydrogens (Table 3.4), obtained from the low order difference map, H(13)iii is found to lie close to a line joining the methyl carbon, C(13), and the carbonyl oxygen, O(2), at position 1 + x, y, z . As there is no further evidence for the formation of a hydrogen bond between C(13) and O(2) this contact must be classed with the “short” carbon to oxygen interatomic distances discussed by Sutor (1963) where perhaps some electrostatic forces are involved. The remaining interatomic distances are not significant in terms of van der Waals radii.

The final anisotropic thermal parameters (Table 3.3) are physically reasonable and indicate that atoms farthest away from the centre of gravity of the molecule have the largest root mean square amplitudes of vibration. This is seen in the aromatic ring where the uncertainty of atomic position, and hence estimated standard deviation, increases the farther atoms in the ring are from its point of attachment to the tricyclic carbon framework.

CHAPTER 4

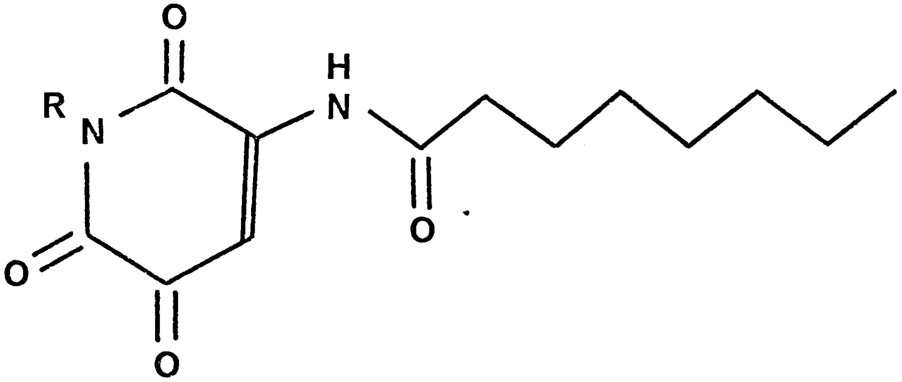
N-p-Iodobenzyl-3-caprylamido-1,2,5,6-tetrahydro pyridine-2,5,6-trione.

4.1 Introduction

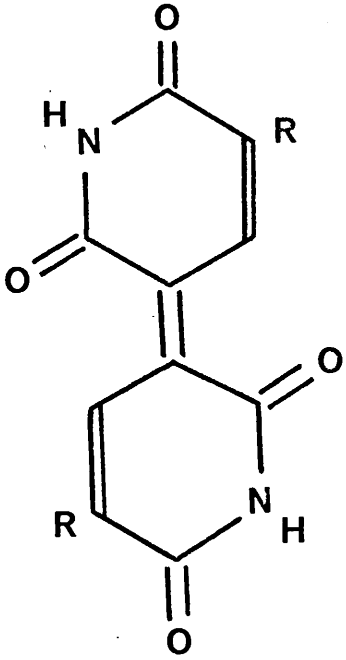
The bacterium Pseudomonas lemmonieri yields an intensely blue quinonoid type pigment which when oxidised with chromic oxide - sulphuric acid gives an optically inactive pale yellow crystalline product of molecular formula $C_{13}H_{18}N_2O_4$. Extensive chemical and spectroscopic work was prevented by the small quantities of material available and Whalley and his co-workers were unable to formulate the structure of either the oxidation product or the pigment. It was possible, however, to prepare an N-p-iodobenzyl derivative of the oxidation product and the three-dimensional X-ray analysis of this compound is described in the following sections.

The derivative is shown to be (I ; R = p-iodobenzyl) and the oxidation product is therefore (I ; R = H) which has obvious similarities with pigments of types (II ; R = -OH) and (II ; R = $-NH_2$) isolated from related micro-organisms by Kuhn, Bauer, Knackmuss, Kuhn and Starr (1964). It thus seemed possible that the original pigment had a structure (II ; R = $-NHCO(CH_2)_6Me$).

I



II



This was synthesised by Whalley and his co-workers but although obviously similar to the metabolite is not identical with it. The chemical implications of these findings have been briefly discussed by Ferguson et al. (1965).

4.2 Experimental

Crystal data

N-p-Iodobenzyl-3-caprylamido-1,2,5,6-tetrahydro pyridine-2,5,6-trione.

$C_{20}H_{23}N_2O_4I$

F.W. = 481.3 .

Orthorhombic, a = 8.68 ± 3, b = 38.71 ± 5, c = 12.53 ± 3 Å

$U = 4210\ \text{Å}^3$, $D_m = 1.51\ \text{gm.cm.}^{-3}$, $Z = 8$, $D_x = 1.52\ \text{gm.cm.}^{-3}$

Linear absorption coefficient $\mu = 126\ \text{cm.}^{-1}$ (Cu K_α).

F(000) = 1928.

Systematic absences,

0kl when k is odd,

h0l when l is odd,

hk0 when h is odd.

Space group Pbca (D_{2h}^{15} , No. 61).

Cell dimensions and space group were determined from rotation and equatorial layer Weissenberg photographs taken about the a and c axes with Cu K_α radiation. The crystal data together with work done in the (100) and (001) projections was provided by D. M. Hawley from his B.Sc. Thesis (Glasgow, 1965).

Intensity data were obtained from multiple film Weissenberg photographs of the 0kl to 7kl reciprocal lattice nets. The crystals were in the form of fragile thin plates not really suitable for accurate intensity measurements. The Weissenberg photographs were of poor quality but as the main objective of the X-ray analysis was the gross chemical structure no attempt was made to obtain better quality crystals. Lorentz, polarisation and rotation factors were applied and the resulting 1004 structure amplitudes were put on a common scale by comparison with reflections from the hk0 zone. Scale factors were determined thereafter from calculated and observed structure factors and finally by least-squares methods. No unobserved reflections were used in the analysis. The relatively small number of structure amplitudes (21 % of the data accessible to Cu K_{α} radiation) was due to the rapid fall off of the data with sin θ / λ .

4.3 Structure Solution and Refinement

The iodine atom coordinates were provided by D. M. Hawley and were confirmed by inspection of the three-dimensional Patterson function. Harker sections arising on account of the three twofold screw axes, that is sections $U = V = W = 1/2$, are shown in Fig. 4.1 . Iodine fractional coordinates used for computing the first set of structure factors were

I(1)	0.0950	0.0752	0.0539 .

An electron-density synthesis, phased from these structure factors ($R = 0.441$), revealed the N-p-iodobenzyl group and other features of the molecule. Nineteen atoms (see course of analysis) apart from the iodine were included in the next structure factor calculation ($R = 0.362$) and a subsequent electron-density distribution showed all atoms in the molecule. Structure factors involving all atoms, except hydrogens, gave $R = 0.292$ and a further electron-density calculation allowed assignment of chemical types.

Using the author's least-squares program, the block diagonal approximation, 0.75 shifts and a weighting scheme such that

$$w = 1 / (A + |Fo| + B\, |Fo|^2)$$

where $A = 2 F$ min. and $B = 2 / F$ max., the value of R was lowered to 0.189 in four cycles of refinement. Inspection of an electron-density distribution then showed the iodine peak to be highly ellipsoidal indicating a high anisotropic vibration for this atom. Refinement was continued using Cruickshank and Smith's 1965 least-squares program and incorporating anisotropic vibration parameters for the iodine atom. Three cycles of block diagonal refinement with a weighting scheme fitted by the method described by McGregor (1967) reduced R to 0.125 . As the distribution of $\langle w \Delta^2 \rangle$ had become uneven during this refinement, McGregor's weighting scheme program was used to readjust the parameters of the weighting scheme. Three further cycles of least-squares calculations reduced the average parameter shift to less than one tenth of the corresponding standard deviation and the refinement was concluded with $R = 0.122$.

A final (Fo - Fc) distribution showed no serious errors in the structural model and gave a fluctuating background in the region of ± 0.3 e/Å^3 , $\sigma(\rho)$ was 0.22 e/Å^3 and the only features on the map greater than $3 \sigma(\rho)$ were associated with the iodine atom and were most likely caused by absorption.

Formfactors used were those of Freeman (1959) for carbon, Berghuis et al. (1955) for nitrogen and oxygen, and Thomas and Umeda (1957) for iodine. Atomic fractional and thermal parameters are given in Table 4.1 , coordinates in Angstroms in Table 4.2 , final observed and calculated structure factors together with an analysis by magnitude of Fo and reciprocal lattice net in Tables 4.3 and 4.4 ; interatomic distances and angles, some non-bonded interatomic distances and finally mean planes are in Tables 4.5 to 4.8 . Standard deviations, when quoted, are in units of the last decimal place.

DIAGRAMS AND TABLES.

COURSE OF ANALYSIS.

I Fourier refinement.

		R
1	Iodine alone.	0.441
2	I, 1 N, 18 C.	0.362
3	I, 4 O, 2 N, 20 C.	0.291

II Least-squares refinement.

Cycles of refinement	Final R	Final R dash	*
1 - 4	0.189	-	(a)
5 - 7	0.125	0.029	(b)
8 - 10	0.122	0.026	(c)

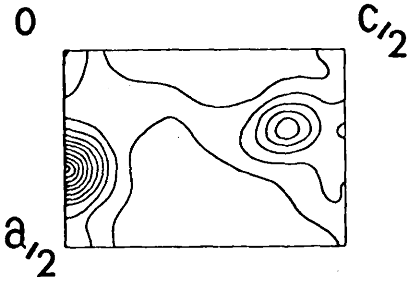
* (a) isotropic, individual layer scale factors.

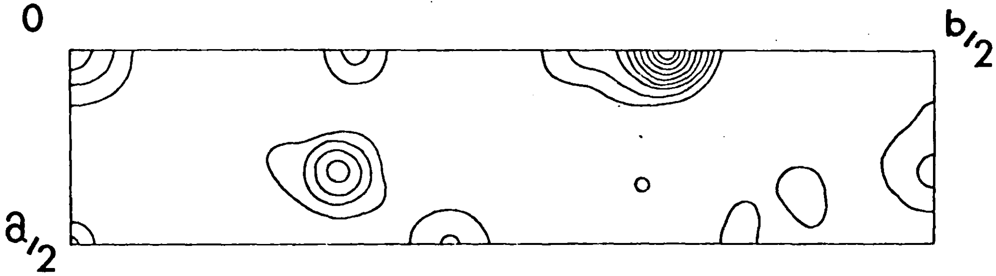
(b) Iodine anisotropic, one overall scale parameter.

(c) as above, weighting scheme changed.

FIG. 4.1

The Harker sections at U = 1/2, V = 1/2 and W = 1/2. Contours are at arbitrary intervals.


0
c/2
a/2


0
b/2
a/2

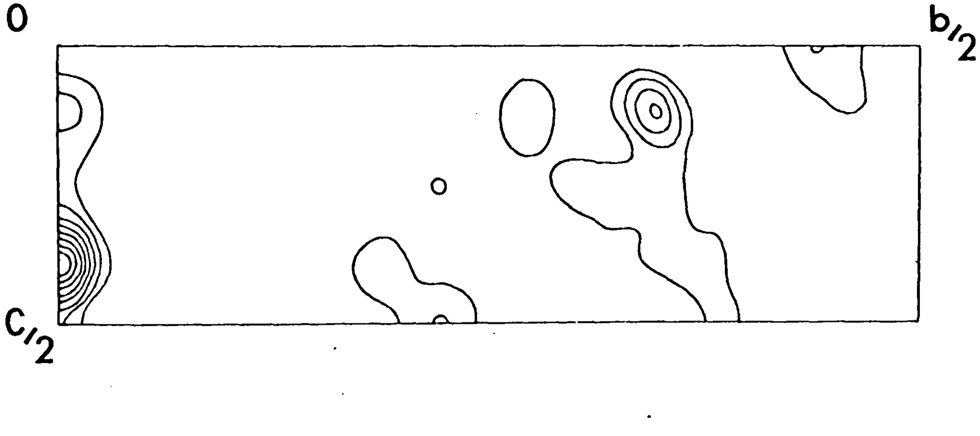

0
b/2
c/2

FIG. 4.2

Final three-dimensional electron-density synthesis viewed down the *a* axis, and corresponding atomic arrangement. Contours are at 1 e/Å^3 intervals starting at 1 e/Å^3 except around the iodine atom which is contoured at 5 e/Å^3 intervals.

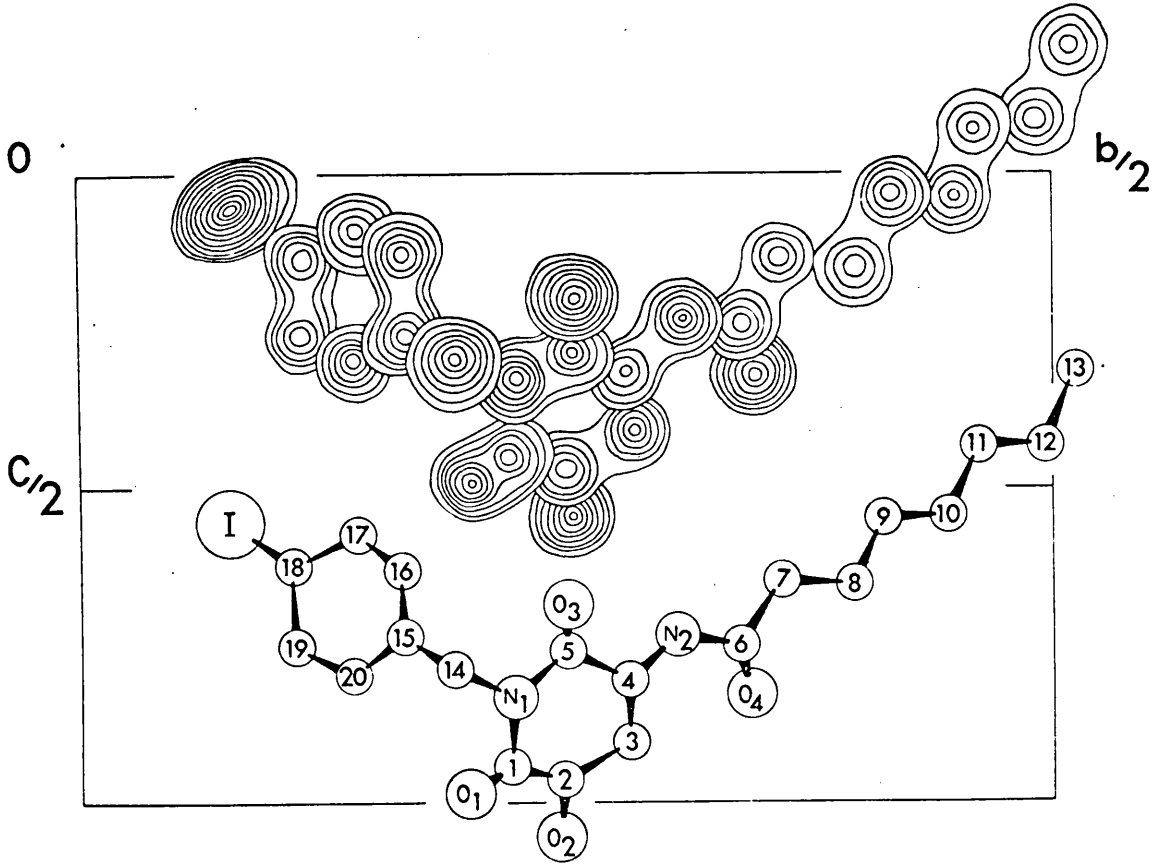

0
b/2
c/2
13
12
11
10
9
8
7
6
O_4
N_2
4
3
5
O_3
2
O_2
1
O_1
N_1
14
15
16
17
18
19
20
I

FIG. 4.3

The molecular packing viewed down the a axis.

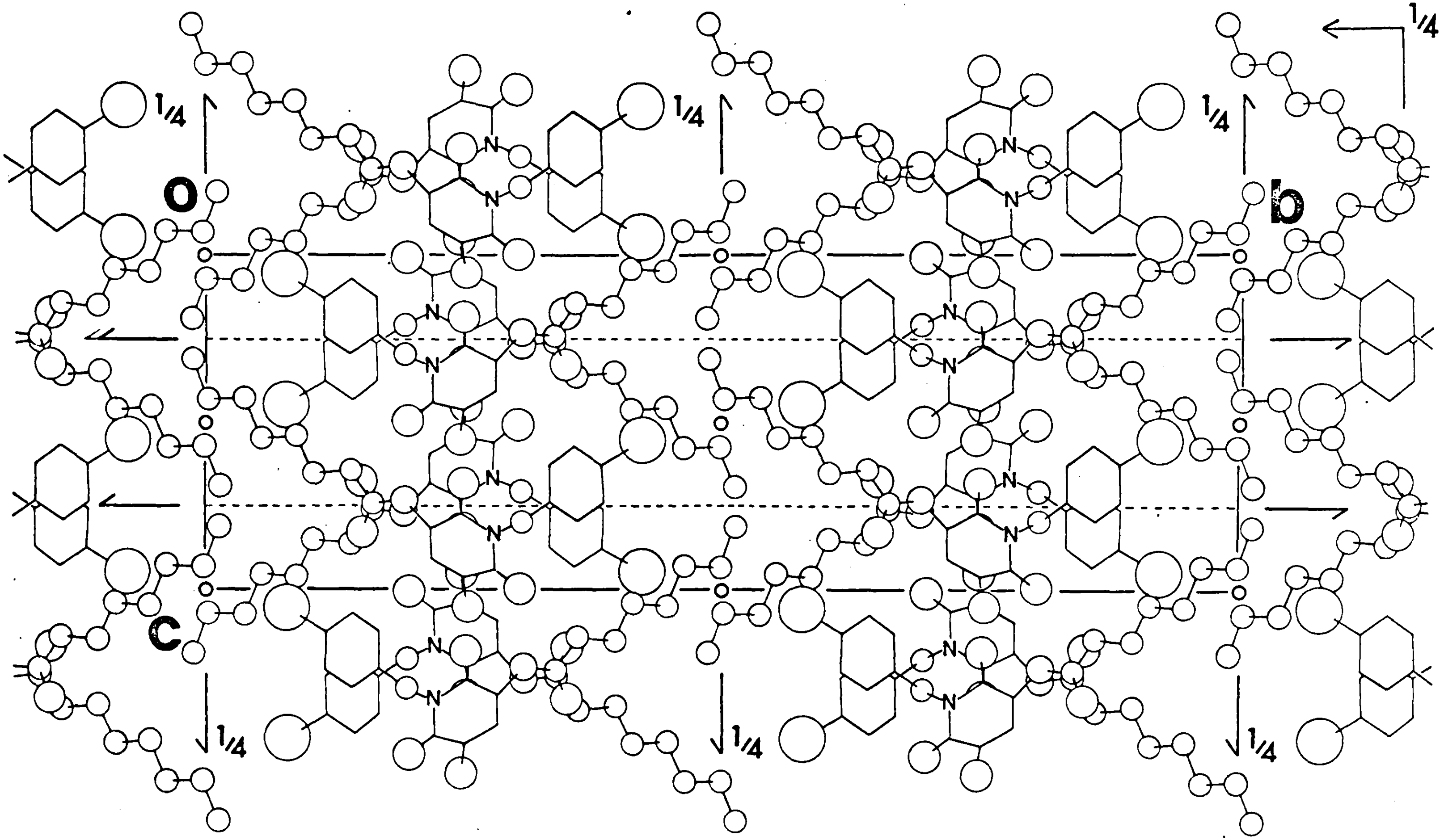

1/4
O
b
c
N
1/4
1/4
1/4
1/4
1/4
1/4

FIG. 4.4

Region around the heterocyclic ring viewed down the b axis. (Roman numerals have the same significance as in Table 4.7)

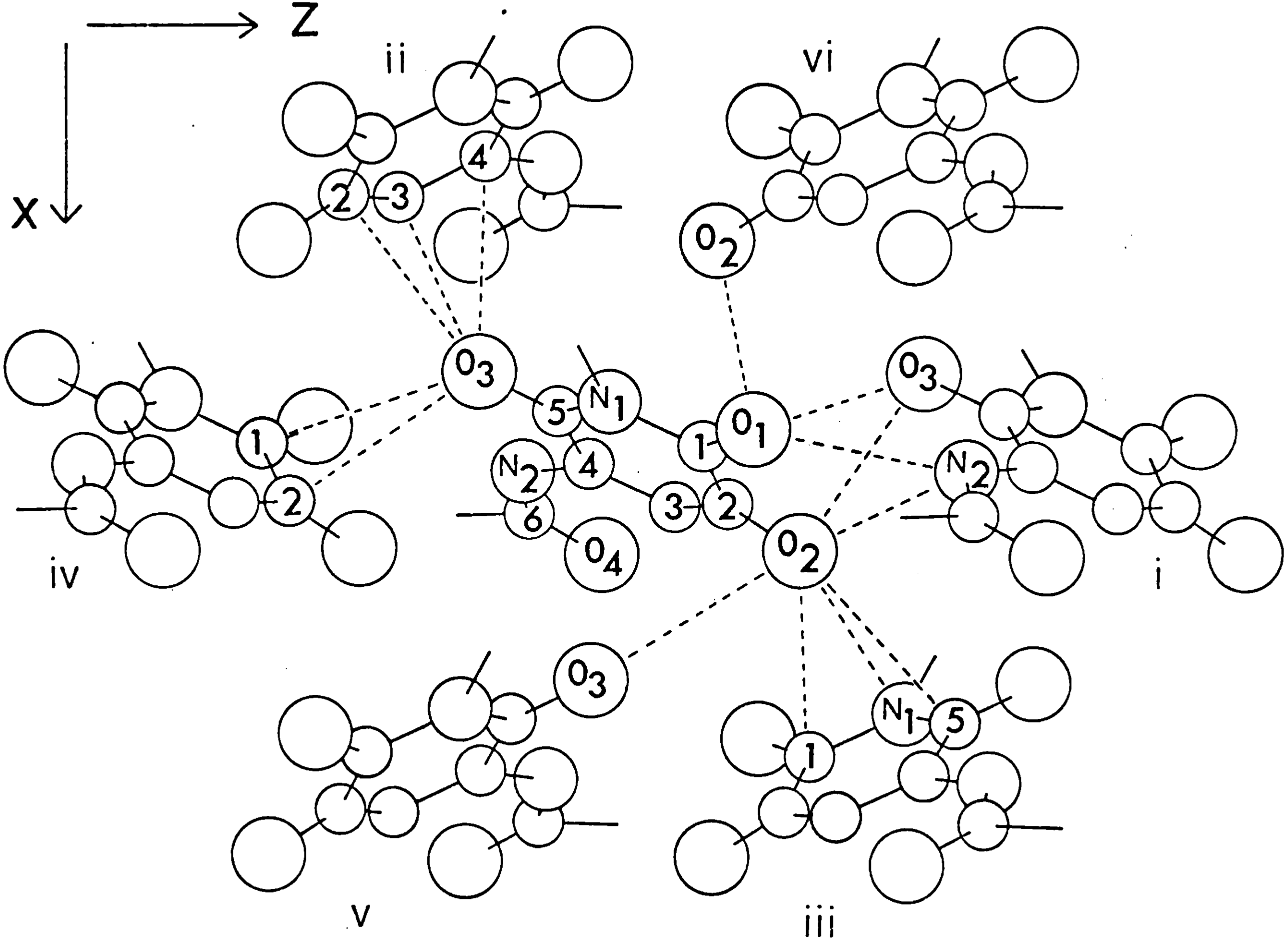

Z
X
ii
vi
iv
i
v
iii
O3
N1
O1
O2
N2
O4
1
2
3
4
5
6

TABLE 4.1

FRACTIONAL COORDINATES AND ISOTROPIC TEMPERATURE FACTORS WITH E.S.D.S.

ATOM	X/a	Y/b	Z/c	U iso
I(1)	0.1007(3)	0.0772(1)	0.0538(3)	*
O(1)	-0.1456(19)	0.1956(4)	0.4941(15)	0.055(5)
O(2)	0.0503(24)	0.2468(5)	0.5484(16)	0.080(6)
O(3)	-0.2330(20)	0.2508(5)	0.1834(15)	0.061(5)
O(4)	0.0586(26)	0.3481(6)	0.3266(19)	0.099(7)
N(1)	-0.1857(22)	0.2209(5)	0.3329(16)	0.044(5)
N(2)	-0.0751(22)	0.3067(5)	0.2349(17)	0.047(6)
C(1)	-0.1151(31)	0.2209(7)	0.4374(21)	0.055(7)
C(2)	-0.0226(32)	0.2515(7)	0.4654(21)	0.057(8)
C(3)	-0.0199(29)	0.2827(6)	0.4063(20)	0.045(7)
C(4)	-0.0875(27)	0.2822(6)	0.3081(19)	0.043(7)
C(5)	-0.1719(28)	0.2524(6)	0.2733(20)	0.044(7)
C(6)	-0.0046(34)	0.3380(7)	0.2435(26)	0.068(8)
C(7)	-0.0067(41)	0.3592(8)	0.1426(26)	0.087(10)
C(8)	0.0758(35)	0.3951(8)	0.1521(26)	0.077(9)
C(9)	0.0776(33)	0.4120(7)	0.0397(23)	0.066(9)
C(10)	0.1522(33)	0.4459(8)	0.0487(23)	0.068(9)
C(11)	0.1570(45)	0.4622(10)	-0.0722(31)	0.108(13)
C(12)	0.2418(41)	0.4976(9)	-0.0683(31)	0.097(11)
C(13)	0.2519(63)	0.5121(13)	-0.1876(46)	0.177(22)

C(14)	-0.2796(27)	0.1930(6)	0.2978(19)	0.042(7)
C(15)	-0.1917(28)	0.1665(6)	0.2394(21)	0.044(7)
C(16)	-0.1856(32)	0.1661(7)	0.1281(22)	0.056(8)
C(17)	-0.0995(32)	0.1418(8)	0.0731(23)	0.067(8)
C(18)	-0.0280(34)	0.1168(7)	0.1264(24)	0.067(9)
C(19)	-0.0374(37)	0.1148(8)	0.2422(30)	0.082(10)
C(20)	-0.1135(34)	0.1398(8)	0.2968(24)	0.073(9)

* The thermal vibration of the Iodine atom is represented by the equation,

$$\exp [- 2 \pi^2 (0.101 \, h^2 a^{*2} + 0.093 \, k^2 b^{*2} + 0.154 \, l^2 c^{*2} - 0.111 \, k \, l \, b^* c^* + 0.052 \, l \, h \, c^* a^* - 0.002 \, h \, k \, a^* b^*)]$$

TABLE 4.2

COORDINATES AND E.S.D.S IN ANGSTROMS WITH RESPECT TO THE CRYSTAL AXES.

ATOM	X	Y	Z
I(1)	0.874 ± 3	2.989 ± 3	0.674 ± 3
O(1)	-1.264 ± 17	7.571 ± 17	6.191 ± 19
O(2)	0.437 ± 21	9.552 ± 21	6.871 ± 20
O(3)	-2.022 ± 18	9.707 ± 18	2.299 ± 19
O(4)	0.508 ± 23	13.476 ± 22	4.092 ± 24
N(1)	-1.612 ± 19	8.550 ± 18	4.171 ± 20
N(2)	-0.652 ± 19	11.873 ± 19	2.943 ± 22
C(1)	-0.999 ± 27	8.551 ± 26	5.480 ± 27
C(2)	-0.196 ± 28	9.735 ± 27	5.832 ± 27
C(3)	-0.173 ± 25	10.941 ± 24	5.091 ± 26
C(4)	-0.759 ± 23	10.922 ± 23	3.860 ± 24
C(5)	-1.492 ± 25	9.772 ± 24	3.424 ± 25
C(6)	-0.040 ± 30	13.082 ± 26	3.051 ± 32
C(7)	-0.058 ± 36	13.903 ± 32	1.786 ± 33
C(8)	0.658 ± 30	15.294 ± 30	1.906 ± 33
C(9)	0.673 ± 29	15.948 ± 28	0.498 ± 28
C(10)	1.321 ± 28	17.259 ± 29	0.610 ± 29
C(11)	1.362 ± 39	17.892 ± 38	-0.905 ± 39
C(12)	2.099 ± 36	19.261 ± 37	-0.855 ± 38
C(13)	2.186 ± 54	19.823 ± 51	-2.351 ± 57

C(14)	-2.426 ± 23	7.470 ± 22	3.731 ± 24
C(15)	-1.663 ± 25	6.445 ± 22	3.000 ± 26
C(16)	-1.610 ± 28	6.431 ± 26	1.605 ± 27
C(17)	-0.864 ± 28	5.490 ± 29	0.916 ± 29
C(18)	-0.243 ± 30	4.520 ± 28	1.584 ± 30
C(19)	-0.325 ± 32	4.443 ± 30	3.035 ± 37
C(20)	-0.985 ± 30	5.411 ± 30	3.719 ± 30

TABLE 4.3

FINAL OBSERVED AND CALCULATED STRUCTURE FACTORS.

H	K	L	Fo	Fc
0	4	0	51.3	-49.0
0	6	0	322.0	-361.0
0	8	0	80.6	-72.7
0	10	0	50.8	-51.6
0	12	0	379.0	334.9
0	14	0	225.2	218.9
0	16	0	59.5	56.0
0	18	0	235.6	-202.5
0	20	0	51.7	-53.3
0	22	0	124.5	-106.8
0	24	0	145.2	130.0
0	26	0	153.3	108.5
0	28	0	106.4	91.9
0	30	0	78.3	-84.7
0	32	0	33.3	-18.6
0	34	0	115.8	-108.5
0	36	0	112.7	103.4
0	42	0	30.4	14.8
0	2	1	112.5	-127.8
0	4	1	106.9	-119.9
0	6	1	16.1	18.1
0	8	1	72.4	62.7
0	10	1	83.2	85.1
0	14	1	22.9	23.6
0	16	1	23.0	-28.3
0	18	1	134.5	-99.0
0	22	1	140.2	110.4
0	24	1	55.1	39.0
0	30	1	38.2	-30.6
0	32	1	29.8	-30.9
0	34	1	37.3	36.6
0	0	2	148.5	163.3
0	2	2	280.5	291.5
0	4	2	38.1	24.2
0	6	2	373.4	-408.7
0	8	2	226.6	-213.0
0	10	2	50.5	61.7
0	12	2	160.7	175.0
0	14	2	164.2	181.7
0	18	2	168.5	-169.6
0	20	2	110.3	-110.6
0	22	2	48.4	32.8
0	24	2	117.7	68.7
0	26	2	98.2	78.6
0	28	2	38.0	26.8
0	30	2	37.4	-40.1
0	32	2	72.0	-58.4
0	38	2	35.3	35.6
0	42	2	28.6	-15.2
0	2	3	196.1	-225.3
0	4	3	171.4	-176.5
0	6	3	129.6	-125.0
0	8	3	79.5	85.7
0	10	3	188.6	202.5
0	12	3	100.5	101.0
0	14	3	92.2	-88.7
0	16	3	180.2	-186.0
0	18	3	91.6	-109.4
0	22	3	112.0	110.3
0	24	3	130.5	116.7
0	26	3	32.2	18.8
0	28	3	79.0	-74.2
0	30	3	70.8	-59.7
0	32	3	65.6	-45.0
0	36	3	41.8	37.3
0	0	4	146.5	151.5
0	2	4	60.8	-68.6
0	4	4	31.8	24.9
0	6	4	72.0	-60.5
0	8	4	87.2	-91.8
0	10	4	80.8	58.3
0	12	4	131.6	114.5
0	14	4	64.9	-60.8
0	16	4	45.6	44.6
0	18	4	151.6	-140.8
0	20	4	47.3	41.3
0	24	4	82.0	69.0
0	26	4	33.2	29.0
0	28	4	73.9	-60.9
0	30	4	43.9	-28.9
0	36	4	47.5	46.6
0	2	5	196.1	-203.0
0	4	5	174.0	-186.7
0	6	5	42.3	36.6
0	8	5	81.5	76.8
0	10	5	98.9	122.4
0	12	5	75.7	73.1
0	14	5	94.7	-84.5
0	16	5	182.0	-171.6
0	18	5	65.3	-53.7
0	20	5	32.8	31.9
0	24	5	52.5	64.3
0	26	5	34.2	31.8
0	28	5	71.4	-67.1
0	30	5	100.7	-81.9
0	32	5	21.6	-17.0
0	36	5	27.2	27.0
0	0	6	67.9	-78.8
0	2	6	83.1	-82.1
0	6	6	25.1	26.8
0	8	6	94.8	78.9
0	10	6	48.5	39.3
0	12	6	46.3	-42.7
0	14	6	60.6	-54.3
0	16	6	61.4	-48.2
0	18	6	22.5	-24.9
0	20	6	82.6	72.8
0	22	6	48.5	49.4
0	26	6	39.4	-34.3
0	28	6	49.3	-41.7
0	30	6	39.3	-28.9
0	34	6	34.6	27.2
0	2	7	30.3	-29.7
0	6	7	57.6	-62.4
0	8	7	106.8	96.4
0	10	7	67.9	59.3
0	14	7	39.7	-28.6
0	16	7	95.0	-98.1
0	20	7	70.0	63.6
0	22	7	59.2	53.0
0	28	7	52.9	-55.6
0	34	7	30.1	30.0
0	0	8	114.8	-114.7
0	2	8	33.8	-37.1
0	4	8	74.0	-59.3
0	6	8	119.9	123.6
0	10	8	52.8	42.5
0	12	8	66.8	-53.8
0	14	8	102.4	-104.7
0	18	8	44.6	48.9
0	20	8	45.5	41.2
0	28	8	63.6	-64.4
0	34	8	19.2	29.7
0	8	9	31.4	30.0
0	14	9	44.1	-37.6
0	22	9	39.9	49.0
0	0	10	73.7	-65.6
0	4	10	32.9	30.5
0	6	10	50.7	47.6
0	12	10	68.8	-61.5
0	4	11	30.5	20.2
0	6	11	37.4	20.9
0	12	11	41.5	-36.3
1	1	1	221.9	198.9
1	2	1	144.1	-131.2
1	3	1	16.3	-7.4
1	4	1	56.8	-63.6
1	5	1	240.1	-213.7
1	6	1	89.8	-90.2
1	7	1	176.1	-165.6
1	8	1	224.0	205.6
1	9	1	79.5	-80.2
1	10	1	124.1	149.6
1	11	1	83.7	108.5
1	12	1	28.8	46.3
1	14	1	39.4	-46.7
1	15	1	110.7	99.8
1	16	1	203.6	-181.1
1	17	1	42.3	-57.3
1	18	1	108.4	-82.7
1	19	1	140.2	-123.4
1	20	1	44.7	37.5
1	21	1	154.8	-144.4
1	22	1	88.6	70.8
1	23	1	32.9	26.3
1	24	1	83.0	74.5
1	25	1	88.9	84.5
1	26	1	25.4	10.3
1	27	1	105.7	84.2
1	28	1	65.3	-66.2
1	30	1	39.4	-21.1
1	31	1	63.3	-59.5
1	33	1	35.6	-34.3
1	34	1	29.2	27.0
1	35	1	20.7	-16.9
1	0	2	102.4	-108.0
1	1	2	117.6	-34.6
1	2	2	26.4	14.7
1	3	2	315.5	-321.4
1	4	2	74.2	39.7
1	5	2	40.7	-35.8
1	6	2	233.9	246.2
1	7	2	101.9	92.2
1	8	2	179.9	159.5
1	9	2	134.9	127.1
1	10	2	60.6	-65.2
1	11	2	126.8	129.5
1	12	2	28.0	10.6
1	13	2	41.2	-46.8
1	14	2	110.7	-126.5
1	15	2	12.8	9.0
1	17	2	140.0	-157.8
1	20	2	90.3	94.0
1	23	2	80.3	73.1
1	24	2	34.5	17.8
1	25	2	43.4	39.2
1	26	2	40.7	-39.4
1	27	2	18.6	-20.4
1	28	2	33.0	-23.4
1	29	2	33.7	-24.5
1	30	2	28.1	21.1
1	1	3	103.1	77.1
1	2	3	58.4	-56.0
1	3	3	44.5	44.2
1	4	3	61.2	-45.6
1	5	3	173.6	-158.5
1	6	3	24.9	14.0
1	7	3	62.0	-58.2
1	8	3	96.0	86.4
1	9	3	11.2	-12.3
1	10	3	114.0	111.1
1	11	3	69.4	74.0
1	12	3	32.5	-30.1
1	13	3	83.3	92.7
1	14	3	29.4	-35.6
1	15	3	45.0	37.7
1	16	3	28.0	-31.0
1	17	3	50.0	-57.4
1	19	3	70.4	-98.8
1	22	3	53.0	61.6
1	23	3	42.2	39.3
1	24	3	30.7	35.3
1	25	3	25.7	24.1
1	26	3	37.2	-30.0
1	27	3	26.9	28.1
1	31	3	45.5	-36.8
1	33	3	90.7	-35.0
1	0	4	170.8	-145.7
1	1	4	150.1	-129.6
1	2	4	69.2	-69.2
1	3	4	233.9	-219.0
1	5	4	129.1	-109.8
1	6	4	210.2	195.3
1	7	4	67.8	63.1
1	8	4	99.6	94.6
1	9	4	78.2	106.6
1	10	4	47.4	44.7
1	11	4	236.2	213.6
1	12	4	167.3	-171.1
1	14	4	44.6	-48.3
1	15	4	154.6	-149.9
1	16	4	39.4	-39.5
1	17	4	101.5	-133.7
1	18	4	54.5	73.5
1	20	4	49.8	59.6
1	21	4	45.0	57.9
1	22	4	49.4	66.6
1	23	4	56.6	76.1
1	24	4	55.0	-54.2
1	29	4	60.4	-65.2
1	1	5	39.4	-33.5
1	2	5	71.9	-60.6
1	3	5	41.8	35.6
1	4	5	105.5	87.0
1	6	5	34.4	-19.6
1	7	5	45.8	29.8
1	8	5	35.6	-26.1
1	10	5	27.9	-25.7
1	12	5	66.7	-59.0
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1	15	5	86.7	-68.7
1	16	5	22.5	-26.2
1	17	5	28.2	-26.7
1	18	5	33.4	32.2
1	20	5	46.2	46.9
1	22	5	31.6	-33.6
1	23	5	32.3	38.5
1	0	6	151.7	-135.4
1	1	6	34.4	-28.2
1	3	6	116.6	-113.8
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1	5	6	96.3	-99.9
1	6	6	91.8	93.5
1	8	6	85.4	80.2
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1	12	6	50.1	-51.6
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1	17	6	81.7	-81.2
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1	21	6	46.0	54.9
1	23	6	33.7	51.8
1	27	6	45.7	-52.3
1	1	7	54.1	-55.9
1	2	7	33.4	39.4
1	4	7	38.7	40.0
1	5	7	31.8	37.2
1	7	7	74.2	66.9
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1	3	8	51.9	-46.5
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1	1	9	37.4	-29.0
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1	12	9	34.2	-38.3
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1	14	9	49.0	50.3
1	15	9	34.9	-48.0
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2	5	0	280.3	-291.9
2	6	0	189.7	-177.6
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2	10	0	23.1	-22.8
2	11	0	250.6	261.9
2	12	0	10.9	6.3
2	13	0	97.4	-76.4
2	14	0	98.9	82.5
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2	16	0	80.3	-49.2
2	17	0	137.5	-152.4
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2	20	0	50.1	-47.0
2	21	0	90.7	88.2
2	22	0	34.6	-33.4
2	23	0	124.6	121.1
2	25	0	51.5	45.5
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2	27	0	65.7	-63.2
2	28	0	33.0	31.5
2	29	0	63.7	-69.8
2	30	0	39.6	-41.9
2	31	0	51.3	-43.3
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2	33	0	35.6	40.8
2	37	0	64.3	67.6
2	1	1	17.3	-7.7
2	2	1	147.5	117.8
2	3	1	135.4	-112.6
2	4	1	228.1	-218.2
2	5	1	23.5	-14.3
2	6	1	92.9	79.1
2	7	1	220.5	195.5
2	8	1	55.2	45.1
2	9	1	33.3	30.1
2	10	1	37.9	-38.0
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2	12	1	31.5	-35.2
2	14	1	43.0	46.6
2	15	1	103.8	-90.9
2	16	1	13.2	-5.6
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2	1	2	198.3	-187.1
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2	5	2	220.1	-218.7
2	6	2	193.8	-207.7
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2	10	2	57.2	-55.0
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2	12	2	134.7	149.8
2	13	2	53.6	-54.8
2	14	2	12.7	-13.5
2	18	2	81.9	-103.2
2	20	2	15.6	-24.1
2	21	2	90.2	91.6
2	23	2	105.9	107.6
2	24	2	58.2	59.3
2	27	2	37.8	-30.7
2	28	2	33.4	32.3
2	29	2	69.6	-70.6
2	1	3	204.9	-167.6
2	2	3	62.1	-73.0
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2	4	3	9.9	7.2
2	5	3	197.5	187.9
2	6	3	23.3	-23.3
2	7	3	125.1	123.7
2	8	3	20.7	-13.5
2	9	3	117.1	104.2
2	10	3	104.7	119.8
2	11	3	87.5	-90.3
2	12	3	17.6	14.1
2	13	3	149.2	-157.0
2	15	3	61.3	-76.4
2	16	3	36.5	-38.5
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2	22	3	29.6	16.4
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2	0	4	36.9	31.7
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2	5	4	36.8	-33.7
2	6	4	52.4	-39.2
2	7	4	73.1	57.2
2	8	4	31.5	-21.7
2	9	4	21.9	21.2
2	11	4	37.7	39.9
2	12	4	72.3	72.0
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2	17	4	29.2	29.7
2	18	4	27.8	-20.5
2	19	4	16.5	-8.4
2	20	4	16.9	-12.8
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2	24	4	37.2	33.7
2	25	4	53.7	-44.5
2	27	4	27.8	-27.6
2	28	4	20.0	-12.7
2	29	4	20.2	-21.5
2	1	5	153.5	-144.5
2	2	5	60.8	-58.9
2	4	5	63.7	-58.7
2	5	5	104.8	104.2
2	6	5	24.9	19.4
2	7	5	139.4	139.9
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2	9	5	43.2	41.1
2	11	5	104.5	-108.8
2	12	5	58.9	61.9
2	13	5	100.8	-108.3
2	14	5	15.5	-7.2
2	15	5	33.7	-51.3
2	16	5	32.5	-44.5
2	17	5	42.4	52.8
2	19	5	77.8	100.2
2	21	5	49.8	64.4
2	25	5	61.9	-31.5
2	26	5	44.4	35.7
2	27	5	63.7	-67.4
2	0	6	51.7	-47.2
2	2	6	61.6	59.0
2	3	6	40.9	35.0
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2	5	6	104.2	105.2
2	6	6	36.3	31.6
2	7	6	21.2	22.7
2	9	6	53.4	-61.5
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2	12	6	22.9	22.4
2	13	6	52.1	-49.5
2	17	6	56.1	58.9
2	19	6	31.9	42.2
2	23	6	27.8	-39.5
2	1	7	37.4	-33.7
2	2	7	16.0	-15.3
2	5	7	74.4	62.8
2	6	7	28.3	-24.5
2	7	7	26.1	29.7
2	10	7	55.3	57.6
2	11	7	58.6	-62.4
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2	17	7	56.5	27.1
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2	7	8	25.4	29.1
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2	9	8	63.3	-75.1
2	11	8	65.3	-60.0
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2	17	8	54.1	70.1
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2	0	10	44.9	-37.1
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3	1	1	18.0	-12.5
3	2	1	160.3	-159.2
3	3	1	18.1	-12.8

H	K	L	Fo	Fc
3	4	1	188.5	-167.7
3	5	1	141.4	122.3
3	6	1	89.3	-67.1
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3	8	1	123.3	107.2
3	9	1	75.0	65.6
3	10	1	226.6	222.2
3	11	1	91.1	-83.1
3	12	1	55.8	84.5
3	13	1	73.0	-99.4
3	14	1	65.8	-75.7
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3	16	1	168.0	-165.9
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3	20	1	19.9	24.1
3	21	1	25.1	24.0
3	22	1	123.8	104.8
3	24	1	90.9	82.1
3	25	1	30.4	-29.3
3	26	1	16.6	-6.9
3	28	1	67.8	-64.8
3	29	1	24.9	-25.2
3	30	1	41.9	-43.5
3	32	1	25.7	-22.6
3	34	1	31.6	32.8
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3	5	2	25.6	24.5
3	6	2	213.4	209.2
3	7	2	52.2	41.6
3	8	2	58.7	46.8
3	9	2	78.8	-81.1
3	11	2	85.7	-94.3
3	12	2	131.0	-150.6
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3	18	2	25.3	35.2
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3	21	3	26.5	29.5
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3	23	3	22.7	-30.3
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3	12	6	123.3	-109.5
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3	18	6	58.3	62.7
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3	9	8	31.2	26.5
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3	2	9	24.4	18.7
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4	12	0	60.1	-40.4
4	13	0	123.9	-108.2
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4	16	0	45.2	30.5
4	17	0	52.2	-51.0
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4	20	0	114.5	98.9
4	21	0	22.5	19.0
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4	26	0	74.1	-62.7
4	1	1	14.6	-8.0
4	2	1	52.4	65.2
4	3	1	17.5	18.9
4	4	1	34.0	38.6
4	5	1	61.7	-71.1
4	6	1	8.3	-0.5
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4	8	1	18.9	24.3
4	9	1	26.4	32.2
4	10	1	33.3	-36.7
4	12	1	69.9	-87.5
4	13	1	41.9	-52.4
4	14	1	47.1	58.2
4	16	1	78.1	62.0
4	21	1	51.4	51.7
4	22	1	36.8	-31.3
4	23	1	44.7	-37.1
4	0	2	184.6	-185.0
4	1	2	59.7	-50.1
4	2	2	80.5	-60.6
4	3	2	89.6	-93.8
4	4	2	74.9	69.3
4	5	2	44.4	-45.8
4	6	2	98.6	83.4
4	7	2	23.2	20.9
4	8	2	123.9	123.5
4	9	2	158.4	160.2
4	10	2	38.9	-30.1
4	11	2	24.8	-11.5
4	12	2	69.9	-72.9
4	14	2	46.3	-58.8
4	15	2	35.9	-45.7
4	18	2	49.6	62.7
4	20	2	46.3	49.8
4	21	2	43.2	42.1
4	23	2	24.3	28.6
4	24	2	35.1	-28.9
4	25	2	31.0	34.7
4	26	2	59.0	-59.9
4	27	2	48.9	-49.3
4	1	3	45.3	-44.6
4	2	3	133.8	117.9
4	3	3	32.0	-27.9
4	4	3	85.4	72.0
4	5	3	66.8	66.6
4	6	3	80.1	75.1
4	7	3	66.0	66.7
4	8	3	119.7	-113.4
4	9	3	31.4	30.8
4	10	3	77.4	-66.7
4	11	3	12.6	-14.9
4	12	3	59.7	-62.2
4	13	3	50.3	-58.2
4	14	3	27.7	23.4
4	15	3	41.6	-44.0
4	16	3	79.8	95.0
4	18	3	31.1	50.1
4	21	3	39.4	41.5
4	22	3	58.2	-62.7
4	24	3	37.9	-43.8
4	26	3	36.9	-29.9
4	28	3	51.7	39.9
4	30	3	33.0	35.0
4	0	4	58.9	-48.7
4	3	4	37.2	-27.6
4	5	4	31.9	-37.6
4	6	4	21.3	16.0
4	7	4	31.9	33.9
4	8	4	12.8	7.5
4	9	4	<13.1	17.8
4	10	4	43.4	-44.9
4	11	4	38.8	35.9
4	13	4	25.0	-25.8
4	14	4	20.9	-20.8
4	15	4	26.3	-28.7
4	17	4	29.8	-31.1
4	18	4	39.9	42.2
4	22	4	25.0	-31.4
4	23	4	25.5	29.4
4	1	5	65.5	-66.7
4	2	5	97.7	86.6
4	4	5	77.2	64.9
4	5	5	50.8	51.0
4	6	5	25.7	23.0
4	7	5	48.3	48.4
4	8	5	74.2	-70.5
4	9	5	53.9	56.9
4	10	5	74.7	-67.9
4	11	5	55.9	-58.4
4	12	5	37.3	-38.6
4	13	5	29.0	-28.6
4	14	5	50.1	57.0
4	15	5	41.2	-43.2
4	16	5	59.2	59.5
4	24	5	26.5	-37.3
4	25	5	26.7	-34.8
4	0	6	46.6	42.3
4	1	6	37.6	-42.6
4	2	6	29.5	27.8
4	6	6	64.4	-61.7
4	10	6	51.6	-51.4
4	11	6	45.7	-47.0
4	12	6	28.4	35.0
4	13	6	16.6	9.3
4	14	6	31.6	28.0
4	16	6	32.6	36.0
4	18	6	33.5	-39.4
4	19	6	18.2	25.7
4	20	6	26.0	-24.7
4	2	7	32.5	26.1
4	3	7	28.2	-30.4
4	4	7	36.6	30.1
4	5	7	30.8	38.0
4	6	7	16.6	-17.7
4	7	7	51.4	52.0
4	8	7	29.2	-28.3
4	9	7	24.0	-25.1
4	0	8	54.0	51.7
4	2	8	43.0	35.4
4	4	8	51.4	-49.4
4	5	8	25.0	25.6
4	6	8	35.6	-36.6
4	7	8	25.3	-31.5
4	8	8	31.1	-25.6
4	14	8	56.0	55.4
4	16	8	32.7	-27.0
4	17	8	32.8	32.8
5	1	1	88.6	-116.6
5	2	1	17.8	17.9
5	3	1	35.0	-33.0
5	4	1	33.2	-33.9
5	5	1	111.0	107.1
5	7	1	116.2	97.7
5	8	1	58.2	-63.7
5	9	1	57.5	54.6
5	10	1	19.2	18.6
5	11	1	38.7	-47.5
5	13	1	100.8	-118.5
5	15	1	72.7	-69.1
5	17	1	46.9	44.5
5	19	1	87.0	84.7
5	21	1	52.6	37.5
5	25	1	55.7	-42.1
5	27	1	51.8	-46.3
5	0	2	35.5	36.4
5	1	2	25.9	22.8
5	3	2	85.3	84.4
5	5	2	100.2	91.6
5	7	2	28.1	-32.0
5	8	2	17.4	16.8
5	9	2	77.3	-84.2
5	11	2	65.2	-50.2
5	13	2	17.3	-10.4
5	14	2	12.6	26.5
5	15	2	42.3	56.9
5	17	2	39.3	50.6
5	19	2	20.8	16.2
5	21	2	46.3	-28.1
5	23	2	42.5	-30.9
5	25	2	37.0	-25.0
5	29	2	34.0	32.2
5	1	3	77.5	-66.8
5	4	3	57.7	-54.9
5	5	3	79.2	69.6
5	7	3	33.4	27.9
5	8	3	19.3	22.7
5	9	3	36.3	33.6
5	11	3	73.8	-73.5
5	12	3	17.8	20.5
5	13	3	25.9	-19.5
5	15	3	27.4	-18.3
5	16	3	14.1	17.0
5	17	3	14.4	17.4
5	19	3	30.3	45.2
5	22	3	27.8	29.8
5	25	3	44.3	-47.3
5	0	4	42.0	-41.2
5	1	4	96.6	86.3
5	2	4	45.9	47.4
5	3	4	118.7	110.3
5	4	4	28.2	-27.0
5	5	4	59.6	50.9
5	6	4	30.3	25.9
5	7	4	22.7	-21.8
5	8	4	58.7	-49.4
5	9	4	74.2	-67.7
5	10	4	61.3	52.4
5	11	4	89.1	-94.4
5	12	4	19.1	-27.4
5	13	4	13.8	-3.4
5	14	4	28.3	28.0
5	15	4	58.8	69.3
5	17	4	69.2	88.8
5	18	4	21.8	33.5
5	20	4	22.6	-17.3
5	21	4	28.1	-31.6
5	22	4	23.2	22.0
5	23	4	51.3	-55.4
5	1	5	12.7	18.3
5	2	5	22.1	-16.7
5	4	5	22.4	-13.1
5	5	5	18.5	-13.7
5	7	5	19.0	13.5
5	9	5	44.7	-42.8
5	10	5	14.0	15.7
5	15	5	51.9	46.7
5	1	6	26.4	25.6
5	3	6	79.7	78.2
5	5	6	47.7	35.3
5	7	6	36.7	-30.5
5	9	6	43.0	-46.0
5	11	6	53.0	-58.4
5	13	6	36.8	31.4
5	15	6	30.1	32.6
5	17	6	46.4	53.9
5	19	6	23.6	-12.7
5	21	6	29.3	-27.9
5	23	6	34.0	-43.8
5	1	7	40.6	35.0
5	5	7	45.3	-42.8
5	7	7	29.4	-21.7
5	9	7	29.8	-28.2
5	11	7	28.0	18.2
5	12	7	16.3	25.8
5	13	7	23.2	30.8
5	14	7	23.4	-32.3
5	15	7	23.6	19.6
5	1	9	41.4	34.4
6	7	0	25.5	-23.7
6	8	0	47.2	39.9
6	9	0	36.9	-39.6
6	10	0	44.6	-48.4
6	11	0	52.1	-54.0
6	12	0	69.9	-66.7
6	13	0	23.2	19.7
6	14	0	74.1	-71.0
6	15	0	13.4	16.3
6	16	0	19.5	-12.2
6	17	0	20.1	11.5
6	18	0	71.7	68.8
6	19	0	43.8	46.7
6	20	0	36.0	29.2
6	21	0	19.2	-20.8
6	23	0	66.1	-57.1
6	26	0	31.1	-40.2
6	1	1	21.1	32.2
6	4	1	36.2	36.3
6	5	1	36.4	-34.4
6	6	1	8.8	-12.6
6	7	1	13.3	-10.6
6	10	1	22.2	-25.2
6	11	1	11.6	10.9
6	12	1	17.1	-29.6
6	13	1	21.8	27.8
6	15	1	19.1	-19.8
6	16	1	32.7	27.8
6	0	2	49.6	-47.2
6	2	2	41.1	-51.1
6	3	2	51.2	60.6
6	4	2	42.3	44.2
6	5	2	22.5	19.6
6	6	2	54.9	62.0
6	8	2	21.3	14.5
6	9	2	37.6	-32.7
6	11	2	24.4	-26.3
6	12	2	69.3	-57.9
6	14	2	46.3	-44.5
6	15	2	24.1	34.6
6	17	2	29.4	25.0
6	20	2	44.3	50.7
6	1	3	49.5	40.7
6	2	3	51.6	50.2
6	4	3	39.2	31.2
6	5	3	45.8	-43.0
6	6	3	34.2	34.8
6	7	3	45.3	-42.0
6	8	3	46.6	-40.4
6	9	3	27.7	-28.2
6	10	3	40.2	-33.8
6	11	3	37.0	39.1
6	12	3	40.3	-33.3
6	13	3	30.9	25.2
6	15	3	25.1	29.1
6	16	3	34.8	50.4
6	18	3	21.3	27.0
6	19	3	31.4	-46.2
6	0	4	24.2	-21.9
6	4	4	21.5	16.8
6	6	4	22.1	19.4
6	10	4	33.7	-29.8
6	1	5	30.4	22.2
6	2	5	56.2	52.6
6	4	5	41.4	43.2
6	5	5	48.2	-43.4
6	7	5	26.6	-22.0
6	8	5	38.1	-33.2
6	9	5	29.2	-18.3
6	10	5	36.2	-29.0
6	11	5	41.1	35.0
6	12	5	30.4	-41.8
6	13	5	21.8	23.4
6	14	5	27.1	31.7
6	3	6	43.4	-36.8
6	7	6	21.6	-17.9
6	8	6	26.7	-22.6
6	9	6	38.1	35.9
6	14	6	32.5	28.9
6	4	7	35.7	23.9
6	10	7	41.7	-39.6
6	0	8	57.1	54.9
7	2	1	42.4	56.3
7	4	1	44.0	53.9
7	6	1	21.7	22.9
7	8	1	35.9	-41.9
7	10	1	32.9	-47.9
7	0	2	35.9	36.7
7	1	2	17.0	18.7
7	2	2	33.6	35.5
7	4	2	41.3	-47.3
7	5	2	23.5	15.8
7	6	2	28.2	-17.5
7	8	2	49.5	-43.5
7	4	3	30.5	21.2
7	8	3	44.3	-36.5
7	0	4	58.2	35.7
7	2	4	29.3	25.0
7	6	4	68.4	-61.7
7	8	4	48.2	-37.2
7	12	4	38.8	36.4
7	0	6	19.9	16.1
7	1	6	19.9	25.6
7	2	6	28.2	20.7
7	8	6	38.2	-33.7

TABLE 4.4

(A) ANALYSIS OF STRUCTURE FACTOR DATA AS A FUNCTION OF LAYER LINE INDEX. ALL FIGURES ARE ON ABSOLUTE SCALE.

h	$\Sigma\lvert F_o\rvert$	$\Sigma\lvert F_c\rvert$	$\Sigma\lvert\Delta\rvert$	N	R	$\Sigma\lvert\Delta\rvert/N$
0	12339	11765	1389	143	0.1125	9.71
1	12523	12044	1581	176	0.1263	8.99
2	12511	12182	1424	182	0.1138	7.82
3	8817	8601	1083	152	0.1229	7.13
4	7618	7576	880	145	0.1155	6.07
5	4406	4294	651	101	0.1477	6.44
6	2926	2886	410	82	0.1402	5.00
7	830	788	166	23	0.2004	7.23
ALL	61970	60136	7584	1004	0.1224	7.55

(B) ANALYSIS OF STRUCTURE FACTORS BY MAGNITUDE OF Fo.

RANGE	$\Sigma\lvert F_o\rvert$	$\Sigma\lvert F_c\rvert$	$\Sigma\lvert\Delta\rvert$	N	R	$\Sigma\lvert\Delta\rvert/N$
0 - 20	1288	1288	417	80	0.3240	5.22
20 - 40	10683	10354	1900	352	0.1779	5.40
40 - 60	11461	11272	1450	233	0.1265	6.22
60 - 80	8281	8049	1024	119	0.1236	8.60
80 - 120	10873	10348	1195	112	0.1099	10.67
120 - 200	11958	11626	1050	78	0.0878	13.46
200 - 450	7427	7197	549	30	0.0739	18.29

TABLE 4.5

BOND LENGTHS AND E.S.D.S IN ANGSTROMS.

I(1) - C(18)	2.10 ± 3	C(6) - C(7)	1.51 ± 5
O(1) - C(1)	1.24 ± 3	C(7) - C(8)	1.57 ± 5
O(2) - C(2)	1.23 ± 3	C(8) - C(9)	1.55 ± 4
O(3) - C(5)	1.25 ± 3	C(9) - C(10)	1.47 ± 4
O(4) - C(6)	1.24 ± 4	C(10) - C(11)	1.64 ± 5
N(1) - C(1)	1.45 ± 3	C(11) - C(12)	1.56 ± 5
N(1) - C(5)	1.44 ± 3	C(12) - C(13)	1.60 ± 7
N(1) - C(14)	1.42 ± 3	C(14) - C(15)	1.47 ± 3
N(2) - C(4)	1.33 ± 3	C(15) - C(16)	1.40 ± 4
N(2) - C(6)	1.36 ± 3	C(15) - C(20)	1.43 ± 4
C(1) - C(2)	1.47 ± 4	C(16) - C(17)	1.38 ± 4
C(2) - C(3)	1.42 ± 4	C(17) - C(18)	1.33 ± 4
C(3) - C(4)	1.36 ± 4	C(18) - C(19)	1.46 ± 5
C(4) - C(5)	1.43 ± 3	C(19) - C(20)	1.36 ± 4

TABLE 4.6

BOND ANGLES AND E.S.D.S IN DEGREES.

C(1)- N(1)- C(5)	116 ± 2	N(2) - C(6) - C(7)	114 ± 3
C(1)- N(1)- C(14)	122 ± 2	C(6) - C(7) - C(8)	114 ± 3
C(5)- N(1)- C(14)	122 ± 2	C(7) - C(8) - C(9)	108 ± 3
C(4)- N(2)- C(6)	128 ± 2	C(8) - C(9) - C(10)	108 ± 2
O(1)- C(1)- N(1)	115 ± 2	C(9) - C(10)- C(11)	107 ± 3
O(1)- C(1)- C(2)	128 ± 3	C(10)- C(11)- C(12)	109 ± 3
N(1)- C(1)- C(2)	117 ± 2	C(11)- C(12)- C(13)	108 ± 3
O(2)- C(2)- C(1)	111 ± 2	N(1) - C(14)- C(15)	113 ± 2
O(2)- C(2)- C(3)	124 ± 3	C(14)- C(15)- C(16)	122 ± 2
C(1)- C(2)- C(3)	125 ± 2	C(14)- C(15)- C(20)	120 ± 2
C(2)- C(3)- C(4)	117 ± 2	C(16)- C(15)- C(20)	119 ± 2
N(2)- C(4)- C(3)	125 ± 2	C(15)- C(16)- C(17)	122 ± 3
N(2)- C(4)- C(5)	114 ± 2	C(16)- C(17)- C(18)	120 ± 3
C(3)- C(4)- C(5)	120 ± 2	I(1) - C(18)- C(17)	124 ± 2
O(3)- C(5)- N(1)	113 ± 2	I(1) - C(18)- C(19)	115 ± 2
O(3)- C(5)- C(4)	122 ± 2	C(17)- C(18)- C(19)	121 ± 3
N(1)- C(5)- C(4)	125 ± 2	C(18)- C(19)- C(20)	120 ± 3
O(4)- C(6)- N(2)	123 ± 3	C(15)- C(20)- C(19)	120 ± 3
O(4)- C(6)- C(7)	122 ± 3		

TABLE 4.7

NON - BONDED DISTANCES.

(A) INTERMOLECULAR DISTANCES LESS THAN 3.5 Å

O(2).....O(3)	i	2.99 Å	O(3).....C(2)	iv	3.29 Å
O(3).....C(3)	ii	3.00	O(2).....N(2)	i	3.31
O(2).....N(1)	iii	3.01	O(3).....C(4)	ii	3.31
O(1).....C(7)	i	3.07	O(2).....C(14)	iii	3.37
O(1).....N(2)	i	3.08	O(4).....C(17)	i	3.40
O(3).....C(2)	ii	3.13	O(2).....C(5)	i	3.42
O(2).....C(1)	iii	3.17	O(3).....C(1)	iv	3.43
O(1).....O(3)	i	3.24	O(2).....O(3)	v	3.46
O(2).....C(5)	iii	3.29	O(1).....O(2)	vi	3.50

The Roman numerals refer to the following transformations applied to the coordinates found in Table 4.1 , the transformation is applied to the second atom of each contact.

i	x,	1/2 - y,	1/2 + z.
ii	-1/2 + x,	y,	1/2 - z.
iii	1/2 + x,	1/2 - y,	1 - z.
iv	x,	1/2 - y,	-1/2 + z.
v	1/2 + x,	y,	1/2 - z.
vi	-1/2 + x,	1/2 - y,	1 - z.

(B) SOME INTRAMOLECULAR DISTANCES.

O(1).....O(2)	2.70 Å	C(7).....C(9)	2.52 Å
O(1).....C(14)	2.72	C(8).....C(10)	2.45
O(3).....N(2)	2.64	C(9).....C(11)	2.49
O(3).....C(14)	2.69	C(10)....C(12)	2.60
O(4).....C(3)	2.81	C(11)....C(13)	2.55
C(6).....C(8)	2.59		

TABLE 4.8

MEAN MOLECULAR PLANES.

PLANE NO.	ATOMS DEFINING PLANE
1	N(1),C(1),C(2),C(3),C(4) and C(5)
2	N(2),O(4),C(6) and C(7)
3	N(2),C(2),C(3),C(4) and C(5)
4	C(15),C(16),C(17),C(18),C(19) and C(20)

PLANE EQUATIONS

PLANE NO.	P	Q	R	S	RMS D *
1	0.8298	-0.3558	-0.4299	-6.1869	0.032
2	0.8620	-0.4167	-0.2887	-6.3606	0.004
3	0.8233	-0.3877	-0.4145	-6.4208	0.056
4	-0.8212	-0.5682	-0.0517	-2.4413	0.019

* These symbols are defined in Table 1.10. The plane equation is

$$PX + QY + RZ = S$$

where X,Y and Z are in Å and refer to the crystal axes.

DEVIATIONS FROM MEAN PLANES (Å).

PLANE NO.	ATOM AND DISTANCE.					
1	N(1)	0.014	C(1)	-0.041	C(2)	0.053
	C(3)	-0.038	C(4)	0.011	C(5)	0.000
	O(1)	-0.217	O(2)	0.197	O(3)	0.067
	N(2)	0.156	C(14)	-0.088		
2	N(2)	0.002	O(4)	0.002	C(6)	-0.006
	C(7)	0.002	C(4)	0.041	C(8)	0.005
3	N(2)	0.061	C(2)	0.068	C(3)	-0.074
	C(4)	-0.039	C(5)	-0.016		
4	C(15)	-0.010	C(16)	0.027	C(17)	-0.017
	C(18)	-0.010	C(19)	0.026	C(20)	-0.017
	C(14)	-0.004	I(1)	-0.009		

DIHEDRAL ANGLES.

PLANE A	PLANE B	∠ AB	PLANE A	PLANE B	∠ AB
1	2	9.0°	2	3	7.7°
1	3	2.1	2	4	62.9
1	4	62.8	3	4	64.3

4.4 Discussion

The structure of the molecule, numbering scheme adopted for the analysis and the final three-dimensional electron density distribution, shown by superimposed contour sections drawn parallel to (100), are in Fig. 4.2 . The most striking feature in the molecule is the substituted heterocyclic ring, the chromophoric groups present undoubtedly causing the deep colour of the original pigment. The heterocyclic ring itself is not quite planar, indeed, as it is not truly aromatic this is not unexpected. The equation of the best plane through the six atoms comprising the ring is

$$0.8298\ X - 0.3558\ Y - 0.4299\ Z = -6.1869\ .$$

Although barely significant, deviations of the atoms in the plane from the plane (Table 4.8) indicate that the ring adopts a flattened chair conformation. This is also seen in the deviations of the five atoms bonded directly to the ring, the carbonyl oxygens O(1) and O(2) are -0.22 and 0.20 Å from the above plane while C(14),O(3) and N(2) are -0.09, 0.07 and 0.16 Å respectively from it. The intramolecular distances between these atoms (Table 4.7) show little or no evidence for neighbouring ortho substituent steric interaction, the deviations from the plane of the ring are then most likely caused by the adopted ring conformation.

Bond distances in the heterocyclic ring are consistent with its formulation as a triketo derivative, carbon to nitrogen bonds, C(1)-N(1), C(5)-N(1) and C(14)-N(1), approach the single bond value of 1.47 Å (Sutton, 1965) indicating no contribution of the type $\overset{-}{O} - \overset{|}{C} = \overset{|+}{N} -$ to the structure. Carbon to carbon bonds are characteristic of the hybridisation state involved, thus C(2)-C(3) and C(4)-C(5) are single bonds from α β unsaturated ketones and are close to the expected value of 1.44 Å (Sutton, 1965); C(1)-C(2) is a $C\ sp^2 - C\ sp^2$ single bond and its value of 1.47 Å compares with a similar bond of 1.48 Å in butadiene (Almenningen, Bastiansen and Traetteberg, 1958); C(3)-C(4) is a conjugated double bond and is in close agreement with the value of 1.36 Å quoted by Sutton (1965). The carbon to oxygen double bonds do not differ significantly from their mean value of 1.24 Å.

At the peptide group, 1.36 Å for the carbon to nitrogen bond, C(6)-N(2), is close to distances found in similar groups, Davies and Pasternak (1956) found 1.333 Å in succinamide and Mootz (1965) quotes 1.368 and 1.335 Å from DL-allantoin. The carbon to nitrogen bond, C(4)-N(2), is significantly shorter, at 1.33 Å , than the expected single bond value of 1.47 Å and must indicate a large degree of conjugation in the region involving the double bonds C(3)-C(4) and C(6)-O(4) . The angle between the planes specified by N(2),O(4),C(6),C(7) and N(2),C(2),C(3), C(4),C(5), is only 7.7° (Table 4.8 , equations 2 and 3) which would not interfere unduly with conjugation effects. An interesting feature of the system is the close contact of 2.81 Å between the carbonyl oxygen O(4) and the ethylenic CH group at C(3).

The C_7H_{15} side chain adopts a staggered zigzag conformation and small deviations from this are almost certainly caused by intermolecular packing forces. The $C\ sp^2 - C\ sp^3$ single bond is 1.51 Å and average $C\ sp^3 - C\ sp^3$ single bond 1.56 Å . I(1) and C(14) are coplanar with the six atoms of the phenyl ring which within experimental error are planar (Table 4.8 , equation 4). The mean carbon to carbon bond distance in the aromatic ring is 1.39 Å , and interbond angle 120° . The carbon to iodine bond of 2.10 Å does not differ significantly from the reported value of 2.05 Å (Sutton, 1965).

Packing in the crystal is shown in Fig. 4.3 and consists of infinite stacks of molecules in the a and c directions. The intermolecular distances (Table 4.7) show only three contacts which may be involved in hydrogen bonding. Carbonyl oxygen, O(1), gives a "bifurcated" interaction with the nitrogen atom N(2) and a methylene group at C(7) ; O(1)...N(2) and O(1)...C(7) are 3.08 and 3.07 Å respectively (hydrogens on C(7) are acidic because of the neighbouring carbonyl group). The other contact of 3.31 Å is from carbonyl oxygen, O(2), to the nitrogen atom N(2). In each of these cases the second atom is from a molecule at a position $x, 1/2 - y, 1/2 + z$. Apart from the above mentioned contacts all others are at normal van der Waals distances. The region involving most of these contacts, that is the heterocyclic ring, is shown enlarged in Fig. 4.4 and some contacts referred to in Table 4.7 are indicated.

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APPENDIX I

Appendix I

The programs in this Appendix are written in the computer language KDF 9 - ALGOL . The language is defined in a manual by Green (1964) and in the ALGOL Users Manual (1964).

DBX009800KP4 is the isotropic least squares program.

DBX021000KP4 is the anisotropic structure factor program.

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```
DBX0098OOKP4→
  begin
    library A0, A6, A7, A8, A9, A12, A13;
    integer i, j, k, n, p, q, f1, f2, f3, f4,
      params, ov params, norm elemt, ls params,
      block count, C, D, E, F, z, cycles, f5, f6, f7, f8;
    real twopi, factor, A part, B part, phi, Fcalc,
      sinphi, cosphi, root w, w, root w del, delta,
      M, Q, eightpisq, a1, a2, a3, a4, a5;
    boolean print, no u, sf only;
    array data[1:20], LS, LX[1:6];
    f1 := format([s-ndd;]);   f2 := format([ss-nddd.dd;]);
    f4 := format([s-nd.ddd;]);   f3 := format([ssndd;c]);
    twopi := 2 × 3.14159265;
    eightpisq := 2.0 × twopi ↑ 2;   z := 0;
    f5 := format([nddddd.dss]);
    f6 := format([s-nd.ddddd;]);   f7 := format([nddddss]);
    f8 := format([s-nd.ddddd;c]);   params := 0;
    open(10);   open(20);   open(30);   find(100,[20]);
    find(101,[DG030003]);   interchange(101);
    print := read(20) > 0.5;   read binary(100, LX, [S1]);
    read binary(100, LS, [LP]);   i := in basic symbol(20);
title:
    i := in basic symbol(20);   out basic symbol(30,i);
    if i ≠ 152 then goto title;
    data[2] := read(20);
    for i := 4 step 1 until 14 do data[i] := read(20);
    no u := data[5]> 1.5;   sf only := data[13]> 0.5;
   .C := LX[4];   D := data[2];   E := LS[2];
    F := data[4];   cycles := data[14];
    begin
      array R[1:12, 1:C], coord[1:D, 1:8], df[1:5],
        H,K,L,T[1:C], X[1:D,1:7], bat scale[1:F];
      boolean array  unrefined[1:D];
      begin
        array RT[1:C, 1:12];
        read binary (100, RT, [RT]);
        for i := 1 step 1 until C do
          for j := 1 step 1 until 12 do
            R[j,i] := RT[i,j] × twopi;
      end;
      for i := 1 step 1 until D do
      begin
        for j := 1 step 1 until 4 do
          coord[i,j] := read(20);
        coord[i,5] := read(20) × (- eightpisq);
        params := params + coord[i,3];
        unrefined[i] := coord[i,3] < 0.5;
```

```
        for j := 6 step 1 until 8 do
          coord[i,j] := read(20);
      end;
      begin
        integer array block[1:params + 1];
        block[1] := ls params := 4 × params + F;
        for i := 2 step 1 until params + 1 do
          block[i] := 4;
        norm elemt := ls params × (ls params + 3) ÷ 2;
        block count := 1;
        if not no u then
        begin
          block[1] := F + 1;   ls params := ls params + 1;
          block count := params + 1;   norm elemt := 0;
          for i := 1 step 1  until block count do
            norm elemt := norm elemt + block[i] ×
            (block[i] + 3) ÷ 2;
        end;
        for i := 1 step 1 until F do
          bat scale[i] := read(20);
        close(20);
        ov params := if no u then F else F + 1;
recycle:
        begin
          array in[1:6 + E], out[1:12 + E], A,
            B[1:D, 1:C], V[1:ls params], total[1:F,
            1:5], element[1:norm elemt];
          procedure out soln;
          begin
            for i := 1 step 1 until D do
            begin
              for j := 1 step 1 until 3 do
                write(30, f1, coord[i,j]);
              for j := 4 step 1 until 7 do
                write(30, f6, coord[i,j]);
              write(30, f8, coord[i,8]);
            end;
            for j := 1 step 1 until F do
              write(30, f8, bat scale[j]);
          end;
          if cycles > 1.5 then goto A1;
          if print then
            write text(30,[[2c4s]H[5s]K[5s]L[7s]
            F*OBS[5s]F*CALC[5s]A*PART[5s]B*PART[6s]
            DEL[6s]DEL*R*W[4s]ROOT*W[ss]BATCH[cc]]);
     A1:  for i := 1 step 1 until ls params do V[i] := 0.0;
          for i := 1 step 1 until norm elemt do
            element[i] := 0.0;
```

```
       for i := 1 step 1 until 5 do df[i] := 0.0;
       for i := 1 step 1 until F do
         for j := 1 step 1 until 5 do total[i,j] := 0.0;
       read binary(100, in, [PLS]);
loop:  if in[1] < -200 then goto EQNS;
       in[4] := in[4] × data[12];
       A part := B part := 0.0;
       for i := 1 step 1 until C do
       begin
         H[i] := R[1,i] × in[1] + R[5,i] × in[2]
           + R[9,i] × in[3];
         K[i] := R[2,i] × in[1] + R[6,i] × in[2]
           + R[10,i] × in[3];
         L[i] := R[3,i] × in[1] + R[7,i] × in[2]
           + R[11,i] × in[3];
         T[i] := R[4,i] × in[1] + R[8,i] × in[2]
           + R[12,i] × in[3];
       end;
       for i := 1 step 1 until D do
       begin
         factor := in[6 + coord[i,2]] ×
           exp(coord[i,5] × in[6]) × coord[i,4]
           × data[11];
         a1 := coord[i,6];   a2 := coord[i,7];
         a3 := coord[i,8];
         for j := 1 step 1 until C do
         begin
           phi := a1 × H[j] + a2 × K[j] + a3 ×
             L[j] + T[j];
           A[i,j] := a4 := cos(phi) × factor;
           B[i,j] := a5 := sin(phi) × factor;
           A part := A part + a4;
           B part := B part + a5;
         end;
       end;
       M := bat scale[in[5]];   p := in[5];
       Q := 1 / M;
       for k := 1 step 1 until 3 do out[k] := in[k];
       out[4] := in[4] × Q;   out[6] := A part;
       if data[11] > 1.5 then
         B part := out[7] := 0.0 else out[7] := B part;
       Fcalc := out[5] := sqrt(A part × A part +
         B part × B part);
       if Fcalc = 0.0 then
       begin
         cosphi := sinphi := 0.0;   goto jump;
       end;
       cosphi := A part/ Fcalc;
```

```
       sinphi := B part/ Fcalc;
jump:  if data[7] < 1.5 then
       begin
         w := 1.0/ (data[8] + in[4] + data[9] ×
           in[4] × in[4] + data[10] × in[4] ×
           in[4] × in[4]);
         root w := sqrt(w);
       end
         else if data[7] < 2.5 then
       begin
         w := 1.0/(1.0 + ((in[4] - data[9])/
           data[8]) ↑ 2);
         root w := sqrt(w);
       end
         else w := root w := 1;
       delta := in[4] - Fcalc × M;
       root w del := root w × delta;
       out[8] := delta × Q;
       if out[8] < 0.0 then
       begin
         out[9] := -out[6];   out[10] := -out[7];
       end
         else
       begin
         out[9] := out[6];   out[10] := out[7];
       end;
       for k := 1 step 1 until E + 2 do
         out[10 + k] := in[4 + k];
       total[p,1] := total[p,1] + out[4];
       total[p,2] := total[p,2] + Fcalc;
       total[p,3] := total[p,3] + abs(out[8]);
       total[p,4] := total[p,4] + w × delta × delta;
       total[p,5] := total[p,5] + 1;
       if cycles ≠ 1 then goto miss;
       write binary(101, out, [Structurefactors]);
       if print then
       begin
         for k := 1 step 1 until 3 do
           write(30,f1,out[k]);
         for k := 4 step 1 until 8 do
           write(30,f2,out[k]);
         write(30, f2, root w del × Q);
         write(30,f4,root w);   write(30, f3, p);
       end;
miss:  if sf only then goto only;
       phi := in[6] × eightpisq;   n := 0;
       for i := 1 step 1 until D do
       begin
```

```
            if unrefined[i] then goto not refined;
            for j := 1 step 1 until C do
            begin
              factor := B[i,j] × cosphi - A[i,j] × sinphi;
              df[1] := df[1] - factor × H[j];
              df[2] := df[2] - factor × K[j];
              df[3] := df[3] - factor × L[j];
              df[4] := df[4] - phi × (A[i,j] ×
                cosphi + B[i,j] × sinphi);
            end;
            for k := ov params + n do
              for q := 1 step 1 until 4 do
                V[k + q] := df[q];
            n := n + 4;
            for k := 1 step 1 until 5 do df[k] := 0.0;
not refined:
          end;
          if not no u then
          begin
            V[1] := V[1] - phi × Fcalc;
            V[p + 1] := V[p + 1] + Fcalc × Q;
          end
            else V[p] := V[p] + Fcalc × Q;
          M := M × root w;
          for i := 1 step 1 until ls params do
            V[i] := V[i] × M;
   only:  read binary(100, in, [PLS]);
          if sf only then goto loop;
          begin
            comment set up normal equations;
            k := 0;   q := 1;
            for j := 1 step 1 until block count do
            begin
              integer row, col, limit;
              limit := k + block[j];
              for i := k + 1 step 1 until limit do
              begin
                element[q] := element[q] + V[i] ×
                  root w del;
                q := q + 1;
              end;
              for row := k + 1 step 1 until limit do
                for col := row step 1 until limit do
              begin
                element[q] := element[q] + V[row] × V[col];
                q := q + 1;
              end;
              k := limit;
```

```
         end;
       end;
       for i := 1 step 1 until ls params do V[i] := 0.0;
       goto loop;
EQNS:  write text(30,[[4c]BATCH[4s]SIG*FO[5s]
         SIG*FC[5s]SIG*DEL[2s]SIG*W*DEL*SQ[s]
         PLANES[5s]R[2c]]);
       for k := 1 step 1 until F do
       begin
         write(30, f7, k);
         for j := 1 step 1 until 4 do
           write(30, f5, total[k,j]);
         write(30, f7, total[k,5]);
         write(30, f6, total[k,3] / total[k,1]);
         newline(30, 1);
       end;
       for i := 1 step 1 until 5 do
         for k := 1 step 1 until F do
           df[i] := df[i] + total[k,i];
       write text(30, [[c]TOTAL[2s]]);
       for k := 1 step 1 until 4 do
         write(30, f5, df[k]);
       write(30, f7, df[5]);
       write(30, format([nd.dddddc]), df[3] / df[1]);
       write text(00, [R*=*]);
       write(00, f6, df[3] / df[1]);
       if cycles = 1 then
       begin
         out[1] := -999;
         write binary(101, out, [Structurefactors]);
         data skip(100);   interchange(100);
         interchange(101);
       end
         else rewind(100);
       if sf only then goto end block;
       p := q := 0;
       for i := 1 step 1 until block count  do
       begin
         n := block[i];
         begin
           real  tot, diag;
           integer  l, j, k;
           array  rhs[1:n];
           real procedure  N(i,j);  value  i, j;
             integer  i,j;
             N := element[p + i × (2 × n - i +
               1) ÷ 2 + j];
           procedure  set N(i, j, x);
```

```
          value  i, j, x;  integer  i, j;  real  x;
          element[p + i × (2 × n -i + 1) ÷ 2
            + j] := x;
        procedure  exchange;
        begin
          procedure change Ns(a, b, c, d);
            value  a, b, c, d;  integer  a, b, c, d;
          begin
            real hold;
            hold := N(a, b);   set N(a, b,N(c, d));
            set N(c, d, hold);
          end;
          k := N(l- 1, l);
          if k≠l then
          begin
            for j:= l step 1 until k-1 do
              change Ns(l,j,j,k);
            for j:= k step 1 until n do
              change Ns(l,j,k,j);
            change Ns(l,l,l,k);
          end;
        end;
        comment store rhs;
        for l := 1 step 1 until n do
          rhs[l] := element[p + l];
invert: for l := 1 step 1 until n do
        begin
          set N(l - 1, l, l);   diag := 0;
          for j := l step 1 until n do
            if abs(N(j, j)) > diag then
          begin
            diag := abs(N(j, j));   set N(l-1,l, j);
          end;
          exchange;
          if diag < 0.000001 then
          begin
            write text(30, [[c]PARAMETER*DROPPED]);
            set N(l, l, 1);
            for j := l + 1 step 1 until n do
            begin
              set N(l, j, 0);   set N(l - 1, j, 0);
            end;
            j := l;
            for k := l - 1 step -1 until 1 do
            begin
              set N(k - 1, j, 0);
              if j = N(k - 1, k) then j := k;
            end;
```

```
                  rhs[j] := 0;   z := z + 1;
                  goto next row;
                end;
                set N(l, l, 1/N(l, l));
                for j := l + 1 step 1 until n do
                  set N(l - 1,j, -N(l, l) × N(l, j));
                for j := l + 1 step 1 until n do
                  for k := j step 1 until n do
                    set N(j,k,N(j,k) + N(l-1,k) × N(l,j));
     next row:
              end;
    restore:  for l := n - 1 step -1 until 1 do
              begin
                for j := l + 1 step 1 until n do
                  set N(l, j, 0);
                for j := l + 1 step 1 until n do
                begin
                  for k := j step 1 until n do
                  begin
                    set N(l,k,N(l,k) + N(l - 1,j) ×
                      N(j,k));
                    if j ≠ k then
                      set N(l,j,N(l,j) + N(l - 1,k)
                      × N(j,k));
                  end;
                  set N(l,l, N(l,l) + N(l - 1,j) × N(l,j));
                end;
                exchange;
              end;
      solve:  for l := 1 step 1 until n do
              begin
                tot := 0;
                for j := 1 step 1 until l - 1 do
                  tot := tot + N(j,l) × rhs[j];
                for j := l step 1 until n do
                  tot := tot + N(l,j) × rhs[j];
                V[q + l] := tot;
              end;
              p := p + n × (n + 3) ÷ 2;   q := q + n;
            end;
          end;
end block:
          if sf only then goto nearly;
          for j := 1 step 1 until ls params do
            V[j] := V[j] × data[6];
          k := 0;
          if not no u then k := 1;
          for n := 1 step 1 until F do
```

```
           bat scale[n] := bat scale[n] + V[k + n];
         k := k + F;
         for j := 1 step 1 until D do
         begin
           coord[j, 5] := coord[j, 5] / (- eightpisq);
           if unrefined[j] then goto removed;
           begin
             for n := 1 step 1 until 3 do
               coord[j, 5 + n] := coord[j, 5 + n]
               + V[k + n];
             coord[j, 5] := coord[j, 5] + V[k + 4];
             k := k + 4;
           end;
 removed:
         end;
         write text(30,[[cc]NEW*PARAMETERS[cc]]);
         gap(10, 100);
         for i := 1 step 1 until D do
         begin
           for j := 1 step 1 until 3 do
             write(10, f1, coord[i,j]);
           for j := 4 step 1 until 7 do
             write(10, f6, coord[i,j]);
           write(10, f8, coord[i,8]);
         end;
         for j := 1 step 1 until F do
           write(10, f8, bat scale[j]);
         char out(10, 61);   gap(10, 100);   out soln;
         cycles := cycles - 1;
         if cycles ≠ 0 then
         begin
           for i := 1 step 1 until D do
             coord[i, 5] := coord[i, 5] × (- eightpisq);
           goto recycle;
         end;
nearly:  for i := 1 step 1 until D do
         begin
           X[i, 1] := coord[i, 2];
           X[i, 2] := coord[i, 1];
           for j := 3, 4, 5 do X[i, j] := coord[i, 3 + j];
           X[i, 6] := 0.0;   X[i, 7] := coord[i, 5];
         end;
         LX[1] := data[2];   LX[2] := 7;
         write binary(100, LX, [LX]);
         write binary(100, X, [X]);
         if sf only then goto nearly there;
         write text (30, [[cc]SHIFTS[cc]]);   k := 0;
         if not no u then k := 1;
```

```
         for n := 1 step 1 until F do
           bat scale[n] := V[k + n];
         k := k + F;
         for j := 1 step 1 until D do
         begin
           coord[j, 4] := 0.0;
           if unrefined[j] then
           begin
             for i := 5 step 1 until 8 do
               coord[j,i] := 0.0;
             goto continue;
           end;
           for n := 1 step 1 until 3 do
             coord[j, 5 + n] := V[k + n];
           coord[j, 5] := V[k + 4];   k := k + 4;
continue:
         end;
         out soln;
         write text(30,[[cc]STANDARD*DEVIATIONS[cc]]);
         begin
           integer count, c, b;
           count := 0;   c := 1;
           for j := 1 step 1 until block count do
           begin
             b := block[j];   c := c + b;
             for i := 1 step 1 until b do
             begin
               count := count + 1;
               V[count] := if element[c] <
                 0.0000001 then 0.0 else
                 sqrt(element[c]);
               c := c + (b - i + 1);
             end;
           end;
         end;
         factor := sqrt(df[4] / (df[5] - ls params + z));
         for n := 1 step 1 until ls params do
           V[n] := V[n] × factor;
         k := 0;
         if not no u then k := 1;
         for n := 1 step 1 until F do
           bat scale[n] := V[k + n];
         k := k + F;
         for j := 1 step 1 until D do
         begin
           if unrefined[j] then goto released;
           for n := 1 step 1 until 3 do
             coord[j, 5 + n] := V[k + n];
```

```
            coord[j, 5] := V[k + 4];   k := k + 4;
 released:
          end;
          out soln;
          for i := 1 step 1 until D do
            for j := 3, 4, 5 do X[i, j] := coord[i, 3 + j];
          write binary(100, X, [ESD]);
nearly there:
          LS[1] := 12 + E;   LS[2] := E;
          write binary(100, LS, [LS]);
  round:  read binary(101, out, [Structurefactors]);
          if out[1] < -256 then goto end at last;
          write binary(100, out, [SFS]);   goto round;
end at last:
          write binary(100, out, [SFS]);
        end;
      end;
    end;
    close(100);   close(101);   close(10);   close(30);
  end→
```

```
DBX021000KP4→
  begin
    library A0, A6, A7, A8, A9, A12, A13;
    integer nats, nf, centre, n, a, b, i, j, fa,
      fb, fc, fd, fe, ff, fg, fh, fi, count, page;
    real f1, f2, f3, f4, f5, f6, pi, factor, A, B, phi, E;
    boolean sfout, cen;
    real array unit cell, S1, LP[1: 6];
    open(20);   open(30);   find(100, [20]);
    find(101, [DG030003]);   interchange(101);   page := 1;
    fg := format([ndd]);   write text(30, [[pcc98s]PAGE]);
    write(30,fg, page);   page := page + 1;
    sfout := read(20) > 0.5;   i := in basic symbol(20);
    newline(30, 1);
title:
    i := in basic symbol(20);
    if i = 152 then goto on;
    out basic symbol(30, i);   goto title;
on:
    newline(30, 4);   nats := read(20);   nf := read(20);
    read binary(100, unit cell, [unitcell]);
    read binary(100, S1, [S1]);
    read binary (100, LP, [LP]);   skip(100, -2);
    centre := S1[1];   n := S1[4];   a := LP[1];
    b := 12 + LP[2];   pi := 3.14159265;   count := 0;
    cen := centre > 1.5;   factor := - 8 × pi ↑ 2;
    begin
      real array PLS[1:a], RT[1:n, 1:12],
        scales[1:nf], coord[1:nats, 1:13],
        SFS[1:b], total[1:4, 1:nf], H, K, L, T, HH,
        KK, LL, HK, KL, LH[1:n];
      for i := 1 step 1 until nf do scales[i] := read(20);
      for i := 1 step 1 until nats do
      begin
        for j := 1 step 1 until 6 do
          coord[i,j] := read(20);
        if coord[i, 6] > 0.5 then
        begin
          for j := 7 step 1 until 13 do
            coord[i,j] := read(20);
        end
          else
        begin
          coord[i, 7] := read(20) × factor;
          coord[i, 13] := read(20);
        end;
      end;
      read binary(100, RT, [RT]);   skip(100, 1);
```

```
for i := 1 step 1 until 4 do
  for j := 1 step 1 until nf do total[i,j] := 0.0;
begin
  real V, al, be, ga, cosal, cosbe, cosga,
    sinal, sinbe, singa;
  al := unit cell[4] × pi / 180;
  be := unit cell[5] × pi / 180;
  ga := unit cell[6] × pi / 180;   cosal := cos(al);
  cosbe := cos(be);   cosga := cos(ga);
  sinal := sin(al);   sinbe := sin(be);
  singa := sin(ga);
  V := sqrt(1 + 2 × cosal × cosbe × cosga -
    cosal ↑ 2 - cosbe ↑ 2 - cosga ↑ 2);
  f1 := (((sinal)/(V × unit cell[1])) ↑ 2) / 2;
  f2 := (((sinbe)/(V × unit cell[2])) ↑ 2) / 2;
  f3 := (((singa)/(V × unit cell[3])) ↑ 2) / 2;
  f4 := ((sinal)/(V × unit cell[1])) ×
    ((sinbe)/(V × unit cell[2])) / 2;
  f5 := ((sinbe)/(V × unit cell[2])) ×
    ((singa)/(V × unit cell[3])) / 2;
  f6 := ((singa)/(V × unit cell[3])) ×
    ((sinal)/(V × unit cell[1])) / 2;
end;
fa := format([s-nd]);   fb := format([s-ndd.dd]);
fc := format([s-ndc]);   fh := format([ndd.ddd]);
fd := format([nddddd.dss]);
fe := format([nddddss]);
ff := format([-nd.ddddc]);
fi := format([s-nd.ddddd]);
write text(30,[[2s]UNIT*CELL*DATA[4c2s]
  A**IN*ANGSTROM*UNITS[10s]]);
write(30, fh, unit cell[1]);
write text(30, [[2c2s]B **IN*ANGSTROM*UNITS[10s]]);
write(30, fh, unit cell[2]);
write text(30, [[2c2s]C**IN*ANGSTROM*UNITS[10s]]);
write(30, fh, unit cell[3]);
write text(30, [[2c2s]ALPHA*IN*DEGREES[14s]]);
write(30, fh, unit cell[4]);
write text(30, [[2c2s]BETA**IN*DEGREES[14s]]);
write(30, fh, unit cell[5]);
write text(30, [[2c2s]GAMMA*IN*DEGREES[14s]]);
write(30, fh, unit cell[6]);
write text(30, [[6c2s]LAUE*GROUP[20s]]);
write(30, fa, S1[3]);
write text(30,[[2c2s]LATTICE*TYPE*GLASGOW*SFLS[5s]]);
write(30, fa, S1[2]);
write text(30,[[pcc98s]PAGE]);   write(30, fg, page);
page := page + 1;   newline(30, 1);
```

```
write text(30,[[ss]SYMMETRY*DATA[2c2s]]);
if S1[1] > 1.5 then
  write text(30, [A*CENTRE*OF*SYMMETRY*IS*
  PRESENT[2c2s]]) else
  write text(30,[NO*CENTRE*OF*SYMMETRY*
  PRESENT[2c2s]]);
write text(30,[SUMMATION*DONE*OVER]);
write(30, fa, S1[4]);
write text(30, [[2s]R*AND*T*MATRICES[2c]]);
for i := 1 step 1 until n do
begin
  for j := 1 step 1 until 3 do
  begin
    write(30, fa, RT[i,j]);   space(30, 2);
  end;
  write(30, ff, RT[i,4]);   newline(30, 1);
  for j := 5 step 1 until 7 do
  begin
    write(30, fa, RT[i,j]);   space(30, 2);
  end;
  write(30, ff, RT[i,8]);   newline(30,1);
  for j := 9 step 1 until 11 do
  begin
    write(30, fa, RT[i,j]);   space(30, 2);
  end;
  write(30, ff, RT[i,12]);   newline(30, 3);
  count := count + 1;
  if count = 6 then
  begin
    write text(30, [[pcc98s]PAGE]);
    write(30, fg, page);   page := page + 1;
    newline(30, 4);   count := 0;
  end;
end;
write text(30, [[pcc98s]PAGE]);
write(30, fg, page);   page := page + 1;
count := 0;
write text(30, [[4c2s]ATOMIC*DATA[4c2s]THERE*ARE*]);
write(30, fg, LP[2]);
write text(30, [[2s]FORMFACTORS*LOADED[2c2s]]);
write text(30, [SUMMATION*DONE*ON*]);
write(30, fg, nats);
write text(30, [[2s]ATOMS[2c2s]
  ATOM*TYPE**X/A[7s]Y/B[7s]Z/C[4s]KEY[3s]
  U11/UISO[2s]U22[7s]U33[7s]2U23[6s]2U31[6s]
  2U12[5s]OCC*NO[cc]]);
for i := 1 step 1 until nats do
begin
```

```
    for j := 1, 2 do write(30, fa, coord[i,j]);
    for j := 3, 4, 5 do write(30, fi, coord[i,j]);
    for j := 6 do write(30, fa, coord[i,j]);
    if coord[i,j] > 0.5 then
    begin
     for j := 7 step 1 until 13 do
      write(30, fi, coord[i,j]);
    end
     else
    begin
     write(30, fi, coord[i, 7] / factor);
     space(30, 50);   write(30, fi, coord[i, 13]);
    end;
    newline(30, 1);   count := count + 1;
    if count = 35  then
    begin
     write text(30, [[pcc98s]PAGE]);
     write(30, fg, page);   page := page + 1;
     count := 0;
     write text(30, [[4c2s]ATOMIC*DATA*
      CONTINUED[4c2s]ATOM*TYPE**X/A[7s]
      Y/B[7s]Z/C[4s]KEY[3s]U11/UISO[2s]
      U22[7s]U33[7s]2U23[6s]2U31[6s]2U12[5s]
      OCC*NO[cc]]);
    end;
   end;
   write text(30, [[pcc98s]PAGE]);   write(30,fg, page);
   page := page + 1;
   if sfout then
    write text(30, [[cc3s]H[3s]K[3s]L[2s]
    F*OBS[3s]F*CALC[2s]A*PART[2s]B*PART[2s]
    DELTA[2s]BATCH[cc]]);
   for i := 1 step 1 until n do
    for j := 1 step 1 until 12 do
     RT[i,j] := RT[i,j] × pi × 2;
   read binary(100, PLS, [PLS]);   count := 0;
loop:
   if abs(PLS[1]) > 200 then goto endsum;
   a := PLS[5];   A := B := 0.0;
   for i := 1 step 1 until 3 do SFS[i] := PLS[i];
   SFS[4] := PLS[4] × scales[a];
   for i := 1 step 1 until n do
   begin
    H[i] := RT[i, 1] × PLS[1] + RT[i, 5] ×
     PLS[2] + RT[i,  9] × PLS[3];
    K[i] := RT[i, 2] × PLS[1] + RT[i, 6] ×
     PLS[2] + RT[i, 10] × PLS[3];
    L[i] := RT[i, 3] × PLS[1] + RT[i, 7] ×
```

```
       PLS[2] + RT[i, 11] × PLS[3];
     T[i] := RT[i, 4] × PLS[1] + RT[i, 8] ×
       PLS[2] + RT[i, 12] × PLS[3];
     HH[i] := H[i] × H[i] × f1;
     KK[i] := K[i] × K[i] × f2;
     LL[i] := L[i] × L[i] × f3;
     HK[i] := H[i] × K[i] × f4;
     KL[i] := K[i] × L[i] × f5;
     LH[i] := L[i] × H[i] × f6;
   end;
   for i := 1 step 1 until nats do
   begin
     factor := PLS[6 + coord[i,2]] × coord[i,
       13] × centre;
     if coord[i, 6] < 0.5 then
       factor := factor × exp(coord[i, 7] × PLS[6]);
     for j := 1 step 1 until n do
     begin
       phi := coord[i, 3] × H[j] + coord[i, 4] ×
         K[j] + coord[i, 5] × L[j] + T[j];
       if coord[i, 6] < 0.5 then goto iso;
       E := exp(-(HH[j] × coord[i, 7] + KK[j] ×
         coord[i, 8] + LL[j] × coord[i, 9] +
         KL[j] × coord[i, 10] + LH[j] × coord[i,
         11] + HK[j] × coord[i, 12]));
       A := A + factor × E × cos(phi);
       B := B + factor × E × sin(phi);   goto jump;
 iso:  A := A + factor × cos(phi);
       B := B + factor × sin(phi);
jump:
     end;
   end;
   for i := 1 step 1 until 2 + LP[2] do
     SFS[10 + i] := PLS[4 + i];
   read binary(100, PLS, [PLS]);   SFS[6] := A;
   if cen then SFS[7] := B := 0.0 else SFS[7] := B;
   SFS[5] := sqrt(A × A + B × B);
   SFS[8] := SFS[4] - SFS[5];
   if sfout then
   begin
     for i := 1 step 1 until 3 do write(30, fa, SFS[i]);
     for i := 4 step 1 until 8 do write(30, fb, SFS[i]);
     write(30, fc, SFS[11]);   count := count + 1;
     if count = 50 then
     begin
       write text(30, [[pcc98s]PAGE]);
       write(30, fg, page);   page := page + 1;
       write text(30, [[cc3s]H[3s]K[3s]L[2s]
```

```
      F*OBS[3s]F*CALC[2s]A*PART[2s]B*PART[2s]
      DELTA[2s]BATCH[cc]]);
     count := 0;
    end;
   end;
   if SFS[8] < 0.0 then
   begin
    SFS[9] := - SFS[6];   SFS[10] := - SFS[7];
   end
    else
   begin
    SFS[9] := SFS[6];   SFS[10] := SFS[7];
   end;
   total[1,a] := total[1,a] + SFS[4];
   total[2,a] := total[2,a] + SFS[5];
   total[3,a] := total[3,a] + abs(SFS[8]);
   total[4,a] := total[4,a] + 1;
   write binary(101, SFS, [SFS]);   goto loop;
endsum:
   SFS[1] := PLS[1];   write binary(101, SFS, [SFS]);
   interchange(101);   dataskip(100);
   interchange(100);
   begin
    real array LX[1:6];
    real hold;
    LX[1] := nats;   LX[2] := 13;
    write binary(100, LX, [LX]);
    for i := 1 step 1 until nats do
    begin
     hold := coord[i, 1];
     coord[i, 1] := coord[i, 2];
     coord[i, 2] := hold;
    end;
    write binary(100, coord, [X]);
    LP[1] := 12 + LP[2];   write binary(100, LP, [LS]);
   end;
   if sfout then
   begin
    write text(30, [[pcc98s]PAGE]);
    write(30, fg, page);
   end;
   write text(30, [[2c2s]RESULTS[4cs]
    BATCH**SUM*F*OBS**SUM*FCALC**SUM*DELTA**
    PLANES[4s]R[cc]]);
   for i := 1 step 1 until nf do
   begin
    write(30, fe, i);
    for j := 1 step 1 until 3 do
```

```
          write(30, fd, total[j,i]);
        write(30, fe, total[4, i]);
        write(30, ff, total[3, i] / total[1, i]);
      end;
      if nf > 1.5 then
      begin
        real array sub[1:4];
        for i := 1 step 1 until 4 do sub[i] := 0.0;
        for i := 1 step 1 until 4 do
          for j := 1 step 1 until nf do
            sub[i] := sub[i] + total[i, j];
        write text(30,[[cs]TOTAL[s]]);
        for i := 1 step 1 until 3 do write(30, fd, sub[i]);
        write(30, fe, sub[4]);
        write(30, ff, sub[3] / sub[1]);
      end;
      write text(30, [[2p]]);
round:
      read binary(101, SFS, [SFS]);
      if abs(SFS[1]) > 200 then goto finished;
      write binary(100, SFS, [SFS]);   goto round;
finished:
      write binary(100, SFS, [SFS]);
    end;
    close(100);   close(101);   close(20);   close(30);
  end→
```

APPENDIX II

Appendix II

The Linear Diffractometer

The Hilger and Watts Y190 linear diffractometer is a device for the automatic collection of integrated intensities of diffracted X-ray beams. It consists of a stabilised X-ray generator, counter equipment, a mechanical analogue of the reciprocal lattice for setting the crystal and the counter, and an output printer and tape punch. Mo radiation, combined with balanced $SrO - ZrO_2$ Ross filters, is normally employed and a scintillation counter, in conjunction with a pulse height analyser, that is an analyser which accepts only pulses within a given energy range, is used as a detector.

The mechanical analogue of the reciprocal lattice consists of three slides to which the motions of the crystal and counter are linked. The crystal is set so that each of its reciprocal axes is parallel to one of the slides. By moving the slides the crystal is rotated so that any crystal plane can be brought into the Bragg reflection condition while at the same time the counter is positioned so as to measure the diffracted beam. In the normal mode the diffractometer measures each reflection in a given reciprocal lattice net by automatic scanning on two slides, the upper layers are brought into the reflection condition by manual setting in the equi-inclination Weissenberg manner.

The integrated intensity is determined by first rotating the crystal by a small amount out of the reflection condition and counting the background for t seconds, the crystal is then rotated at a constant speed for 2t seconds through the reflection condition and finally the background is counted again for t seconds. The intensity is then the difference between the second count and the sum of the first and third. The counting cycle is repeated at least once for each filter and the final intensity is that of the Zirconium count less that of the Strontium.