X-RAY STRUCTURAL ANALYSIS OF AFLATOXIN G_1, ISOCOLUMBIN, AND OTHER COMPLEX MOLECULES.

A thesis
submitted to the University of Glasgow
for the degree of Doctor of Philosophy
in the Faculty of Science
by
Kung Kai Cheung.

Chemistry Department. September, 1965.

In memory
of
my beloved father.

PREFACE

I wish to thank Professor G.A. Sim, and Professor J. Monteath Robertson, F.R.S., for their instructive supervision throughout the period of my research.

I am grateful to Professor D.W.J. Cruickshank, Drs. J.S. Rollett, J.G. Sime, W.E. Oberhansli, Messrs. J.G.F. Smith, K.W. Muir, W.S. McDonald, D.R. McGregor, and Miss A. Kerr for the use of their computing programmes.

The award to me of a Li Po Chun Studentship (1962-64) from Hong Kong, a travel grant from the British Council, and the appointment of an Assistantship from the Glasgow University are all gratefully acknowledged.

SUMMARY

This thesis contains an account of the work on the crystal structure analysis of three natural product derivatives and a coordination complex by x-ray diffraction methods.

Part I includes outlines of the methods involved in the course of the work.

The elucidation of the structure of the groundnut toxin, aflatoxin G_1, in the form of its three isomorphous solvates is described in Part II. Little was known about the chemical constitution of this highly toxic metabolite when this work was begun. But just before the crystal structure was solved by the method of isomorphous replacement, there were two different proposals of its structure, both on the basis of chemical and spectroscopic investigations. The present result agrees with one of these proposed structures.

All three isomorphous solvates have been refined to the same extent. Since there are two molecules in an asymmetric unit, there are six measures of each bond length and angle so that the average values should give an accurate picture of the molecule.

Part III gives an account of the structure and stereochemistry of isocolumbin in the form of its iodo-derivative, established by the heavy atom method. The result thus obtained agrees with the conclusion drawn from chemical and optical rotatory dispersion studies.

The absolute configuration around the sulphoxide grouping of a naturally occurring sulphoxide mustard oil is described in Part IV. The result is useful in that it is also valid for other members of a homologous series of sulphoxide mustard oils. The structure was also solved by the heavy atom method and refined by the Fourier and least squares methods.

Part V describes the structure of the bis(mercuric chloride) adduct of 1,6-dithiacyclodeca-3,8-diene. Instead of both mercury atoms each having a three-fold coordination, one mercury atom is almost linearly coordinated to two chlorine atoms while the other mercury atom is tetrahedrally coordinated to two chlorine and two sulphur atoms.

TABLE OF CONTENTS

PART I

Some Methods of X-ray Crystal Structure Analysis

PART II

The Crystal Structure of Aflatoxin G_1

PART III

The Stereochemistry of Isocolumbin:

X-ray Analysis of 1-p-iodophenyl,3-phenyl Pyrazoline Adduct of Isocolumbin Monoacetone Solvate.

PART IV

The Structure and Absolute Configuration of N-(α-phenylethyl) N'-(ω-methylsulphinylpropyl) Thiourea.

PART V

The Structure of 1,6-dithiacyclodeca-cis-3,cis-8-diene Bis(mercuric chloride).

PART I

Some Methods of X-ray Crystal Structure Analysis.

1.1. INTRODUCTION

The study of the structure of crystals by the method of x-ray diffraction has developed rapidly since the discovery in 1912 by Von Laue of the diffraction effects when x-rays were passed through a crystal.

A vast number of crystal structures from the very simple in the case of diamond to the highly complex structures of the proteins, haemoglobin and myoglobin has so far been determined.

The procedures of a structure determination employed in this thesis involved (a) recording the diffractions of the x-radiations by the crystal photographically and estimating their intensities visually; (b) determining the structure either by the heavy atom method or by the isomorphous replacement method; and (c) refining the parameters of the atoms in the molecule by the Fourier and least squares methods.

Brief accounts of the methods involved are given in the following sections.

1.2. DIFFRACTION BY A CRYSTAL.

A crystal consists of a large number of repetitions in three-dimensions of a basic pattern of atoms. This basic pattern can be enclosed by a parallelopiped formed by three non-planar vectors. This parallelopiped is called a unit cell. In order to provide the lattice with means to scatter radiation, it is assumed that an electron is situated at each lattice point. The position of these electrons can be specified by a set of vectors $\underline{r}$, such that

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

where $\underline{a}$, $\underline{b}$, and $\underline{c}$ are the vectors defining the unit cell of the space lattice, and u, v, and w are integers.

In Fig. 1-1, A_1 and A_2 are two lattice points in a parallel beam of x-rays of wave-length λ. The direction of radiation is noted by the vector $\underline{s}_0$, with magnitude $1/\lambda$. The scattered wave is denoted by the vector $\underline{s}$, also with magnitude as $\underline{s}_0$. The path difference between the scattered waves from the two lattice points is given by

$$A_1N - A_2M = (\underline{r}.\underline{s} - \underline{r}.\underline{s}_0) = \underline{r}.\underline{S},$$

where $\underline{S} = \underline{s} - \underline{s}_0$

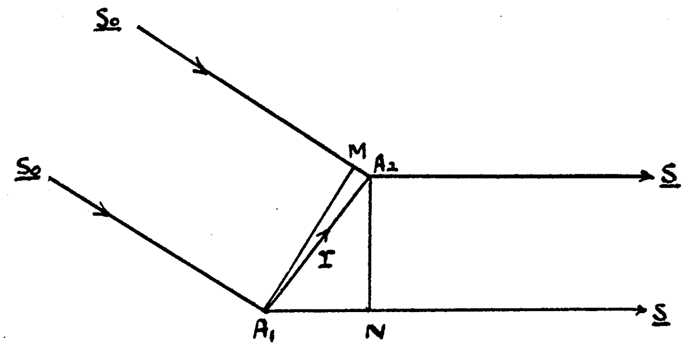


Fig. 1-1.

The condition for the waves to be in phase is that the path difference should equal an integral number of wave-lengths.

$$\text{i.e. } (u\underline{a} + v\underline{b} + w\underline{c}).\underline{S} = \text{integer}.$$

Since u, v, and w are integers, any change in them is also integral, therefore each of the products separately must be integral;

hence
$$\underline{a}.\underline{S} = h,\qquad \underline{b}.\underline{S} = k,\qquad \underline{c}.\underline{S} = \ell, \qquad \ldots (1.1)$$

where h, k, and l are integers. These are conditions for a diffracted beam to be of maximum intensity. They are known as the Laue's equations.

Rewriting Laue's equations in the form

$$\frac{a}{h}\,.\underline{S} = 1, \frac{b}{k}\,.\underline{S} = 1, \frac{c}{\ell}\,.\underline{S} = 1,$$

it can be seen that $\underline{S}$ is perpendicular to the plane defined by the intercepts $\underline{a}/h$, $\underline{b}/k$, and $\underline{c}/\ell$, i.e. the plane with Miller indices h k ℓ, as illustrated in Fig. 1-2(a).

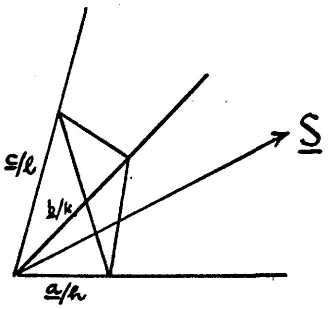


Fig. 1-2(a)

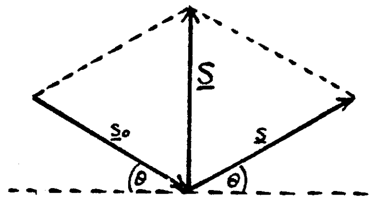


Fig. 1-2(b)

$|s_0| = 1/\lambda$

$|S| = 2\,|s_0|\sin\theta = 2\sin\theta/\lambda$

Since $\underline{S}$ bisects the incident and diffracted beams. The spacing d (h k ℓ) between planes h k ℓ is the perpendicular distance of the plane from the origin, and is equal to the projection of $\underline{a}/h$, $\underline{b}/k$, or $\underline{c}/\ell$ on the vector $\underline{S}$;

$$\text{i.e.}\qquad d = \frac{\underline{a}/h\ .\underline{S}}{|S|}$$

Substituting the values of $\underline{a}/h\ .\underline{S}$ and $|S|$ from Fig. 1-2(b) we have

$$\lambda = 2\,d \sin\theta \qquad \ldots (1.2)$$

This relation, known as Bragg's Law, gives the angle of incidence of the x-ray beams for a reflection from a lattice point to take place.

1.3. THE ATOMIC SCATTERING FACTOR, f.

The scattering power of an atom is expressed in terms of the scattering for a single, free electron. The maximum scattering by an atom is equal to its atomic number, Z, and this occurs at $\sin\theta/\lambda = 0$. Owing to the destructive interference between the wavelets scattered by these electrons, which are distributed throughout the volume of the atom, f will fall off as $\sin\theta/\lambda$ increases.

Using a function giving the distribution of electrons in an atom put forward by Hartree, the atomic scattering factors for most atoms have been calculated and listed in Volume III of the International Tables For X-Ray Crystallography, from where all the atomic scattering factors of the atoms carbon, oxygen, nitrogen, sulphur, chlorine, bromine, iodine, and mercury employed in the work described in this thesis are obtained.

1.4. THERMAL VIBRATION.

The atomic scattering factors have been calculated on the assumption that the atoms are at rest. In practice, atoms have thermal vibrations and the effect of this vibration is to smear the electron distribution and so decrease the intensities of the wavelets scattered.

If fo is the scattering factor calculated for atom at rest, then the temperature corrected factor f is given by

$$f = f_o e^{-B(\sin \theta/\lambda)^2} \qquad \ldots (1.3)$$

the value of B is shown by Debye to be $8\pi\bar{u}^2$ where $\bar{u}^2$ is the mean square amplitude of vibration of the atom about its mean position. B is called the Debye factor or commonly, the temperature factor.

In general, the thermal vibration of an atom is not isotropic but varies with direction. Cruickshank (1956) describes the anisotropic thermal vibration in terms of a symmetric tensor with six independent components Uij. The Uij's are defined with respect to the reciprocal axes <u>a</u>*, <u>b</u>*, <u>c</u>*. At a reciprocal lattice point <u>S</u> = (ha*, kb*, ℓc*), the temperature factor is represented by

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{23}k\ell b^*c^* + 2U_{31}\ell hc^*a^* + 2U_{12}hka^*b^*)\right] \qquad \ldots (1.4)$$

so that, for instance, U_{11} is the mean square amplitude of vibration of the atom parallel to the reciprocal axis a*.

1.5. THE STRUCTURE FACTOR, F.

The wave scattered in each reflection h k ℓ is characterized by the complex quantity F(h k ℓ) called the structure factor. It has a magnitude $|F(h\ k\ \ell)|$ and a phase $\alpha(h\ k\ \ell)$. $|F(h\ k\ \ell)|$ is directly related to the intensity of the reflection but

$\alpha(h\,k\,\ell)$ cannot be recorded directly.

If the atomic positions in a structure are known, it is possible to calculate a set of structure factors.

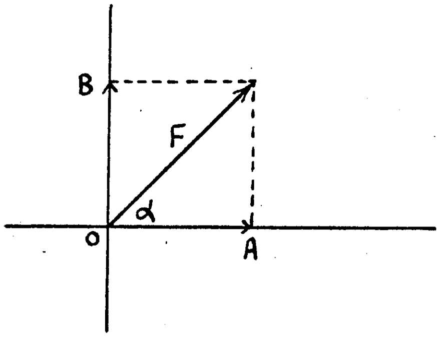
Let the coordinates of the jth atom be (x_j, y_j, z_j); the phase ϕ_j for each reflection $h\,k\,\ell$ to which this atom contributes is

$$\phi_j = 2\pi(hx_j + ky_j + \ell z_j),$$

and the structure-factor contribution due to this atom is equal to $f_j \exp 2\pi i\,(hx_j + ky_j + \ell z_j)$, where f_j is the atomic scattering factor.

The expression for the complete wave scattered by all N atoms in the unit cell is

$$F(hk\ell) = \sum_{j=1}^{N} f_j \exp 2\pi i(hx_j + ky_j + \ell z_j) \quad \ldots (1.5)$$



The complex quantity $F(h\,k\,\ell)$ can be resolved into its real and imaginary components and written as

$$F(hk\ell) = A(hk\ell) + iB(hk\ell) \quad \ldots(1.6)$$

such that

$$A(hk\ell) = \sum f_j \cos 2\pi(hx_j + ky_j + \ell z_j)$$

and

$$B(hk\ell) = \sum f_j \sin 2\pi(hx_j + ky_j + \ell z_j) \quad \ldots(1.7)$$

and $$\tan\alpha(hk\ell) = \frac{B(hk\ell)}{A(hk\ell)} \quad \ldots (1.8)$$

The Structure factor equation can also be expressed in terms of the electron density $\rho(x\,y\,z)$ at a point (x,y,z). The amount of scattering matter in the volume element $Vd\,x\,dydz$ is $\rho(x\,y\,z)\,Vd\,x\,dydz$, and

$$F(hk\ell) = V \int_0^1\int_0^1\int_0^1 \rho(xyz) \exp 2\pi i \,(hx + ky + \ell z)dx\, dy\, dz \quad \ldots (1.9)$$

1.6. THE FOURIER SERIES.

Since a crystal is periodic in space, its electron density can be represented by a three-dimensional Fourier series.

The electron density $\rho(x\ y\ z)$ can be written

$$\rho(xyz) = \sum\sum\sum_{-\infty}^{+\infty} A(pqr) \exp 2\pi i(px + qy + rz) \quad \ldots (1.11)$$

where p, q, and r are integers and A(p q r) the coefficient of the general term of the series.

Substituting $\rho(x\ y\ z)$ in the expression for the structure factor in equation (9), we have,

$$F(hk\ell) = V \int_0^1\int_0^1\int_0^1 \Big[\sum\sum\sum_{-\infty}^{+\infty} A(pqr) \exp 2\pi i(px + qy + rz) \times$$

$$\exp 2\pi i(hx + ky + \ell z)dx\, dy\, dz \quad \ldots (1.12)$$

On integrating, each term is zero except the ones for which $p = -h$, $q = -k$, $r = -\ell$ and this gives

$$F(hk\ell) = V\int_0^1\int_0^1\int_0^1 A(\bar{h}\bar{k}\bar{\ell})\, dx\, dy\, dz \;=\; VA(\bar{h}\bar{k}\bar{\ell}) \quad \ldots (1.13)$$

therefore, $$A(\bar{h}\bar{k}\bar{\ell}) = \frac{F(hk\ell)}{V}$$

Therefore the Fourier series which represents the electron density distribution at every point in the crystal may be written as

$$\rho(xyz) = \frac{1}{V}\sum\sum\sum_{-\infty}^{+\infty} F(hk\ell) \exp \left[-2\pi i(hx + ky + \ell z)\right] \ldots (1.14)$$

or, after some algebraic manipulation,

$$\rho(xyz) = \frac{1}{V} \sum\sum\sum_{-\infty}^{+\infty} |F(hk\ell)| \cos\left[2\pi(hx + ky + \ell z) - \alpha(hk\ell)\right] \quad \ldots (1.15)$$

1.7. THE PHASE PROBLEM AND SOME METHODS OF ITS SOLUTION.

The amplitude, $|F|$ of the structure factor can be obtained directly from the measured intensity of a reflection. But in order to use equation (15) to calculate the Fourier representation of the electron distribution in the crystal, we have to know the phase constant, α, for each reflection. But the phase cannot be obtained experimentally and is therefore unknown. This constitutes what is called the Phase Problem in x-ray crystallography.

The phase problem may be overcome by several methods widely used by crystallographers in structure determinations. Some of them are outlined as follows:

1.7.1. THE VECTOR METHOD.

Patterson (1935) defined a function P(u v w) such that

$$P(uvw) = V\int_0^1\int_0^1\int_0^1 \rho(xyz)\,\rho(x + u,\ y + v,\ z + w)\ dx\ dy\ dz \quad \ldots (1.16)$$

Substituting the values for the electron densities given in (14), we have

$$P(uvw) = \frac{1}{V}\int_0^1\int_0^1\int_0^1 \sum_{h}\sum_{k}\sum_{\ell}\sum_{h'}\sum_{k'}\sum_{\ell'}{}_{-\infty}^{+\infty} \Big\{F(hk\ell)\exp\left[-2\pi i(hx + ky + \ell z)\right]$$
$$\times F(h'k'\ell')\exp\left[-2\pi i(h'x+k'y+\ell'z)\right]\exp\left[-2\pi i(h'u+k'v+\ell'zv)\right]\Big\}\ dx\ dy\ dz \quad \ldots (1.17)$$

On integrating, every term is zero except when

$$h = -h', \; k = -k', \; \ell = -\ell'.$$

hence,

$$\rho(uvw) = \frac{1}{V} \sum_{h'} \overset{+\infty}{\underset{-\infty}{\sum_{k'}}} \sum_{\ell'} F(hk\ell)F(\overline{h}\overline{k}\overline{\ell}) \exp\left[-2\pi i(h'u+k'v+\ell'w)\right] \quad \ldots (1.18)$$

and, as $F(hk\ell)$ and $F(\overline{h}\overline{k}\overline{\ell})$ are complex conjugates,

$$P(uvw) = \frac{1}{V}\sum\sum\sum\left|F(hk\ell)\right|^2 \exp 2\pi i(hu + kv + \ell w) \quad \ldots (1.19)$$

This is known as the Patterson function.

Each Patterson peak corresponds to two atoms in the crystal structure. If the coordinates of the two atoms are (x_1, y_1, z_1) and (x_2, y_2, z_2), then the Patterson peaks corresponding to the vectors between these two atoms have coordinates $(x_2-x_1, y_2-y_1, z_2-z_1)$ and $(-x_2+x_1, -y_2+y_1, -z_2+z_1)$. This means that the Patterson function is always centrosymmetrical. For a crystal containing N atoms per unit cell, the total number of distinct peaks in the Patterson function is $N(N-1)/2$.

A characteristic of the Patterson function is the presence of the origin peak which represents all the N vectors between atoms and themselves. The height of a Patterson peak between two atoms containing Z_1 and Z_2 electrons respectively is approximately proportional to $Z_1 Z_2$. It follows from this that a heavy atom (e.g. I atom) of significantly higher atomic number in presence of a large number of light atoms (e.g. carbon and oxygen atoms) of lower atomic numbers will be represented by outstanding peaks at the end of the vectors between the heavy atoms at symmetry related equivalent positions.

For simple structures, it is possible to assign atomic positions to all the atoms in the unit cell from a knowledge of the interatomic vectors from the Patterson function. But in more complex molecules, it can seldom be used to work out the complete structures, except to locate the positions of a heavy atom that may be present.

1.7.2. THE HEAVY ATOM METHOD.

This offers a very practical procedure in working out the structure of a molecule, and was first employed by Robertson et al. (1937, 1940) in the analysis of the structure of phthalocyanine. For if a compound contains a few atoms whose atomic numbers are considerably greater than those of the remaining atoms, their positions can generally be determined from the Patterson function.

Now, the structure factor may be written as

$$F = F_H + F_L \qquad \ldots (1.20)$$

F_H and F_L are the contributions of the heavy atoms and light atoms respectively.

Very often the magnitudes of F_H are considerable and may approximate the magnitudes of F. Under this condition, the phase constants, α_H, due to the heavy atoms alone may be good approximations to the true phase constants, α, due to the whole molecule.

If a Fourier synthesis is computed using the observed structure amplitudes $|Fobs|$ and the phase constants, α_H, the resulting electron-density distribution generally will reveal part or whole of the structure. Lipson and Cochran (1953) have suggested that, as a rough guide, the square of the atomic number of the heavy atom should be equal approximately to the sum of the squares of the atomic numbers of the light atoms.

Sim (1957, 1961) has employed a function

$$r = (\Sigma f_H^2 / \Sigma f_L^2)^{1/2},$$

where Σf_H^2 is the sum of the squares of the atomic scattering factors of the heavy atom and Σf_L^2 is the sum of the squares of the atomic scattering factors of the light atoms, and showed that for the centrosymmetrical case of a molecule with one heavy atom in general position in the space group $P\bar{1}$, 80-90% of the F will have the same sign as the F_H when r = 1 to 2.

For non-symmetrical space groups, Sim concludes that when r = 1; 38% of the errors (equal to $\alpha - \alpha_H$) will lie within $\pm 20^0$ of α, and that the fraction increases to 67% when r = 2.

Woolfson (1956) has worked out a weighting function, applied to the Fobs for the centrosymmetrical case in order to minimize these errors and hence improve the resolution of the light atoms in the Fourier synthesis; while Sim (1959) has worked out an analogous weighting function for the non-centrosymmetrical case.

The structures of the iodo-derivative of the diterpene isocolumbin, the naturally occurring sulphoxide derivative and the mercuric chloride adduct described in this thesis were established by using the heavy atom method.

1.7.3. THE ISOMORPHOUS REPLACEMENT METHOD.

Centrosymmetrical Case.

This method was first applied by Robertson (1935, 1936) also in the analysis of phthalocyanine. This requires two isomorphous derivatives of the type (A + B) and (A + C) so that only part of the structures are different. B and C are generally heavy atoms whose position can be obtained from the Patterson function. The observed structure amplitudes in this case must be on the absolute scale.

Assuming exact isomorphism, we have

$$F_{(A + B)} = F_A + F_B \qquad \ldots (1.21)$$

$$F_{(A + C)} = F_A + F_C \qquad \ldots (1.22)$$

The structure factors F_B and F_C can be calculated. Therefore, by comparing the differences of F_B and F_C with the differences of $F_{(A + B)}$ and $F_{(A + C)}$, it is possible to assign signs to both $F_{(A + B)}$ and $F_{(A + C)}$.

Other examples are found in the work on camphor by Wiebenga and Krom (1946) and on sucrose derivatives by Beevers and Cochran (1947).

Non-Centrosymmetrical Case.

Bijvoet and his co-workers (1951) have pointed out the possibility of determining the phase constants uniquely by a graphical method in acentric space groups ~~by a graphical method~~ using a series of three isomorphous derivatives. The applicability of this procedure has been elucidated in detail by

Harker (1956). Perutz (1960, 1963) and Kendrew (1958, 1960) and their co-workers have achieved outstanding successes by applying this method in the field of protein structures.

The structure of aflatoxin G_1 described in this thesis was also established by using this method. Its procedure will be described in more detail later.

1.8. SOME METHODS OF STRUCTURE REFINEMENT.

1.8.1. THE FOURIER METHOD.

A Fo Fourier synthesis computed from the observed structure amplitudes and the calculated phase constants from a set of approximate atomic coordinates in a postulated structure, will generally lead to a set of improved coordinates. By this procedure of successive refinement by approximation, a set of accurate atomic coordinates can be attained.

In the centrosymmetrical case when the phases are either 0 or 180°, convergence of the successive Fo synthesis is reached when there is no more sign change between two successive sets of calculated structure factors.

In the non-centrosymmetrical case, when the phase constants can vary from 0 to 360°, the calculated phase constants will only approach the correct values gradually. Shoemaker et al. (1950) proposed an n-shift rule which may accelerate the refinement. By this rule, instead of just using the shifts in coordinates indicated between two successive Fo syntheses, one uses a value n times the shifts, where $n = p + 2(1-p)$, p being the fraction of the observed structure factors which are restricted to 0 or 180° and the remainder which may have any value.

Earlier, Cruickshank (1950) has introduced the double-shift rule for the most general case in the space group P1 where p = 0.

In most analyses, only a finite number of reflections can be observed, whereas the Fourier summation in equation (15) ranges from $-\infty$ to $+\infty$. This effect, due to the cutting-short of part of the observed $|Fo|$ while they are still of appreciable magnitude, is called the series-termination effect and can be seen in the resulting electron-density distribution when peaks are surrounded by diffraction ripples. This has the effect of displacing the peaks from their true positions.

Booth (1946) has suggested a method for correcting the series-termination effects by computing another Fourier synthesis using the calculated structure factors (the Fc synthesis). This Fc synthesis is subjected to the same series-termination effects and shifts in the atomic coordinates can be evaluated. These shifts can then be applied to the improved coordinates evaluated from the Fo synthesis. This is called back-shift correction and can be summarized as

$$x_t = x_{obs} - (x_{cal} - x') \qquad \ldots (1.23)$$

where x_t is the true coordinate, x_{obs} and x_{cal} are the new coordinates obtained in the Fo and Fc syntheses respectively and x' is the coordinates used to calculate the set of F cal which is in turn used to calculate the Fc synthesis.

The series-termination effects can also be eliminated by using the (Fo - Fc) - synthesis (Cochran, 1951).

1.8.2. THE LEAST SQUARES METHOD.

One way of assessing the accuracy of a structure determination is to compare the $|Fo|$ with the $|Fc|$ of the proposed structure.

Hughes (1941) first applied the theory of observation and employed the method of least squares to refine the structure parameters.

In equation

$$R = \sum_{hk\ell} w(|Fo| - |Fc|)^2 \qquad \ldots (1.24)$$

$|Fo|$ and $|Fc|$ are the observed and calculated structure amplitudes and w is a weight for each reflection.

If $p_1, p_2, \ldots, p_n$ are the n parameters occurring in the Fc whose values are to be determined, then the condition for R to be a minimum is

$$\frac{\partial R}{\partial p_j} = 0 \quad (j = 1, \ldots, n) \qquad \ldots (1.25)$$

i.e. $$\sum_{hk\ell} w\Delta \frac{\partial |Fc|}{\partial p_j} = 0, \text{ where } \Delta = |Fo| - |Fc| \qquad \ldots (1.26)$$

If the correct value of p_i is $(p_i + \epsilon_i)$, where ϵ_i is a small correction, then by expanding as a function of the parameters by a Taylor series to the first order, we have

$$\Delta(p + \epsilon) = \Delta(p) - \sum_{i=1}^{n} \epsilon_i \frac{\partial |Fc|}{\partial p_i} \qquad \ldots (1.27)$$

substituting equation (27) into (26), we have

$$\sum_{i=1}^{n} \left\{ \sum_{hk\ell} w \frac{\partial |Fc|}{\partial p_i} \frac{\partial |Fc|}{\partial p_j} \right\} \epsilon_i = \sum_{hk\ell} w\Delta \frac{\partial |Fc|}{\partial p_j} \qquad \ldots (1.28)$$

There are n of these equations for j = 1, ..., n and they are known as the normal equations.

Upon solving these normal equations for the n unknowns, we obtain a set of improved coordinates.

There can either be four parameters (three positional and one isotropic temperature factor) or nine parameters (three positional and six anisotropic factors) for every atom to be refined. The solution of the normal equations for a structure containing a large number of atoms (say 20 or more) would be a major task even with the aid of modern computers. Therefore, approximations have to be made in solving the normal equations in addition to the full matrix solution. The block-diagonal approximation neglects the interactions between the atoms and involves only parts of the equation for one atom. Thus, in the case of the benzene and bromothiophen solvates of aflatoxin G_1 described in this thesis, the atomic parameters have been refined by solving a series of 9 x 9 matrices; and in the case of the iodo-derivative of isocolumbin, a series of 4 x 4 matrices. Another approximation, employed in the mercuric chloride complex involves the solution of a series of 3 x 3 matrices for positional parameters and 6 x 6 matrices for the anisotropic temperature factors.

The estimated standard deviation of the parameters, $\sigma(p_i)$, can be calculated from the least squares totals. The equation used in deriving the e.s.d. is

$$\sigma^2(p_j) = \frac{\sum w\Delta^2}{(m-n)\sum w\left(\frac{\partial|Fc|}{\partial p_j}\right)^2} \qquad \ldots (1.29)$$

Where m is the number of independent observations and n is the number of parameters to be refined.

1.9. ACCURACY OF A STRUCTURE.

The most commonly-employed index to show the correctness of a structure is the discrepancy factor $R \left(= \frac{\Sigma \left| |Fo| - |Fc| \right|}{\Sigma |Fo|}\right)$, where $|Fo|$ and $|Fc|$ are the observed and calculated structure amplitudes. For a correct proposed structure to compare with a set of faultless data, there should be no discrepancy at all. But this is never attained and the usual final discrepancy in most structures published ranges from 5 - 15%.

A more reliable assessment of accuracy would be to compare the molecular dimensions such as bond lengths and angles with values derived from theoretical consideration and with results obtained from other experiments, both x-ray diffraction method or other physical methods.

In the structures described in this thesis, the estimated standard deviation, $\sigma(\ell)$, of a bond length, ℓ, between two atoms with coordinates (x_1, y_1, z_1), (x_2, y_2, z_2) referred to a set of orthogonal axes has been calculated using the equation

$$\sigma(\ell) = \frac{1}{\ell} \left\{ (x_1-x_2)^2 \left[\sigma^2(x_1) + \sigma^2(x_2)\right]^2 + (y_1-y_2)^2 \left[\sigma^2(y_1) + \sigma^2(y_2)\right]^2 + (z_1-z_2)^2 \left[\sigma^2(z_1) + \sigma^2(z_2)\right]^2 \right\}^{\frac{1}{2}} \qquad \ldots (1.30)$$

The estimated standard deviations of the bond angles have been calculated using the equations worked out by Darlow (1960).

In comparing two bond lengths ℓ_1 and ℓ_2, the difference is considered to be significant if it is greater than three times the value $\left[\sigma^2(\ell_1)+\sigma^2(\ell_2)\right]^{\frac{1}{2}}$.

PART II

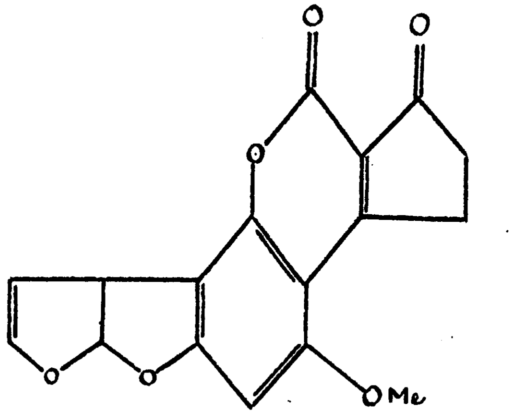
The Crystal Structure of Aflatoxin G_1.

2.1. INTRODUCTION.

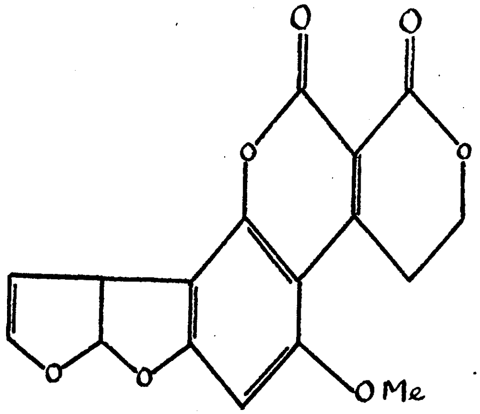
Aflatoxin G_1 is one of the mixture of structurally related metabolites extracted from groundnuts infected with the toxic strain *Aspergillus flavus*. The study of these metabolites follows the outbreaks in 1960 among young turkeys of a disease named 'turkey X disease' which caused vast numbers of them to die (Blount, 1961), and the subsequent isolation of a toxic fraction from some samples of groundnut meals upon which they were fed (Sargeant et al., 1961). It was found (Lancaster et al., 1961) that the toxin causes hepatic changes in turkey poults, ducklings, cattle, pigs, and sheep. Further work by Nesbitt et al. (1962), using chromatography on alumina, first indicated that this toxin is a mixture of two substances, aflatoxins B and G. They were separated from one another by the counter-current technique. It was subsequently found by Hartley et al. (1963) that the mixture contains four substances, aflatoxins B_1 and G_1; and their dihydro-derivatives, B_2 and G_2.

On the basis of chemical and spectroscopic (in particular nuclear magnetic resonance) investigations, Büchi et al. (1963, 1965) have proposed structures (I) and (II) for aflatoxins B_1 and G_1, while van der Merive, Fourie and Scott (1963) have proposed the alternative formulae (III) and (IV) for these compounds.

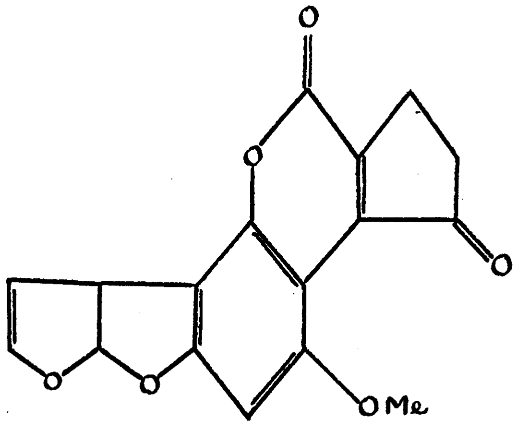
In the x-ray crystal analysis of aflatoxin G_1, the materials were kindly supplied by Dr. B.F. Nesbitt of the Tropical Products Institute, London. They consist of the aflatoxin G_1-benzene



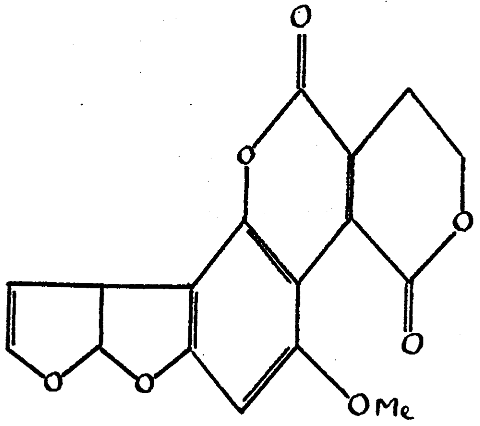
(I)



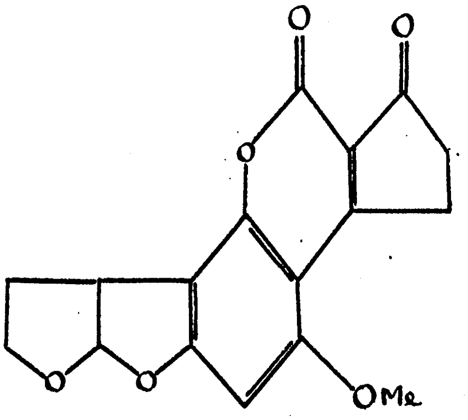
(II)



(III)



(IV)



(V)

solvate, obtained by crystallizing aflatoxin G_1 from benzene; and the corresponding bromobenzene and bromothiophen solvates.

The work began with the bromobenzene solvate, with a view to establishing the structure by the heavy-atom method. However, the presence of pseudo-symmetry in the Fo Fourier synthesis with phases based on the bromine atom alone made it difficult to interpret the electron-density maps.

Finally the method of isomorphous replacement was employed and the phase constants were determined graphically. From the resulting Fourier synthesis, it was possible to establish the structure, agreeing with the structure (II) proposed by Büchi et al.

The structure of dihydro-aflatoxin B_1 (V) has also been determined by x-ray methods by van Soest and Peerdeman (1964).

2.2. EXPERIMENTAL DATA.

THE BROMOBENZENE SOLVATE.

Rotation, oscillation, and Weissenberg photographs were taken with Copper K α radiation. The cell-dimensions were obtained from the rotation and Weissenberg photographs. The only systematic absence of reflections is (0k0) when k is odd. The space group is therefore either $P2_1(C_2^2)$ or $P2_1/m(C_{2h}^2)$. It cannot be $P2_1/m$ because the compound is optically active. Intensity data were obtained from equatorial and equi-inclination upper layer Weissenberg photographs taken from a crystal rotated about its unique axis. The multiple-film technique (Robertson,

1943) was employed. In the nine layers of $h0\ell$,..., $h8\ell$, a total of 3,057 reflections was recorded. The intensities were estimated visually by comparison with a calibrated step-wedge. Lorentz, polarization and rotation factors for upper-layers (Tunell, 1939) were applied. No correction for absorption was made.

THE BENZENE SOLVATE.

Rotation and Weissenberg photographs were taken from a crystal rotated also about the unique axis. The crystal is also monoclinic with space group $P2_1$. The Weissenberg photograph exhibits a similar pattern of the distribution of reflections. The two non-unique axes were chosen to correspondence with the same set for the bromobenzene solvate. The cell dimensions thus obtained were almost identical ~~as~~ with those for the bromobenzene solvate. Eight equi-inclination upper-layer Weissenberg photographs were taken and a total of 2,833 reflections were estimated visually. Lorentz, polarization and rotation factors (for the upper layers) were applied.

THE BROMOTHIOPHEN SOLVATE.

The crystals of this solvate are also prismatic. Rotation and Weissenberg photographs taken from a crystal rotated about the elongated crystal axis revealed the same space group and almost equal, unique axis. When the other axes were chosen in accordance with the previous two cases, the unit-cell dimensions were again almost identical. The crystal data for all these three solvates are listed in Table 2-1. A total of 3,035 reflections was estimated.

2.3. DETERMINATION OF THE STRUCTURE.

A. Approach by the heavy-atom method using the bromobenzene solvate.

With one bromine atom weighted against 54 carbon and oxygen atoms (the value of r in section I. on pp. 11 is 0.7), it was expected from the start that a Fourier synthesis with the phase constants based on the heavy atom alone would have a number of spurious peaks. However, it was hoped that under the other favourable conditions, it might still be possible to establish the structure.

For the space group $P2_1$, and with one bromine atom in an asymmetric unit, there is only one Br-Br vector in the Patterson function. This was determined from the Patterson projection P(uw) shown in Fig. 2-1(a), and confirmed by calculating the Harker section $P(u\frac{1}{2}w)$, shown in Fig. 2-1(b), using all three-dimensional data.

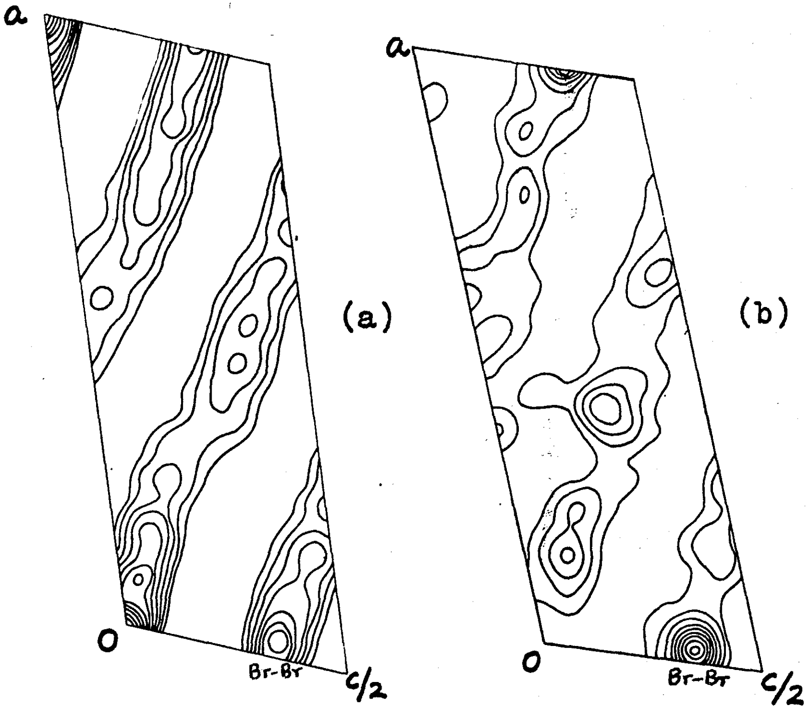
The coordinates of the Br atom were x = 0.012, z = 0.176. As there is no fixed origin in $P2_1$, we selected as origin the centre of symmetry relating the bromine atoms, and the y-coordinate was therefore arbitrarily assigned the value 0.250.

When a set of structure factors was calculated using an isotropic temperature factor of $B = 4.5\text{Å}^2$, the usual discrepancy

Table 2-1 Crystal data

	Bromobenzene solvate	Bromothiophen solvate	Benzene solvat
Molecular formula	$C_{40}H_{29}O_{14}Br$	$C_{38}H_{27}O_{14}SBr$	$C_{40}H_{30}O_{14}$
Molecular weight	813.5	819.6	734.6
Space group	$P2_1$ (C_2^2)	$P2_1$ (C_2^2)	$P2_1$ (C_2^2)
a	15.27 Å	15.73 Å	15.75 Å
b	10.48	10.53	10.47
c	11.35	10.88	10.96
β	110°27'	107°57'	109°29'
Cell volume	1702 $Å^3$	1714 $Å^3$	1704 $Å^3$
Density(cal.)	1.587 gm cm^{-3}	1.587 gm cm^{-3}	1.431 gm cm^{-3}
Absorption coeff. μ for x-rays(λ=1.542A)	24.9 cm^{-1}	29.9 cm^{-1}	9.3 cm^{-1}
Composition of asymmetric unit	$C_6H_5Br+2(C_{17}H_{12}O_7)$	$C_4H_3SBr+2(C_{17}H_{12}O_7)$	C_6H_6+ $2(C_{17}H_{12}O_7)$
F(000)	832	836	764
No. of observed structure amplitudes	3057	3019	2833

Fig.2-1. Aflatoxin G_1 bromobenzene solvate. (a) Patterson projection, P(uw), (b) Section at $V=\frac{1}{2}$ through the three-dimensional Patterson synthesis. Contour scale arbitrary.



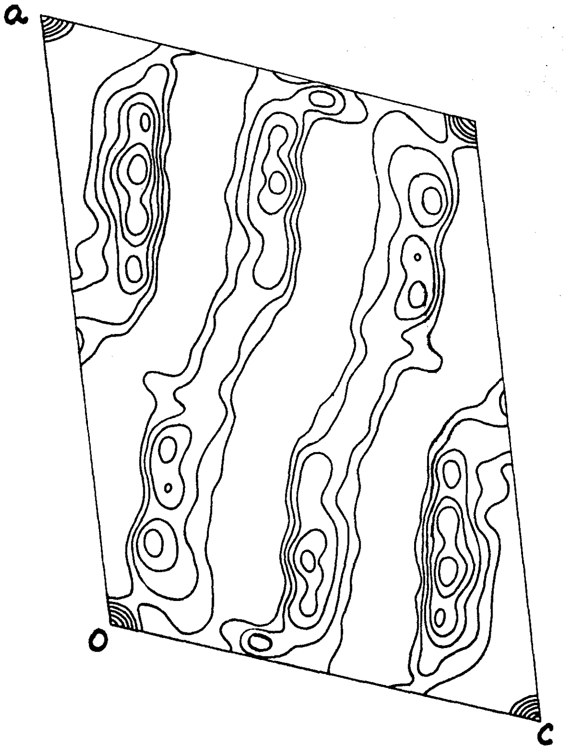


Fig.2-2. Difference Patterson projection along the *b*-axis calculated with coefficients $(|F_{brbz}|^2-|F_{bz}|^2)$. Contour scale arbitrary.

R over all the observed structure amplitudes was 46.2%.

With the exception of 380 terms for which $|Fo| > 2|Fc|$, all other $|Fo|$ were included in a Fourier synthesis, using the signs of the corresponding Fc.

Since the bromine atoms are linked by a centre of symmetry, a Fourier synthesis based on the phase constants of the bromine atom alone (which are restricted to 0 or 180°) creates the unavoidable situation of introducing a false centre of symmetry into the rest of the structure as well, and peaks appear not only at the true atomic positions but also at position related to these by the false centre of symmetry.

Besides this pseudo-symmetry, the presence of spurious peaks and their 'ghost' peaks made the situation even more confusing and it was not possible to interpret the Fourier map.

B. Difference Patterson projection and Fourier projection using the bromobenzene and benzene solvates.

The almost identical cell dimensions of these two solvates suggested that they are at least approximately isomorphous. To confirm this, difference Patterson projection along the unique axis using various coefficients were computed. This would require the observed structure amplitudes of both solvates to be on the absolute scale.

The absolute scale, K_1, for the $h0\ell|Fo|$ of the bromobenzene solvate was determined using the statistical method of Wilson (1942), and the scale factor, K_2, for the benzene solvate was determined by the relation:

$$\frac{(K_1F_{o,brbz})^2}{(K_2F_{o,bz})^2} = \frac{\Sigma f^2_{brbz}}{\Sigma f^2_{bz}} . \qquad \ldots (2.1)$$

where $F_{o,\,brbz}^2$ and $F_{o,\,bz}^2$ are the mean values of the square of the observed structure amplitudes of the bromobenzene and benzene solvates over a range of $\sin^2\theta$, and Σf^2_{brbz} and Σf^2_{bz} are the sum of the squares of the atomic scattering factors over the same range of $\sin^2\theta$.*

Thus, even if the absolute scale for the bromobenzene solvate might not be accurate, the scale factor thus obtained for the benzene solvate would mean that the two sets of Fo will have the same relative scale.

A difference Patterson projection proposed by Kartha and Ramachandran (1955) using $(|F_{brbz}| - |F_{bz}|)^2$ as coefficients was computed and this gave the same x- and z- coordinates for the Br atom. Another difference Patterson projection with coefficients $(|F_{brbz}|^2 - |F_{bz}|^2)$ was also computed. According to Perutz (1956), this function gave two kinds of peaks: those due to interaction between the Br - Br vector, and those due to interaction between the Br atom on the one hand, and the light atoms of the aflatoxin molecule on the other.

---

* The subscripts brbz, bz and brthio will be used throughout to refer to the bromobenzene and benzene, and bromothiophene solvates respectively.

The close resemblance of this difference Patterson (Fig. 2-2) with the Patterson projection shown in Fig. 2-1(a) suggested that the two solvates are isomorphous.

Since the (010) projection is centro-symmetrical, it is possible to determine the signs of the F_{brbz}^{obs} and F_{bz}^{obs} in the following equation

$$F_{brbz}^{obs} = F_{bz}^{obs} + F_{br}^{cal} \qquad \ldots (2.2)$$

Where F_{br}^{cal} is the contribution of the bromine atoms.

Fig. 2-3(a) shows the Fourier projection of bromobenzene solvate calculated with 290 terms whose signs were thus determined; while Fig. 2-3(b) shows the Fourier projection of the benzene solvate calculated with 245 terms. The close resemblance of these two Fourier projections clearly indicate that the two solvates are at least approximately isomorphous.

C. The function of $(|F_{brbz}|^2 - |F_{bz}|^2)|F_{br}^{cal}| \exp i\, \alpha_{br}$.

This is one of the eight coefficients proposed by Ramachandran and Raman (1959) for use in a Fourier synthesis to give electron-density distribution. They suggested that with such coefficients, the background would be low and the number of spurious peaks would be limited. As a trial, the (010) Fourier projection was calculated using this cubic function. The fact that this projection (shown in Fig. 2-4) gave features almost identical with the two Fourier projections (Figs. 2-3 (a) and (b)) led us to compute a three-dimensional synthesis.

Before the synthesis, the absolute scales of the Fo for

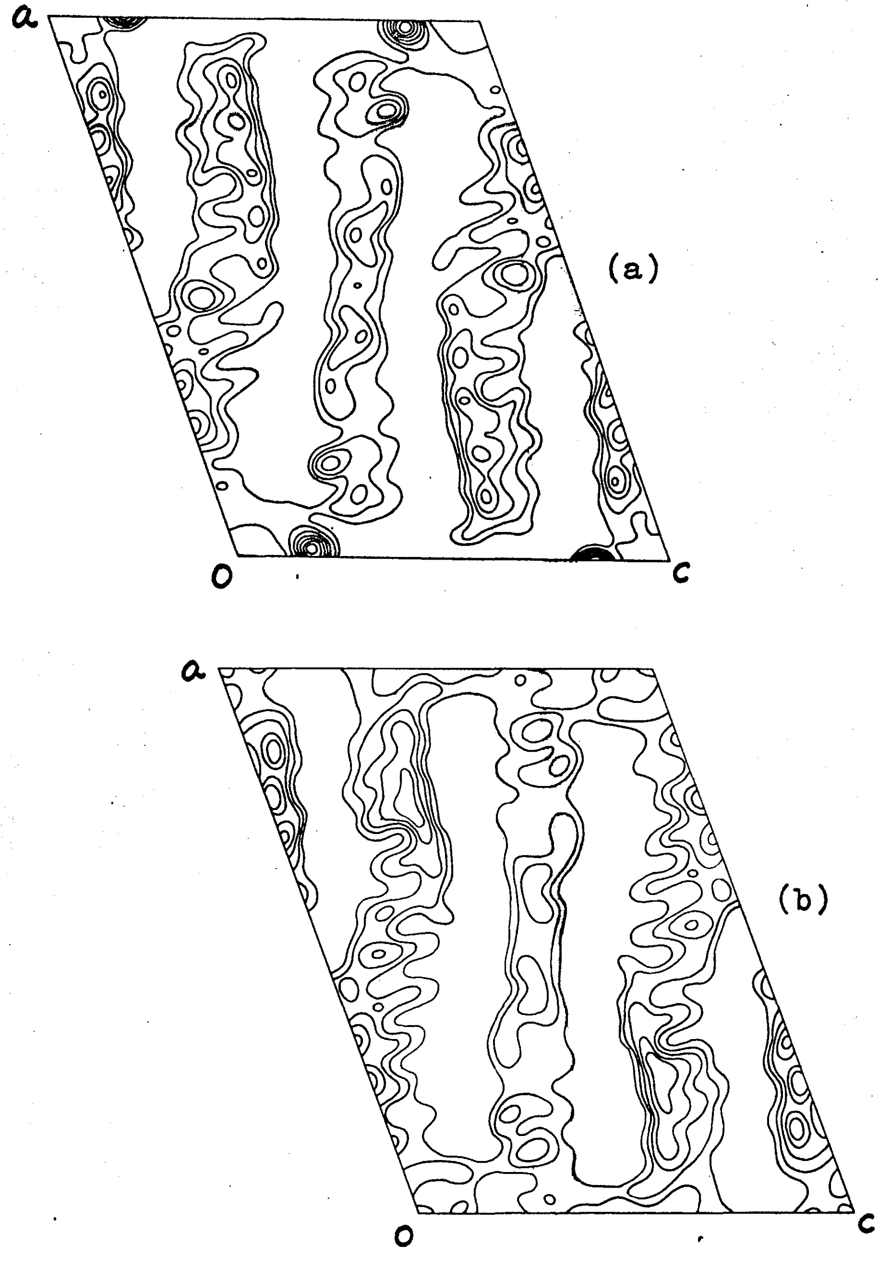


Fig.2-3. Electron-density projection along the b-axis
(a) for aflatoxin G_1 bromobenzene solvate,
(b) for aflatoxin G_1 benzene solvate.
Contour scale arbitrary.

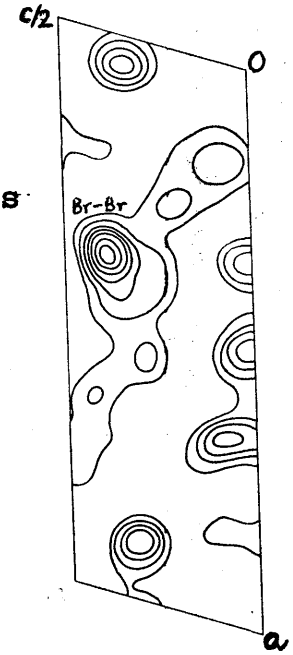


Fig.2-5. Section at $V=\frac{1}{2}$ through the three-dimensional Patterson synthesis for aflatoxin G_1 bromothiophen solvate. Contour scale arbitrary.

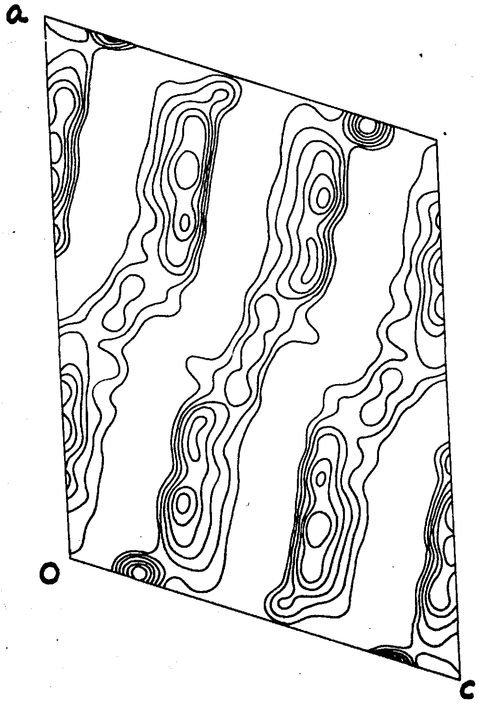


Fig.2-4. Electron-density projection along the b-axis calculated with coefficients $(|F_{brbz}|^2-|F_{bz}|^2)\,|F_{br}^{cal}|\exp(i\alpha_{br})$. Contour scale arbitrary.

each of the $h1\ell$, ..., $h8\ell$, layers of the bromobenzene solvate were determined, and the corresponding scales for the benzene solvate were calculated using equation 2-1.

The pseudo-symmetry was expected to be present because we were still using the signs of the calculated structure factors based on the Br atom alone. However, by comparing with the original heavy-atom Fourier maps, we could pick only the peaks which occurred in both syntheses and rule out some spurious peaks. Thus, only genuine peaks and their 'ghost' peaks should be present. It was hoped that the structure might be visible at this stage. But the situation of the resulting electron-density distribution was still very confusing and it was decided that it was not practicable to pick some atoms and carry on the analysis on a trial basis.

D. The Bromothiophen Solvate.

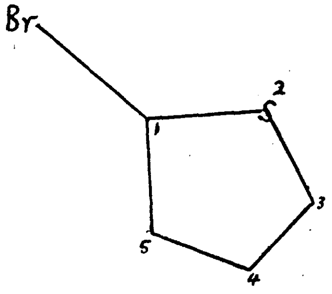
By this time the bromothiophen solvate had been supplied by Dr. Nesbitt.

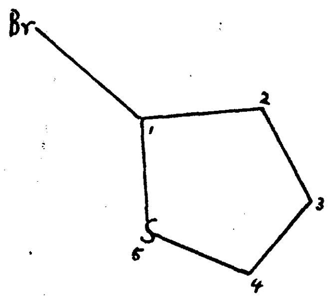
In this case, since there is a second moderately heavy sulphur atom, it was hoped that with the Br and S atoms, we might still be able to establish the structure by the heavy-atom method. For, though the sulphur atoms are also connected by a centre of symmetry, it is rather unlikely that this centre will coincide with that of the bromine atoms. If their centres do not coincide, then the overall symmetry may be annulled.

The x- and z- coordinates of the bromine atom were determined from the three-dimensional Patterson synthesis. The

Harker section P(u½w) is shown in Fig. 2-5. But it was not possible to assign outstanding peaks corresponding to the Br-S or the S-S vectors.

It was later found, when the structure has been established, that the thiophene molecule actually assumes two configurations by rotating about the Br-C bond so that at positions 2 and 5, the atoms have effectively a weight of ½(C+S).





E. The Method of Isomorphous Replacement.

As the bromothiophen solvate is also isomorphous with the bromobenzene and the benzene solvates, it was decided to apply the method of isomorphous replacement in this non-centrosymmetric structure. (Bijvoet et al., 1951, Harker, 1956).

The structure amplitudes of the bromothiophen solvate were now scaled to those of the bromobenzene solvate so that all the observed structure amplitudes were on approximately the same absolute scale.

In order to apply the isomorphous replacement method to determine the phase constants, it is necessary to ensure that the same origin of coordinates in the unit cell is employed for all three solvates. The origin assigned to the bromobenzene solvate was chosen as the common origin and the y-coordinate of the bromine atom in the bromothiophen solvate was referred to this origin by evaluating two- and three- dimensional difference Patterson syntheses with coefficients $(|F^{obs}_{brthio}| - |F^{obs}_{brbz}|)^2$. This function, proposed by Rossmann(1960), displays positive peak (self-Patterson) corresponding to the Br-Br vector in the bromobenzene solvate, and positive peak (self-Patterson) corresponding to the Br-Br vector in the bromothiophen solvate, as well as negative peaks (cross-Patterson) corresponding to vectors between the replaceable atoms (that is, Br) in the two crystals. The c-axis projection is shown in Fig. 2-6, and the composite view down the unique b-axis is shown in Fig. 2-7.

The coordinates adopted for the bromine atom in the bromothiophene solvate are x = 0.298, y = 0.125, z = 0.308.

Assuming the three solvates are ideally isomorphous, the following conditions will hold for any reflection $(h,k,\ell,)$

$$F^{obs}_{brbz} = F^{obs}_{bz} + F^{cal}_{br\ in\ brbz} \quad \ldots (2.3)$$

$$F^{obs}_{brthio} = F^{obs}_{bz} + F^{cal}_{br\ in\ brthio} \quad \ldots (2.4)$$

$$F^{obs}_{brbz} = F^{obs}_{brthio} + (F^{cal}_{br\ in\ brbz} - F^{cal}_{br\ in\ brthio}) \ldots (2.5)$$

Combining (2.3) and (2.5), it is possible to determine the phase constants for all the three solvates. The graphical method used is illustrated in Fig. 2-8.

The phase circles were drawn on graph papers. Some of them are shown in Fig. 2-9. It is seen that they seldom intersect at a point because the three solvates cannot be expected to be exactly isomorphous and experimental inaccuracy and errors are also present. The best point is chosen to satisfy all three cases. Only the phase angles for the bromobenzene solvate were read off for use to compute a Fourier synthesis.

It must also be pointed out that because the y-coordinate of the Br atom in the bromothiophen solvate is 0.125, for the layers h4ℓ and h8ℓ, the imaginary part of the contribution of the bromine atom is also zero so that it was not possible to determine the phase constants unambiguiously. Instead, the mean vectors were employed. This is illustrated in the reflection 1,4,5, shown in Fig. 2-9.

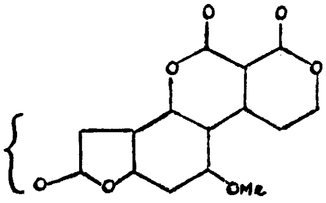
The signs of the F^{obs} in the centro-symmetrical layer h0ℓ had been determined previously.

In this way, it was possible to obtain the phase constants of 2268 reflections, and these were used to derive a three-dimensional electron-density distribution which was displayed as contoured sections drawn on sheets of glass and stacked in a metal frame.

Nine of the 21 Fourier sections are shown alongside the corresponding sections of the final Fourier in Fig. 2-10.

Examination of the three-dimensional electron density clearly revealed for aflatoxin G_1 the part structure Vl, though without distinction between carbon and oxygen atoms. The benzene ring in the bromobenzene solvent was indistinct.

(VI)



Difference Patterson syntheses with $(|F^{obs}_{brthio}| - |F^{obs}_{brbz}|)^2$ as coefficients.

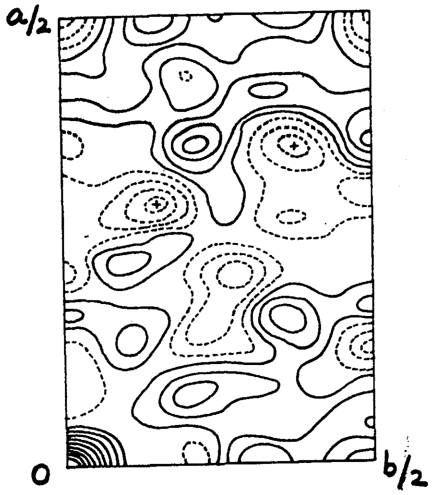


Fig.2-6. Difference Patterson projection along the c-axis derived with data for the bromobenzene and bromothiophen solvates. Contour scale arbitrary. The positions of the Br-Br vectors are indicated by crosses.

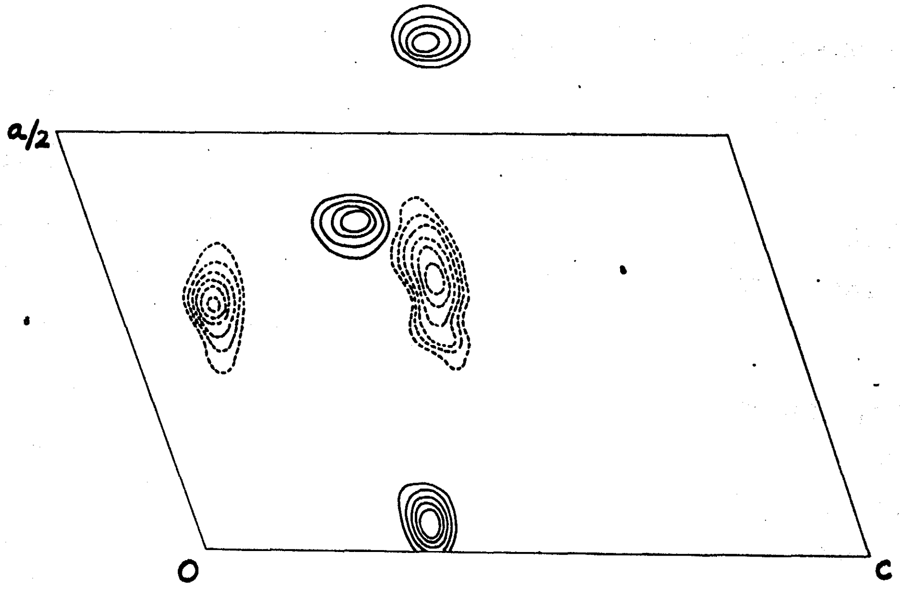


Fig.2-7. Composite view down the unique axis of the three-dimensional difference Patterson synthesis, showing positive and negative peaks corresponding to the Br-Br vectors. Contour scale arbitrary. Negative peaks are drawn in broken lines.

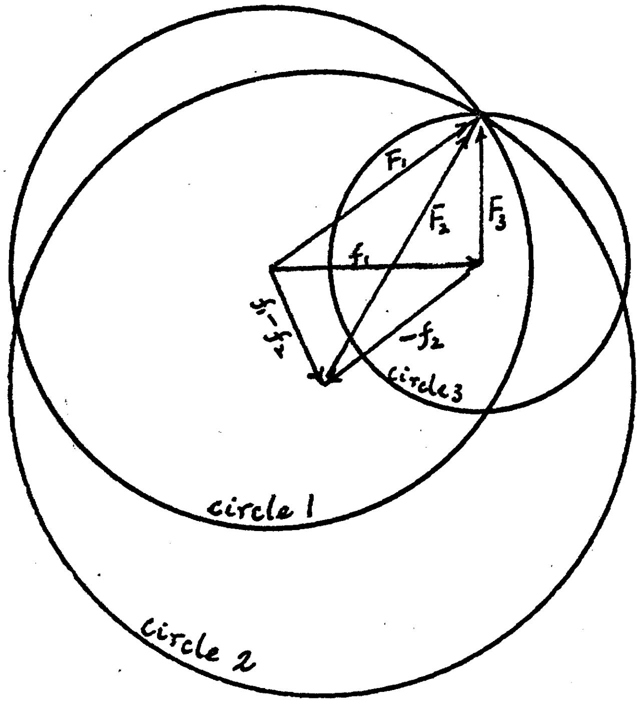


Fig.2-8. The symbols F_1, F_2, F_3, f_1, and f_2 used here are equivalent respectively to F^{obs}_{brbz}, F^{obs}_{brthio}, F^{obs}_{bz}, $F^{cal}_{br\ in\ brbz}$, and $F^{cal}_{br\ in\ brthio}$ used in equations (2.3) to (2.5) on pp. 28. The subscripts 1, 2, and 3 used in Fig.2-9 refer to the brbz, brthio, and bz solvates respectively.

The circle 3 (radius $|F_3|$) is drawn using the end of the vector f_1 drawn from the centre of circle 1 (radius $|F_1|$). The circle 2 (radius $|F_2|$) is drawn using the end of the vector $-f_2$ which is drawn from the centre of circle 3, as centre.

At the point of intersection of the three circles the equations (2.3), (2.4), and (2.5) are satisfied so that the directions of F_1, F_2, and F_3 are known and their phase angles can be read off.

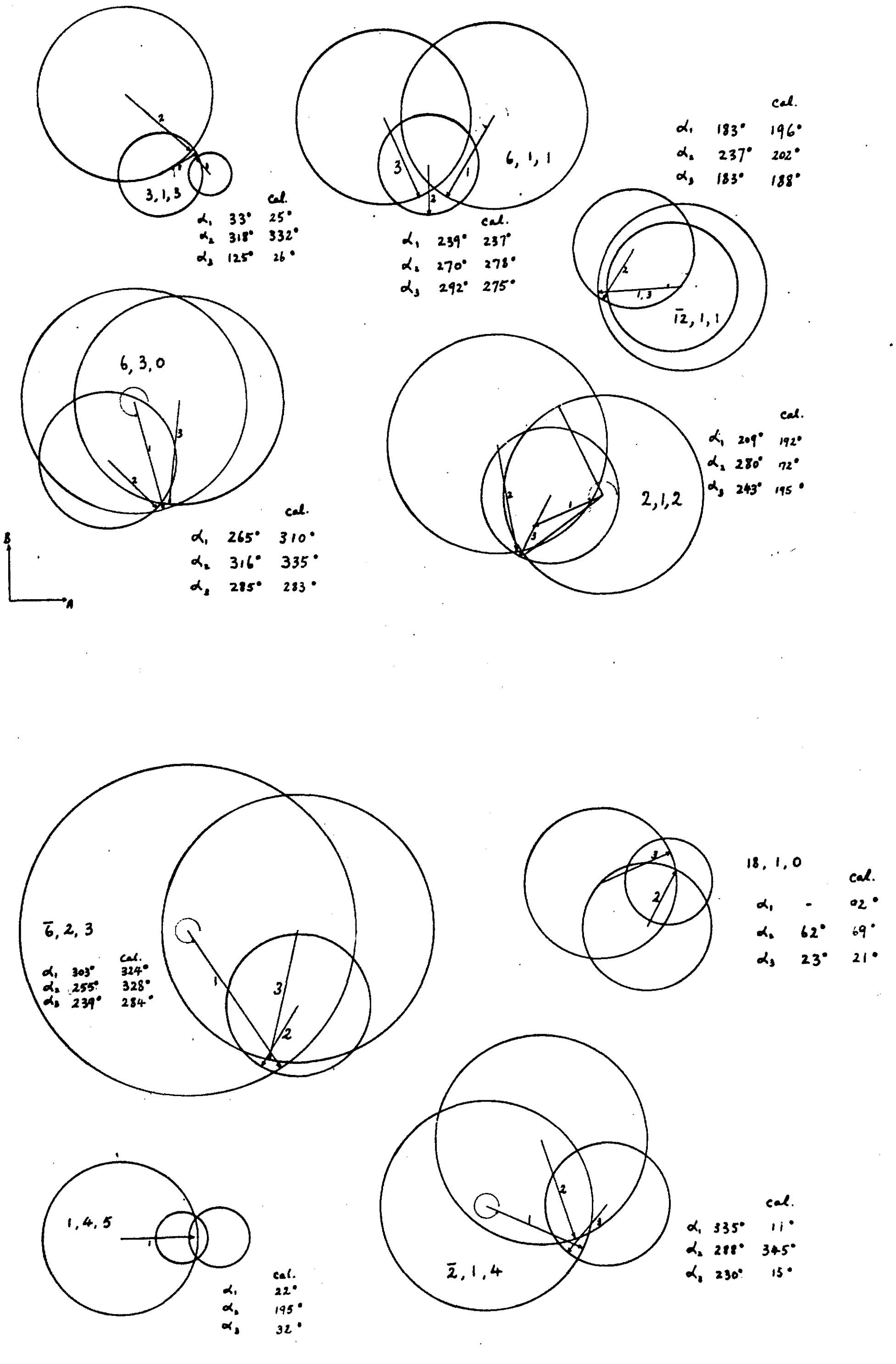
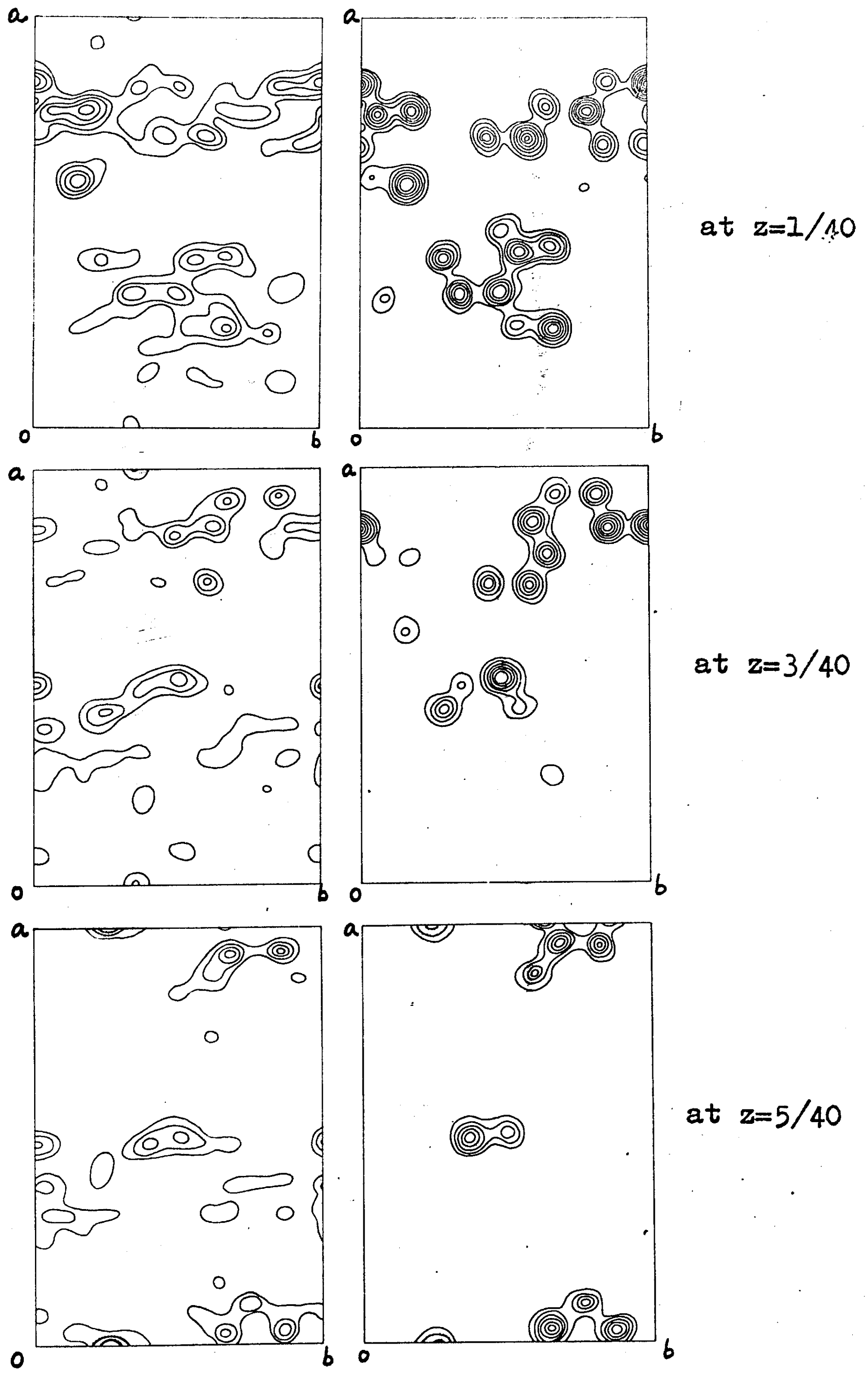


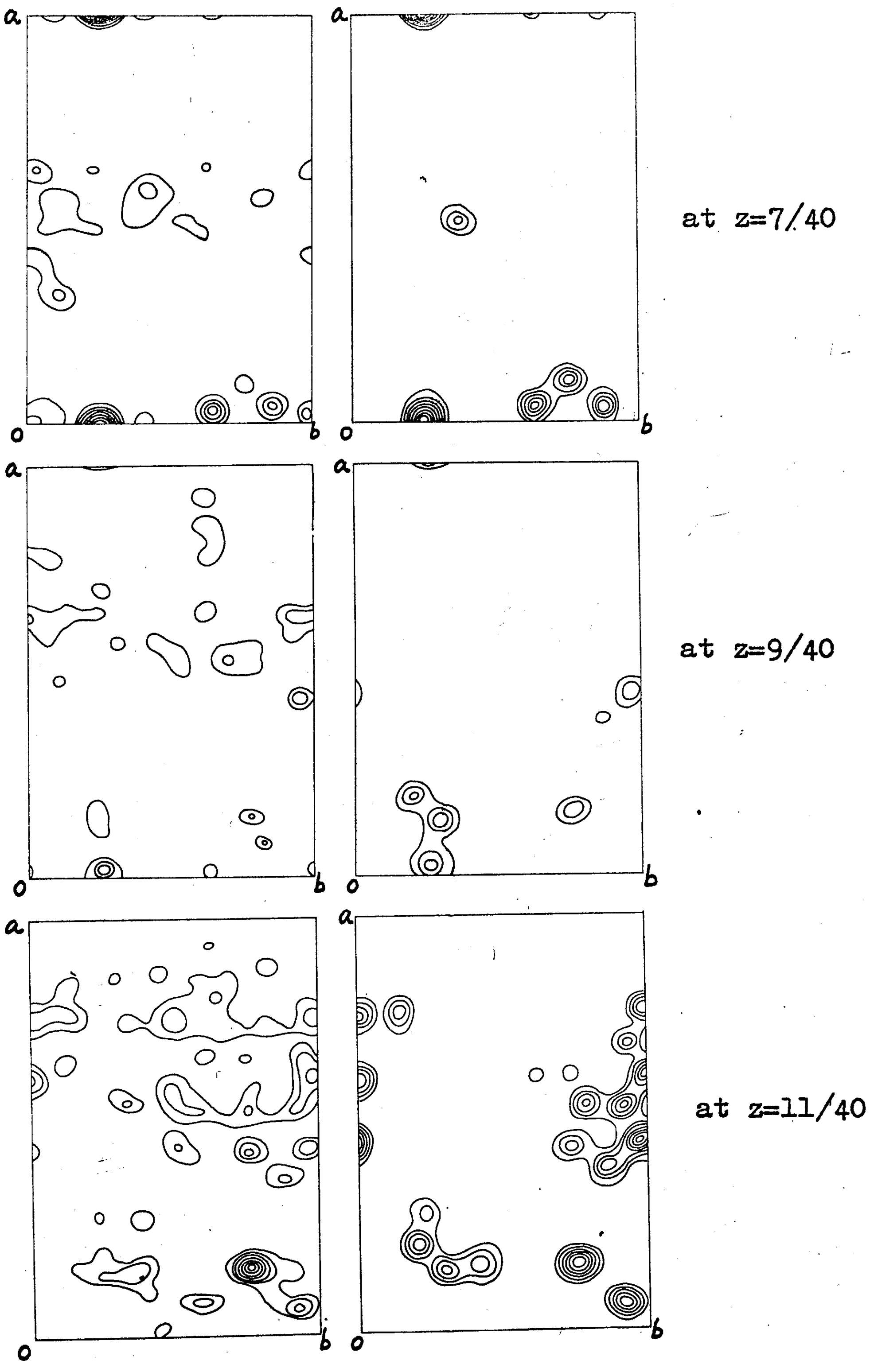
Fig.2-9. Some of the phase circles. The phase angles derived are compared with the calculated values in the final structure factors.

Fig.2-10. Aflatoxin G_1 bromobenzene solvate.



Left. Sections of the three-dimensional electron-density distribution derived by the method of isomorphous replacement.

Right. Corresponding sections of the final election-density distribution.



at z=7/40

at z=9/40

at z=11/40

Fig.2-10. (cont.)

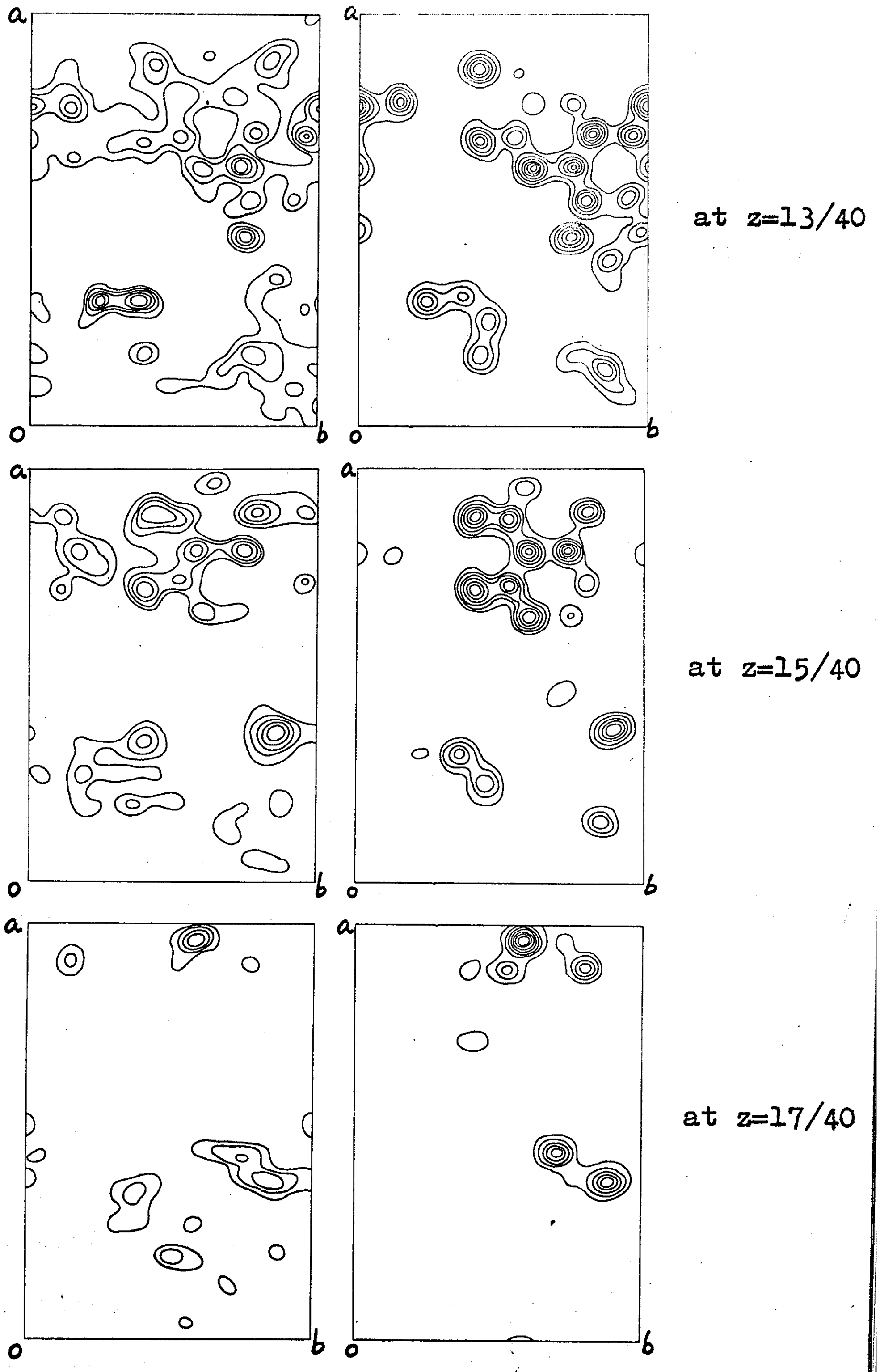


Fig.2-10. (cont.)

Forty-two atoms (all weighted as carbon) were included with the bromine atom in the calculation of a set of structure factors for the bromobenzene solvate. The discrepancy R was 0.356.

The revised values of $\alpha(hk\ell)$ were used in the calculation of a second three-dimensional electron-density distribution. This established structure (II) for aflatoxin G_1, agreeing with the result of Buchi et al. The peaks of the oxygen atoms were significantly higher than the peaks for the carbon atoms and the positions of the carbon atoms in the bromobenzene molecule were also revealed. On calculating a set of structure factors, the discrepancy R fell to 0.286. Two more rounds of Fourier and structure factor calculations further reduced R to 0.186.

2.4. REFINEMENT OF THE STRUCTURE OF THE BROMOBENZENE SOLVATE.

One least squares refinement cycle was carried out on the Deuce computer using Dr. J.S. Rollett's program.

The subsequent least squares refinement was carried out by Professor G.A. Sim in the University of Illinois using the IBM 7094 computer and the least squares program written by Gantzel, Sparks and Trueblood (1961).

Full matrix refinement was employed throughout. Since only about 130 parameters could be refined at a time and only about two thousand reflections could be included, the refinement has to be done by rotation. In the beginning, the atoms were refined isotropically. As there were some anomalous bond lengths, it was decided to allow the atoms to refine anisotropically.

The weighting scheme used is:

$$\sqrt{w} = F^* \,/\, |Fo| \quad \text{for} \quad |Fo| \geqslant F^* \quad \text{and}$$

$$\sqrt{w} = |Fo| \,/\, F^* \quad \text{for} \quad |Fo| < F^* \quad \text{where } F^* = 7.0.$$

2.5. RESULTS OF THE STRUCTURE OF THE BROMOBENZENE SOLVATE.

The final atomic coordinates and their estimated standard deviations are listed in Table 2-2. The anisotropic thermal parameters are listed in Table 2-3.

Table 2-4 lists the observed and calculated values of structure factors of the 3057 observed planes. The overall discrepancy R is 0.093.

The bond lengths and bond angles with their estimated standard deviations are listed in Tables 2-5 and 2-6.

Intramolecular bonded and non-bonded distances and bond angles involving the bromobenzene molecule are listed in Table 2-7.

Intramolecular non-bonded distances less than 3.5Å are given in Table 2-8, and intermolecular distances less than 4Å are given in Table 2-9.

Superimposed contour sections drawn taken from the fourth electron-density distribution and covering the region of one of the aflatoxin G_1 molecules in the asymmetric crystal unit are shown in Fig. 2-11.

The packing of the molecules as viewed down the unique axis and the c-axis are shown in Fig. 2-12, and Fig. 2-13, respectively.

The perspective view of one asymmetric unit is shown in Fig. 2-14.

The arrangement within an asymmetric unit is 'sandwich-like' with the bromobenzene molecule inbetween. The end furanose ring of molecule 1 bends downwards from the rest of the molecule to avoid the Br atom. Molecule 1' can effectively be obtained by turning molecule 1 through 180° and moving up about 7.5Å along the c-axis so that the solvent molecule is about 3.7Å from either of the aflatoxin molecules.

Intermolecular distances between atoms of the bromobenzene molecule and atoms of the two aflatoxin molecules are all greater than the sum of the van der Waals radii for the atoms. The shortest contact involving the bromine atom is 3.68Å for Br...O(17). There is a very close contact of 3.16Å for $C(30)...O(17)_{IV}$. *

The shortest intermolecular distances between molecule 1 and molecule 1' are 3.26Å for $O(2)...O(8')_{VI}$; 3.33Å for $O(18)...O(18')_{III}$; both greater than twice the value of 1.40Å for the van der Waals radius for oxygen. The shortest C-C contacts have values of 3.38Å for $C(12)...C(1')_{I}$; 3.43Å for $C(7)...c(24')_{I}$; while the closest C-O contacts are 3.36Å for

---

* The subscript here and for the atoms mentioned later are defined at the end of Table 2-9.

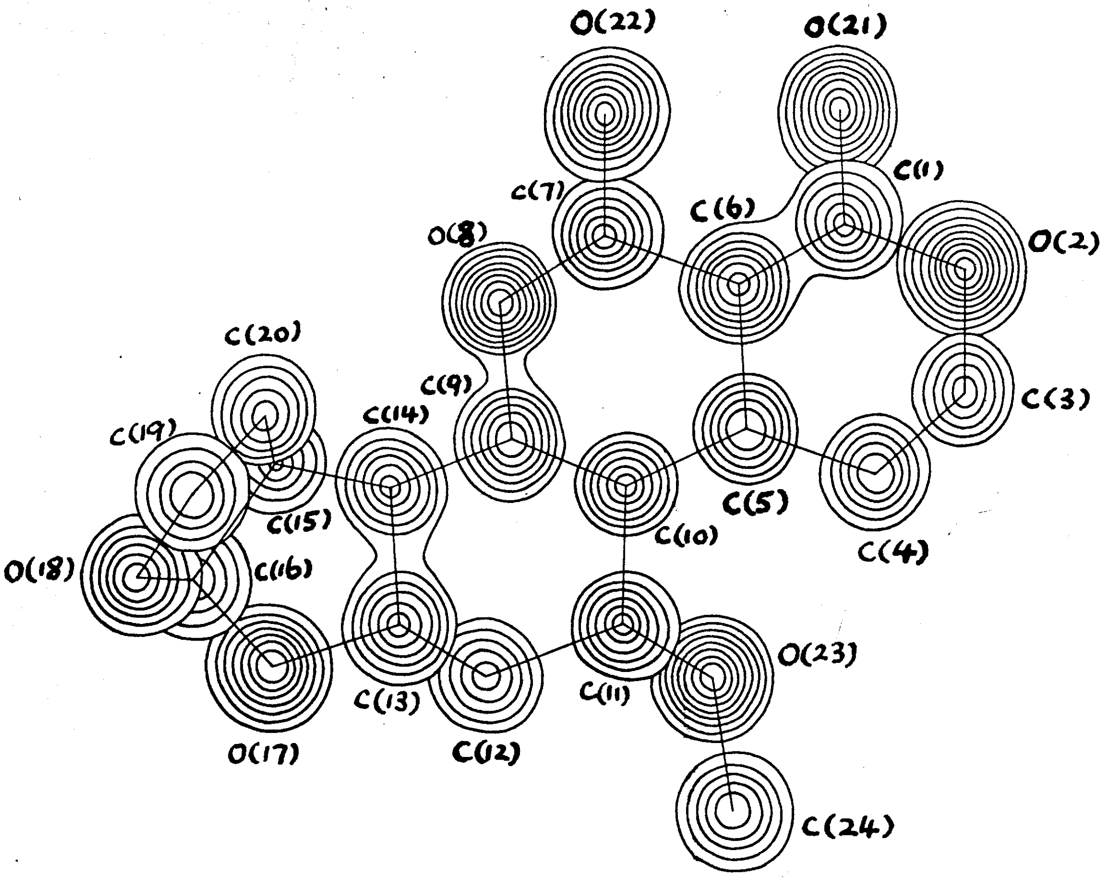


Fig.2-11. Superimposed contour sections drawn parallel to (001) showing that part of the fourth electron-density distribution which covers the region of one of the aflatoxin G_1 molecules in the bromobenzene solvate crystal. Contour interval $1eA^{-3}$.

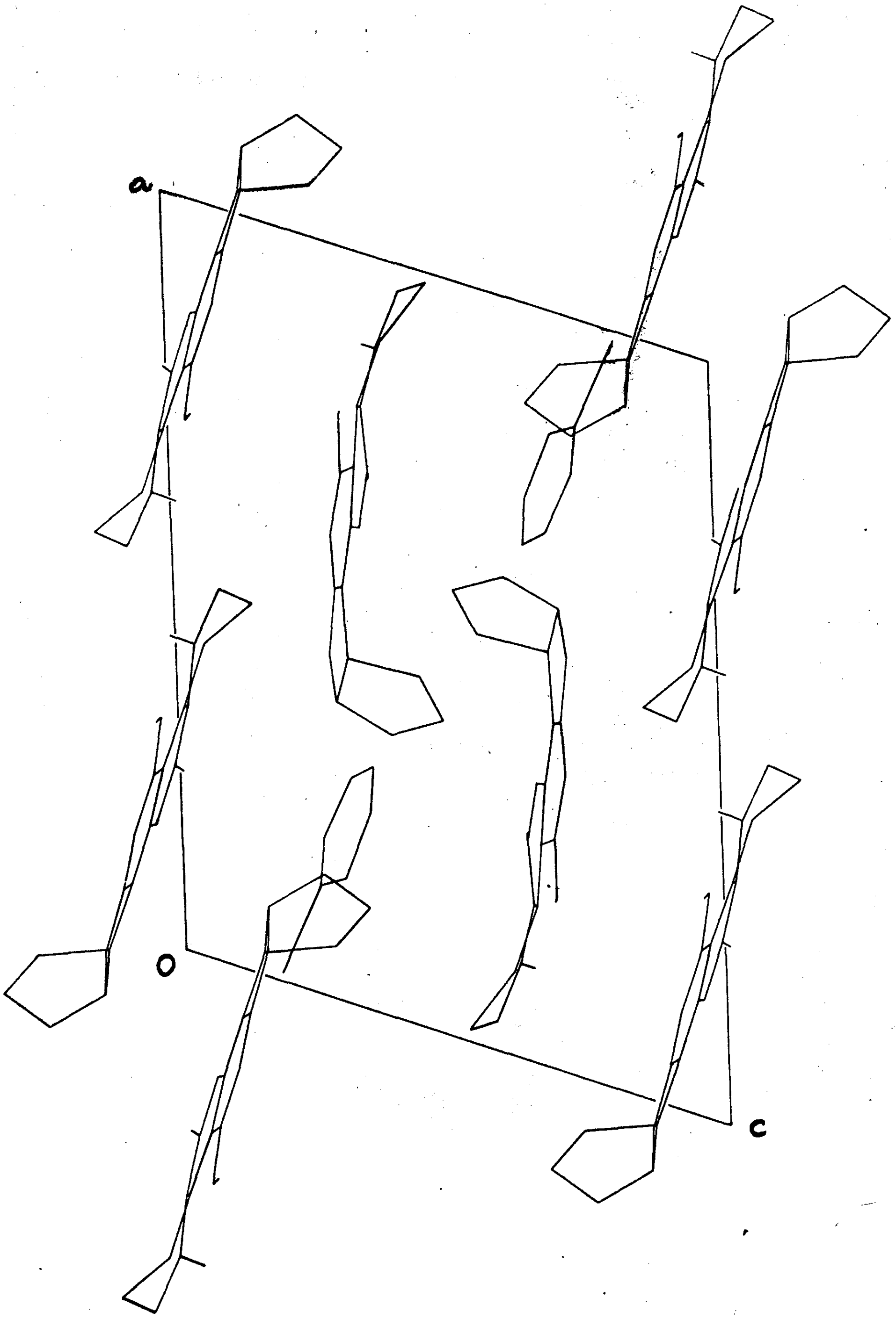
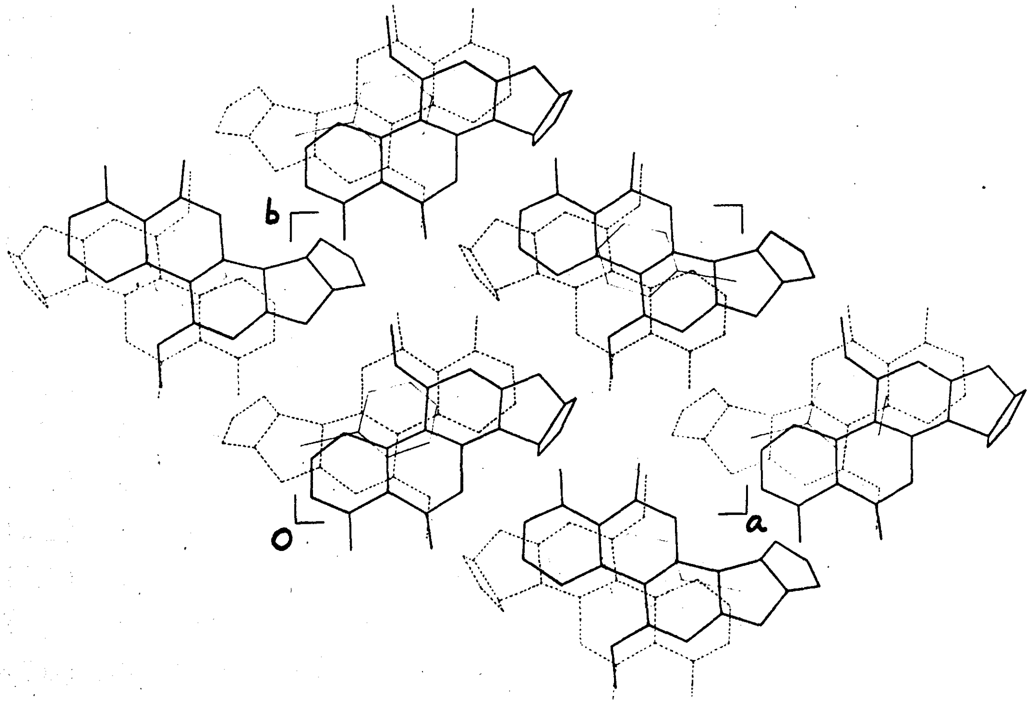


Fig.2-12. The arrangement of the molecules in the crystal of aflatoxin G_1 bromobenzene solvate as viewed along the b-axis.

Fig.2-13. The arrangement of the molecules in the crystal of aflatoxin G_1 bromobenzene solvate as viewed along the c-axis.



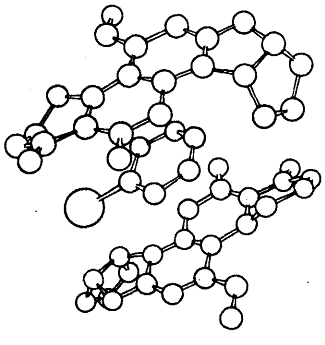


Fig.2-14. Aflatoxin G_1 bromobenzene solvate. Perspective view of one asymmetric unit.

Table 2-2 Aflatoxin G_1 bromobenzene solvate

Final fractional atomic coordinates and their estimated standard deviations in Å

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	0.4062	0.5514	0.0242	0.012	0.017	0.013
O(2)	0.4892	0.4933	0.0753	0.010	0.013	0.011
C(3)	0.4874	0.3687	0.1330	0.016	0.019	0.017
C(4)	0.4122	0.2868	0.0440	0.011	0.014	0.014
C(5)	0.3195	0.3502	0.0065	0.013	0.015	0.013
C(6)	0.3228	0.4859	0.0121	0.013	0.016	0.013
C(7)	0.2399	0.5559	-0.0121	0.014	0.016	0.014
O(8)	0.1548	0.4944	-0.0501	0.008	0.010	0.009
C(9)	0.1514	0.3612	-0.0623	0.009	0.012	0.010
C(10)	0.2303	0.2917	-0.0365	0.009	0.011	0.009
C(11)	0.2167	0.1472	-0.0554	0.013	0.014	0.012
C(12)	0.1299	0.1013	-0.0912	0.011	0.014	0.011
C(13)	0.0559	0.1798	-0.1140	0.011	0.016	0.012
C(14)	0.0639	0.3135	-0.0999	0.012	0.016	0.012
C(15)	-0.0317	0.3661	-0.1451	0.012	0.016	0.014
C(16)	-0.0914	0.2522	-0.1526	0.013	0.023	0.014
O(17)	-0.0346	0.1397	-0.1470	0.010	0.012	0.010
O(18)	-0.1617	0.2532	-0.2778	0.010	0.015	0.011
C(19)	-0.1377	0.3486	-0.3415	0.013	0.017	0.014
C(20)	-0.0661	0.4163	-0.2746	0.013	0.018	0.013
O(21)	0.4090	0.6633	-0.0118	0.010	0.013	0.011
O(22)	0.2341	0.6709	0.0035	0.010	0.013	0.012
O(23)	0.2975	0.0839	-0.0377	0.010	0.011	0.010
C(24)	0.2896	-0.0541	-0.0445	0.014	0.017	0.014
C(25)	0.1427	0.2917	0.2526	0.017	0.019	0.014
C(26)	0.2073	0.2015	0.2635	0.017	0.019	0.014
C(27)	0.2974	0.2333	0.3141	0.018	0.023	0.019
C(28)	0.3211	0.3562	0.3556	0.019	0.023	0.018
C(29)	0.2570	0.4450	0.3499	0.019	0.024	0.021
C(30)	0.1642	0.4174	0.3009	0.021	0.022	0.020
Br	0.0106	0.2500	0.1769	0.002	0	0.002

Table 2-2 (cont.)

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1')	0.1234	0.0266	0.6157	0.013	0.017	0.013
O(2')	0.0394	0.0840	0.5726	0.010	0.012	0.010
C(3')	0.0356	0.2085	0.5208	0.014	0.015	0.015
C(4')	0.1112	0.2954	0.6048	0.011	0.014	0.012
C(5')	0.2054	0.2323	0.6335	0.010	0.014	0.010
C(6')	0.2054	0.1000	0.6262	0.012	0.015	0.011
C(7')	0.2900	0.0289	0.6414	0.012	0.015	0.012
O(8')	0.3690	0.1011	0.6570	0.008	0.010	0.008
C(9')	0.3721	0.2352	0.6735	0.011	0.017	0.010
C(10')	0.2901	0.3010	0.6647	0.013	0.014	0.011
C(11')	0.3024	0.4376	0.6883	0.012	0.016	0.011
C(12')	0.3920	0.4974	0.7139	0.016	0.021	0.014
C(13')	0.4592	0.4202	0.7079	0.011	0.018	0.012
C(14')	0.4543	0.2862	0.6917	0.011	0.012	0.012
C(15')	0.5467	0.2375	0.6874	0.011	0.020	0.012
C(16')	0.6064	0.3673	0.7105	0.017	0.021	0.018
O(17')	0.5465	0.4669	0.7231	0.013	0.014	0.013
O(18')	0.6272	0.3855	0.6058	0.012	0.015	0.012
C(19')	0.5864	0.2864	0.5157	0.021	0.022	0.022
C(20')	0.5407	0.2034	0.5573	0.012	0.018	0.014
O(21')	0.1239	-0.0891	0.6447	0.010	0.012	0.011
O(22')	0.2989	-0.0846	0.6405	0.011	0.012	0.011
O(23')	0.2261	0.4990	0.6908	0.010	0.012	0.010
C(24')	0.2217	0.6389	0.6879	0.016	0.018	0.015

Table 2-3 Aflatoxin G_1 bromobenzene solvate

Anisotropic temperature factors

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	0.037	0.042	0.051	0.003	0.041	-0.001
O(2)	0.045	0.052	0.074	-0.012	0.053	-0.021
C(3)	0.052	0.035	0.064	0.007	0.035	-0.008
C(4)	0.025	0.039	0.055	-0.010	0.002	0.016
C(5)	0.044	0.014	0.046	-0.003	0.021	0.004
C(6)	0.045	0.044	0.042	-0.015	0.029	0.017
C(7)	0.052	0.019	0.066	0.009	0.059	0.011
O(8)	0.032	0.028	0.052	-0.005	0.044	0.008
C(9)	0.023	0.024	0.045	0.004	0.026	0.009
C(10)	0.024	0.029	0.033	-0.007	0.018	-0.012
C(11)	0.050	0.030	0.046	-0.008	0.029	0.007
C(12)	0.038	0.047	0.037	0.007	0.024	-0.020
C(13)	0.035	0.039	0.044	0.004	0.038	-0.012
C(14)	0.037	0.045	0.036	0.003	0.035	-0.004
C(15)	0.028	0.036	0.053	-0.011	0.019	0.001
C(16)	0.038	0.062	0.054	0.013	0.026	-0.011
O(17)	0.043	0.049	0.055	0.004	0.024	-0.017
O(18)	0.052	0.073	0.068	0.009	0.008	-0.033
C(19)	0.047	0.050	0.052	0.014	0.019	0
C(20)	0.045	0.054	0.053	0.012	0.043	0.007
O(21)	0.051	0.047	0.075	-0.005	0.055	-0.018
O(22)	0.054	0.044	0.094	-0.002	0.075	-0.005
O(23)	0.048	0.027	0.066	-0.007	0.034	0.016
C(24)	0.057	0.026	0.060	0.010	0.050	0.012
C(25)	0.076	0.061	0.043	0.014	0.053	-0.004
C(26)	0.080	0.048	0.067	-0.001	0.076	0.014
C(27)	0.080	0.065	0.076	0.013	0.065	0.009
C(28)	0.075	0.079	0.074	0.055	0.052	0.026
C(29)	0.080	0.050	0.079	0.029	0.030	-0.002
C(30)	0.082	0.057	0.076	0.038	0.033	0.023
Br	0.074	0.081	0.061	0.031	0.048	-0.001

Table 2-3 (cont.)

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1')	0.057	0.035	0.046	-0.010	0.048	-0.011
O(2')	0.048	0.048	0.057	-0.005	0.031	-0.005
C(3')	0.054	0.041	0.052	-0.007	0.023	0.012
C(4')	0.040	0.043	0.042	-0.002	0.030	0.004
C(5')	0.045	0.038	0.032	0.008	0.028	0.009
C(6')	0.050	0.037	0.037	0.005	0.038	0.005
C(7')	0.047	0.041	0.047	0	0.038	0.009
O(8')	0.046	0.034	0.054	0.012	0.049	0.012
C(9')	0.051	0.058	0.033	-0.001	0.028	-0.010
C(10')	0.053	0.042	0.033	0.007	0.030	0.004
C(11')	0.054	0.039	0.046	0.004	0.055	0.003
C(12')	0.078	0.063	0.050	-0.004	0.053	-0.040
C(13')	0.036	0.092	0.041	-0.016	0.030	-0.042
C(14')	0.043	0.026	0.049	0.002	0.030	-0.002
C(15')	0.043	0.071	0.057	0.017	0.046	-0.003
C(16')	0.065	0.069	0.067	-0.004	0.046	-0.018
O(17')	0.072	0.057	0.071	-0.017	0.065	-0.057
O(18')	0.092	0.088	0.088	0.012	0.119	-0.032
C(19')	0.070	0.047	0.089	-0.012	0.064	-0.007
C(20')	0.047	0.073	0.057	0.012	0.056	0.012
O(21')	0.061	0.044	0.065	0	0.047	-0.026
O(22')	0.075	0.034	0.071	-0.006	0.056	0.028
O(23')	0.065	0.037	0.067	-0.004	0.053	0.006
C(24')	0.077	0.039	0.064	-0.002	0.050	-0.003

Table 2-4 Aflatoxin G_1 bromobenzene solvate.

H	K	L	Fo	Fc	α
2	0	0	14.6	14.9	0
3	0	0	30.3	36.7	0
4	0	0	40.6	52.1	0
5	0	0	37.8	42.0	0
6	0	0	25.5	31.5	0
7	0	0	53.5	52.4	0
8	0	0	21.5	20.1	0
9	0	0	10.7	10.2	0
10	0	0	16.3	14.3	0
11	0	0	25.6	24.0	180
12	0	0	21.0	18.2	180
13	0	0	23.9	24.9	0
14	0	0	4.7	5.8	0
15	0	0	5.5	2.7	180
16	0	0	4.9	6.0	180
17	0	0	3.4	3.9	0
0	0	1	52.8	56.4	0
1	0	1	63.6	69.7	0
2	0	1	6.6	3.8	180
3	0	1	49.2	37.6	180
4	0	1	56.0	54.8	0
7	0	1	28.2	24.6	180
8	0	1	17.6	16.7	180
9	0	1	20.0	20.6	180
11	0	1	19.6	17.2	180
12	0	1	21.3	18.2	0
13	0	1	17.2	14.9	180
15	0	1	9.5	7.8	180
16	0	1	11.8	10.9	180
-1	0	1	11.1	5.6	180
-2	0	1	35.6	37.0	0
-3	0	1	73.3	68.8	0
-4	0	1	46.9	44.6	0
-5	0	1	27.1	33.1	0
-6	0	1	24.5	23.2	0
-7	0	1	66.5	53.9	0
-8	0	1	41.6	38.8	0
-9	0	1	24.6	20.0	0
-10	0	1	18.1	16.5	0
-11	0	1	34.7	32.4	180
-12	0	1	54.7	48.4	0
-13	0	1	48.3	41.1	0
-14	0	1	15.8	13.7	0
-15	0	1	9.7	7.7	0
-16	0	1	7.3	4.8	0
-17	0	1	6.9	7.3	0
0	0	2	28.6	29.0	180
1	0	2	62.0	66.6	180
2	0	2	44.9	43.7	180
3	0	2	16.2	17.9	180
4	0	2	43.2	44.6	180
5	0	2	45.5	51.3	180
6	0	2	55.5	56.5	180
7	0	2	23.9	22.0	180
8	0	2	10.9	11.9	180
9	0	2	21.4	20.6	180
10	0	2	18.7	18.6	180
11	0	2	77.7	70.0	180
12	0	2	12.2	11.2	180
13	0	2	19.4	17.6	180
14	0	2	5.4	5.9	180
15	0	2	10.1	8.6	180
17	0	2	2.8	0.1	0
-1	0	2	12.0	13.0	180
-2	0	2	42.2	45.8	180
-3	0	2	26.5	19.8	0
-4	0	2	25.6	21.1	0
-5	0	2	23.1	18.4	0
-6	0	2	16.6	12.1	180
-8	0	2	9.9	9.5	180
-10	0	2	29.3	25.5	0
-11	0	2	6.1	8.0	0
-12	0	2	41.9	39.3	0
-16	0	2	13.8	10.7	180
-17	0	2	9.9	8.4	0
-18	0	2	5.1	4.0	180
0	0	3	152.8	176.5	0
1	0	3	43.4	46.6	180
2	0	3	78.8	80.4	180
3	0	3	47.3	48.7	180
4	0	3	48.4	45.3	180
5	0	3	45.4	44.9	180
6	0	3	37.7	36.7	180
7	0	3	7.4	7.4	0
9	0	3	4.5	3.6	180
11	0	3	18.2	17.5	180
12	0	3	17.1	18.1	180
14	0	3	4.1	3.9	0
-1	0	3	193.3	225.0	180
-2	0	3	303.7	323.1	180
-3	0	3	66.0	68.4	180
-4	0	3	84.2	83.9	180
-5	0	3	35.1	34.4	180
-6	0	3	35.1	38.8	180
-7	0	3	27.7	24.6	180
-8	0	3	53.3	54.3	180
-10	0	3	17.8	16.0	180
-11	0	3	26.5	24.8	180
-12	0	3	21.6	20.2	180
-13	0	3	45.2	42.6	0
-15	0	3	10.0	7.7	180
-17	0	3	10.2	8.4	0
-18	0	3	3.3	0.7	180
0	0	4	24.8	24.1	180
1	0	4	6.4	2.6	0
2	0	4	28.0	26.3	180
3	0	4	36.1	31.5	180
4	0	4	8.9	5.7	0
5	0	4	33.3	36.8	0
6	0	4	4.0	6.6	180
7	0	4	12.8	11.3	0
8	0	4	13.2	12.5	180
9	0	4	12.0	12.1	0
11	0	4	5.8	5.8	0
12	0	4	17.6	14.4	0
13	0	4	4.2	3.7	0
14	0	4	9.3	9.2	0
15	0	4	6.1	5.9	0
-1	0	4	125.7	128.3	180
-2	0	4	24.1	27.3	180
-3	0	4	18.9	17.3	180
-4	0	4	12.7	15.9	180
-5	0	4	26.7	23.6	0
-6	0	4	52.0	49.9	180
-7	0	4	8.4	8.2	0
-8	0	4	36.7	36.6	180
-9	0	4	21.7	19.5	180
-10	0	4	23.2	24.5	180
-11	0	4	44.2	41.8	180
-12	0	4	28.8	23.7	0
-13	0	4	30.8	28.4	0
-14	0	4	42.2	37.2	180
-15	0	4	12.0	12.1	180
-16	0	4	13.0	14.3	180
-18	0	4	4.9	4.8	180
-19	0	4	4.2	2.4	180
0	0	5	6.1	6.5	180
1	0	5	20.2	24.7	0
2	0	5	19.9	22.8	0
3	0	5	37.6	42.3	0
4	0	5	37.4	37.0	0
5	0	5	35.3	33.5	0
6	0	5	18.9	19.7	0
7	0	5	22.5	25.4	0
8	0	5	24.5	25.5	0
9	0	5	40.7	35.9	0
10	0	5	27.9	28.1	0
12	0	5	10.3	11.0	0
13	0	5	4.5	3.1	0
14	0	5	5.2	6.0	0
-1	0	5	45.5	50.8	0
-2	0	5	44.4	36.6	180
-3	0	5	62.9	67.7	0
-4	0	5	22.1	18.0	180
-5	0	5	11.3	8.8	0
-6	0	5	4.1	4.1	180
-9	0	5	7.9	7.2	0
-10	0	5	10.6	9.5	0
-12	0	5	15.6	12.8	0
-13	0	5	24.7	25.3	180
-14	0	5	18.2	20.1	180
-15	0	5	12.0	9.4	180
-17	0	5	6.4	5.8	0
-19	0	5	4.2	3.2	0
0	0	6	63.5	71.1	0
1	0	6	41.4	43.8	0
2	0	6	25.5	24.4	0
3	0	6	43.5	43.9	0
4	0	6	16.5	17.6	0
5	0	6	10.4	10.3	0
7	0	6	17.1	17.7	0
8	0	6	9.0	9.1	0
9	0	6	10.5	11.2	0
10	0	6	7.4	8.6	0
11	0	6	7.2	7.4	0
-1	0	6	33.5	31.7	180
-2	0	6	32.3	32.4	180
-3	0	6	85.8	87.8	0
-4	0	6	63.2	63.6	0
-5	0	6	39.5	45.2	0
-6	0	6	43.1	45.0	0
-7	0	6	24.5	27.8	0
-8	0	6	13.1	14.7	0
-9	0	6	45.2	40.4	0
-10	0	6	31.0	34.4	0
-11	0	6	4.5	1.9	180
-12	0	6	11.9	1.4	0
-13	0	6	27.4	25.3	0
-14	0	6	7.6	7.8	180
-15	0	6	14.7	13.3	180
-16	0	6	3.1	4.0	0
-17	0	6	11.3	9.9	0
-18	0	6	4.2	2.1	180
-19	0	6	2.6	0.9	180
0	0	7	17.6	16.6	0
1	0	7	13.1	13.0	0
2	0	7	12.5	12.3	0
3	0	7	16.3	14.5	180
6	0	7	7.6	5.8	180
7	0	7	4.7	6.7	180
8	0	7	10.9	12.2	180
9	0	7	3.1	2.8	0
10	0	7	11.5	12.0	180
-1	0	7	23.9	21.7	180
-2	0	7	48.8	49.4	0
-3	0	7	36.8	39.7	0
-4	0	7	31.6	33.4	0
-5	0	7	34.3	35.1	0
-6	0	7	15.1	14.3	180
-9	0	7	7.5	7.9	0
-10	0	7	9.6	8.6	0
-11	0	7	6.5	5.4	0
-12	0	7	22.0	23.3	0
-13	0	7	27.1	28.2	0
-14	0	7	39.0	36.7	180
-16	0	7	3.0	6.2	0
-17	0	7	4.7	6.9	0
-18	0	7	9.8	9.0	0
0	0	8	15.9	15.5	180
1	0	8	15.3	15.8	180
2	0	8	18.5	20.2	180
3	0	8	23.1	23.8	180
4	0	8	22.1	26.1	180
5	0	8	13.9	13.3	180
6	0	8	17.3	19.9	180
7	0	8	17.6	17.4	180
8	0	8	14.5	14.5	180
11	0	8	8.9	8.0	180
-1	0	8	5.3	5.0	0
-2	0	8	24.4	21.1	180
-3	0	8	13.4	12.6	0
-4	0	8	6.7	12.1	180
-5	0	8	17.8	18.0	180
-7	0	8	13.8	13.9	180
-8	0	8	4.4	1.7	180
-9	0	8	4.5	3.4	180
-10	0	8	5.7	5.4	180
-11	0	8	13.4	12.2	180
-12	0	8	5.9	5.3	0
-13	0	8	25.0	25.5	180
-14	0	8	5.7	5.2	180
-15	0	8	4.4	4.9	0
-16	0	8	8.7	9.9	0
-17	0	8	5.8	6.3	0
-18	0	8	5.3	2.6	180
0	0	9	20.8	20.2	180
1	0	9	18.4	18.7	180
2	0	9	14.0	15.1	180
3	0	9	5.8	7.2	180
4	0	9	7.5	6.2	180
5	0	9	12.5	13.8	180
7	0	9	6.4	6.7	180
8	0	9	5.1	4.0	180
9	0	9	4.3	5.0	180
10	0	9	5.1	5.7	180
-1	0	9	21.6	22.5	180
-2	0	9	15.6	16.0	180
-3	0	9	17.7	15.4	0
-4	0	9	12.5	10.7	180
-5	0	9	24.0	23.3	180
-6	0	9	17.2	16.6	180
-7	0	9	18.0	19.1	180
-8	0	9	19.1	19.7	180
-9	0	9	6.8	8.1	180
-10	0	9	10.1	9.3	180
-11	0	9	20.9	22.6	180
-12	0	9	15.0	15.0	180
-14	0	9	14.2	13.3	180
-15	0	9	8.4	6.9	180
-16	0	9	8.1	9.3	0
-18	0	9	6.5	5.9	180
2	0	10	4.5	4.6	0
3	0	10	4.5	2.7	0
4	0	10	4.3	4.4	0
5	0	10	5.0	5.4	0
6	0	10	2.6	3.9	0
-1	0	10	5.9	7.4	180
-2	0	10	12.2	11.6	0
-4	0	10	8.9	9.3	180
-5	0	10	10.6	8.5	180
-6	0	10	18.5	22.3	180
-7	0	10	9.0	12.1	180
-8	0	10	8.3	7.3	180
-14	0	10	19.5	20.7	180
-16	0	10	14.4	13.5	0
0	0	11	9.1	9.6	0
1	0	11	10.3	11.6	0
2	0	11	12.3	14.4	0
3	0	11	7.9	7.6	0
4	0	11	4.5	3.3	0
5	0	11	8.3	9.6	0
6	0	11	5.4	5.3	0
-1	0	11	9.3	8.9	0
-2	0	11	3.3	2.1	180
-3	0	11	6.6	4.9	0
-4	0	11	7.5	7.6	0
-5	0	11	4.7	4.6	180
-6	0	11	10.0	10.7	0
-8	0	11	9.4	11.3	0
-9	0	11	6.6	7.1	0
-12	0	11	6.1	6.9	0
-14	0	11	4.6	5.5	0
-15	0	11	15.8	16.5	0
-16	0	11	7.4	6.9	0
1	0	12	2.6	3.6	0
2	0	12	3.4	4.2	0
3	0	12	7.5	8.5	0
-1	0	12	8.7	10.4	0
-2	0	12	7.9	8.1	0
-3	0	12	11.0	11.0	0
-6	0	12	5.4	5.6	0
-7	0	12	10.3	10.9	0
-8	0	12	8.7	9.4	0
-9	0	12	8.0	7.1	0
-10	0	12	8.3	9.3	0
-11	0	12	4.0	2.4	0
-12	0	12	6.6	6.5	0
-13	0	12	5.6	4.9	0
-14	0	12	6.7	7.7	0
-16	0	12	9.5	12.4	0
1	0	13	4.6	4.6	180
-3	0	13	6.3	8.0	180
-4	0	13	4.5	5.4	180
-5	0	13	4.6	5.4	180
-7	0	13	5.2	6.1	0
-10	0	13	6.1	6.8	0
-11	0	13	7.3	3.8	0
-14	0	13	2.7	2.5	180
-1	0	14	2.7	5.2	180
-2	0	14	1.5	5.9	180
-5	0	14	5.2	6.0	180
-7	0	14	2.8	2.5	180
-8	0	14	3.4	1.5	180
-1	0	14	3.5	5.6	180
-11	0	14	3.8	3.8	180
2	1	0	27.2	27.9	143
3	1	0	38.9	43.7	53
4	1	0	77.6	75.3	131
5	1	0	12.8	13.3	251
6	1	0	17.8	17.1	192
7	1	0	44.8	39.1	162
8	1	0	13.0	12.0	116
9	1	0	9.8	9.1	211
10	1	0	14.3	14.5	202
11	1	0	34.6	30.5	134
12	1	0	21.7	18.5	102
13	1	0	25.5	25.6	281
14	1	0	4.2	5.0	233
15	1	0	6.8	6.7	218
17	1	0	6.4	6.9	146
18	1	0	4.0	2.9	92
0	1	1	28.8	46.6	355
1	1	1	79.5	93.4	237
2	1	1	18.8	17.8	87
3	1	1	68.7	81.7	222
4	1	1	42.0	47.4	163
5	1	1	46.3	41.0	111
6	1	1	43.0	41.8	237
7	1	1	46.2	43.0	191
8	1	1	36.5	35.3	212
9	1	1	25.4	25.7	189
10	1	1	32.5	32.4	203
11	1	1	8.2	7.6	62
12	1	1	17.2	15.3	134
13	1	1	5.2	4.5	85
14	1	1	8.6	7.9	175
15	1	1	10.6	9.9	184
16	1	1	8.6	7.0	192
17	1	1	6.3	5.7	186
-1	1	1	47.1	50.9	143
-2	1	1	15.9	15.1	96
-3	1	1	53.6	48.7	124
-4	1	1	84.8	85.4	190
-5	1	1	42.9	42.0	150
-6	1	1	63.7	65.3	187
-7	1	1	27.5	27.0	198
-8	1	1	27.6	29.2	180
-9	1	1	31.1	33.5	135
-10	1	1	31.1	29.2	183
-11	1	1	8.2	7.2	181
-12	1	1	39.0	36.7	196
-13	1	1	26.1	22.8	99
-14	1	1	8.4	8.7	213
-15	1	1	7.0	5.6	140
-16	1	1	5.9	5.1	262
-18	1	1	5.7	4.3	323
0	1	2	68.4	68.3	179
1	1	2	57.8	49.6	50
2	1	2	91.1	99.9	192
3	1	2	21.9	29.2	218
4	1	2	32.1	33.3	183
5	1	2	37.6	36.6	166
6	1	2	27.8	30.0	166
7	1	2	28.0	31.8	146
8	1	2	23.6	23.6	176
9	1	2	9.7	9.6	195
10	1	2	17.4	17.5	208
11	1	2	19.0	18.6	125
12	1	2	10.8	10.0	37
13	1	2	10.1	8.5	262
16	1	2	3.5	2.6	289
17	1	2	4.6	3.3	301
-1	1	2	62.9	65.7	67
-2	1	2	92.6	45.3	246
-3	1	2	50.8	45.0	106
-4	1	2	45.4	45.9	271
-5	1	2	65.9	72.4	187
-6	1	2	39.4	39.8	172
-7	1	2	28.5	23.3	140
-8	1	2	38.7	39.1	193
-9	1	2	28.7	27.2	175
-10	1	2	24.1	23.5	206
-11	1	2	51.4	42.1	194
-12	1	2	18.2	15.3	229
-13	1	2	15.2	13.5	147
-14	1	2	11.2	8.7	205
-15	1	2	13.0	9.7	190
-16	1	2	8.6	7.4	174
-17	1	2	8.3	6.6	189
-18	1	2	6.9	6.6	207
-19	1	2	3.3	3.4	117
0	1	3	114.2	118.6	306
1	1	3	35.4	33.0	9
2	1	3	35.7	30.0	347
3	1	3	19.0	22.3	25
4	1	3	10.1	9.2	299
5	1	3	27.1	24.7	10
6	1	3	13.9	15.7	343
7	1	3	6.1	7.4	142
8	1	3	22.6	21.8	15
9	1	3	24.7	24.6	355
10	1	3	21.9	20.8	292
11	1	3	19.0	18.5	109
12	1	3	20.7	19.5	110
13	1	3	8.9	9.8	304
14	1	3	3.6	2.7	332
15	1	3	3.1	2.2	324
16	1	3	4.0	3.8	318
-1	1	3	120.7	114.8	27
-2	1	3	47.2	45.0	207
-3	1	3	32.1	30.2	235
-4	1	3	69.7	71.8	287
-5	1	3	49.8	49.3	90
-6	1	3	31.6	33.6	120
-7	1	3	11.3	12.5	158
-8	1	3	17.5	17.3	147
-9	1	3	19.8	18.8	192
-10	1	3	20.9	17.3	170
-11	1	3	12.4	11.7	238
-12	1	3	18.9	17.2	165
-13	1	3	29.5	27.5	115
-14	1	3	6.7	6.7	43
-15	1	3	7.8	6.1	217
-16	1	3	5.5	6.3	199
-17	1	3	10.3	10.8	236
-18	1	3	8.6	7.5	187
-19	1	3	5.2	4.5	154
0	1	4	67.9	61.8	14
1	1	4	57.3	52.6	329
2	1	4	23.5	25.5	33
3	1	4	30.7	30.9	321
4	1	4	18.2	16.2	39
5	1	4	45.6	48.5	45
6	1	4	35.2	36.2	351
7	1	4	13.7	14.1	36
8	1	4	19.0	18.9	332
9	1	4	5.9	6.4	360
10	1	4	22.4	21.1	258
11	1	4	11.9	10.7	334
13	1	4	13.5	14.1	31
14	1	4	6.0	4.8	343
15	1	4	5.0	2.9	24
-1	1	4	69.6	73.1	80
-2	1	4	48.8	45.7	17
-3	1	4	14.8	17.3	328
-4	1	4	18.0	50.8	3
-5	1	4	22.6	23.5	349
-6	1	4	23.6	27.2	346
-7	1	4	44.5	44.8	334
-8	1	4	27.3	29.7	352
-9	1	4	28.7	29.6	26
-10	1	4	13.1	12.7	328
-11	1	4	26.7	25.2	2
-12	1	4	14.0	12.4	313
-13	1	4	26.9	25.6	24
-14	1	4	24.2	21.2	351
-15	1	4	5.9	6.2	350
-16	1	4	11.6	11.4	325
-19	1	4	4.3	4.3	195
0	1	5	34.1	30.8	51
1	1	5	24.4	27.3	25
2	1	5	3.1	4.4	20
3	1	5	20.1	22.1	304
4	1	5	30.9	32.8	333
5	1	5	11.0	9.5	18
6	1	5	11.3	12.4	353
7	1	5	8.1	8.9	42
8	1	5	3.0	5.8	27
9	1	5	7.4	7.6	2
10	1	5	13.9	14.3	230
11	1	5	11.0	9.5	115
14	1	5	3.6	3.4	121
-1	1	5	12.3	14.4	328
-2	1	5	40.7	39.6	337
-3	1	5	13.0	14.6	351
-4	1	5	42.7	44.2	335
-5	1	5	9.3	7.5	30
-6	1	5	31.3	32.6	2
-7	1	5	42.9	40.6	350
-8	1	5	21.9	21.8	17
-9	1	5	23.6	21.1	339
-10	1	5	25.3	26.0	355
-11	1	5	15.9	15.1	327
-12	1	5	34.3	30.8	357
-13	1	5	36.8	32.8	26
-14	1	5	7.9	7.7	338
-15	1	5	8.8	8.6	344
-16	1	5	13.3	11.1	28
-17	1	5	6.0	7.0	355
-18	1	5	7.5	6.2	35
-19	1	5	2.9	4.8	47
0	1	6	55.0	53.5	235
1	1	6	7.8	4.9	148
2	1	6	23.5	24.5	132
3	1	6	14.9	14.1	203
4	1	6	17.8	18.5	210
5	1	6	5.6	6.7	281
6	1	6	8.2	6.7	159
7	1	6	24.9	26.5	187
8	1	6	7.4	7.8	83
10	1	6	16.6	17.4	296
11	1	6	8.2	8.2	173
12	1	6	10.4	10.2	132
13	1	6	4.5	3.9	227
-1	1	6	48.2	43.8	110
-2	1	6	89.1	90.7	172
-3	1	6	49.5	48.9	211
-4	1	6	16.9	18.5	322
-5	1	6	37.3	36.6	105
-6	1	6	30.8	30.7	82
-8	1	6	17.8	18.8	20
-9	1	6	20.7	20.7	305
-10	1	6	7.7	7.2	263
-11	1	6	9.8	9.5	345
-12	1	6	15.0	15.6	42
-14	1	6	27.3	25.5	357
-15	1	6	11.3	12.0	249
-16	1	6	6.7	4.7	345
-17	1	6	10.2	8.5	298
-18	1	6	2.1	4.4	60
-19	1	6	8.1	7.5	28
0	1	7	18.5	18.8	170
1	1	7	21.4	21.8	214
2	1	7	21.5	19.8	172
3	1	7	18.7	21.2	207
4	1	7	19.1	17.1	171
5	1	7	11.4	10.5	156
6	1	7	7.4	6.4	78
7	1	7	8.9	11.0	155
8	1	7	8.2	7.6	111
9	1	7	6.7	6.4	132
10	1	7	8.3	10.0	217
11	1	7	7.9	7.5	264
12	1	7	5.3	6.1	179
-1	1	7	67.5	69.0	148
-2	1	7	35.5	36.8	207
-3	1	7	46.1	48.9	219
-4	1	7	9.4	11.4	165
-5	1	7	36.1	34.6	193
-6	1	7	44.1	38.2	184
-7	1	7	12.0	14.7	214
-8	1	7	17.6	16.2	174
-9	1	7	13.1	13.6	176
-10	1	7	20.4	21.4	162
-11	1	7	7.7	6.8	169
-12	1	7	16.3	14.9	189
-14	1	7	13.6	12.4	158
-15	1	7	13.7	13.5	193
-16	1	7	10.7	10.3	201
-17	1	7	2.4	3.2	173
-18	1	7	6.7	6.8	116
-19	1	7	2.6	2.7	213
0	1	8	20.8	19.7	115
1	1	8	4.0	3.5	86
2	1	8	16.4	17.8	143
3	1	8	7.8	6.8	212
4	1	8	8.5	9.1	268
5	1	8	4.2	4.3	180
6	1	8	5.1	5.1	253
7	1	8	4.0	4.5	180
8	1	8	8.0	8.1	35
9	1	8	5.9	5.8	18
10	1	8	4.2	4.3	274
11	1	8	3.2	2.7	69
-1	1	8	20.8	20.3	236
-2	1	8	8.9	7.7	230
-3	1	8	22.0	21.8	198
-4	1	8	16.9	17.5	159
-5	1	8	25.3	24.2	163
-6	1	8	20.1	20.1	164
-7	1	8	11.0	12.6	226
-8	1	8	31.3	32.1	156
-9	1	8	11.5	11.7	192
-10	1	8	16.8	16.9	182
-11	1	8	9.9	10.5	172
-12	1	8	9.9	9.6	201
-13	1	8	19.8	19.4	123
-14	1	8	24.8	25.8	190
-15	1	8	20.5	18.8	193
-16	1	8	4.4	4.3	137
-17	1	8	6.8	6.9	179
-18	1	8	5.3	4.1	157
-19	1	8	3.0	3.2	168
0	1	9	18.2	18.4	290
1	1	9	16.1	17.4	3
2	1	9	16.7	17.4	56
3	1	9	7.9	7.6	3
4	1	9	4.2	4.2	291
5	1	9	14.0	14.6	352
6	1	9	4.7	3.6	24
8	1	9	5.9	6.4	78
9	1	9	6.3	6.5	40
10	1	9	8.1	10.8	336
-1	1	9	21.5	24.1	40
-2	1	9	5.8	7.4	134
-3	1	9	26.6	27.2	318
-4	1	9	34.0	36.9	6
-5	1	9	22.3	21.2	61
-6	1	9	5.0	7.7	24
-8	1	9	7.8	9.3	340
-9	1	9	13.6	12.4	257
-10	1	9	10.8	12.1	205
-11	1	9	7.9	8.3	136
-12	1	9	7.9	9.1	92
-13	1	9	11.6	12.0	213
-15	1	9	13.1	13.2	194
-16	1	9	10.4	9.5	172
-18	1	9	7.3	7.9	138
0	1	10	12.0	11.8	352
1	1	10	13.6	14.1	341
2	1	10	6.5	5.3	340
3	1	10	7.5	6.2	348
5	1	10	5.7	4.7	307
6	1	10	8.0	8.1	6
8	1	10	4.8	6.2	40
-1	1	10	13.7	15.1	65
-2	1	10	26.8	28.7	326
-3	1	10	31.3	35.1	338
-4	1	10	29.8	32.7	18
-5	1	10	14.3	15.8	8
-6	1	10	4.2	3.3	16
-7	1	10	23.0	22.8	5
-8	1	10	15.3	13.6	4
-9	1	10	11.6	12.4	356
-10	1	10	8.9	9.6	69
-11	1	10	13.5	13.9	16
-12	1	10	7.6	6.4	335
-13	1	10	11.4	11.5	329
-14	1	10	7.8	8.1	86
-15	1	10	9.0	7.5	212
-16	1	10	6.4	6.2	344
-17	1	10	4.4	4.0	20
-18	1	10	3.0	5.8	35
0	1	11	6.3	6.2	33
1	1	11	6.1	6.0	353
2	1	11	5.9	7.1	90
6	1	11	5.2	5.7	226
-1	1	11	10.0	10.1	288
-2	1	11	11.3	11.6	337
-4	1	11	11.5	11.7	41
-5	1	11	9.4	8.6	20
-6	1	11	10.3	10.4	359
-7	1	11	15.1	13.5	325
-8	1	11	4.1	4.9	45
-9	1	11	8.2	8.5	349
-10	1	11	9.0	9.1	344
-11	1	11	3.9	5.9	16
-12	1	11	9.3	9.7	339
-13	1	11	8.8	9.6	66
-15	1	11	11.7	10.5	321
-16	1	11	11.1	13.2	6
-17	1	11	7.1	7.8	11
0	1	12	9.2	9.9	218
1	1	12	4.0	4.7	204
2	1	12	4.8	5.7	132
3	1	12	6.3	6.8	170
4	1	12	6.0	6.9	197
-1	1	12	4.4	5.6	150
-2	1	12	8.3	10.0	155
-3	1	12	8.1	10.1	248
-4	1	12	7.2	7.8	62
-5	1	12	8.2	11.1	157
-6	1	12	8.2	9.6	215
-7	1	12	6.1	7.4	218
-9	1	12	7.1	7.3	242
-10	1	12	4.5	6.7	184
-11	1	12	5.0	4.7	92
-12	1	12	3.3	4.1	40
-13	1	12	3.1	4.4	267
-14	1	12	5.9	6.9	340
0	1	13	4.5	5.0	182
1	1	13	4.9	3.9	207
-1	1	13	7.9	9.2	147
-2	1	13	5.7	5.9	242
-4	1	13	12.7	14.3	140
-5	1	13	14.3	16.8	174
-6	1	13	14.0	16.2	190
-7	1	13	4.1	5.5	163
-8	1	13	4.6	3.5	217
-9	1	13	10.1	10.3	181
-10	1	13	6.1	6.3	146
-11	1	13	3.6	3.8	149
-12	1	13	5.7	6.4	242
-13	1	13	4.7	6.9	227
-14	1	13	6.3	6.9	130
-3	1	14	6.6	8.9	159
-4	1	14	4.0	4.5	136
-5	1	14	4.2	3.5	183
-6	1	14	4.6	4.7	217
-8	1	14	6.2	6.9	182
-9	1	14	2.9	1.9	108
-10	1	14	3.0	3.4	185
1	2	0	53.5	93.0	160
2	2	0	81.6	86.8	187
3	2	0	39.1	40.7	166
4	2	0	50.6	52.8	127
5	2	0	95.2	99.7	140
6	2	0	31.4	30.7	242
7	2	0	37.3	37.1	239
8	2	0	10.5	10.3	164
9	2	0	32.5	30.4	166
10	2	0	12.3	11.9	171
11	2	0	18.4	17.0	194
12	2	0	14.5	13.3	228
13	2	0	10.6	10.7	233
14	2	0	4.2	3.8	181
15	2	0	6.9	4.9	216
16	2	0	3.5	2.3	307
17	2	0	2.9	2.7	139
0	2	1	44.2	41.6	98
1	2	1	34.6	26.6	96
2	2	1	28.9	30.2	281
3	2	1	35.7	40.3	147
4	2	1	35.7	29.2	247
5	2	1	45.7	42.8	197
6	2	1	18.5	15.0	32
7	2	1	16.4	17.2	28
8	2	1	17.6	15.3	29
9	2	1	14.6	12.4	112
10	2	1	15.6	13.3	21
11	2	1	14.0	13.8	330
12	2	1	6.8	4.7	267
13	2	1	5.3	4.6	356
14	2	1	6.7	8.6	340
15	2	1	4.6	6.3	29
17	2	1	8.6	9.0	112
-1	2	1	113.8	133.9	204
-2	2	1	54.8	51.1	216
-3	2	1	49.5	45.9	201
-4	2	1	31.5	32.9	193
-5	2	1	27.0	28.1	149
-6	2	1	79.7	71.6	288
-7	2	1	36.1	29.8	111
-8	2	1	49.7	44.1	167
-9	2	1	29.5	26.4	187
-10	2	1	36.1	28.8	156
-11	2	1	20.6	18.0	193
-12	2	1	10.8	9.5	160
-13	2	1	14.0	11.1	161
-14	2	1	18.3	16.7	174
-15	2	1	10.5	10.6	173
-16	2	1	7.0	6.9	164
-17	2	1	8.2	7.2	151
-18	2	1	4.6	4.9	141
0	2	2	69.7	80.1	57
1	2	2	45.0	42.5	326
2	2	2	30.7	26.8	41
3	2	2	25.8	27.0	327
4	2	2	23.4	20.4	43
5	2	2	11.1	9.9	90
6	2	2	83.0	86.1	319
7	2	2	28.4	29.6	24
8	2	2	26.1	24.2	341
9	2	2	12.5	11.6	299
10	2	2	12.1	11.9	9
11	2	2	17.2	15.4	13
12	2	2	22.9	20.4	347
13	2	2	14.6	13.9	14
14	2	2	12.1	11.7	331
15	2	2	4.9	5.4	4
16	2	2	8.0	6.2	328
17	2	2	3.1	3.6	121
-1	2	2	61.0	66.7	21
-2	2	2	22.9	13.6	123
-3	2	2	12.0	10.4	359
-4	2	2	14.8	14.2	33
-5	2	2	20.7	13.7	247
-6	2	2	70.1	63.2	39
-7	2	2	40.2	36.4	20
-8	2	2	3.2	4.4	64
-9	2	2	18.1	15.8	138
-10	2	2	11.5	9.6	299
-11	2	2	22.3	17.1	295
-12	2	2	14.2	13.8	197
-13	2	2	8.6	7.2	167
-14	2	2	4.3	2.5	195
-15	2	2	8.9	8.3	151
-16	2	2	6.1	5.5	155
-17	2	2	8.7	8.5	146
-18	2	2	4.7	6.5	290
0	2	3	94.1	97.5	344
1	2	3	48.9	49.5	348
2	2	3	40.8	40.6	323
3	2	3	63.8	61.6	345
4	2	3	42.5	44.4	20
5	2	3	38.2	39.2	70
6	2	3	4.8	6.7	302
7	2	3	28.8	30.2	330
8	2	3	22.7	21.3	14
9	2	3	7.1	7.1	10
10	2	3	16.3	16.8	53
11	2	3	11.1	11.0	70
12	2	3	11.7	11.6	24
13	2	3	4.9	4.4	289
16	2	3	3.5	2.0	306
-1	2	3	81.2	85.3	333
-2	2	3	63.8	65.6	8
-3	2	3	53.6	53.1	351
-4	2	3	36.1	39.6	20
-5	2	3	46.5	45.0	64
-6	2	3	83.0	76.5	324
-7	2	3	49.7	52.0	309
-8	2	3	26.6	27.3	54
-9	2	3	13.4	14.4	3
-10	2	3	22.8	22.6	15
-11	2	3	18.8	18.4	316
-12	2	3	7.8	8.1	30
-13	2	3	14.9	14.0	3
-14	2	3	11.5	10.9	77
-15	2	3	7.3	5.1	334
-17	2	3	5.0	3.5	355
-18	2	3	2.1	1.3	152
-19	2	3	5.6	5.6	21
0	2	4	16.2	16.1	70
1	2	4	26.4	28.7	3
2	2	4	19.4	26.1	320
3	2	4	4.3	2.5	233
4	2	4	3.2	3.9	266
5	2	4	30.7	30.7	213
6	2	4	18.2	16.8	176
7	2	4	16.2	14.8	199
8	2	4	4.1	3.4	109
9	2	4	7.4	8.0	202
10	2	4	7.5	6.0	90
11	2	4	4.3	2.9	280
12	2	4	6.4	5.8	162
13	2	4	2.6	2.9	225
15	2	4	3.0	2.9	287
-1	2	4	30.5	29.7	250
-2	2	4	38.3	33.2	331
-3	2	4	56.6	57.7	0
-4	2	4	31.6	28.8	45
-5	2	4	43.2	36.7	355
-6	2	4	45.0	38.4	325
-7	2	4	25.5	22.0	61
-8	2	4	29.1	32.8	124
-9	2	4	29.0	28.1	7
-10	2	4	10.7	12.3	38
-11	2	4	27.4	24.7	342
-12	2	4	19.4	18.4	21
-13	2	4	23.2	20.2	1
-14	2	4	3.0	1.2	97
-15	2	4	12.0	9.9	340
-16	2	4	10.1	9.6	348
-17	2	4	5.7	4.9	358
-18	2	4	5.2	5.4	339
-19	2	4	8.4	8.9	321
0	2	5	26.8	26.7	124
1	2	5	11.9	14.1	231
2	2	5	24.7	25.7	154
3	2	5	16.7	15.7	201
4	2	5	45.5	47.2	152
5	2	5	34.5	34.0	156
6	2	5	15.2	17.7	221
7	2	5	9.8	9.5	141
8	2	5	10.5	10.8	168
9	2	5	17.9	21.2	218
10	2	5	20.2	19.0	168
11	2	5	11.5	10.3	164
12	2	5	7.9	8.1	162
13	2	5	6.4	6.2	157
14	2	5	5.3	4.7	189
-1	2	5	28.7	29.0	199
-2	2	5	28.2	27.5	170
-3	2	5	25.6	25.8	278
-4	2	5	10.4	11.7	223
-5	2	5	12.5	12.0	214
-6	2	5	17.5	15.7	197
-7	2	5	4.6	3.5	112
-8	2	5	50.5	50.7	214
-9	2	5	21.0	19.9	173
-10	2	5	12.5	14.0	252
-11	2	5	13.4	12.2	302
-12	2	5	7.9	7.5	137
-13	2	5	4.3	4.7	70
-15	2	5	5.2	5.3	75
-16	2	5	3.9	2.0	275
-17	2	5	7.6	5.1	42
-18	2	5	6.8	6.9	297
-19	2	5	6.1	5.4	322
0	2	6	34.6	38.3	178
1	2	6	20.9	20.9	186
2	2	6	36.7	39.2	195
3	2	6	15.5	17.1	200
4	2	6	7.9	10.0	207
5	2	6	21.7	23.0	137
6	2	6	21.6	19.9	202
7	2	6	3.0	5.1	214
8	2	6	3.0	2.7	273
9	2	6	7.9	9.0	245
10	2	6	7.5	6.9	186
11	2	6	8.2	8.3	135
12	2	6	5.0	5.0	75
13	2	6	3.1	3.5	175
-1	2	6	18.4	18.2	221
-2	2	6	68.4	68.0	156
-3	2	6	43.1	43.1	161
-4	2	6	59.7	55.8	183
-5	2	6	33.6	35.6	134
-6	2	6	23.4	23.1	237
-7	2	6	43.2	42.2	213
-8	2	6	52.1	47.7	150
-9	2	6	20.2	20.2	188
-10	2	6	12.6	14.4	199
-11	2	6	13.4	11.8	183
-12	2	6	12.5	12.6	175
-13	2	6	12.6	9.7	92
-14	2	6	6.8	6.8	178
-15	2	6	5.9	4.6	222
-16	2	6	4.8	5.3	262
-17	2	6	4.3	3.3	176
-18	2	6	3.6	2.7	169
-19	2	6	2.1	4.6	92
0	2	7	6.4	10.5	135
1	2	7	11.2	11.7	85
2	2	7	2.8	4.7	301
3	2	7	13.9	13.9	7
4	2	7	8.9	9.3	24
5	2	7	17.7	19.9	356
7	2	7	4.3	5.4	60
9	2	7	2.8	3.9	284
10	2	7	2.5	2.3	325
11	2	7	3.8	3.6	252
12	2	7	2.4	3.1	70
-1	2	7	17.8	17.8	129
-2	2	7	3.6	2.2	207
-3	2	7	11.5	10.9	80
-4	2	7	14.7	15.0	161
-5	2	7	6.2	5.4	175
-6	2	7	19.8	18.7	232
-7	2	7	36.3	31.7	140
-8	2	7	20.6	20.0	166
-9	2	7	6.9	6.2	87
-10	2	7	12.1	11.7	231
-11	2	7	16.5	17.7	219
-12	2	7	9.2	8.9	154
-13	2	7	9.7	9.6	225
-14	2	7	14.8	14.9	179
-15	2	7	5.0	3.4	244
-16	2	7	4.6	2.8	220
-17	2	7	5.3	4.2	141
-18	2	7	5.3	5.2	169
-19	2	7	3.1	2.0	238
0	2	8	14.4	13.2	3
1	2	8	10.2	11.9	10
2	2	8	20.1	22.0	347
3	2	8	20.4	21.0	349
4	2	8	14.4	14.4	14
5	2	8	11.8	13.0	39
6	2	8	10.4	11.2	348
7	2	8	12.8	14.6	19
8	2	8	10.4	11.8	5
9	2	8	13.0	11.5	319
11	2	8	5.7	5.8	2
-2	2	8	11.5	11.9	2
-3	2	8	24.8	26.4	345
-4	2	8	9.6	10.3	345
-5	2	8	9.2	9.6	57
-6	2	8	4.8	3.2	267
-7	2	8	14.8	14.7	15
-8	2	8	4.1	4.9	274
-9	2	8	16.0	16.4	26
-10	2	8	14.5	12.9	24
-11	2	8	15.9	16.2	1
-12	2	8	7.5	7.8	102
-13	2	8	7.4	5.6	24
-14	2	8	6.6	5.8	162
-15	2	8	2.8	2.0	75
-16	2	8	5.1	4.2	330
-17	2	8	3.9	2.6	47
-18	2	8	5.2	3.6	334
0	2	9	8.6	9.7	48
1	2	9	17.9	19.0	356
2	2	9	9.2	8.9	9
3	2	9	10.6	11.6	354
4	2	9	9.5	9.4	334
5	2	9	5.8	6.7	47
6	2	9	6.7	6.7	254
7	2	9	3.5	3.9	71
8	2	9	3.8	5.1	8
9	2	9	5.5	5.6	319
-1	2	9	20.2	22.6	338
-2	2	9	13.4	15.5	71
-3	2	9	10.7	12.1	354
-4	2	9	17.3	18.0	328
-5	2	9	29.8	32.7	12
-6	2	9	28.6	26.5	353
-7	2	9	17.5	17.6	325
-8	2	9	17.1	19.1	34
-9	2	9	20.5	21.7	354
-10	2	9	27.3	26.9	347
-11	2	9	7.5	8.7	35
-12	2	9	16.0	16.5	5
-13	2	9	5.9	5.9	63
-14	2	9	5.6	6.2	329
-15	2	9	4.5	3.2	248
-16	2	9	4.7	5.3	337
-17	2	9	3.5	2.1	123
1	2	10	6.7	7.3	159
2	2	10	4.2	4.9	234
3	2	10	7.6	7.7	206
4	2	10	3.8	2.3	185
5	2	10	4.4	5.6	326
7	2	10	4.4	4.6	105
8	2	10	3.7	5.0	119
-1	2	10	7.5	5.6	66
-2	2	10	8.1	7.9	230
-3	2	10	10.6	9.8	269
-4	2	10	6.1	4.6	281
-6	2	10	4.3	6.2	331
-7	2	10	9.2	11.0	143
-8	2	10	15.0	14.1	12
-9	2	10	11.8	11.3	337
-10	2	10	9.6	9.8	339
-11	2	10	8.4	9.6	276
-12	2	10	7.1	7.6	56
-13	2	10	8.8	9.3	321
-15	2	10	5.9	6.4	5
0	2	11	4.0	3.8	170
1	2	11	11.4	14.1	180
2	2	11	8.4	8.7	184
3	2	11	7.8	9.8	213
4	2	11	7.1	7.9	177
5	2	11	7.4	7.4	176
6	2	11	7.1	8.3	179
-1	2	11	12.1	14.4	197
-2	2	11	3.0	2.9	266
-3	2	11	5.2	5.0	271
-4	2	11	5.2	7.7	185
-5	2	11	10.9	12.3	165
-6	2	11	9.5	10.9	189
-7	2	11	3.0	3.0	187
-8	2	11	3.0	3.5	149
-9	2	11	2.9	3.9	129
-10	2	11	2.9	3.0	154
-11	2	11	4.8	4.6	190
-12	2	11	6.0	6.4	183
-13	2	11	2.5	3.6	213
-14	2	11	6.6	6.2	163
-15	2	11	5.5	4.2	231
-16	2	11	4.3	4.3	316
0	2	12	7.8	9.0	168
1	2	12	5.6	4.9	176
2	2	12	5.2	4.4	121
3	2	12	4.1	4.0	162
4	2	12	1.5	1.8	93
-1	2	12	5.1	5.9	227
-2	2	12	6.5	6.4	139
-3	2	12	6.6	6.0	192
-4	2	12	2.7	4.1	253
-5	2	12	4.8	4.7	110
-6	2	12	6.8	8.1	162
-7	2	12	11.4	14.3	194
-8	2	12	8.6	9.0	[illegible]
-9	2	12	6.4	9.6	184
-10	2	12	6.4	7.6	202
-11	2	12	12.2	13.8	164
-12	2	12	5.7	6.4	166
-13	2	12	6.5	8.8	167
-14	2	12	7.5	8.3	205
0	2	13	1.8	2.8	38
-1	2	13	4.4	5.6	24
-2	2	13	4.7	5.3	191
-3	2	13	5.5	6.3	308
-4	2	13	4.6	5.8	30
-5	2	13	5.2	5.3	73
-7	2	13	2.3	4.4	150
-8	2	13	3.3	4.0	8
-9	2	13	2.2	2.9	248
-10	2	13	4.8	3.9	183
-11	2	13	5.7	6.6	166
-12	2	13	2.6	3.3	125
-13	2	13	2.8	2.0	172
-3	2	14	4.2	7.0	35
-4	2	14	2.7	3.1	54
-5	2	14	3.3	4.8	85
-6	2	14	2.9	4.6	359
-7	2	14	2.9	3.5	8
-8	2	14	4.7	6.4	8
1	3	0	15.1	13.9	339
2	3	0	8.6	8.3	245
3	3	0	18.4	18.0	16
4	3	0	23.7	25.3	27
5	3	0	43.9	43.0	36
6	3	0	61.4	56.3	310
7	3	0	14.4	13.4	146
8	3	0	17.9	17.4	348
9	3	0	10.4	10.1	329
10	3	0	27.1	22.7	11
11	3	0	16.0	16.1	352
12	3	0	10.1	7.9	9
13	3	0	3.8	4.3	359
15	3	0	4.9	5.0	358
16	3	0	3.0	3.1	304
17	3	0	5.7	4.1	330
0	3	1	43.3	49.8	4
1	3	1	41.9	39.6	11
2	3	1	39.5	45.9	352
3	3	1	73.7	81.4	2
4	3	1	18.6	19.7	355
5	3	1	75.4	86.2	16
6	3	1	61.8	62.1	29
7	3	1	35.3	33.7	319
8	3	1	30.3	30.2	11
9	3	1	21.8	20.5	1
10	3	1	24.0	18.6	356
11	3	1	5.3	5.8	263
12	3	1	6.7	4.5	31
13	3	1	12.0	12.7	15
14	3	1	6.7	5.6	350
15	3	1	7.3	7.3	21
-1	3	1	55.1	64.3	332
-2	3	1	81.5	85.9	26
-3	3	1	65.9	65.7	358
-4	3	1	53.3	51.6	11
-5	3	1	8.4	7.8	10
-6	3	1	33.0	28.8	257
-7	3	1	20.1	19.5	35
-8	3	1	18.1	16.7	283
-9	3	1	10.4	9.4	77
-10	3	1	17.5	15.8	289
-11	3	1	10.6	8.1	8
-12	3	1	8.5	7.3	153
-13	3	1	6.1	5.4	149
-14	3	1	7.6	7.1	333
-15	3	1	5.0	4.1	310
-16	3	1	3.2	3.7	32
-17	3	1	6.1	4.5	308
-18	3	1	7.2	7.6	184
0	3	2	56.8	60.4	17
1	3	2	61.6	58.0	47
2	3	2	25.6	26.7	305
3	3	2	14.8	12.8	280
4	3	2	16.0	16.5	282
5	3	2	20.0	17.4	337
6	3	2	24.5	26.2	20
7	3	2	28.4	27.9	343
8	3	2	7.5	10.1	27
9	3	2	18.3	17.4	325
10	3	2	4.6	3.3	135
11	3	2	7.7	6.7	189
12	3	2	6.1	7.8	271
13	3	2	7.8	7.2	331
15	3	2	2.1	2.2	359
16	3	2	6.7	5.4	191
-1	3	2	54.5	58.3	1
-2	3	2	30.9	28.7	9
-3	3	2	29.4	28.8	13
-4	3	2	52.4	56.7	351
-5	3	2	35.8	39.3	340
-6	3	2	41.1	41.9	324
-7	3	2	82.0	88.7	30
-8	3	2	28.4	28.7	15
-9	3	2	31.9	32.1	12
-10	3	2	27.4	24.9	344
-11	3	2	23.8	19.5	349
-12	3	2	11.3	9.6	358
-13	3	2	5.4	6.1	255
-14	3	2	4.7	2.9	356
-15	3	2	8.2	8.3	4
-16	3	2	9.2	7.3	15
-17	3	2	7.4	6.6	27
-18	3	2	2.8	1.8	264
0	3	3	22.9	21.5	243
1	3	3	19.5	17.5	41
2	3	3	27.6	28.0	227
3	3	3	4.3	0.8	142
4	3	3	36.8	40.1	160
5	3	3	31.0	32.1	69
6	3	3	12.3	12.6	230
7	3	3	27.0	25.1	186
8	3	3	19.9	16.3	203
9	3	3	26.4	26.3	193
10	3	3	12.1	11.5	157
11	3	3	9.4	9.1	177
12	3	3	11.5	10.0	176
13	3	3	6.5	7.4	183
14	3	3	7.6	7.6	185
15	3	3	4.8	3.2	157
16	3	3	7.0	7.6	211
-1	3	3	12.6	11.1	143
-2	3	3	2.7	0.9	68
-3	3	3	15.5	11.5	196
-4	3	3	5.6	6.2	184
-5	3	3	27.2	23.1	351
-6	3	3	37.2	37.2	33
-7	3	3	55.8	52.1	350
-8	3	3	35.6	32.3	321
-9	3	3	16.4	16.3	54
-10	3	3	15.7	14.1	268
-11	3	3	5.1	2.3	17
-12	3	3	13.3	12.5	340
-13	3	3	3.8	4.1	330
-14	3	3	10.5	11.2	348
-15	3	3	4.5	2.3	133
-16	3	3	4.2	3.7	36
-18	3	3	7.1	6.3	235
0	3	4	42.0	38.7	188
1	3	4	53.6	53.0	174
2	3	4	34.5	34.1	205
3	3	4	25.3	27.9	183
4	3	4	51.4	54.4	209
5	3	4	27.0	25.8	146
6	3	4	19.0	19.3	184
7	3	4	22.6	23.7	192
8	3	4	23.9	23.3	185
9	3	4	14.6	13.9	132
11	3	4	12.4	12.3	193
12	3	4	6.1	6.3	189
13	3	4	3.9	2.9	222
14	3	4	3.8	2.8	192
15	3	4	1.9	2.5	1
-1	3	4	52.7	54.6	178
-2	3	4	35.5	37.0	196
-3	3	4	38.2	33.8	178
-4	3	4	35.7	38.9	185
-5	3	4	61.0	60.9	170
-6	3	4	45.0	46.8	193
-7	3	4	10.0	1.9	80
-8	3	4	16.0	15.3	150
-9	3	4	19.8	17.7	177
-10	3	4	6.8	6.0	290
-11	3	4	6.7	7.0	119
-12	3	4	3.8	4.4	147
-13	3	4	7.7	5.6	110
-14	3	4	6.6	5.5	311
-15	3	4	4.5	3.3	266
-16	3	4	4.9	3.9	168
-17	3	4	2.2	2.6	270
-18	3	4	5.9	5.8	184
-19	3	4	5.9	5.3	66
0	3	5	11.1	9.6	44
1	3	5	31.4	31.9	108
2	3	5	14.9	14.7	194
3	3	5	20.1	19.0	163
4	3	5	31.1	33.1	187
5	3	5	25.6	27.3	182
6	3	5	19.7	21.7	170
7	3	5	8.0	7.9	140
8	3	5	6.6	7.0	135
9	3	5	3.8	2.5	56
10	3	5	3.7	4.9	38
11	3	5	7.5	7.3	114
12	3	5	5.0	4.0	267
13	3	5	5.4	3.8	29
14	3	5	3.2	2.7	352
-1	3	5	33.3	33.1	197
-2	3	5	32.4	27.4	191
-3	3	5	40.9	42.4	197
-4	3	5	12.7	11.5	207
-5	3	5	32.4	28.5	195
-6	3	5	24.7	25.7	201
-7	3	5	47.1	42.9	96
-8	3	5	30.4	33.0	189
-9	3	5	26.9	26.7	163
-10	3	5	24.6	26.5	245
-11	3	5	21.1	20.8	186
-12	3	5	16.3	13.9	171
-13	3	5	17.3	16.4	178
-14	3	5	10.1	9.7	165
-15	3	5	4.5	2.5	154
-16	3	5	4.8	5.0	184
-17	3	5	6.1	5.1	173
-18	3	5	7.8	6.7	234
-19	3	5	3.1	3.0	279
0	3	6	19.9	20.0	309
1	3	6	10.2	11.9	53
2	3	6	27.5	30.1	330
3	3	6	9.7	8.9	287
5	3	6	24.3	25.6	36
6	3	6	14.7	16.2	16
7	3	6	13.4	13.7	4
8	3	6	15.8	15.6	339
9	3	6	9.8	9.3	344
10	3	6	10.2	11.4	5
11	3	6	9.9	10.6	24
12	3	6	6.8	7.2	19
13	3	6	4.0	2.7	16
-1	3	6	6.6	6.0	126
-2	3	6	5.5	3.1	73
-3	3	6	14.0	11.8	105
-4	3	6	8.3	11.0	179
-5	3	6	15.6	14.1	30
-6	3	6	4.4	5.3	5
-7	3	6	12.0	10.7	200
-8	3	6	16.0	15.1	194
-9	3	6	31.6	27.7	157
-10	3	6	19.0	20.0	170
-11	3	6	3.7	5.4	198
-12	3	6	11.2	11.8	83
-13	3	6	3.8	3.5	188
-14	3	6	3.8	4.3	76
-15	3	6	4.5	4.9	189
-16	3	6	3.4	6.3	148
-17	3	6	3.0	3.9	230
-18	3	6	4.6	4.9	198
-19	3	6	2.9	3.1	23
0	3	7	14.4	14.2	17
1	3	7	20.3	19.2	18
2	3	7	15.6	14.9	21
3	3	7	21.0	23.2	7
4	3	7	18.7	17.3	315
5	3	7	11.9	13.4	24
6	3	7	19.3	19.8	356
7	3	7	5.3	5.9	351
8	3	7	5.8	6.3	323
9	3	7	5.9	6.5	56
10	3	7	10.6	10.7	5
12	3	7	4.8	4.9	359
-1	3	7	24.0	25.3	10
-2	3	7	29.8	32.3	352
-3	3	7	34.9	36.4	350
-4	3	7	30.6	30.6	27
-5	3	7	13.8	13.5	337
-6	3	7	13.2	12.2	30
-7	3	7	41.3	39.7	340
-8	3	7	20.6	17.8	58
-9	3	7	7.3	7.8	288
-10	3	7	23.8	22.5	351
-11	3	7	11.1	10.8	24
-12	3	7	14.9	14.2	3
-13	3	7	6.1	5.5	3
-14	3	7	6.5	6.0	11
-15	3	7	6.2	5.4	277
-16	3	7	3.2	3.4	95
-17	3	7	3.5	2.6	307
-18	3	7	4.0	2.3	110
0	3	8	20.7	22.7	1
1	3	8	8.3	9.6	19
2	3	8	21.5	22.0	1
3	3	8	17.6	18.0	341
4	3	8	7.7	8.6	17
5	3	8	10.8	11.2	314
6	3	8	2.6	3.3	232
7	3	8	5.6	6.3	352
8	3	8	3.2	3.7	219
9	3	8	4.1	3.8	263
11	3	8	2.5	2.5	153
-1	3	8	12.0	10.1	280
-2	3	8	13.1	12.6	347
-3	3	8	22.5	19.9	337
-4	3	8	21.3	21.4	14
-5	3	8	14.5	14.7	355
-6	3	8	14.2	11.9	24
-7	3	8	18.6	14.9	44
-8	3	8	5.2	5.3	288
-9	3	8	10.3	10.1	277
-10	3	8	16.0	16.0	325
-11	3	8	7.7	7.5	330
-12	3	8	10.5	11.7	65
-13	3	8	11.0	10.6	360
-14	3	8	6.3	4.9	53
-15	3	8	13.0	14.8	349
-16	3	8	3.8	4.9	47
-17	3	8	6.5	7.1	335
-18	3	8	1.5	1.8	8
0	3	9	12.2	12.5	176
1	3	9	9.4	10.5	161
3	3	9	12.3	13.9	212
4	3	9	10.8	12.6	191
5	3	9	5.6	5.5	170
6	3	9	8.9	9.5	144
7	3	9	4.8	4.7	188
8	3	9	5.0	5.6	200
9	3	9	5.2	7.3	220
-1	3	9	13.5	13.8	193
-2	3	9	14.5	15.1	122
-3	3	9	5.3	4.8	212
-4	3	9	4.6	6.2	142
-5	3	9	11.9	14.2	319
-6	3	9	2.7	3.7	330
-7	3	9	10.1	10.1	255
-8	3	9	2.7	4.2	202
-10	3	9	4.7	4.4	232
-11	3	9	11.2	11.6	342
-12	3	9	4.6	6.6	37
-13	3	9	2.6	4.2	281
-14	3	9	3.4	5.1	326
-15	3	9	4.5	5.2	277
-16	3	9	2.0	0.8	30
-18	3	9	1.1	3.1	8
0	3	10	14.0	14.0	181
1	3	10	9.2	9.3	182
2	3	10	12.1	14.1	143
3	3	10	3.5	3.7	180
4	3	10	5.3	6.1	202
5	3	10	4.3	5.0	219
7	3	10	3.6	3.4	197
8	3	10	3.0	6.4	187
-1	3	10	6.1	5.7	168
-2	3	10	10.6	10.3	208
-3	3	10	8.2	9.0	208
-4	3	10	18.7	18.8	166
-5	3	10	13.7	16.2	193
-6	3	10	18.5	16.9	178
-8	3	10	13.1	11.6	136
-9	3	10	17.1	17.3	197
-10	3	10	7.6	7.7	326
-11	3	10	7.8	9.6	169
-12	3	10	5.1	5.2	190
-13	3	10	9.9	12.3	188
-14	3	10	10.1	9.9	168
-15	3	10	5.4	6.7	207
-16	3	10	3.5	4.0	158
0	3	11	6.1	4.8	173
1	3	11	9.0	9.4	163
4	3	11	5.4	5.8	91
6	3	11	2.9	2.4	129
-1	3	11	6.2	8.1	168
-2	3	11	7.8	8.5	173
-3	3	11	4.5	4.3	174
-4	3	11	10.2	10.3	146
-5	3	11	13.3	13.9	179
-6	3	11	8.4	10.0	152
-7	3	11	7.9	8.4	195
-8	3	11	7.4	6.5	201
-9	3	11	4.5	3.9	238
-11	3	11	3.4	4.0	91
-12	3	11	4.6	6.3	146
-13	3	11	3.8	3.3	160
-14	3	11	3.4	4.8	194
-15	3	11	3.9	2.1	250
-16	3	11	3.6	4.0	174
0	3	12	4.7	5.0	2
1	3	12	2.7	3.9	23
2	3	12	7.9	8.7	18
3	3	12	2.1	3.0	327
-2	3	12	8.9	10.1	18
-3	3	12	4.7	5.1	344
-4	3	12	4.1	4.1	325
-5	3	12	4.8	6.6	351
-6	3	12	2.4	2.9	342
-8	3	12	2.4	2.6	7
-9	3	12	5.6	7.3	88
-10	3	12	3.1	1.9	345
-11	3	12	4.3	7.1	64
-12	3	12	2.0	1.7	84
-13	3	12	1.8	2.0	137
-14	3	12	2.7	3.1	240
0	3	13	3.2	4.8	305
-1	3	13	5.9	8.0	9
-2	3	13	4.7	6.1	328
-3	3	13	4.6	4.4	319
-4	3	13	3.9	3.2	24
-5	3	13	4.4	5.0	7
-6	3	13	4.0	5.1	12
-7	3	13	6.3	9.2	21
-8	3	13	4.4	5.9	21
-9	3	13	3.8	2.7	298
-10	3	13	8.9	9.4	341
-11	3	13	3.8	4.2	45
-4	3	14	1.1	1.2	329
-6	3	14	2.7	4.2	350
-7	3	14	6.1	5.6	330
-8	3	14	2.9	4.1	337
-9	3	14	2.4	3.6	21
1	4	0	16.9	18.5	26
2	4	0	36.1	38.7	0
3	4	0	6.7	7.8	111
4	4	0	48.2	50.8	326
5	4	0	20.1	22.7	353
6	4	0	20.0	19.0	1
7	4	0	32.8	32.2	15
8	4	0	21.7	17.6	38
9	4	0	21.2	20.1	356
10	4	0	17.3	15.1	15
11	4	0	22.4	22.4	343
12	4	0	17.5	17.0	14
13	4	0	12.4	12.0	345
14	4	0	5.4	4.6	32
17	4	0	5.6	4.6	33
0	4	1	9.7	12.0	61
1	4	1	4.5	7.9	17
2	4	1	2.8	5.1	280
3	4	1	11.6	13.6	5
4	4	1	2.9	3.8	15
5	4	1	10.6	11.5	77
6	4	1	35.0	32.2	7
7	4	1	19.4	17.4	151
8	4	1	15.5	14.5	330
9	4	1	6.4	7.1	129
10	4	1	8.3	7.8	288
11	4	1	4.0	4.5	312
12	4	1	7.0	8.2	56
14	4	1	3.6	2.7	179
15	4	1	3.2	3.1	176
16	4	1	1.9	1.6	180
-1	4	1	26.2	25.9	303
-2	4	1	38.2	36.2	341
-3	4	1	22.8	20.3	342
-4	4	1	11.7	9.6	128
-5	4	1	33.1	34.0	304
-6	4	1	8.6	8.8	326
-7	4	1	18.2	18.5	5
-8	4	1	19.9	18.4	6
-9	4	1	6.5	7.1	343
-10	4	1	13.9	11.6	31
-11	4	1	13.9	12.3	10

Measured and calculated values of structure factors.
The phase angles are in degrees.

Table 2-4 (cont.)

H	K	L	Fo	Fc	α
-12	4	1	21.6	21.2	352
-13	4	1	15.7	14.7	50
-14	4	1	12.7	10.6	342
-15	4	1	6.8	7.5	21
-16	4	1	6.8	5.6	0
-17	4	1	4.5	4.0	34
-18	4	1	3.7	4.9	313
0	4	2	50.3	50.7	143
1	4	2	42.5	39.7	194
2	4	2	6.9	7.3	149
3	4	2	25.6	27.6	200
4	4	2	19.9	25.5	164
5	4	2	16.4	16.8	240
6	4	2	17.7	19.4	227
7	4	2	4.8	5.4	152
8	4	2	10.6	11.3	248
9	4	2	11.3	10.4	180
10	4	2	12.0	12.8	148
11	4	2	17.0	16.1	201
12	4	2	4.9	4.3	275
13	4	2	10.0	9.0	171
14	4	2	4.2	4.0	157
15	4	2	5.0	4.5	172
16	4	2	5.1	5.5	154
-1	4	2	5.5	4.5	97
-2	4	2	16.2	14.7	252
-3	4	2	10.8	12.6	117
-4	4	2	24.3	21.1	201
-5	4	2	36.0	36.5	157
-6	4	2	10.3	10.8	208
-7	4	2	35.5	36.4	182
-8	4	2	17.3	14.7	238
-9	4	2	22.3	21.0	167
-10	4	2	5.8	6.4	269
-11	4	2	5.5	5.7	227
-12	4	2	11.4	8.7	74
-13	4	2	7.0	6.4	170
-14	4	2	2.8	3.9	231
-15	4	2	5.9	4.9	354
-16	4	2	3.3	3.7	345
-17	4	2	3.5	4.6	346
-18	4	2	2.5	2.3	316
0	4	3	26.1	26.3	193
1	4	3	37.1	35.9	164
2	4	3	23.6	21.8	124
3	4	3	41.8	41.4	166
4	4	3	12.1	9.3	120
5	4	3	25.0	25.9	188
6	4	3	14.8	17.8	179
7	4	3	5.1	4.8	199
8	4	3	12.7	13.1	180
9	4	3	11.6	11.1	128
10	4	3	14.1	13.0	178
11	4	3	7.5	7.6	174
12	4	3	4.7	4.0	216
13	4	3	3.5	4.0	201
14	4	3	2.2	2.4	116
15	4	3	6.4	5.9	214
-1	4	3	41.4	38.6	183
-2	4	3	37.9	34.6	189
-3	4	3	43.2	41.7	164
-4	4	3	13.1	12.6	252
-5	4	3	22.5	21.4	168
-6	4	3	16.5	18.0	138
-7	4	3	35.2	33.7	181
-8	4	3	25.4	26.2	173
-9	4	3	40.9	36.5	207
-10	4	3	21.0	22.0	193
-11	4	3	12.5	11.4	203
-12	4	3	22.0	20.9	186
-13	4	3	11.8	10.2	187
-14	4	3	19.6	16.7	197
-15	4	3	7.1	7.0	169
-16	4	3	5.5	5.9	187
-18	4	3	1.6	1.7	308
0	4	4	27.0	25.4	342
1	4	4	5.7	7.7	199
2	4	4	8.8	7.5	340
3	4	4	6.4	5.3	226
4	4	4	5.1	4.2	139
5	4	4	9.8	9.4	26
6	4	4	22.0	20.8	359
7	4	4	6.1	5.3	105
8	4	4	6.9	5.9	2
9	4	4	2.9	2.6	153
10	4	4	6.4	6.9	206
11	4	4	8.2	10.0	229
12	4	4	5.1	5.0	80
13	4	4	2.2	1.9	310
14	4	4	4.0	4.0	38
-1	4	4	5.3	6.4	237
-2	4	4	13.6	12.1	237
-3	4	4	27.1	25.5	109
-4	4	4	23.2	24.1	177
-5	4	4	2.7	2.7	202
-6	4	4	11.4	9.1	164
-7	4	4	10.0	8.9	110
-8	4	4	15.1	16.1	143
-9	4	4	17.0	17.8	204
-10	4	4	28.6	27.4	158
-11	4	4	11.0	11.1	156
-12	4	4	19.7	17.4	159
-13	4	4	11.8	12.6	157
-14	4	4	17.0	16.4	209
-15	4	4	9.3	7.8	193
-16	4	4	9.8	8.8	175
-17	4	4	4.8	5.1	193
-18	4	4	7.7	5.9	196
0	4	5	20.1	20.6	78
1	4	5	33.9	32.1	22
2	4	5	22.8	24.2	34
3	4	5	9.6	12.3	3
4	4	5	19.1	19.4	37
5	4	5	6.5	6.1	359
6	4	5	13.5	14.7	358
7	4	5	6.3	5.6	65
8	4	5	11.8	14.0	313
9	4	5	7.0	7.7	31
10	4	5	4.7	4.0	39
11	4	5	4.4	2.4	249
12	4	5	9.0	8.7	319
13	4	5	4.1	3.4	306
14	4	5	4.9	6.3	356
-1	4	5	53.4	55.7	10
-2	4	5	20.8	20.3	326
-3	4	5	18.3	18.1	23
-4	4	5	6.8	5.4	73
-5	4	5	15.6	17.1	332
-6	4	5	16.5	15.4	44
-7	4	5	35.2	33.1	340
-8	4	5	21.0	20.2	27
-9	4	5	16.6	14.9	10
-1	4	5	14.9	13.9	342
-11	4	5	9.6	8.3	355
-12	4	5	4.0	1.3	77
-13	4	5	4.0	4.2	127
-14	4	5	8.9	9.0	233
-15	4	5	9.6	8.6	20
-16	4	5	2.4	3.2	152
0	4	6	21.3	19.3	337
1	4	6	29.1	27.7	324
2	4	6	20.4	22.3	355
3	4	6	21.2	20.7	331
4	4	6	13.1	14.2	39
5	4	6	8.4	7.2	352
6	4	6	7.6	7.7	176
7	4	6	9.1	9.1	322
8	4	6	5.6	6.7	256
9	4	6	6.6	7.2	29
10	4	6	2.5	2.4	51
11	4	6	3.1	4.2	62
13	4	6	1.6	1.8	42
-1	4	6	31.7	32.1	320
-2	4	6	11.1	10.7	334
-3	4	6	39.7	39.2	20
-4	4	6	30.6	29.2	352
-5	4	6	33.7	35.7	7
-6	4	6	31.5	29.1	350
-7	4	6	16.0	14.8	7
-8	4	6	22.2	20.1	357
-9	4	6	19.6	18.5	347
-10	4	6	20.0	19.7	7
-11	4	6	27.7	28.1	12
-12	4	6	5.0	4.9	19
-13	4	6	16.4	17.2	12
-14	4	6	5.6	3.9	347
-15	4	6	10.8	11.3	25
-16	4	6	5.4	6.2	39
-17	4	6	5.1	5.8	355
-18	4	6	2.3	3.0	56
0	4	7	10.4	11.6	275
2	4	7	6.7	6.6	239
3	4	7	15.1	15.4	220
4	4	7	16.4	17.0	174
5	4	7	7.0	6.8	184
6	4	7	4.0	3.1	231
9	4	7	7.6	8.2	24
10	4	7	6.4	4.9	204
11	4	7	3.0	3.3	249
-1	4	7	7.2	7.0	85
-2	4	7	21.4	22.7	191
-3	4	7	17.2	16.3	72
-4	4	7	4.3	4.2	184
-5	4	7	10.1	10.0	301
-6	4	7	9.3	8.6	39
-7	4	7	15.1	14.5	310
-8	4	7	11.1	11.1	57
-9	4	7	18.7	18.9	323
-10	4	7	8.5	8.4	61
-11	4	7	23.6	21.7	6
-12	4	7	9.1	5.5	10
-13	4	7	20.8	19.1	340
-14	4	7	14.4	14.8	295
-15	4	7	7.6	8.4	344
-16	4	7	7.3	6.8	17
-17	4	7	4.4	4.4	338
-18	4	7	5.5	5.0	1
0	4	8	21.6	22.2	180
2	4	8	16.4	17.5	169
3	4	8	10.4	10.8	207
4	4	8	12.4	13.2	179
5	4	8	8.4	9.1	199
6	4	8	6.6	6.8	142
7	4	8	8.0	8.7	161
8	4	8	4.1	4.2	203
9	4	8	4.9	4.1	79
10	4	8	5.9	5.6	109
-1	4	8	10.3	10.7	237
-2	4	8	28.4	28.5	219
-3	4	8	24.9	24.0	198
-4	4	8	20.3	22.0	206
-5	4	8	11.8	13.2	239
-6	4	8	19.6	20.7	178
-7	4	8	4.8	2.7	309
-8	4	8	13.8	13.7	154
-9	4	8	15.3	15.4	191
-10	4	8	10.7	9.1	244
-11	4	8	8.1	8.2	180
-13	4	8	3.9	4.4	134
-14	4	8	2.6	3.7	271
-15	4	8	6.9	6.9	38
-16	4	8	2.2	2.2	59
-17	4	8	1.8	2.9	192
0	4	9	16.2	16.9	168
1	4	9	11.5	11.8	173
2	4	9	6.4	5.6	211
3	4	9	7.9	7.7	240
4	4	9	6.0	5.2	77
5	4	9	2.5	1.6	54
6	4	9	6.3	5.6	133
8	4	9	5.1	6.4	245
9	4	9	1.8	2.9	338
-1	4	9	15.7	14.9	168
-2	4	9	21.7	22.6	163
-3	4	9	21.5	21.3	101
-4	4	9	11.4	12.1	162
-5	4	9	19.9	19.1	193
-6	4	9	10.7	11.6	182
-7	4	9	28.8	31.5	181
-8	4	9	20.7	20.2	165
-9	4	9	20.3	20.2	189
-10	4	9	15.4	13.9	179
-11	4	9	8.0	7.8	166
-12	4	9	12.6	13.1	196
-13	4	9	6.4	6.3	146
-14	4	9	5.5	5.7	218
-15	4	9	5.5	6.0	173
-16	4	9	3.4	3.6	135
-17	4	9	5.2	6.1	227
0	4	10	3.9	4.2	17
1	4	10	6.1	6.4	54
2	4	10	6.5	8.4	18
3	4	10	4.4	5.4	305
5	4	10	3.0	2.6	50
6	4	10	3.2	3.0	351
7	4	10	2.1	2.4	23
-1	4	10	4.9	5.7	90
-2	4	10	4.9	5.6	98
-3	4	10	4.0	7.4	44
-4	4	10	7.0	7.7	8
-5	4	10	5.7	5.4	234
-7	4	10	7.0	6.7	212
-8	4	10	2.8	3.8	103
-9	4	10	7.9	7.3	166
-10	4	10	6.7	5.1	179
-11	4	10	6.0	7.1	153
-12	4	10	7.3	8.1	195
-13	4	10	6.4	9.0	209
-14	4	10	11.0	10.5	218
-15	4	10	9.8	11.0	146
-16	4	10	4.7	4.8	97
-17	4	10	3.2	5.3	162
0	4	11	5.6	5.6	343
1	4	11	9.9	10.8	18
2	4	11	8.5	9.4	19
3	4	11	6.6	7.0	4
4	4	11	7.8	8.0	343
5	4	11	4.9	6.4	317
-1	4	11	5.8	5.9	20
-2	4	11	10.6	10.5	308
-3	4	11	7.6	8.7	52
-4	4	11	14.4	15.3	5
-5	4	11	13.4	13.0	26
-6	4	11	11.2	12.7	49
-7	4	11	10.8	10.6	37
-8	4	11	10.7	11.2	42
-9	4	11	5.2	5.4	249
-10	4	11	5.7	6.0	344
-11	4	11	3.5	3.4	63
-12	4	11	3.3	3.4	24
-13	4	11	3.0	3.0	96
-15	4	11	2.3	2.0	77
0	4	12	3.6	3.5	39
1	4	12	3.2	3.0	55
2	4	12	4.0	3.7	339
3	4	12	2.5	2.6	270
-1	4	12	3.1	3.5	339
-2	4	12	6.0	6.6	8
-3	4	12	9.4	12.4	16
-4	4	12	9.9	11.2	336
-5	4	12	9.1	11.6	288
-6	4	12	7.6	9.7	349
-7	4	12	4.2	4.2	356
-8	4	12	5.8	7.2	352
-9	4	12	10.6	12.5	345
-10	4	12	8.6	10.5	347
-11	4	12	7.9	9.6	11
-12	4	12	6.7	8.1	349
-13	4	12	4.2	6.0	53
-14	4	12	4.2	5.1	331
0	4	13	1.2	2.8	248
-1	4	13	2.1	1.5	214
-3	4	13	2.5	2.3	275
-4	4	13	2.6	4.2	257
-5	4	13	3.9	6.6	220
-8	4	13	5.4	5.2	20
-1	4	13	4.5	4.8	318
-11	4	13	3.8	3.4	355
1	5	0	65.0	65.3	254
2	5	0	6.2	7.0	80
3	5	0	13.5	17.6	269
4	5	0	12.6	13.5	199
5	5	0	16.2	15.6	172
6	5	0	29.0	28.6	170
7	5	0	13.0	11.8	107
8	5	0	25.1	24.6	181
9	5	0	20.0	19.6	195
10	5	0	19.8	18.2	198
11	5	0	22.8	21.0	193
12	5	0	14.0	15.7	136
13	5	0	7.3	6.1	116
14	5	0	5.2	5.1	222
15	5	0	4.0	3.6	195
17	5	0	2.9	3.1	182
0	5	1	39.9	41.5	113
1	5	1	37.2	34.4	206
2	5	1	25.9	27.5	234
3	5	1	47.9	53.3	177
4	5	1	20.1	23.7	162
5	5	1	29.8	33.9	177
6	5	1	23.6	25.3	164
7	5	1	23.1	24.3	178
8	5	1	33.8	32.7	191
9	5	1	10.8	11.6	116
10	5	1	19.5	15.8	196
11	5	1	6.4	5.0	94
12	5	1	12.6	12.5	163
13	5	1	6.6	6.0	212
14	5	1	5.5	4.7	169
15	5	1	7.9	8.4	250
16	5	1	3.2	1.9	123
-1	5	1	80.9	79.2	165
-2	5	1	54.0	44.4	213
-3	5	1	39.7	40.8	202
-4	5	1	28.0	26.4	192
-5	5	1	22.6	22.4	176
-6	5	1	18.8	18.1	201
-7	5	1	16.3	15.2	168
-8	5	1	19.7	20.1	148
-9	5	1	3.6	1.6	270
-10	5	1	10.2	11.7	89
-11	5	1	5.6	6.1	299
-12	5	1	19.0	17.2	270
-13	5	1	11.3	10.9	32
-14	5	1	2.7	0.7	301
-15	5	1	3.4	3.6	120
-16	5	1	2.9	2.7	243
-17	5	1	2.7	2.1	68
0	5	2	70.0	61.6	200
1	5	2	31.2	30.0	216
2	5	2	32.4	35.0	175
3	5	2	39.4	39.3	166
4	5	2	22.6	23.9	172
5	5	2	26.2	26.9	180
6	5	2	14.0	14.7	144
7	5	2	10.6	9.8	219
8	5	2	7.1	7.1	29
9	5	2	5.6	5.8	167
10	5	2	7.0	7.1	322
11	5	2	7.5	6.4	3
12	5	2	6.7	6.5	48
15	5	2	2.6	2.6	26
-1	5	2	60.2	60.6	158
-2	5	2	31.4	31.9	172
-3	5	2	33.5	26.6	218
-4	5	2	57.0	51.1	194
-5	5	2	35.0	34.9	174
-6	5	2	35.4	33.0	196
-7	5	2	26.5	24.5	137
-8	5	2	11.8	10.4	209
-9	5	2	19.2	19.2	186
-10	5	2	11.4	10.6	202
-11	5	2	17.8	14.8	158
-12	5	2	2.8	6.2	328
-13	5	2	11.7	11.8	117
-14	5	2	6.1	7.2	154
-15	5	2	6.2	7.0	127
-16	5	2	5.0	4.6	222
-17	5	2	3.1	2.5	221
0	5	3	23.6	19.2	137
1	5	3	19.2	15.2	216
2	5	3	15.1	14.8	33
3	5	3	20.2	19.1	359
4	5	3	16.6	17.2	327
5	5	3	15.2	16.1	312
6	5	3	10.6	13.4	345
7	5	3	24.9	26.9	29
8	5	3	15.7	17.5	356
9	5	3	13.1	14.0	10
10	5	3	21.6	21.8	340
11	5	3	7.3	7.3	305
12	5	3	8.2	8.2	14
13	5	3	3.3	4.7	34
14	5	3	3.4	2.7	13
-1	5	3	91.6	76.0	75
-2	5	3	23.8	21.0	262
-3	5	3	17.8	16.5	80
-4	5	3	27.5	22.6	206
-5	5	3	11.7	9.8	228
-6	5	3	13.9	11.7	147
-7	5	3	29.2	26.8	169
-8	5	3	7.1	6.4	148
-9	5	3	14.4	14.3	178
-10	5	3	5.4	6.4	109
-11	5	3	5.6	6.2	181
-12	5	3	11.4	12.7	231
-13	5	3	14.3	15.4	178
-14	5	3	11.0	10.7	141
-15	5	3	3.6	2.2	65
-16	5	3	6.8	6.1	215
-18	5	3	2.0	1.9	171
0	5	4	41.5	39.8	43
1	5	4	22.3	23.4	11
2	5	4	40.3	40.7	333
3	5	4	24.6	26.1	6
4	5	4	21.8	22.6	351
5	5	4	21.9	23.7	358
6	5	4	19.9	20.7	2
7	5	4	19.7	20.2	17
8	5	4	11.8	11.4	357
9	5	4	18.4	18.6	15
10	5	4	9.2	9.7	327
11	5	4	12.1	13.0	32
12	5	4	6.3	4.3	6
13	5	4	5.0	4.8	44
14	5	4	2.6	0.9	70
-1	5	4	3.8	0.8	248
-2	5	4	43.9	40.8	317
-3	5	4	38.3	36.4	340
-4	5	4	31.4	29.9	353
-5	5	4	32.4	29.1	14
-6	5	4	19.0	18.3	4
-7	5	4	14.9	14.2	40
-8	5	4	8.1	8.0	109
-9	5	4	6.3	7.6	3
-11	5	4	11.9	11.6	334
-12	5	4	18.3	20.5	252
-13	5	4	21.2	20.0	60
-14	5	4	5.5	5.2	111
-15	5	4	2.6	1.2	26
-17	5	4	1.9	1.7	347
-18	5	4	2.3	2.3	77
0	5	5	24.2	22.6	329
1	5	5	25.8	24.0	354
2	5	5	16.2	16.7	336
3	5	5	8.7	8.4	354
4	5	5	12.0	13.6	354
5	5	5	7.2	8.0	28
6	5	5	6.3	6.5	297
7	5	5	11.4	11.8	327
8	5	5	6.4	5.6	272
9	5	5	4.8	3.8	185
10	5	5	7.4	6.9	225
12	5	5	3.4	2.4	244
13	5	5	1.5	2.1	227
-1	5	5	35.4	32.6	32
-2	5	5	52.4	48.0	342
-3	5	5	41.0	40.6	357
-4	5	5	15.8	13.5	17
-5	5	5	34.0	33.4	22
-6	5	5	21.9	20.4	3
-7	5	5	36.9	32.0	15
-8	5	5	12.7	13.9	342
-9	5	5	14.3	14.8	343
-10	5	5	3.9	2.5	50
-11	5	5	4.9	3.2	39
-12	5	5	11.8	11.0	13
-13	5	5	6.4	7.0	89
-14	5	5	2.7	4.1	158
-15	5	5	7.3	8.1	333
-16	5	5	7.6	8.4	297
-17	5	5	5.1	5.7	319
-18	5	5	1.9	1.2	345
0	5	6	16.0	16.8	170
1	5	6	13.2	12.3	175
2	5	6	9.8	11.5	171
3	5	6	11.5	12.1	75
4	5	6	8.8	8.8	176
5	5	6	8.0	9.8	211
6	5	6	10.7	11.8	180
7	5	6	10.6	11.4	183
8	5	6	11.6	12.2	169
9	5	6	10.3	12.0	164
11	5	6	5.4	6.3	183
12	5	6	4.1	5.3	231
-1	5	6	17.9	16.2	39
-2	5	6	22.0	20.7	274
-3	5	6	17.9	16.7	293
-4	5	6	13.5	14.3	210
-5	5	6	19.6	17.8	309
-6	5	6	3.5	4.1	348
-7	5	6	8.1	6.0	357
-8	5	6	21.3	19.7	303
-9	5	6	8.2	7.9	277
-10	5	6	10.5	11.0	351
-11	5	6	2.8	2.5	227
-12	5	6	5.7	5.7	280
-13	5	6	11.5	10.9	175
-14	5	6	5.4	5.9	53
-15	5	6	9.4	9.8	334
-17	5	6	3.2	3.4	343
-18	5	6	1.2	2.3	52
0	5	7	18.5	17.0	152
1	5	7	22.0	22.7	175
2	5	7	8.8	9.5	208
3	5	7	10.6	9.8	193
4	5	7	14.0	15.6	183
5	5	7	11.4	12.2	202
6	5	7	15.1	17.1	198
7	5	7	11.4	12.0	170
8	5	7	10.6	11.5	179
9	5	7	6.0	6.2	187
10	5	7	7.2	8.4	210
11	5	7	3.8	3.9	156
-1	5	7	25.0	25.6	195
-2	5	7	19.1	21.5	224
-3	5	7	7.8	9.9	159
-4	5	7	17.7	16.3	177
-5	5	7	15.1	14.5	142
-6	5	7	16.2	16.1	135
-7	5	7	13.8	15.1	183
-8	5	7	11.4	11.1	142
-9	5	7	15.2	17.0	227
-10	5	7	2.8	1.8	94
-11	5	7	7.6	8.1	260
-12	5	7	8.5	9.2	201
-13	5	7	8.3	10.5	95
-14	5	7	5.2	5.9	114
-15	5	7	9.0	10.1	257
-16	5	7	3.7	3.4	161
-17	5	7	4.0	4.3	236
-18	5	7	1.3	2.9	122
0	5	8	2.8	3.1	107
1	5	8	7.0	6.5	129
2	5	8	7.6	7.6	171
3	5	8	9.5	9.9	176
4	5	8	6.3	6.1	113
5	5	8	12.2	13.2	119
6	5	8	5.8	5.4	140
7	5	8	2.4	3.3	30
8	5	8	3.0	2.3	122
9	5	8	1.8	3.0	111
-1	5	8	9.7	9.3	166
-2	5	8	3.9	3.2	65
-3	5	8	22.7	24.0	197
-4	5	8	30.4	31.0	141
-5	5	8	19.7	20.0	197
-6	5	8	14.6	14.7	162
-7	5	8	11.6	12.2	212
-8	5	8	13.0	12.5	184
-9	5	8	7.0	6.5	198
-10	5	8	6.4	9.1	160
-11	5	8	6.4	8.7	194
-13	5	8	8.0	7.8	172
-14	5	8	6.6	6.7	203
-15	5	8	6.0	7.6	290
-16	5	8	3.9	4.8	265
-17	5	8	4.1	4.2	154
0	5	9	6.4	6.2	23
1	5	9	6.3	5.4	263
2	5	9	2.8	2.3	355
3	5	9	5.4	5.8	1
4	5	9	5.2	5.8	28
5	5	9	6.4	7.0	359
6	5	9	7.0	7.0	39
7	5	9	5.1	6.2	304
8	5	9	2.6	3.5	51
-1	5	9	8.1	8.6	327
-2	5	9	5.7	6.8	330
-4	5	9	7.6	8.4	145
-5	5	9	5.7	5.2	68
-7	5	9	7.0	6.3	102
-9	5	9	6.4	7.8	140
-1	5	9	12.9	13.7	65
-11	5	9	5.5	6.7	126
-12	5	9	5.9	5.9	51
-13	5	9	5.0	6.2	158
-14	5	9	6.6	7.6	28
-15	5	9	6.8	7.5	330
-16	5	9	3.4	4.0	128
0	5	10	8.1	9.5	17
1	5	10	2.6	3.8	72
2	5	10	7.1	8.7	0
3	5	10	6.3	7.6	354
4	5	10	6.2	7.3	23
5	5	10	7.1	7.0	343
6	5	10	4.6	5.7	344
-1	5	10	5.5	6.3	343
-2	5	10	11.5	9.8	13
-3	5	10	11.2	11.8	11
-4	5	10	8.5	9.0	42
-5	5	10	8.5	9.3	358
-6	5	10	8.0	9.0	43
-7	5	10	6.8	7.2	320
-8	5	10	7.3	7.9	341
-9	5	10	6.1	6.6	283
-10	5	10	7.0	9.6	7
-11	5	10	6.3	7.2	6
-12	5	10	5.4	5.0	22
-13	5	10	6.0	6.5	33
-14	5	10	6.5	6.2	2
-15	5	10	6.1	7.1	295
-16	5	10	2.2	2.4	110
0	5	11	6.7	8.1	344
1	5	11	3.9	4.1	326
2	5	11	2.1	2.6	281
3	5	11	6.5	8.6	267
4	5	11	4.4	5.1	308
-1	5	11	5.5	4.7	277
-2	5	11	3.6	4.5	336
-3	5	11	5.8	5.6	231
-5	5	11	7.4	8.3	335
-6	5	11	6.4	6.8	336
-7	5	11	7.3	8.2	344
-8	5	11	4.4	5.1	354
-9	5	11	5.0	5.9	5
-10	5	11	4.8	4.3	357
-11	5	11	3.2	2.6	318
-12	5	11	3.0	4.3	11
-13	5	11	3.4	3.8	22
-14	5	11	2.9	4.2	282
-15	5	11	6.6	9.0	355
2	5	12	3.4	4.4	206
-1	5	12	2.8	3.4	185
-2	5	12	4.2	4.1	173
-3	5	12	2.2	3.6	120
-4	5	12	2.2	2.7	165
-6	5	12	4.5	6.0	279
-8	5	12	2.2	2.2	133
-9	5	12	2.1	2.3	197
-10	5	12	2.8	1.9	225
-11	5	12	3.2	3.4	226
-12	5	12	3.4	4.6	265
-13	5	12	3.4	4.3	235
-2	5	13	2.3	4.0	164
-4	5	13	5.4	6.3	191
-6	5	13	4.2	6.0	212
-7	5	13	2.9	1.7	149
-8	5	13	4.0	5.9	246
-9	5	13	4.4	6.4	179
-10	5	13	2.4	2.2	163
-11	5	13	2.1	3.6	150
1	6	0	23.6	31.5	173
2	6	0	20.6	21.9	166
3	6	0	36.8	40.4	172
4	6	0	13.3	12.8	203
5	6	0	12.7	10.2	176
6	6	0	22.6	21.8	145
7	6	0	28.7	28.0	191
8	6	0	10.6	9.5	136
9	6	0	12.0	11.5	227
10	6	0	14.8	13.2	189
11	6	0	24.4	25.0	185
12	6	0	4.7	5.0	357
13	6	0	2.9	3.1	183
14	6	0	4.5	3.7	230
16	6	0	2.9	3.6	196
0	6	1	32.2	36.6	345
1	6	1	21.8	21.7	254
2	6	1	19.5	18.0	40
3	6	1	12.1	11.4	194
4	6	1	10.3	12.0	177
5	6	1	11.1	11.6	8
6	6	1	14.2	14.0	343
7	6	1	18.5	16.6	179
8	6	1	11.7	9.9	93
9	6	1	11.9	9.8	168
10	6	1	9.6	8.4	188
11	6	1	19.0	19.2	191
12	6	1	9.3	8.0	94
13	6	1	6.1	6.0	23
14	6	1	5.9	6.2	312
-1	6	1	18.8	18.5	146
-2	6	1	27.7	27.8	206
-3	6	1	18.6	18.0	198
-4	6	1	5.4	7.3	171
-5	6	1	12.4	11.3	160
-6	6	1	11.6	13.9	175
-7	6	1	18.1	17.7	179
-8	6	1	25.9	23.2	196
-9	6	1	14.5	12.0	203
-10	6	1	11.5	13.4	181
-11	6	1	9.6	11.1	118
-12	6	1	4.3	4.5	120
-13	6	1	7.7	8.5	193
-14	6	1	10.1	8.4	184
-15	6	1	7.9	7.9	185
-16	6	1	2.7	3.7	171
0	6	2	41.9	43.7	11
1	6	2	28.4	26.9	14
2	6	2	25.6	25.0	354
3	6	2	17.6	19.7	24
4	6	2	11.4	11.6	24
5	6	2	6.5	8.9	319
6	6	2	28.4	31.3	11
7	6	2	16.9	16.7	23
9	6	2	8.6	8.1	1
10	6	2	9.1	9.4	285
11	6	2	8.4	8.0	28
12	6	2	11.6	10.4	353
13	6	2	6.7	6.2	21
14	6	2	5.3	4.4	347
15	6	2	3.8	3.5	333
-1	6	2	4.8	4.5	273
-2	6	2	20.0	21.8	74
-3	6	2	27.3	20.9	210
-4	6	2	11.9	9.4	136
-5	6	2	15.4	12.6	23
-6	6	2	10.6	9.3	14
-7	6	2	4.9	1.9	63
-9	6	2	7.5	4.7	45
-10	6	2	13.9	13.0	240
-11	6	2	3.0	6.3	96
-12	6	2	11.8	9.1	344
-13	6	2	12.2	12.4	24
-14	6	2	8.8	9.1	267
-16	6	2	2.1	3.8	187
-17	6	2	2.1	2.8	160
0	6	3	22.3	18.5	332
1	6	3	13.6	11.9	5
2	6	3	17.1	17.0	350
3	6	3	19.0	17.5	354
4	6	3	17.5	17.5	301
5	6	3	16.1	17.6	6
6	6	3	11.5	9.5	19
7	6	3	4.2	3.9	29
8	6	3	14.8	13.9	43
9	6	3	13.3	14.6	337
10	6	3	6.0	4.8	17
11	6	3	6.4	9.4	206
12	6	3	10.5	10.2	14
13	6	3	4.5	3.5	13
-1	6	3	44.4	40.2	12
-2	6	3	23.4	22.0	350
-3	6	3	12.7	12.0	53
-4	6	3	34.0	34.9	331
-5	6	3	35.4	35.1	3
-6	6	3	16.2	14.5	338
-7	6	3	21.3	22.9	14
-8	6	3	23.8	23.8	350
-9	6	3	25.8	25.2	7
-10	6	3	16.7	15.9	359
-11	6	3	5.3	4.6	342
-12	6	3	23.9	22.7	13
-13	6	3	7.9	8.2	350
-14	6	3	4.9	3.7	42
-15	6	3	5.7	5.8	35
-16	6	3	4.9	5.0	171
-17	6	3	1.6	1.8	22
0	6	4	23.3	21.6	337
1	6	4	8.9	6.7	258
2	6	4	7.3	6.4	326
3	6	4	9.9	8.3	75
4	6	4	15.3	16.8	189
5	6	4	13.3	13.5	345
6	6	4	14.5	13.3	328
7	6	4	6.1	7.1	254
8	6	4	8.1	8.6	14
9	6	4	6.7	6.8	8
10	6	4	6.4	6.0	325
11	6	4	10.6	9.6	197
12	6	4	7.4	5.9	158
13	6	4	1.9	3.2	105
-1	6	4	27.5	23.2	111
-2	6	4	31.8	27.2	199
-3	6	4	14.9	12.5	352
-4	6	4	17.2	16.4	26
-5	6	4	16.5	14.9	35
-6	6	4	13.9	11.1	215
-7	6	4	11.6	11.2	287
-8	6	4	7.3	7.2	12
-9	6	4	23.7	23.5	344
-10	6	4	17.9	16.8	24
-11	6	4	20.4	21.4	18
-12	6	4	12.2	11.8	343
-13	6	4	11.5	10.9	316
-14	6	4	2.8	3.2	312
-16	6	4	3.2	1.8	1
-17	6	4	5.2	6.6	348
0	6	5	20.6	17.2	145
1	6	5	16.6	16.3	230
2	6	5	14.0	13.9	213
3	6	5	13.1	9.6	133
4	6	5	29.2	33.1	182
5	6	5	27.4	29.7	192
6	6	5	7.5	6.4	273
7	6	5	6.1	5.7	103
8	6	5	7.9	5.9	176
9	6	5	5.7	4.4	102
10	6	5	6.5	5.6	190
11	6	5	5.2	3.6	196
12	6	5	4.2	3.9	189
-1	6	5	36.6	34.3	196
-2	6	5	25.2	24.1	182
-3	6	5	25.7	27.0	216
-4	6	5	12.5	12.1	189
-5	6	5	5.1	5.1	49
-6	6	5	13.3	12.1	193
-7	6	5	13.3	12.6	329
-8	6	5	19.6	17.6	204
-9	6	5	4.1	1.6	271
-10	6	5	9.5	10.3	197
-11	6	5	4.3	6.0	40
-12	6	5	8.0	7.9	338
-13	6	5	6.6	7.4	357
-14	6	5	4.0	4.4	195
-16	6	5	3.1	2.2	118
0	6	6	18.5	18.6	166
1	6	6	23.3	23.5	161
2	6	6	17.4	17.2	165
4	6	6	13.5	13.8	197
5	6	6	6.8	6.2	190
6	6	6	8.0	8.2	211
7	6	6	3.0	3.4	137
8	6	6	5.6	6.5	135
9	6	6	2.6	5.2	17
10	6	6	2.3	2.2	196
11	6	6	5.6	7.0	201
-1	6	6	6.6	6.3	59
-2	6	6	20.4	19.3	167
-3	6	6	21.9	20.4	188
-4	6	6	7.6	6.3	144
-5	6	6	11.2	11.5	226
-6	6	6	17.8	19.9	149
-7	6	6	11.8	10.9	175
-8	6	6	25.4	24.5	177
-9	6	6	20.6	19.0	178
-10	6	6	13.6	14.5	176
-11	6	6	21.2	20.6	200
-12	6	6	6.7	7.6	154
-13	6	6	12.3	13.5	191
-14	6	6	7.7	8.9	180
-15	6	6	5.0	4.1	177
-16	6	6	7.1	7.5	199
0	6	7	6.3	8.2	65
1	6	7	9.9	10.7	237
2	6	7	3.0	3.4	104
3	6	7	5.6	9.7	53
4	6	7	12.5	12.4	149
5	6	7	5.2	6.9	6
6	6	7	4.1	3.6	63
7	6	7	3.8	4.7	249
8	6	7	4.3	5.9	60
9	6	7	4.8	5.3	348
10	6	7	7.3	8.5	20
-1	6	7	12.1	11.2	156
-2	6	7	18.8	18.5	182
-3	6	7	11.9	12.2	356
-4	6	7	10.4	10.5	16
-5	6	7	12.3	11.2	89
-6	6	7	18.1	19.1	223
-7	6	7	7.3	8.8	185
-8	6	7	9.0	9.2	105
-9	6	7	4.3	1.5	312
-10	6	7	8.1	8.7	120
-11	6	7	12.5	11.3	179
-12	6	7	12.9	12.5	181
-13	6	7	10.9	13.8	203
-14	6	7	9.1	9.4	144
-15	6	7	9.5	9.2	170
-16	6	7	2.0	3.9	152
-17	6	7	2.0	3.0	338
0	6	8	15.0	16.7	39
1	6	8	15.0	13.8	342
2	6	8	16.4	19.4	352
3	6	8	22.6	25.3	12
4	6	8	7.1	7.7	85
5	6	8	7.8	6.8	202
6	6	8	7.3	7.4	331
7	6	8	4.7	3.0	358
8	6	8	2.8	1.9	303
9	6	8	3.6	3.2	18
-1	6	8	3.0	3.7	63
-2	6	8	14.2	13.3	341
-3	6	8	16.0	14.9	9
-4	6	8	18.9	19.5	29
-5	6	8	14.8	16.9	16
-6	6	8	9.1	10.1	51
-7	6	8	24.0	22.7	345
-8	6	8	3.0	4.6	115
-9	6	8	4.3	2.7	77
-1	6	8	9.0	9.3	81
-11	6	8	3.0	2.3	43
-12	6	8	2.8	4.0	299
-13	6	8	3.8	2.3	95
-14	6	8	6.1	7.2	155
-16	6	8	1.8	1.6	339
0	6	9	10.8	10.5	8
2	6	9	5.7	4.5	10
3	6	9	11.6	12.5	359
4	6	9	5.8	6.1	13
6	6	9	5.6	6.2	271
-1	6	9	21.4	20.6	334
-2	6	9	9.6	9.5	351
-3	6	9	11.8	10.7	348
-4	6	9	14.9	15.0	6
-5	6	9	6.1	6.9	324
-6	6	9	11.0	8.8	11
-7	6	9	9.6	9.7	1
-8	6	9	7.9	7.9	226
-9	6	9	6.9	8.8	33
-1	6	9	14.8	15.2	353
-11	6	9	7.9	7.9	341
-12	6	9	7.6	6.5	1
-13	6	9	4.3	5.1	68
-14	6	9	6.4	7.3	338
-15	6	9	5.4	6.7	34
-16	6	9	3.6	5.2	333
0	6	10	2.7	2.5	327
1	6	10	5.9	7.2	240
2	6	10	4.3	5.0	246
3	6	10	2.3	1.9	38
4	6	10	5.1	6.5	177
5	6	10	1.7	1.0	259
-1	6	10	2.8	1.5	95
-2	6	10	6.4	8.3	251
-3	6	10	9.6	10.9	14
-4	6	10	7.1	8.3	325
-5	6	10	5.1	6.1	123
-6	6	10	11.7	11.1	254
-7	6	10	4.1	4.2	187
-8	6	10	7.0	8.9	30
-9	6	10	7.7	8.3	336
-10	6	10	7.6	9.1	343
-11	6	10	2.5	1.8	228
-12	6	10	3.4	4.1	335
-13	6	10	3.1	2.8	320
-14	6	10	6.0	7.6	14
-15	6	10	1.4	1.8	314
0	6	11	6.1	6.9	144
1	6	11	9.4	10.3	195
2	6	11	5.1	5.8	246
3	6	11	4.3	5.5	356
-1	6	11	9.4	12.2	182
-2	6	11	6.2	7.9	238
-3	6	11	5.2	5.1	169
-4	6	11	5.9	7.1	184
-5	6	11	9.1	9.8	197
-6	6	11	9.1	11.8	204
-8	6	11	9.6	10.6	198
-9	6	11	8.6	8.9	171
-11	6	11	5.0	5.5	251
-12	6	11	4.0	5.9	269
-13	6	11	1.7	2.8	39
-14	6	11	1.2	1.5	159
0	6	12	2.2	2.0	160
-1	6	12	1.8	1.0	235
-2	6	12	3.9	4.6	177
-3	6	12	7.2	8.1	150
-4	6	12	4.3	5.4	145
-5	6	12	9.2	10.8	170
-6	6	12	5.3	6.7	161
-7	6	12	5.3	5.3	207
-8	6	12	11.5	13.8	174
-9	6	12	2.8	4.4	87
-10	6	12	4.2	4.7	6
-11	6	12	3.7	3.7	220
-12	6	12	2.9	3.6	187
-6	6	13	2.3	1.3	236
-8	6	13	2.0	4.9	60
-9	6	13	1.3	2.1	327
1	7	0	22.2	23.9	271
2	7	0	15.6	16.8	183
3	7	0	6.5	3.9	108
4	7	0	24.2	22.3	228
5	7	0	9.9	10.4	39
6	7	0	18.2	18.1	325
7	7	0	16.0	15.8	279
8	7	0	9.9	8.4	276
9	7	0	11.6	11.9	26
10	7	0	6.1	6.9	333
11	7	0	7.4	9.2	341
12	7	0	6.5	6.9	55
13	7	0	2.7	4.3	354
14	7	0	3.3	3.3	18
15	7	0	4.2	4.9	316
0	7	1	23.1	20.2	43
1	7	1	29.3	34.0	1
2	7	1	18.0	18.7	323
3	7	1	13.1	13.9	332
4	7	1	23.8	25.9	321
5	7	1	27.1	30.1	89
6	7	1	31.9	29.5	94
7	7	1	14.0	13.7	325
8	7	1	14.4	15.4	18
9	7	1	11.7	12.2	328
10	7	1	13.9	12.8	26
11	7	1	6.6	6.7	335
12	7	1	2.8	2.8	24
13	7	1	5.6	5.6	15
14	7	1	4.2	4.3	338
15	7	1	4.1	4.0	3
-1	7	1	14.0	13.3	67
-2	7	1	29.7	28.9	333
-3	7	1	25.5	22.7	356
-4	7	1	25.1	26.4	342
-5	7	1	4.9	3.9	199
-6	7	1	38.6	39.2	306
-7	7	1	43.2	40.2	26
-8	7	1	22.0	17.6	114
-9	7	1	8.3	7.5	22
-10	7	1	13.9	13.4	273
-11	7	1	8.0	8.1	221
-12	7	1	4.1	4.3	308
-13	7	1	3.9	2.6	118
-14	7	1	3.5	4.1	60
-16	7	1	5.6	4.3	3
0	7	2	24.9	24.5	310
1	7	2	27.1	26.1	341
2	7	2	23.1	22.8	2
3	7	2	11.8	11.7	334
4	7	2	24.5	23.0	294
5	7	2	33.2	36.0	1
6	7	2	20.5	20.4	79
7	7	2	22.4	19.9	179
8	7	2	24.8	23.3	292
9	7	2	9.1	8.9	225
10	7	2	5.2	3.3	138
11	7	2	4.9	4.8	46
12	7	2	3.7	3.0	109
13	7	2	3.2	3.4	11
14	7	2	1.8	2.1	291
-1	7	2	6.9	5.4	112
-2	7	2	23.3	22.5	340
-3	7	2	30.5	31.4	33
-4	7	2	24.1	22.4	347
-5	7	2	41.6	40.7	329
-6	7	2	31.2	28.3	299
-7	7	2	44.6	43.6	95
-8	7	2	11.2	9.5	73
-9	7	2	10.5	9.5	5
-10	7	2	5.2	3.7	240
-11	7	2	12.5	11.0	22
-12	7	2	12.3	11.8	7
-13	7	2	6.3	6.0	36
-14	7	2	5.8	5.6	22
-15	7	2	3.9	4.1	12
-16	7	2	7.2	6.3	1
0	7	3	8.9	7.7	120
1	7	3	14.7	11.5	321
2	7	3	6.5	6.9	177
3	7	3	14.1	15.5	112
4	7	3	18.0	20.0	222
5	7	3	13.3	13.6	112
6	7	3	7.7	6.8	3
7	7	3	13.8	14.2	149
8	7	3	12.5	11.6	219
9	7	3	6.7	7.8	173
10	7	3	8.2	10.3	212
11	7	3	6.0	4.7	161
12	7	3	2.3	2.4	205
-1	7	3	19.2	15.0	82
-2	7	3	11.0	9.5	319
-3	7	3	14.5	12.4	215
-4	7	3	4.8	4.3	59
-5	7	3	22.5	15.7	244
-6	7	3	23.9	20.5	171
-7	7	3	21.9	20.3	63
-8	7	3	22.6	21.9	288
-9	7	3	17.4	17.3	271
-10	7	3	6.7	6.3	286
-11	7	3	4.3	4.4	52
-12	7	3	9.9	10.9	305
-13	7	3	4.9	4.2	344
-14	7	3	5.9	4.0	67
-15	7	3	3.2	4.9	12
-16	7	3	3.6	3.7	329
0	7	4	22.0	21.4	154
2	7	4	18.6	18.8	207
3	7	4	10.5	11.6	227
4	7	4	24.5	25.2	265
5	7	4	8.3	8.1	98
6	7	4	20.9	21.2	175
7	7	4	14.3	15.3	148
8	7	4	7.9	7.9	162
9	7	4	7.1	6.9	192
10	7	4	4.7	5.0	176
11	7	4	5.4	5.9	187
12	7	4	2.8	3.9	143
-1	7	4	18.8	19.4	160
-2	7	4	21.3	20.2	230
-3	7	4	19.5	20.1	200
-4	7	4	25.2	23.7	216
-5	7	4	31.7	30.8	167
-6	7	4	11.4	9.0	214
-7	7	4	14.5	12.3	85
-8	7	4	27.4	25.5	144
-9	7	4	19.6	17.7	242
-10	7	4	7.4	5.8	170
-11	7	4	13.9	11.7	228
-12	7	4	5.9	5.8	98
-13	7	4	4.0	3.1	165
-14	7	4	4.5	3.6	121
-16	7	4	3.2	3.4	288
0	7	5	16.1	16.6	156
1	7	5	10.5	9.3	173
2	7	5	17.2	18.2	90
3	7	5	13.6	12.7	146
4	7	5	12.6	11.5	227
5	7	5	20.3	18.9	355
6	7	5	12.5	13.6	77
7	7	5	10.8	9.8	156
8	7	5	4.0	4.1	323
10	7	5	3.4	2.5	148
-1	7	5	23.2	20.9	177
-2	7	5	11.2	10.3	163
-3	7	5	15.2	13.8	144
-4	7	5	12.5	13.2	173
-5	7	5	15.3	14.3	216
-6	7	5	32.5	32.0	186
-7	7	5	48.4	47.5	127
-8	7	5	10.5	10.1	188
-9	7	5	23.6	22.0	280
-10	7	5	15.2	15.9	195
-11	7	5	6.0	5.4	1
-13	7	5	6.3	5.8	172
-14	7	5	6.4	6.2	172
-15	7	5	7.9	8.0	222
-16	7	5	3.7	4.6	199
0	7	6	10.6	9.9	78
1	7	6	13.8	15.1	341
2	7	6	11.6	11.1	56
3	7	6	5.2	5.0	355
4	7	6	7.4	8.0	228
6	7	6	12.8	14.4	1
7	7	6	9.3	10.6	67
8	7	6	6.4	7.7	359
9	7	6	6.6	5.4	345
10	7	6	2.7	2.2	282
-1	7	6	8.5	7.6	24
-2	7	6	8.4	8.3	346
-3	7	6	7.9	7.0	137
-4	7	6	4.0	2.6	76
-5	7	6	11.7	9.8	269
-6	7	6	22.1	20.3	145
-7	7	6	23.4	22.2	40
-8	7	6	19.9	17.0	302
-9	7	6	12.5	13.1	261
-10	7	6	14.5	13.5	87
-11	7	6	7.9	8.7	93
-12	7	6	5.8	4.7	37
-14	7	6	6.2	7.1	113
-15	7	6	3.8	2.3	185
0	7	7	11.3	11.1	49
1	7	7	11.3	11.5	17
2	7	7	5.2	4.5	55
4	7	7	14.2	14.5	312
5	7	7	14.3	16.1	352
6	7	7	6.6	6.8	300
7	7	7	5.6	6.2	35
8	7	7	5.4	6.6	15
9	7	7	5.1	5.7	347
-1	7	7	14.0	12.7	11
-2	7	7	9.9	10.4	18
-3	7	7	11.1	11.7	320
-4	7	7	13.3	14.7	1
-6	7	7	24.1	23.6	23
-7	7	7	13.9	13.5	359
-8	7	7	8.0	8.3	18
-9	7	7	12.5	10.6	295
-10	7	7	11.6	11.4	40
-11	7	7	4.1	3.4	352
-12	7	7	7.9	7.4	20
-13	7	7	2.6	1.4	10
-14	7	7	4.8	5.0	347
-15	7	7	2.0	2.6	15
-16	7	7	3.0	3.0	300
0	7	8	6.0	5.0	268
1	7	8	11.2	11.4	259
2	7	8	6.6	7.1	73
3	7	8	5.7	6.1	175
4	7	8	7.7	9.0	197
5	7	8	7.7	8.4	332
6	7	8	3.3	3.5	348
7	7	8	3.5	2.8	219
-1	7	8	6.8	6.6	284
-2	7	8	12.9	14.0	311
-3	7	8	7.4	7.1	32
-4	7	8	15.2	16.1	339
-5	7	8	10.1	10.9	28
-6	7	8	5.2	5.6	323
-7	7	8	16.3	15.1	59
-8	7	8	24.1	24.6	334
-9	7	8	24.6	27.3	305
-10	7	8	7.7	8.6	81
-11	7	8	8.4	10.0	23
-12	7	8	7.5	8.5	35
-14	7	8	3.8	2.6	358
-15	7	8	2.6	4.3	283
0	7	9	4.9	5.4	189
1	7	9	4.8	4.3	211
2	7	9	3.8	4.6	74
4	7	9	7.0	8.5	202
5	7	9	7.2	8.5	199
6	7	9	2.4	2.5	254
-1	7	9	8.3	9.2	214
-2	7	9	2.9	3.6	266
-3	7	9	8.9	9.9	187
-4	7	9	4.2	4.0	260
-5	7	9	10.3	10.6	270
-6	7	9	5.9	6.1	149
-7	7	9	12.1	14.0	10
-8	7	9	12.6	14.7	291
-9	7	9	9.8	10.7	214
-1	7	9	9.5	10.1	115
-11	7	9	5.2	4.7	112
-12	7	9	4.2	4.2	253
-13	7	9	2.2	2.0	192
-14	7	9	2.7	3.1	237
-15	7	9	1.9	3.4	287
0	7	10	3.6	5.4	179
1	7	10	2.4	2.8	181
2	7	10	6.3	7.0	127
3	7	10	9.6	10.3	165
4	7	10	2.9	3.4	186
-1	7	10	2.6	4.1	178
-2	7	10	9.3	10.3	173
-3	7	10	6.7	8.9	213
-4	7	10	3.9	4.7	222
-5	7	10	6.2	7.0	144
-6	7	10	3.9	3.9	160
-7	7	10	7.2	8.7	218
-8	7	10	8.8	10.0	191
-9	7	10	7.3	9.1	198
-1	7	10	7.0	8.1	143
-11	7	10	5.7	6.1	195
-12	7	10	4.2	4.9	230
-13	7	10	5.7	6.8	198
-14	7	10	1.8	2.7	272
0	7	11	2.0	1.4	305
1	7	11	1.8	3.4	303
2	7	11	2.7	4.6	35
-1	7	11	3.0	3.9	78
-3	7	11	4.0	5.4	112
-4	7	11	4.8	5.3	87
-5	7	11	6.8	7.7	126
-6	7	11	5.8	6.6	174
-7	7	11	3.3	5.4	188
-8	7	11	4.0	4.6	297
-9	7	11	8.5	10.6	220
-10	7	11	12.8	15.9	129
-11	7	11	5.2	5.9	149
-12	7	11	3.1	4.7	179
-2	7	12	2.9	5.2	15
-3	7	12	2.3	3.0	82
-4	7	12	2.5	2.2	355
-5	7	12	1.8	2.9	28
-6	7	12	1.9	3.5	123
-7	7	12	4.3	7.4	38
-8	7	12	1.7	2.5	200
-9	7	12	5.0	7.2	155
-10	7	12	4.3	9.5	104
1	8	0	13.4	14.3	13
2	8	0	19.7	21.8	356
3	8	0	9.0	9.4	279
4	8	0	4.0	4.0	326
5	8	0	21.2	21.9	14
6	8	0	39.8	39.8	325
7	8	0	5.2	4.2	74
8	8	0	21.5	19.7	13
9	8	0	9.6	6.7	20
10	8	0	6.7	5.7	34
11	8	0	6.5	4.0	323
12	8	0	6.6	7.6	42
13	8	0	3.8	4.1	354
0	8	1	7.4	9.0	4
1	8	1	14.3	15.0	25
2	8	1	19.2	21.8	5
3	8	1	14.3	14.9	199
4	8	1	18.5	18.0	152
5	8	1	38.1	38.6	112
6	8	1	43.4	43.4	311
7	8	1	14.9	14.0	250
8	8	1	9.2	7.3	221
10	8	1	3.8	2.0	228
11	8	1	5.2	5.3	160
13	8	1	7.0	6.8	208
14	8	1	4.2	5.1	179
-1	8	1	14.3	14.9	336
-2	8	1	23.4	21.4	26
-3	8	1	13.0	12.8	10
-4	8	1	25.1	27.8	22
-5	8	1	23.3	19.5	80
-6	8	1	17.1	18.2	19
-7	8	1	37.3	33.2	327
-8	8	1	14.5	14.7	31
-9	8	1	7.3	6.6	9
-10	8	1	10.4	8.7	287
-11	8	1	11.5	12.4	327
-12	8	1	7.7	7.4	9
-13	8	1	4.7	4.0	358
0	8	2	23.3	23.6	188
1	8	2	26.9	27.2	287
2	8	2	12.8	13.9	204
3	8	2	16.4	17.6	211
4	8	2	16.9	19.7	202
5	8	2	39.3	39.9	150
6	8	2	14.6	14.8	129
7	8	2	7.3	6.9	225
8	8	2	7.9	7.5	174
9	8	2	9.1	8.9	161
10	8	2	4.5	5.2	177
11	8	2	6.0	5.8	223
12	8	2	3.7	4.7	106
13	8	2	2.9	3.3	173
-1	8	2	3.6	4.6	221
-2	8	2	4.1	3.2	132
-3	8	2	15.9	15.0	194
-4	8	2	15.9	11.9	165
-5	8	2	20.1	17.9	5
-6	8	2	60.4	61.6	298
-7	8	2	10.5	8.7	281
-8	8	2	24.8	20.3	132
-9	8	2	8.7	7.2	60
-1	8	2	10.4	9.7	120
-11	8	2	6.7	5.9	9
-13	8	2	2.4	4.9	270
-14	8	2	3.0	0.9	50
-15	8	2	4.0	4.9	347
0	8	3	21.8	18.3	164
1	8	3	20.6	19.6	169
2	8	3	14.4	15.5	139
3	8	3	19.1	22.2	211
4	8	3	31.4	35.6	161
5	8	3	11.3	11.9	183
6	8	3	14.0	14.0	238
7	8	3	13.1	12.9	188
8	8	3	4.8	3.0	164
9	8	3	4.6	2.8	166
10	8	3	7.4	7.6	177
11	8	3	8.0	6.9	208
12	8	3	1.8	1.9	171
-1	8	3	13.1	12.9	153
-2	8	3	18.6	17.7	193
-3	8	3	22.5	19.0	196
-4	8	3	11.7	11.4	172
-5	8	3	12.8	10.4	31
-6	8	3	16.0	10.9	303
-7	8	3	23.3	21.4	152
-8	8	3	19.6	17.7	147
-9	8	3	11.0	8.9	165
-10	8	3	15.0	13.0	185
-11	8	3	2.7	1.7	244
-12	8	3	7.0	6.5	171
-13	8	3	3.5	4.1	246
-14	8	3	3.7	3.5	196
-15	8	3	1.7	1.0	337
0	8	4	17.6	15.8	168
1	8	4	5.9	5.4	18
2	8	4	8.7	7.7	7
3	8	4	15.7	13.3	295
4	8	4	13.5	13.4	173
5	8	4	27.1	30.8	109
6	8	4	15.0	15.7	349
7	8	4	2.7	1.2	232
8	8	4	2.6	0.6	138
9	8	4	5.0	4.3	26
10	8	4	3.9	3.6	39
11	8	4	1.9	1.4	45
12	8	4	2.4	5.3	39
-1	8	4	8.5	7.9	223
-2	8	4	19.5	19.1	167
-3	8	4	10.7	9.2	214
-4	8	4	5.3	6.1	159
-5	8	4	15.6	13.0	140
-6	8	4	21.4	22.2	194
-7	8	4	19.6	15.2	284
-8	8	4	21.6	20.9	140
-9	8	4	18.4	17.3	187
-10	8	4	16.9	16.7	209
-11	8	4	4.7	5.7	273
-12	8	4	9.5	9.0	115
-13	8	4	3.5	4.5	162
-14	8	4	7.8	6.9	158
-15	8	4	5.0	5.3	120
0	8	5	6.8	5.9	83
1	8	5	16.4	16.8	336
2	8	5	15.9	17.3	356
3	8	5	23.8	26.6	345
4	8	5	14.4	14.9	300
5	8	5	2.8	3.9	71
6	8	5	11.9	11.8	57
7	8	5	11.1	10.5	11
8	8	5	3.5	3.2	260
9	8	5	4.4	4.1	12
10	8	5	3.2	3.4	350
11	8	5	2.5	4.3	331
-1	8	5	17.5	15.1	38
-2	8	5	12.8	11.6	61
-3	8	5	6.1	6.4	32
-4	8	5	8.4	8.4	71
-5	8	5	2.5	1.8	347
-6	8	5	35.5	28.3	302
-7	8	5	10.5	8.7	105
-8	8	5	41.7	39.6	115
-9	8	5	2.8	1.0	351
-10	8	5	3.9	6.2	132
-11	8	5	7.7	7.3	344
-12	8	5	4.5	4.1	274
-13	8	5	4.8	4.4	206
-15	8	5	1.7	1.6	29
0	8	6	12.2	11.9	9
1	8	6	14.3	14.9	1
2	8	6	20.8	21.6	23
3	8	6	19.5	20.5	339
4	8	6	10.7	11.8	77
5	8	6	6.6	6.9	350
6	8	6	6.3	7.4	316
7	8	6	4.8	3.2	340
8	8	6	3.7	4.3	10
9	8	6	5.7	6.0	359
-1	8	6	17.3	17.2	346
-2	8	6	13.4	11.9	324
-3	8	6	9.7	9.0	319
-4	8	6	13.5	13.3	347
-5	8	6	10.9	11.8	27
-6	8	6	10.6	10.8	324
-7	8	6	19.6	14.4	120
-8	8	6	7.4	6.3	206
-9	8	6	8.3	8.6	352
-10	8	6	9.5	9.9	314
-11	8	6	10.6	10.9	350
-13	8	6	3.3	3.3	283
-14	8	6	4.5	5.3	44
-15	8	6	1.6	2.9	35
1	8	7	2.8	2.9	92
2	8	7	3.9	4.7	9
3	8	7	14.2	16.8	311
4	8	7	6.4	8.4	245
5	8	7	5.5	7.6	137
6	8	7	5.1	3.6	338
7	8	7	2.8	3.3	221
8	8	7	2.8	3.7	240
-1	8	7	2.8	2.1	315
-2	8	7	3.9	4.2	283
-3	8	7	6.2	5.6	327
-4	8	7	10.0	9.3	356
-5	8	7	2.8	4.3	35
-6	8	7	7.4	6.4	211
-7	8	7	14.4	14.2	307
-8	8	7	10.0	10.4	63
-9	8	7	4.7	5.6	257
-1	8	7	13.3	15.3	322
-11	8	7	10.2	11.6	4
-12	8	7	6.7	8.7	32
0	8	8	9.7	10.8	173
1	8	8	9.2	11.7	174
2	8	8	5.1	4.7	162
4	8	8	6.5	9.8	252
5	8	8	9.3	8.9	206
6	8	8	1.8	2.0	112
7	8	8	2.5	3.2	121
-1	8	8	7.2	6.6	163
-2	8	8	6.7	6.7	155
-3	8	8	2.7	3.0	244
-4	8	8	3.9	3.8	227
-5	8	8	12.0	13.3	217
-6	8	8	9.5	10.3	271
-7	8	8	10.9	9.9	107
-8	8	8	18.0	17.3	130
-9	8	8	15.0	14.9	283
-10	8	8	8.0	8.0	294
-11	8	8	5.6	8.0	333
-12	8	8	3.8	4.2	200
-13	8	8	3.9	3.5	198
0	8	9	9.2	11.3	196
1	8	9	7.9	9.5	163
2	8	9	4.4	5.7	163
4	8	9	8.0	9.8	127
5	8	9	2.8	3.0	184
-1	8	9	7.6	8.3	183
-2	8	9	5.2	6.5	209
-3	8	9	6.9	6.9	164
-4	8	9	2.6	3.3	160
-5	8	9	9.0	9.9	147
-6	8	9	4.5	4.6	148
-7	8	9	9.6	8.7	147
-8	8	9	8.3	8.6	216
-9	8	9	5.4	5.3	314
-10	8	9	3.2	3.6	320
-11	8	9	2.1	1.8	127
-12	8	9	5.4	6.6	147
-13	8	9	2.8	4.3	185
0	8	10	2.0	3.1	125
1	8	10	7.1	7.7	154
2	8	10	5.7	6.8	126
3	8	10	2.4	4.5	333
-2	8	10	2.2	2.9	135
-4	8	10	2.3	3.6	52
-6	8	10	2.3	4.4	192
-7	8	10	3.9	3.4	310
-8	8	10	4.4	5.2	85
-9	8	10	2.1	3.4	105
-10	8	10	3.4	4.1	313
-11	8	10	7.0	7.8	92
-12	8	10	4.8	7.1	151
-1	8	11	2.1	4.3	14
-2	8	11	1.7	2.5	53
-3	8	11	5.4	6.9	7
-4	8	11	2.6	2.9	352
-6	8	11	3.7	4.8	34
-7	8	11	8.7	10.0	37
-8	8	11	1.7	3.8	93
-9	8	11	7.9	10.7	340
-10	8	11	4.2	5.9	7

Table 2-5 Aflatoxin G_1 bromobenzene solvate

Bond lengths and their estimated standard deviations

	Molecule 1		Molecule 1'		Average
C(1)-O(2)	1.342	±0.016	1.346	±0.017	1.344
C(1)-C(6)	1.410	0.018	1.438	0.018	1.424
C(1)-O(21)	1.247	0.021	1.255	0.020	1.251
O(2)-C(3)	1.465	0.022	1.423	0.019	1.444
C(3)-C(4)	1.505	0.021	1.518	0.018	1.512
C(4)-C(5)	1.485	0.017	1.510	0.015	1.498
C(5)-C(6)	1.425	0.022	1.389	0.020	1.407
C(5)-C(10)	1.416	0.016	1.415	0.017	1.415
C(6)-C(7)	1.405	0.019	1.449	0.017	1.427
C(7)-O(8)	1.378	0.016	1.382	0.015	1.380
C(7)-O(22)	1.226	0.021	1.197	0.019	1.212
O(8)-C(9)	1.402	0.015	1.417	0.019	1.410
C(9)-C(10)	1.350	0.013	1.402	0.017	1.376
C(9)-C(14)	1.348	0.015	1.313	0.016	1.331
C(10)-C(11)	1.534	0.018	1.456	0.021	1.495
C(11)-C(12)	1.333	0.016	1.439	0.020	1.386
C(12)-C(13)	1.347	0.017	1.327	0.022	1.337
C(13)-C(14)	1.410	0.025	1.415	0.022	1.413
C(13)-O(17)	1.366	0.016	1.373	0.017	1.370
C(14)-C(15)	1.475	0.017	1.516	0.016	1.496
C(15)-C(16)	1.486	0.025	1.607	0.027	1.547
C(15)-C(20)	1.475	0.019	1.490	0.018	1.483
C(16)-O(17)	1.452	0.023	1.427	0.023	1.440
C(16)-O(18)	1.451	0.016	1.347	0.020	1.399
O(18)-C(19)	1.356	0.020	1.438	0.025	1.397
C(19)-C(20)	1.302	0.020	1.303	0.026	1.303
C(11)-O(23)	1.352	0.016	1.340	0.016	1.346
O(23)-C(24)	1.451	0.020	1.467	0.022	1.459

Table 2-6 Aflatoxin G_1 bromobenzene solvate

Bond angles and their estimated standard deviations

	Molecule 1		Molecule 1'		Average
O(2)C(1)O(21)	115.7	±1.1°	116.5	±1.2°	116.1°
C(6)C(1)O(21)	124.0	1.2	124.7	1.2	124.4
C(6)C(1)O(2)	120.4	1.4	118.8	1.4	119.6
C(1)O(2)C(3)	115.8	1.1	117.2	1.1	116.5
O(2)C(3)C(4)	109.7	1.2	112.2	1.1	111.0
C(3)C(4)C(5)	111.3	1.2	109.4	1.1	110.4
C(4)C(5)C(6)	114.8	1.1	116.3	1.0	115.6
C(6)C(5)C(10)	117.6	1.1	120.3	1.0	119.0
C(4)C(5)C(10)	127.7	1.3	123.4	1.2	125.6
C(5)C(6)C(7)	119.8	1.2	121.7	1.1	120.8
C(1)C(6)C(5)	120.5	1.2	121.4	1.1	121.0
C(1)C(6)C(7)	119.1	1.4	116.7	1.3	117.9
C(6)C(7)O(22)	126.1	1.3	127.5	1.2	126.8
O(8)C(7)O(22)	113.4	1.2	116.6	1.1	115.0
C(6)C(7)O(8)	120.3	1.4	115.9	1.2	118.1
C(7)O(8)C(9)	119.7	0.9	123.2	0.9	121.5
O(8)C(9)C(10)	121.1	0.8	119.4	1.1	120.3
O(8)C(9)C(14)	113.5	1.0	114.3	1.1	113.9
C(10)C(9)C(14)	125.4	1.2	126.3	1.5	125.9
C(9)C(10)C(5)	121.4	1.1	119.0	1.3	120.2
C(9)C(10)C(11)	115.8	0.9	114.6	1.1	115.2
C(5)C(10)C(11)	122.9	1.0	126.5	1.1	124.7
C(10)C(11)C(12)	118.1	1.1	121.2	1.2	119.7
C(10)C(11)O(23)	113.1	1.0	114.8	1.1	114.0
C(12)C(11)O(23)	128.8	1.3	123.9	1.4	126.4
C(11)C(12)C(13)	121.0	1.3	114.9	1.7	118.0
C(12)C(13)C(14)	123.5	1.1	127.2	1.3	125.4
C(12)C(13)O(17)	124.4	1.4	120.7	1.7	122.6
C(14)C(13)O(17)	112.1	1.1	112.0	1.2	112.1
C(13)C(14)C(9)	116.2	1.1	115.4	1.1	115.8
C(15)C(14)C(9)	136.2	1.4	135.0	1.4	135.6
C(13)C(14)C(15)	107.2	1.1	109.3	1.1	108.3
C(14)C(15)C(16)	103.4	1.3	101.0	1.3	102.2
C(14)C(15)C(20)	116.4	1.0	112.1	0.9	114.3
C(16)C(15)C(20)	103.4	1.3	101.5	1.2	102.5
C(15)C(16)O(17)	107.7	1.0	106.8	1.2	107.3
C(15)C(16)O(18)	106.2	1.3	105.8	1.4	106.0
O(17)C(16)O(18)	106.0	1.3	110.0	1.5	108.0
C(13)O(17)C(16)	107.1	1.2	110.8	1.4	109.0
C(16)O(18)C(19)	106.4	1.2	110.4	1.5	108.4
O(18)C(19)C(20)	114.8	1.2	112.2	1.6	113.5
C(19)C(20)C(15)	108.7	1.3	110.2	1.6	109.5
C(11)O(23)C(24)	115.3	1.0	120.9	1.1	118.6

Table 2-7

Bond lengths and angles of the bromobenzene molecule and their estimated standard deviations

C(25)-C(26)	1.341	±0.025	C(29)-C(30)	1.360	±0.027
C(26)-C(27)	1.334	0.023	C(30)-C(25)	1.421	0.028
C(27)-C(28)	1.376	0.031	Br - C(25)	1.946	0.016
C(28)-C(29)	1.336	0.029			

C(26)C(25)Br	119.9	±1.4°	C(26)C(27)C(28)	119.3	±1.8°
C(30)C(25)Br	116.3	1.3	C(27)C(28)C(29)	122.4	1.7
C(26)C(25)C(30)	123.7	1.6	C(28)C(29)C(30)	120.8	2.1
C(25)C(26)C(27)	118.6	1.8	C(29)C(30)C(25)	115.0	1.8

Intramolecular non-bonded distances of the bromobenzene molecule

C(25)...C(27)	2.30	C(26)...Br	2.86
C(25)...C(29)	2.35	C(27)...C(29)	2.38
C(25)...C(28)	2.65	C(27)...C(30)	2.77
C(26)...C(28)	2.34	C(28)...C(30)	2.34
C(26)...C(30)	2.43	C(30)...Br	2.87
C(26)...C(29)	2.74		

Bond lengths and angles in
aflatoxin G_1 bromobenzene solvate.

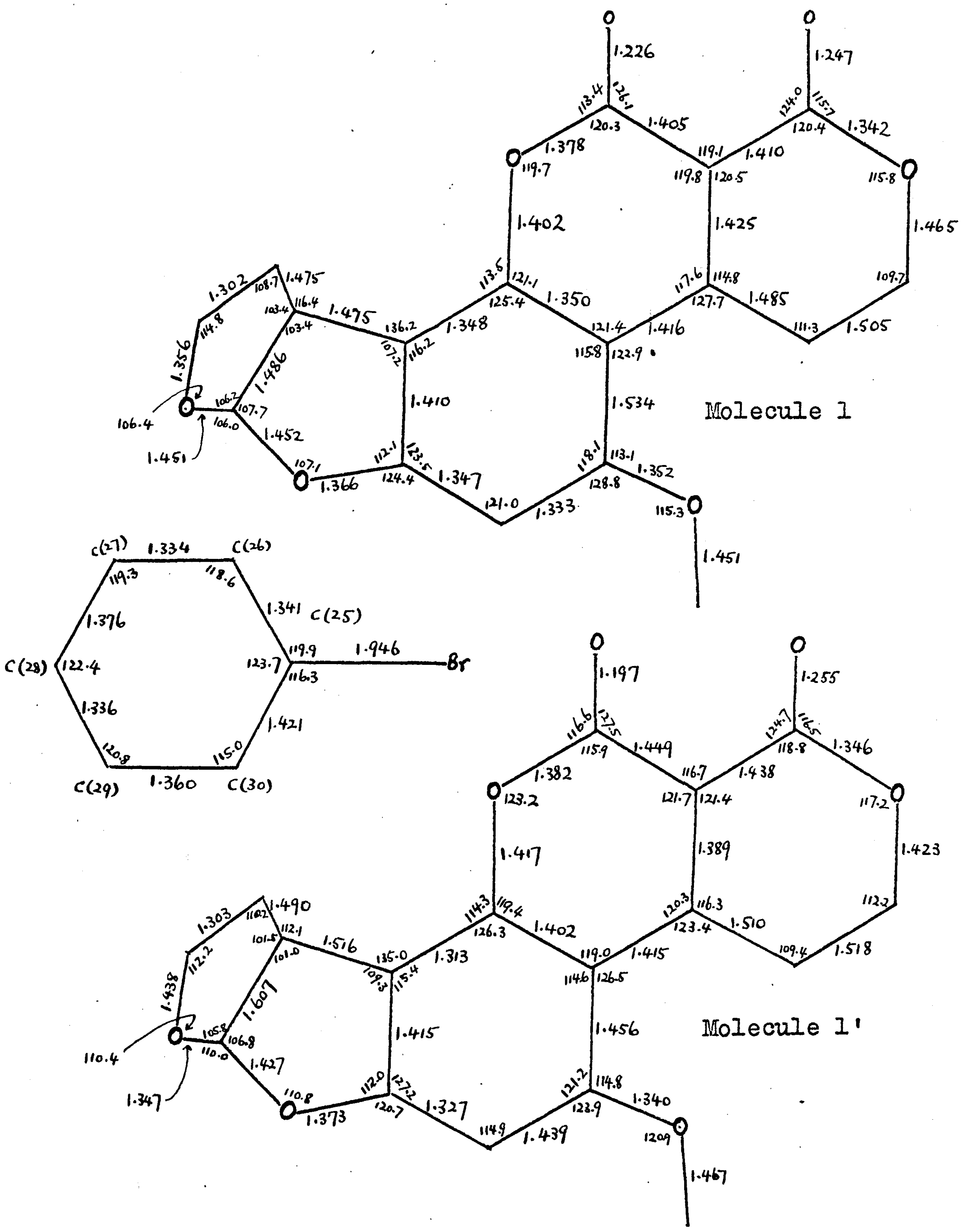


Table 2-8 Aflatoxin G_1 bromobenzene solvate

Intramolecular non-bonded distances $\leqslant 3.5$Å

	Mol. 1	Mol. 1'
C(1)...C(3)	2.38	2.36
C(1)...C(7)	2.43	2.46
C(1)...C(5)	2.46	2.47
C(1)...C(4)	2.78	2.82
C(1)...O(22)	2.85	2.84
O(2)...O(21)	2.19	2.21
O(2)...C(6)	2.39	2.40
O(2)...C(4)	2.43	2.44
C(3)...C(5)	2.47	2.47
C(3)...C(6)	2.70	2.70
C(3)...O(21)	3.51	3.49
C(4)...C(6)	2.45	2.46
C(4)...C(10)	2.60	2.57
C(4)...O(23)	2.71	2.72
C(4)...C(11)	3.16	3.12
C(5)...C(9)	2.41	2.43
C(5)...C(7)	2.45	2.48
C(5)...C(11)	2.59	2.56
C(5)...O(8)	2.81	2.78
C(5)...O(23)	2.85	2.86
C(6)...O(21)	2.35	2.37
C(6)...O(22)	2.35	2.39
C(6)...O(8)	2.41	2.40
C(6)...C(10)	2.43	2.43
C(6)...C(9)	2.78	2.80
C(7)...C(9)	2.40	2.46
C(7)...C(10)	2.78	2.86
C(7)...O(21)	2.82	2.83
O(8)...O(22)	2.18	2.20
O(8)...C(14)	2.30	2.29
O(8)...C(10)	2.40	2.43
O(8)...C(15)	2.99	2.98
O(8)...C(20)	3.54	3.37
C(9)...C(13)	2.34	2.31
C(9) ...C(11)	2.44	2.40
C(9) ...C(15)	2.62	2.61
C(9) ...C(12)	2.75	2.79
C(9) ...C(20)	3.40	3.29
C(9) ...O(22)	3.47	3.51
C(9) ...O(17)	3.53	3.50
C(10)...C(14)	2.40	2.42
C(10)...O(23)	2.41	2.36
C(10)...C(12)	2.46	2.52
C(10)...C(13)	2.76	2.75
C(11)...C(13)	2.33	2.33
C(11)...C(24)	2.37	2.44
C(11)...C(14)	2.81	2.81
C(12)...O(17)	2.40	2.35
C(12)...O(23)	2.42	2.45
C(12)...C(14)	2.43	2.46
C(12)...C(24)	2.82	2.92
C(13)...C(16)	2.27	2.30
C(13)...C(15)	2.32	2.39
C(13)...C(20)	3.25	3.18
C(13)...O(18)	3.28	3.33
C(14)...O(17)	2.30	2.31
C(14)...C(16)	2.32	2.41
C(14)...C(20)	2.51	2.49
C(14)...C(19)	3.35	3.30
C(14)...O(18)	3.39	3.29
C(15)...C(19)	2.29	2.29
C(15)...O(18)	2.35	2.36
C(15)...O(17)	2.37	2.44
C(16)...C(19)	2.25	2.29
C(16)...C(20)	2.32	2.40
O(17)...O(18)	2.32	2.27
O(17)...C(19)	3.12	3.24
O(17)...C(20)	3.20	3.32
O(18)...C(20)	2.24	2.28

Table 2-9 Aflatoxin G_1 bromobenzene solvate

Intermolecular distances ≤ 4Å

O(22)...C(24)XI	3.11
C(4) ...O(21)X	3.16
C(3) ...O(21)X	3.25
C(16)...O(22)IV	3.30
O(21)...C(24)XI	3.43
O(2) ...C(24)IX	3.55
O(2) ...O(23)IX	3.56
O(8) ...O(17)II	3.68
O(18)...O(22)IV	3.75
O(2) ...C(4)IX	3.87
O(8)...C(16)II	3.88
O(17)...O(22)IV	3.94
O(2) ...O(21)X	3.96
C(3) ...C(24)IX	3.96
C(1) ...C(4)IX	3.98
C(7) ...C(16)II	3.98

O(21')...C(24')XII	3.18
C(19')...O(22')VI	3.20
O(22')...C(24')XII	3.24
C(3') ...O(21')VII	3.28
O(18')...O(22')VI	3.38
O(8') ...O(18')V	3.76
C(7')...O(18')V	3.77
C(3') ...C(24')VIII	3.88
O(17')...C(20')VI	3.88
O(2') ...C(4')VIII	3.91
C(4') ...O(21')VII	3.91
C(7') ...C(19')V	3.94

C(30)...O(17)II	3.16
Br . . .C(16)	3.51
C(30)...O(18)II	3.53
Br . . .C(14)	3.57
Br . . .O(8)IV	3.62
C(26)...C(10)	3.67
Br . . .O(22)IV	3.67
Br . . .C(13)	3.68
Br . . .O(17)	3.68
Br . . .C(20)IV	3.68
C(25)...C(9)	3.69
Br . . .C(15)	3.69
C(26)...C(20)IV	3.71
C(26)...C(11)	3.72
C(29)...O(18)II	3.52

C(25)...C(14)	3.76
C(27)...C(10)	3.80
C(27)...C(5)	3.82
C(26)...C(9)	3.87
C(30)...C(16)II	3.88
C(26)...C(12)	3.92
C(29)...O(17)II	3.94
C(25)...C(10)	3.95
C(28)...C(5)	3.95

C(28)...C(9')	3.64
C(29)...C(11')	3.66
C(28)...C(14')	3.72
C(28)...C(10')	3.75
C(29)...C(10')	3.75
Br . . .O(21')VII	3.75
C(27)...O(17')V	3.79
Br . . . C(3')	3.81
C(27)...C(9')	3.83
C(27)...O(18')V	3.83
C(28)...C(13')	3.87
C(28)...C(19')	3.89
C(29)...C(12')	3.96
C(29)...C(20')VI	3.96

O(2) ...O(8')VI	3.26
O(18)...O(18')III	3.33
C(7) ...O(23')I	3.36
C(12)...C(1')I	3.38
C(3) ...O(22')VI	3.41
C(6) ...O(23')I	3.43
C(7) ...C(24')I	3.43
C(19)...O(18')III	3.45
O(8) ...O(23')I	3.48
C(1) ...C(12')I	3.50
C(19)...O(22')II	3.50
O(21)...C(12')I	3.50
C(3) ...O(8')VI	3.57
C(24)...O(21')I	3.57
C(12)...O(21')I	3.58
C(12)...O(2')I	3.58
C(19)...O(21')II	3.58
O(23)...C(6')I	3.58
C(11)...C(5')I	3.59

Table 2-9 (cont.)

C(11)...C(6')I	3.59
O(21)...C(15')VI	3.59
C(6) ...C(11')I	3.61
O(21)...C(24')I	3.61
C(13)...O(2')I	3.62
C(24)...O(17')V	3.64
C(24)...O(22')I	3.64
C(5) ...C(11')I	3.65
O(23)...C(7')I	3.65
C(1) ...C(15')VI	3.66
C(20)...C(4')I	3.66
C(24)...C(16')V	3.66
C(9) ...O(23')I	3.67
C(14)...C(4')I	3.67
C(24)...C(7')I	3.67
C(9) ...C(4')I	3.68
C(10)...C(5')I	3.68
O(18)...C(16')III	3.70
C(19)...C(1')II	3.70
C(5) ...O(23')I	3.71
C(11)...C(1')I	3.72
O(21)...C(14')VI	3.73
O(23)...O(17')V	3.74
C(10)...O(23')I	3.76
O(18)...C(19')III	3.76
C(1) ...C(11')I	3.77
O(2) ...O(22')VI	3.77
C(12)...C(6')I	3.77
C(13)...C(4')I	3.77
O(2) ...C(7')VI	3.78
C(5) ...C(10')I	3.78
O(8) ...C(24')I	3.78
O(17)...O(2')I	3.78
C(6) ...C(24')I	3.81
C(10)...C(10')I	3.81
C(19)...C(3')I	3.81
O(23)...C(5')I	3.83
O(2) ...C(12')I	3.84
C(10)...C(4')I	3.84
C(20)...C(1')II	3.84
O(2) ...C(9')VI	3.85
C(3) ...C(7')VI	3.85
C(1) ...O(23')I	3.86
C(6) ...C(12')I	3.88
C(19)...C(7')II	3.88
C(20)...C(3')I	3.88
O(2) ...C(15')VI	3.89
O(22)...C(16')VI	3.90
C(24)...C(6')I	3.90
C(24)...C(1')I	3.90
C(13)...C(1')I	3.91
C(12)...C(4')I	3.93
O(2) ...C(14')VI	3.94
C(11)...C(4')I	3.94
C(12)...C(5')I	3.94
O(22)...O(23')I	3.94
O(21)...O(23')I	3.95
O(23)...C(1')I	3.95
C(10)...C(11')I	3.96
C(20)...O(2')II	3.96
O(22)...C(15')VI	3.97
C(1) ...C(24')I	3.98
C(20)...O(21')II	3.98
O(21)...C(11')I	3.99
O(23)...O(8')I	3.99

The Roman numerals refer to the following equivalent positions:

I: $x, y, -1+z$
II: $-x, \frac{1}{2}+y, -z$
III: $-1+x, y, -1+z$
IV: $-x, -\frac{1}{2}+y, -z$
V: $1-x, -\frac{1}{2}+y, 1-z$
VI: $1-x, \frac{1}{2}+y, 1-z$
VII: $-x, \frac{1}{2}+y, 1-z$
VIII: $-x, -\frac{1}{2}+y, 1-z$
IX: $1-x, \frac{1}{2}+y, -z$
X: $1-x, -\frac{1}{2}+y, -z$
XI: $x, 1+y, z$
XII: $x, -1+y, z$

C(7)...O(23')$_{I}$; and 3.41Å for C(3)...O(22')$_{VI}$.

The shortest intermolecular distances between symmetry related molecules 1 include 3.11Å for O(22)...C(24)$_{XI}$; 3.16Å for C(4)...O(21)$_{X}$; 3.25Å for C(3)...O(21)$_{X}$; and 3.30Å for C(16)...O(22)$_{IV}$. Similar distances between symmetry-related molecules 1' include 3.18Å for O(21')...C(24')$_{XII}$; 3.20Å for C(19')...O(22')$_{VI}$; 3.24Å for O(22')...C(24')$_{XII}$; 3.28Å for C(3')...O(21')$_{VII}$; and 3.38Å for O(18')...O(22')$_{VI}$. These are probably responsible for the attractive forces holding the molecules together.

Very short intermolecular contacts involving oxygen are often found in other structures, e.g. there are contacts of 3.09Å and 3.14Å in dihydro-β-erythroidine hydrobromide (Hanson, 1963); 3.11Å and 3.14Å in cedrelone iodoacetate (Grant et al., 1963); and 3.11Å and 3.12Å in clerodin bromo-lactone (Paul et al., 1962).

2.6. AFLATOXIN G$_1$ BENZENE SOLVATE.

Using the coordinates of the two molecules of aflatoxin G$_1$ in the structure of the bromobenzene solvate (at R = 0.186), structure factors for the benzene solvate were calculated. The R factor was 0.315. The calculated phase constants were used in an Fo Fourier synthesis.

The molecule of benzene showed up clearly in the three-dimensional electron-density distribution. After three more rounds of structure factor and Fourier calculations, the R factor became 0.227.

Refinement by the least squares method was carried out on the KDF 9 computer using an Algol program written by Mr. J.G.F. Smith and Professor D.W.J. Cruickshank. During the seven cycles of block-diagonal least squares refinement, the atomic thermal parameters were refined isotropically in the first five cycles and anisotropically in the last two cycles. The observed structure factors for each of the nine layers (h 0 ℓ to h 8 ℓ) were scaled to the calculated structure factore before the least squares refinement and after the 5th least squares cycle.

The weights, $\sqrt{w}$, were applied according to an equation suggested by Cruickshank et al. (1961) as follows:

$$\sqrt{w} = \frac{1}{(p_1 + |F| + p_2|F|^2 + p_3|F|^3)^{\frac{1}{2}}}$$

where $p_1 = 2 \times F$ min. = 1.90,

$p_2 = 2/F$ max. = 0.0075

$p_3 = 0$

The shifts of most of the parameters in the last cycle are less than their standard deviations.

A final set of structure factors calculated has an R factor of 0.098.

The course of analysis is shown in Table 2-10.

The observed and calculated structure factors for the 2833 observed reflections are listed in Table 2-11.

The final atomic coordinates and their estimated standard deviations and the anisotropic thermal parameters as listed in

Table 2-10

Course of Analysis: Benzene Solvate

1.	1st S.F. calculation. 34C and 14 0 atoms Individual isotropic temperature factors	R = 0.315
2.	1st Fo synthesis using 2727 terms	
3.	2nd S.F. calculation all atoms:- 40 C and 14 C	R = 0.280
4.	2nd Fo synthesis using all 2833 terms	
5.	3rd S.F. Calculations all atoms	R = 0.238
6.	3rd Fo synthesis	
7.	4th S.F. calculation, all atoms	R = 0.227
8.	1st S.F.L.S. cycle	R = 0.230
9.	2nd S.F.L.S. cycle	R = 0.197
10.	3rd S.F.L.S. cycle	R = 0.166
11.	4th S.F.L.S. cycle	R = 0.153
12.	5th S.F.L.S.	R = 0.152
13.	Layer scaling applied.	
14.	6th S.F.L.S. cycle	R = 0.136
15.	7th S.F.L.S. cycle all atoms anisotropic	R = 0.132
16.	8th S.F.L.S. cycle	R = 0.102
17.	Final S.F. calculations	R = 0.098

Table 2-11 Aflatoxin G_1 benzene solvate.

H	K	L	Fo	Fc	α
2	0	0	46.2	50.8	180
3	0	0	8.4	7.4	180
4	0	0	7.2	10.6	0
5	0	0	11.5	10.1	180
6	0	0	14.3	10.1	180
7	0	0	16.6	19.2	0
8	0	0	7.0	7.5	0
9	0	0	5.1	1.6	0
10	0	0	2.7	3.4	180
11	0	0	24.0	24.9	180
12	0	0	8.2	8.5	180
13	0	0	16.3	18.0	0
16	0	0	4.1	4.0	180
17	0	0	8.8	9.2	0
0	0	1	35.1	31.6	0
1	0	1	47.8	51.8	0
2	0	1	8.8	12.1	180
3	0	1	52.3	57.7	180
4	0	1	50.1	50.0	0
5	0	1	4.9	4.4	0
6	0	1	6.8	7.2	0
7	0	1	16.6	14.7	180
8	0	1	11.0	8.7	180
9	0	1	27.5	23.7	180
10	0	1	2.8	3.3	0
12	0	1	41.9	44.9	0
14	0	1	8.4	7.7	0
15	0	1	3.0	2.6	180
16	0	1	3.8	4.0	180
-1	0	1	35.3	33.2	180
-3	0	1	33.6	35.8	0
-4	0	1	8.1	9.7	0
-6	0	1	9.5	5.5	180
-7	0	1	34.0	32.2	0
-9	0	1	11.3	7.5	0
-10	0	1	9.3	7.6	0
-11	0	1	26.1	29.3	180
-12	0	1	35.2	34.8	0
-13	0	1	41.2	41.0	0
-16	0	1	4.2	4.7	0
-17	0	1	5.4	5.6	0
-18	0	1	3.2	3.4	180
0	0	2	32.0	39.6	0
1	0	2	30.1	32.6	180
2	0	2	12.4	13.0	180
3	0	2	28.3	23.9	0
4	0	2	5.4	2.7	0
5	0	2	7.8	9.6	180
6	0	2	17.9	19.9	180
8	0	2	2.6	2.9	180
9	0	2	15.6	15.6	180
10	0	2	3.0	3.0	0
11	0	2	40.9	40.9	180
15	0	2	8.5	8.2	180
-1	0	2	38.5	42.6	0
-2	0	2	11.4	9.0	180
-3	0	2	40.8	44.7	0
-4	0	2	32.7	36.4	0
-5	0	2	38.9	42.5	0
-6	0	2	4.2	5.9	180
-7	0	2	8.2	6.0	0
-8	0	2	6.7	9.1	180
-10	0	2	29.6	24.4	0
-11	0	2	10.2	8.5	0
-12	0	2	40.9	44.5	0
-13	0	2	4.4	4.1	180
-14	0	2	14.3	13.1	180
-16	0	2	8.1	7.9	180
-17	0	2	6.4	7.8	0
-18	0	2	3.4	2.7	180
-19	0	2	1.9	2.4	0
0	0	3	197.5	224.5	0
1	0	3	28.8	29.5	0
2	0	3	36.3	31.2	180
3	0	3	9.2	8.5	180
4	0	3	8.3	2.2	0
5	0	3	16.6	17.7	180
6	0	3	22.3	24.3	180
7	0	3	11.7	8.0	0
8	0	3	16.1	16.0	0
9	0	3	13.0	10.7	0
10	0	3	6.2	2.9	0
11	0	3	11.5	13.0	180
12	0	3	7.8	7.8	180
13	0	3	6.2	5.6	180
14	0	3	2.9	2.7	180
15	0	3	5.1	5.3	180
16	0	3	3.0	2.4	180
-1	0	3	206.4	195.7	180
-2	0	3	229.2	242.4	180
-3	0	3	12.2	8.8	0
-4	0	3	21.7	25.0	180
-5	0	3	10.0	6.2	0
-6	0	3	8.8	5.8	0
-7	0	3	15.5	10.5	0
-9	0	3	25.6	28.7	0
-11	0	3	4.0	4.9	180
-12	0	3	5.2	4.4	0
-13	0	3	31.7	34.0	0
-14	0	3	6.4	4.2	180
-15	0	3	6.4	5.1	180
-16	0	3	5.3	5.0	0
-17	0	3	14.6	14.9	0
-19	0	3	2.9	2.7	0
1	0	4	13.0	16.2	180
3	0	4	35.3	33.9	180
4	0	4	9.9	7.8	180
5	0	4	17.1	24.6	0
6	0	4	11.6	12.0	180
7	0	4	10.9	8.7	0
8	0	4	9.4	8.6	180
9	0	4	6.2	6.3	180
10	0	4	18.1	20.1	180
11	0	4	22.0	23.6	180
12	0	4	7.6	6.1	0
13	0	4	2.9	3.3	180
14	0	4	2.6	1.2	0
16	0	4	2.3	2.0	180
-1	0	4	79.1	83.6	180
-2	0	4	9.0	4.0	180
-3	0	4	10.5	12.6	180
-4	0	4	3.7	2.8	0
-5	0	4	45.3	40.9	0
-6	0	4	22.3	26.4	180
-7	0	4	32.3	33.7	0
-8	0	4	20.9	19.7	180
-10	0	4	8.9	7.1	180
-11	0	4	24.9	24.3	180
-12	0	4	30.6	32.6	0
-13	0	4	28.7	31.2	0
-14	0	4	46.7	44.7	180
-15	0	4	7.1	6.3	180
-16	0	4	4.4	4.7	180
-17	0	4	5.0	4.8	0
-18	0	4	2.5	3.4	180
-19	0	4	2.1	1.2	0
-20	0	4	2.9	4.1	0
0	0	5	22.0	22.5	180
1	0	5	13.5	13.1	180
2	0	5	27.0	23.6	180
4	0	5	4.5	5.3	0
5	0	5	6.1	7.6	0
6	0	5	2.9	2.3	0
7	0	5	5.2	5.9	0
8	0	5	3.1	4.1	0
9	0	5	3.2	4.1	0
10	0	5	5.5	4.4	0
11	0	5	8.8	9.0	180
14	0	5	2.2	2.2	0
-1	0	5	16.8	16.5	0
-2	0	5	68.6	72.4	180
-3	0	5	32.6	29.8	0
-4	0	5	46.8	49.5	180
-5	0	5	2.1	2.8	180
-6	0	5	19.9	17.5	180
-7	0	5	14.2	11.5	180
-8	0	5	11.9	7.5	180
-10	0	5	3.9	5.5	0
-12	0	5	8.1	8.1	0
-13	0	5	28.4	30.1	180
-14	0	5	20.8	21.8	180
-15	0	5	3.2	4.1	180
-18	0	5	2.5	2.4	0
-20	0	5	2.1	2.3	0
0	0	6	19.9	20.5	0
1	0	6	13.0	13.5	0
2	0	6	9.2	11.9	180
3	0	6	14.6	10.8	0
5	0	6	4.2	4.2	0
6	0	6	13.9	14.8	180
8	0	6	3.2	1.0	0
9	0	6	10.1	9.7	0
11	0	6	4.0	3.5	0
14	0	6	1.6	1.3	180
-1	0	6	84.8	90.4	180
-2	0	6	58.0	59.7	180
-3	0	6	54.8	50.5	0
-4	0	6	21.5	19.8	0
-5	0	6	3.4	4.4	0
-6	0	6	7.4	6.4	0
-7	0	6	10.9	9.4	180
-8	0	6	7.9	5.7	180
-9	0	6	18.9	17.9	0
-10	0	6	2.9	3.2	0
-11	0	6	19.3	21.1	180
-12	0	6	5.4	3.5	0
-14	0	6	17.3	18.9	180
-15	0	6	15.1	16.1	180
-16	0	6	4.3	3.1	0
-17	0	6	4.8	4.4	0
-18	0	6	9.9	9.6	180
-20	0	6	2.4	2.9	180
1	0	7	7.5	5.2	0
2	0	7	14.0	13.7	0
3	0	7	7.9	8.7	180
4	0	7	3.1	1.6	0
5	0	7	10.5	9.5	0
7	0	7	7.8	6.8	0
8	0	7	5.4	5.3	0
9	0	7	12.7	13.6	0
10	0	7	2.8	3.0	180
11	0	7	4.9	3.8	180
12	0	7	2.1	1.8	0
-1	0	7	24.8	23.6	180
-2	0	7	18.5	16.8	0
-4	0	7	15.2	17.5	0
-5	0	7	12.0	13.2	0
-6	0	7	24.9	24.3	180
-7	0	7	6.2	5.4	180
-8	0	7	12.8	12.8	180
-9	0	7	5.1	4.8	180
-10	0	7	3.0	3.6	180
-12	0	7	15.9	14.6	0
-13	0	7	9.6	9.3	0
-14	0	7	44.1	45.7	180
-16	0	7	9.3	9.0	0
-19	0	7	3.8	3.9	180
0	0	8	6.0	7.0	0
1	0	8	8.1	8.7	0
4	0	8	7.2	6.8	180
5	0	8	4.9	4.1	0
10	0	8	4.6	4.2	0
-1	0	8	13.0	13.0	0
-2	0	8	9.7	8.3	180
-3	0	8	21.4	22.2	0
-5	0	8	4.1	3.7	0
-6	0	8	11.0	8.2	0
-7	0	8	6.0	6.7	180
-8	0	8	3.0	0.3	180
-11	0	8	5.5	6.1	180
-12	0	8	5.6	5.2	0
-13	0	8	19.2	19.5	180
-15	0	8	12.0	11.9	0
-16	0	8	5.6	6.8	0
5	0	9	4.3	5.4	180
7	0	9	2.7	2.5	180
9	0	9	4.1	3.9	0
10	0	9	1.5	2.9	180
-1	0	9	6.3	4.1	180
-2	0	9	7.0	7.9	0
-3	0	9	39.4	36.7	0
-4	0	9	8.2	8.1	0
-5	0	9	4.4	0.8	0
-9	0	9	7.1	7.4	0
-10	0	9	3.2	2.8	180
-11	0	9	9.1	8.8	180
-13	0	9	5.4	4.4	0
-14	0	9	6.0	5.2	180
-16	0	9	14.9	14.9	0
-18	0	9	1.9	[illegible]	180
4	0	10	4.9	3.7	180
6	0	10	2.4	2.9	180
7	0	10	3.7	3.3	180
-2	0	10	12.8	10.5	0
-3	0	10	4.5	4.4	0
-4	0	10	8.5	9.0	0
-6	0	10	7.8	8.9	180
-9	0	10	3.2	3.7	0
-12	0	10	4.3	5.0	0
-14	0	10	13.3	13.6	180
-15	0	10	12.3	11.7	0
-16	0	10	16.1	16.3	0
4	0	11	4.6	4.9	180
-2	0	11	5.3	4.6	180
-3	0	11	6.2	5.6	0
-5	0	11	4.4	3.9	180
-6	0	11	3.1	3.4	0
-7	0	11	6.2	5.3	180
-14	0	11	4.2	4.2	0
-15	0	11	12.7	12.8	0
3	0	12	3.6	3.3	0
-1	0	12	4.5	3.3	0
-3	0	12	3.9	4.2	0
-4	0	12	7.9	7.6	180
-5	0	12	7.9	7.0	180
-6	0	12	2.8	3.4	180
-7	0	12	2.8	1.8	0
-8	0	12	2.8	0.4	0
-11	0	12	2.5	2.9	180
-12	0	12	2.4	2.2	0
-3	0	13	4.3	4.7	180
-5	0	13	4.5	5.4	180
-9	0	13	2.2	2.2	0
-10	0	13	2.1	2.5	0
2	1	0	30.9	32.9	123
3	1	0	47.6	51.9	40
4	1	0	65.8	71.5	115
5	1	0	23.2	20.0	327
6	1	0	6.1	8.5	228
7	1	0	30.9	35.1	188
8	1	0	18.5	18.9	37
9	1	0	15.3	12.5	352
10	1	0	2.5	4.3	343
11	1	0	19.9	19.5	72
12	1	0	15.5	15.9	84
13	1	0	30.3	29.2	185
14	1	0	5.8	5.8	254
16	1	0	5.8	4.7	359
18	1	0	7.5	6.4	21
0	1	1	87.1	99.8	356
1	1	1	68.9	76.9	275
2	1	1	60.9	64.9	16
3	1	1	40.5	44.9	262
4	1	1	18.5	15.2	93
5	1	1	39.9	37.5	62
6	1	1	40.2	38.4	295
7	1	1	12.7	9.0	283
8	1	1	16.7	15.1	303
9	1	1	11.8	10.6	320
10	1	1	6.9	7.1	293
11	1	1	15.7	16.9	8
12	1	1	2.9	2.4	42
13	1	1	7.2	5.3	304
14	1	1	5.0	5.0	27
16	1	1	3.5	3.6	100
18	1	1	5.0	4.9	110
-1	1	1	29.4	34.7	75
-2	1	1	48.8	55.1	18
-3	1	1	37.2	39.9	64
-4	1	1	45.6	43.8	197
-5	1	1	26.5	24.3	107
-6	1	1	34.8	40.1	197
-7	1	1	4.2	5.6	222
-8	1	1	4.6	7.9	189
-9	1	1	18.9	19.0	100
-10	1	1	19.3	18.9	199
-11	1	1	6.4	6.4	323
-12	1	1	30.2	29.9	188
-13	1	1	25.4	26.1	82
-14	1	1	14.0	12.5	294
-15	1	1	5.7	3.6	358
-16	1	1	5.4	5.2	285
-18	1	1	2.0	1.4	202
-19	1	1	1.5	1.3	134
0	1	2	19.2	17.0	188
1	1	2	79.7	87.4	23
2	1	2	63.5	65.3	195
3	1	2	16.2	16.8	273
4	1	2	8.1	6.2	165
5	1	2	9.3	7.5	159
6	1	2	5.4	5.2	99
7	1	2	7.3	7.5	123
8	1	2	5.3	5.7	168
9	1	2	5.8	5.1	239
10	1	2	11.3	11.8	234
11	1	2	19.0	17.9	112
12	1	2	21.5	18.8	14
13	1	2	20.7	21.4	304
14	1	2	6.8	7.0	16
17	1	2	1.8	1.9	194
-1	1	2	94.1	100.3	32
-2	1	2	39.5	48.9	318
-3	1	2	63.2	65.5	43
-4	1	2	65.6	75.3	316
-5	1	2	13.7	20.5	196
-6	1	2	3.4	5.1	166
-7	1	2	20.7	18.6	34
-8	1	2	18.2	16.3	247
-9	1	2	4.5	4.4	17
-10	1	2	12.5	11.7	306
-11	1	2	12.1	10.4	215
-12	1	2	15.8	16.9	332
-13	1	2	9.5	11.8	27
-14	1	2	9.3	8.0	276
-16	1	2	4.8	4.5	321
-19	1	2	4.3	3.1	132
0	1	3	108.3	116.2	304
1	1	3	19.9	17.1	321
2	1	3	14.4	13.6	323
3	1	3	9.8	14.6	26
4	1	3	9.5	7.2	236
5	1	3	20.1	21.5	21
6	1	3	12.2	10.4	278
7	1	3	32.1	28.8	182
8	1	3	4.4	2.9	319
9	1	3	10.9	10.1	4
10	1	3	11.7	11.4	262
11	1	3	21.4	19.3	76
12	1	3	19.3	20.6	96
13	1	3	8.9	9.7	312
14	1	3	5.2	4.7	237
15	1	3	4.0	3.1	177
16	1	3	1.9	2.4	193
-1	1	3	110.0	113.7	30
-2	1	3	46.4	51.4	212
-3	1	3	27.1	28.4	250
-4	1	3	60.0	61.3	292
-5	1	3	53.3	57.0	88
-6	1	3	29.2	28.0	87
-7	1	3	5.1	3.2	29
-8	1	3	10.8	10.2	122
-9	1	3	15.9	13.2	207
-10	1	3	5.9	4.8	115
-11	1	3	12.9	12.0	294
-12	1	3	7.2	6.3	16
-13	1	3	22.9	22.6	77
-14	1	3	14.0	13.5	7
-15	1	3	8.7	8.5	267
-16	1	3	6.2	5.6	5
-17	1	3	6.8	6.2	278
-19	1	3	1.8	1.7	87
0	1	4	27.9	22.5	45
1	1	4	38.3	36.2	293
2	1	4	12.0	10.0	143
3	1	4	15.8	13.1	278
4	1	4	14.2	13.5	155
5	1	4	30.7	38.7	96
6	1	4	8.1	6.1	267
7	1	4	18.0	16.0	136
8	1	4	9.1	7.8	241
9	1	4	5.7	4.4	158
10	1	4	13.7	12.5	231
11	1	4	8.3	8.0	35
12	1	4	4.9	5.3	202
13	1	4	2.6	2.7	34
14	1	4	3.4	3.8	242
16	1	4	1.4	2.0	252
-1	1	4	63.9	68.3	109
-2	1	4	17.9	17.0	15
-3	1	4	21.6	23.8	210
-4	1	4	22.8	22.4	3
-5	1	4	15.0	13.1	206
-6	1	4	13.5	10.0	299
-7	1	4	21.5	23.1	322
-8	1	4	6.5	8.9	8
-9	1	4	7.7	8.8	34
-10	1	4	11.8	9.5	238
-11	1	4	13.1	14.0	352
-12	1	4	2.7	2.0	339
-13	1	4	20.9	18.9	24
-14	1	4	23.4	22.7	343
-15	1	4	6.5	5.3	247
-16	1	4	5.6	4.5	310
-18	1	4	4.0	4.5	113
-20	1	4	1.8	2.1	98
0	1	5	16.3	14.2	63
1	1	5	26.6	22.3	41
2	1	5	6.5	8.5	158
3	1	5	15.1	15.2	231
4	1	5	12.4	11.1	304
5	1	5	9.4	8.6	103
6	1	5	2.6	2.7	121
7	1	5	2.8	3.1	88
9	1	5	4.1	3.0	33
10	1	5	16.5	15.6	232
11	1	5	18.7	17.9	138
12	1	5	6.5	6.5	125
13	1	5	4.7	4.7	269
15	1	5	1.5	1.6	94
-1	1	5	6.7	4.4	215
-2	1	5	10.8	9.1	7
-3	1	5	18.1	15.3	182
-4	1	5	23.1	24.8	281
-5	1	5	30.7	28.7	183
-6	1	5	7.5	4.8	158
-7	1	5	10.4	9.3	341
-8	1	5	11.7	9.8	105
-9	1	5	6.8	6.4	279
-10	1	5	5.7	6.3	36
-11	1	5	5.3	5.5	229
-12	1	5	12.5	10.5	357
-13	1	5	18.0	17.7	37
-14	1	5	13.7	13.1	195
-16	1	5	5.6	4.9	36
-18	1	5	5.1	4.5	61
-19	1	5	1.9	0.9	53
0	1	6	30.8	30.4	259
1	1	6	9.9	10.2	307
2	1	6	16.9	18.7	115
3	1	6	3.5	3.4	182
4	1	6	7.0	7.9	208
5	1	6	10.6	7.6	332
6	1	6	11.7	9.2	16
7	1	6	19.3	20.6	189
8	1	6	4.1	3.0	330
9	1	6	5.0	4.5	240
10	1	6	12.5	13.1	268
11	1	6	2.6	3.7	144
12	1	6	9.3	10.0	113
13	1	6	1.9	1.4	21
14	1	6	1.4	1.7	278
-1	1	6	55.3	56.6	89
-2	1	6	72.5	74.0	175
-3	1	6	31.8	32.6	229
-4	1	6	21.3	21.9	4
-5	1	6	39.2	41.7	100
-6	1	6	18.5	19.7	71
-7	1	6	4.7	5.2	272
-8	1	6	12.7	12.2	23
-9	1	6	16.5	15.6	282
-10	1	6	4.6	4.6	196
-11	1	6	7.8	4.6	341
-12	1	6	8.6	7.8	36
-13	1	6	8.2	7.8	148
-14	1	6	9.3	9.4	358
-15	1	6	15.8	15.8	213
-16	1	6	3.9	4.4	35
-17	1	6	4.4	4.3	264
-18	1	6	5.0	4.7	129
-19	1	6	3.7	3.7	27
0	1	7	13.4	12.7	99
1	1	7	12.2	11.5	261
2	1	7	6.0	4.6	207
3	1	7	11.7	11.4	256
4	1	7	8.0	6.4	318
5	1	7	7.1	5.9	55
6	1	7	8.3	8.3	17
7	1	7	5.1	4.6	83
8	1	7	4.0	3.1	52
10	1	7	6.1	6.4	208
12	1	7	2.6	2.6	166
-1	1	7	42.9	42.7	135
-2	1	7	19.2	19.7	168
-3	1	7	25.7	24.2	232
-4	1	7	10.1	9.1	30
-5	1	7	14.2	13.7	198
-6	1	7	8.7	9.2	161
-7	1	7	12.8	10.4	342
-8	1	7	5.3	4.3	110
-9	1	7	2.7	2.3	266
-10	1	7	8.4	8.4	133
-11	1	7	8.6	8.4	11
-12	1	7	4.1	4.2	162
-13	1	7	7.7	7.4	2
-14	1	7	8.7	8.5	143
-15	1	7	17.6	16.4	196
-16	1	7	7.5	6.9	186
-17	1	7	3.4	2.9	68
-18	1	7	8.4	6.2	119
-19	1	7	6.0	2.3	319
0	1	8	10.4	10.1	132
1	1	8	7.6	8.1	13
2	1	8	9.6	7.9	135
3	1	8	5.1	4.4	257
4	1	8	8.6	9.2	288
5	1	8	4.1	3.3	30
7	1	8	3.9	4.3	187
8	1	8	5.8	6.0	22
9	1	8	4.8	3.6	7
10	1	8	7.8	8.0	283
11	1	8	3.8	4.1	148
-1	1	8	14.5	13.1	246
-2	1	8	6.0	4.8	147
-3	1	8	18.2	17.8	219
-5	1	8	8.9	9.5	175
-6	1	8	7.6	5.4	101
-7	1	8	10.6	9.3	346
-8	1	8	14.5	13.9	119
-9	1	8	11.4	9.6	327
-10	1	8	2.9	3.5	163
-12	1	8	4.1	2.9	310
-13	1	8	13.0	12.0	97
-14	1	8	16.4	17.3	194
-15	1	8	7.2	7.3	204
-16	1	8	3.6	3.9	26
-17	1	8	4.6	3.9	155
0	1	9	10.9	8.4	257
1	1	9	8.3	8.6	309
2	1	9	8.8	9.6	75
3	1	9	2.9	3.9	43
4	1	9	4.9	5.2	183
5	1	9	8.3	9.0	338
6	1	9	4.5	4.4	42
7	1	9	2.4	2.9	170
8	1	9	2.2	2.5	78
10	1	9	5.0	5.7	297
-1	1	9	21.6	20.9	100
-2	1	9	21.4	22.2	197
-3	1	9	23.2	22.6	292
-4	1	9	21.0	20.8	16
-5	1	9	13.4	12.8	95
-6	1	9	8.1	5.9	227
-7	1	9	5.0	5.6	244
-8	1	9	7.1	7.1	306
-9	1	9	13.7	13.7	242
-10	1	9	7.2	7.1	178
-11	1	9	5.1	4.4	107
-12	1	9	2.9	2.3	43
-13	1	9	6.3	6.4	192
-15	1	9	7.2	7.1	205
-17	1	9	2.1	2.5	351
-18	1	9	5.9	5.8	145
0	1	10	5.0	5.0	92
1	1	10	4.9	4.9	356
2	1	10	2.8	2.6	174
3	1	10	3.8	4.2	210
4	1	10	2.6	2.5	235
5	1	10	4.2	4.0	225
6	1	10	3.4	4.1	34
-1	1	10	8.3	7.7	107
-2	1	10	18.3	18.8	296
-3	1	10	10.1	10.4	323
-4	1	10	8.8	9.5	44
-6	1	10	5.1	5.6	146
-7	1	10	7.8	9.0	350
-8	1	10	4.1	4.4	219
-9	1	10	2.9	2.9	242
-10	1	10	7.6	7.2	131
-11	1	10	4.9	4.7	9
-12	1	10	5.5	5.1	238
-14	1	10	7.5	7.3	131
-15	1	10	13.8	13.4	215
-16	1	10	8.1	7.4	352
-17	1	10	3.9	3.7	36
0	1	11	2.7	1.8	52
2	1	11	3.5	4.1	113
3	1	11	2.3	1.4	155
4	1	11	3.6	3.4	216
-1	1	11	5.4	5.3	279
-2	1	11	2.8	4.6	5
-3	1	11	2.8	3.4	232
-4	1	11	8.0	8.5	31
-5	1	11	2.8	2.8	53
-6	1	11	4.0	3.6	22
-7	1	11	2.8	3.4	317
-8	1	11	5.6	4.6	129
-9	1	11	4.8	4.1	267
-11	1	11	2.6	2.9	164
-12	1	11	6.2	2.6	304
-13	1	11	5.8	5.6	124
-14	1	11	9.7	6.9	221
-15	1	11	8.2	8.1	308
0	1	12	3.1	2.3	230
1	1	12	2.1	3.3	268
2	1	12	1.9	2.0	60
3	1	12	1.6	2.2	112
4	1	12	2.7	3.8	174
-1	1	12	4.0	4.4	100
-2	1	12	2.6	3.5	257
-3	1	12	12.9	12.6	315
-4	1	12	13.1	13.9	41
-5	1	12	3.6	4.1	132
-6	1	12	4.4	4.0	224
-7	1	12	4.4	2.0	301
-9	1	12	4.9	4.8	226
-10	1	12	5.3	5.2	118
-11	1	12	4.6	4.5	66
-12	1	12	2.1	2.2	341
-13	1	12	2.0	2.0	212
-14	1	12	3.5	3.6	345
-2	1	13	6.6	6.5	306
-4	1	13	5.3	6.0	105
-5	1	13	2.0	2.6	148
-6	1	13	2.0	2.5	183
-7	1	13	2.1	3.2	345
-8	1	13	2.0	2.8	264
-9	1	13	4.0	4.1	156
-10	1	13	3.3	3.6	59
-12	1	13	3.5	4.0	275
-13	1	13	1.3	1.3	149
1	2	0	31.0	36.5	120
2	2	0	38.7	35.5	204
3	2	0	9.9	11.2	54
4	2	0	46.0	49.5	57
5	2	0	56.8	61.3	114
6	2	0	37.9	35.9	317
7	2	0	29.2	28.7	284
8	2	0	13.7	11.1	52
9	2	0	13.8	14.4	143
10	2	0	9.1	8.2	309
11	2	0	7.4	7.5	278
12	2	0	6.0	4.6	209
13	2	0	6.2	5.1	191
14	2	0	5.6	5.3	126
15	2	0	6.4	5.5	196
16	2	0	5.6	5.8	309
0	2	1	30.1	37.2	59
1	2	1	26.2	28.1	59
2	2	1	34.4	36.1	307
3	2	1	24.1	27.8	114
4	2	1	20.9	19.2	270
5	2	1	39.2	34.6	197
6	2	1	27.4	23.9	357
7	2	1	24.2	19.0	71
8	2	1	11.9	11.6	53
9	2	1	13.3	11.9	102
10	2	1	14.7	12.6	37
11	2	1	9.9	10.8	294
12	2	1	8.7	9.5	201
13	2	1	4.4	4.0	121
14	2	1	3.5	3.6	237
17	2	1	6.3	6.6	103
18	2	1	2.8	2.7	316
-1	2	1	96.4	106.9	208
-2	2	1	22.8	26.4	262
-3	2	1	15.6	20.2	223
-4	2	1	11.7	9.1	224
-5	2	1	24.0	20.9	40
-6	2	1	85.1	89.8	307
-7	2	1	32.6	32.1	48
-8	2	1	26.8	29.2	134
-9	2	1	11.6	10.0	202
-10	2	1	16.9	15.0	120
-11	2	1	5.6	4.2	224
-12	2	1	5.4	6.6	30
-13	2	1	8.1	5.9	97
-14	2	1	12.1	12.3	133
-15	2	1	4.3	3.6	195
-16	2	1	4.6	4.7	130
-17	2	1	4.1	4.2	96
-19	2	1	5.1	5.3	318
0	2	2	65.0	71.9	83
1	2	2	20.7	22.0	233
2	2	2	28.4	26.6	120
3	2	2	26.5	23.3	215
4	2	2	26.1	23.7	159
5	2	2	43.1	39.4	145
6	2	2	65.9	72.7	302
7	2	2	16.8	19.4	33
8	2	2	10.1	8.9	345
9	2	2	14.4	12.8	215
10	2	2	4.1	4.4	230
11	2	2	3.5	4.5	205
12	2	2	15.8	15.0	308
14	2	2	6.3	5.5	333
16	2	2	1.9	2.1	320
17	2	2	4.2	4.5	119
-1	2	2	27.4	29.8	19
-2	2	2	32.9	33.9	161
-3	2	2	10.7	12.6	213
-4	2	2	9.7	12.0	134
-5	2	2	24.9	24.0	213
-6	2	2	58.3	53.0	37
-7	2	2	22.8	22.7	29
-8	2	2	14.6	14.0	209
-9	2	2	8.3	9.0	106
-10	2	2	7.3	5.7	283
-11	2	2	19.8	19.5	290
-12	2	2	8.3	8.9	169
-13	2	2	2.5	3.2	67
-14	2	2	2.5	1.9	257
-17	2	2	6.1	6.5	122
-18	2	2	3.7	4.1	282
-19	2	2	5.2	5.2	298
0	2	3	44.6	42.4	328
1	2	3	15.9	12.2	323
2	2	3	20.7	31.9	238
3	2	3	19.8	15.7	291
4	2	3	17.4	17.7	39
5	2	3	28.8	28.2	92
6	2	3	7.6	6.2	178
7	2	3	20.7	20.5	304
8	2	3	7.8	9.0	16
9	2	3	12.1	9.8	169
10	2	3	6.1	5.3	79
11	2	3	2.5	2.1	106
12	2	3	8.3	7.9	9
13	2	3	2.4	3.2	249
14	2	3	2.2	1.6	96
-1	2	3	36.0	34.6	296
-2	2	3	11.8	13.3	25
-3	2	3	8.8	9.2	269
-4	2	3	22.9	19.5	127
-5	2	3	41.7	40.7	122
-6	2	3	55.2	51.7	284
-7	2	3	34.3	34.1	254
-8	2	3	29.6	32.3	120
-9	2	3	12.7	10.5	216
-10	2	3	8.5	7.1	325
-11	2	3	10.8	12.1	263
-12	2	3	12.4	13.2	129
-13	2	3	9.3	7.3	39
-14	2	3	6.7	7.7	116
-15	2	3	5.6	4.6	304
-17	2	3	3.8	3.5	14
-18	2	3	1.9	2.5	244
-19	2	3	2.2	2.1	11
0	2	4	8.0	8.7	153
1	2	4	13.5	11.8	354
2	2	4	21.5	21.2	312
3	2	4	4.7	3.2	211
4	2	4	9.8	7.1	51
5	2	4	25.1	24.8	216
6	2	4	12.7	11.5	267
7	2	4	13.6	13.3	200
8	2	4	7.9	6.5	138
9	2	4	5.5	4.8	177
10	2	4	10.7	11.2	58
11	2	4	8.7	10.2	343
14	2	4	2.0	2.8	306
16	2	4	1.6	0.9	323
-1	2	4	34.5	34.9	233
-2	2	4	12.9	12.5	338
-3	2	4	28.5	30.6	16
-4	2	4	17.3	17.2	94
-5	2	4	18.2	20.7	341
-6	2	4	23.3	23.5	295
-7	2	4	27.3	25.8	127
-8	2	4	44.2	47.0	143
-9	2	4	8.6	7.6	344
-10	2	4	8.4	9.4	169
-11	2	4	13.0	11.9	323
-12	2	4	6.8	6.4	13
-13	2	4	7.0	6.1	354
-14	2	4	5.7	6.1	176
-15	2	4	9.7	9.4	295
-16	2	4	5.9	5.5	350
-18	2	4	3.9	3.8	273
-19	2	4	4.5	4.3	299
0	2	5	23.3	25.8	49
1	2	5	19.4	22.5	326
2	2	5	14.9	13.9	59
3	2	5	15.7	13.7	331
4	2	5	21.2	20.8	142
5	2	5	24.6	24.8	131
6	2	5	13.1	12.8	255
7	2	5	8.4	6.2	44
8	2	5	14.6	11.3	63
9	2	5	5.7	5.2	238
10	2	5	9.0	8.1	111
11	2	5	3.4	3.4	114
12	2	5	2.2	2.8	328
13	2	5	2.8	2.6	233
15	2	5	1.6	1.8	329
-1	2	5	15.1	18.1	292
-2	2	5	6.5	7.8	352
-3	2	5	30.6	33.7	309
-4	2	5	8.4	9.0	334
-5	2	5	9.3	7.6	338
-6	2	5	3.7	3.0	290
-7	2	5	6.9	5.3	12
-8	2	5	45.3	48.1	210
-9	2	5	12.7	13.3	121
-10	2	5	8.6	9.5	257
-11	2	5	12.4	12.3	297
-12	2	5	8.4	8.7	102
-13	2	5	3.5	3.3	1
-14	2	5	4.4	3.7	227
-15	2	5	4.3	3.9	29
-16	2	5	3.4	2.9	251
-17	2	5	5.8	6.0	63
-18	2	5	5.5	5.3	271
-19	2	5	3.9	3.5	318
0	2	6	4.0	3.8	151
1	2	6	11.2	12.4	356
2	2	6	16.2	14.5	225
3	2	6	10.8	8.4	216
4	2	6	4.6	3.4	193
5	2	6	15.1	14.1	109
6	2	6	12.3	12.1	177
7	2	6	4.3	3.4	330
8	2	6	7.6	7.5	338
9	2	6	5.5	5.0	287
11	2	6	4.4	3.7	117
12	2	6	7.3	7.8	30
13	2	6	1.6	2.0	284
14	2	6	1.1	1.3	52
-1	2	6	21.9	20.7	327
-2	2	6	36.7	35.0	130
-3	2	6	4.2	4.9	103
-4	2	6	17.0	18.4	174
-5	2	6	26.2	25.8	52
-6	2	6	28.1	29.2	294
-7	2	6	13.9	13.2	240
-8	2	6	29.6	27.3	107
-9	2	6	9.2	9.1	307
-10	2	6	9.8	10.1	309
-11	2	6	2.4	3.3	54
-12	2	6	4.3	3.9	114
-13	2	6	12.4	12.5	41
-14	2	6	3.5	3.6	280
-15	2	6	3.5	3.7	306
-16	2	6	7.0	7.4	312
-17	2	6	2.1	2.7	89
-18	2	6	2.7	2.3	294
-19	2	6	2.1	2.1	107
0	2	7	3.1	1.7	121
1	2	7	6.4	7.1	74
2	2	7	4.7	3.7	266
3	2	7	6.8	8.0	345
4	2	7	10.2	9.4	105
5	2	7	8.7	10.5	4
6	2	7	2.5	3.5	252
7	2	7	3.5	3.5	314
8	2	7	4.9	4.3	176
9	2	7	4.6	5.8	212
10	2	7	4.8	4.9	125
12	2	7	1.5	2.7	123
-1	2	7	13.2	12.9	99
-2	2	7	8.3	6.0	39
-3	2	7	18.7	16.2	63
-4	2	7	2.1	2.7	234
-5	2	7	10.1	7.5	359
-6	2	7	13.6	13.5	258
-7	2	7	23.7	22.9	122
-8	2	7	5.1	4.9	140
-9	2	7	10.0	9.0	360
-10	2	7	10.8	10.1	277
-11	2	7	6.5	6.2	248
-12	2	7	7.1	7.2	34
-13	2	7	5.0	4.9	236
-14	2	7	4.3	4.5	178
-15	2	7	4.8	4.9	321
-16	2	7	2.2	2.3	84
-17	2	7	2.9	3.1	86
-18	2	7	4.4	2.0	213
0	2	8	3.4	4.6	184
1	2	8	4.2	3.7	166
2	2	8	5.5	5.5	316
3	2	8	5.0	4.9	319
4	2	8	3.5	3.8	311
5	2	8	7.1	6.7	103
6	2	8	5.5	6.3	262
7	2	8	2.4	3.0	29
8	2	8	2.2	2.0	42
9	2	8	4.9	4.5	311
10	2	8	4.2	4.8	172
11	2	8	3.6	3.5	78
-1	2	8	10.6	12.7	168
-2	2	8	2.3	1.6	211
-3	2	8	6.2	5.4	311
-4	2	8	9.0	9.0	172
-5	2	8	10.4	9.7	129
-6	2	8	9.1	7.9	186
-7	2	8	4.8	4.0	31
-8	2	8	10.0	10.0	180
-9	2	8	13.3	13.3	44
-10	2	8	8.6	8.4	5
-11	2	8	10.4	10.6	346
-12	2	8	8.0	7.7	106
-13	2	8	4.3	3.2	327
-14	2	8	7.3	6.6	152
-15	2	8	2.3	1.6	69
-16	2	8	2.1	1.7	353
-17	2	8	2.7	2.7	100
-18	2	8	2.8	2.5	322
-19	2	8	2.0	1.6	211
0	2	9	5.6	6.8	142
1	2	9	6.7	7.1	357
3	2	9	5.0	4.9	341
4	2	9	4.2	3.8	290
5	2	9	5.8	5.9	45
6	2	9	5.4	6.2	202
7	2	9	3.5	4.1	119
9	2	9	3.4	3.5	278
10	2	9	2.9	4.2	239
-1	2	9	6.6	7.1	315
-2	2	9	20.4	20.2	125
-3	2	9	7.0	7.0	228
-4	2	9	5.5	4.0	267
-5	2	9	13.6	14.3	27
-6	2	9	6.1	6.8	281
-7	2	9	6.6	5.7	204
-8	2	9	11.8	12.5	84
-9	2	9	8.8	7.1	312
-10	2	9	9.8	9.5	332
-11	2	9	7.5	8.5	135
-12	2	9	5.0	4.8	40
-13	2	9	5.4	6.4	136
-14	2	9	2.3	2.8	208
-15	2	9	3.8	4.2	194
-17	2	9	4.2	4.9	131
-18	2	9	1.9	1.2	175
0	2	10	2.5	2.3	26
1	2	10	4.9	5.1	148
3	2	10	3.2	3.8	253
4	2	10	2.2	2.5	121
5	2	10	4.0	3.6	15
7	2	10	4.2	4.3	9
-1	2	10	7.5	7.5	112
-2	2	10	9.1	9.0	200
-3	2	10	4.4	4.7	259
-4	2	10	6.2	6.1	249
-5	2	10	3.5	3.1	257
-6	2	10	2.5	3.1	237
-7	2	10	9.8	9.6	149
-8	2	10	9.1	9.6	9
-9	2	10	7.9	7.1	290
-10	2	10	4.3	3.6	331
-13	2	10	6.8	3.4	265
-13	2	10	4.5	3.4	265
-14	2	10	3.0	3.2	140
-15	2	10	1.9	1.9	75
-16	2	10	2.5	1.9	145
-17	2	10	2.9	2.8	154
0	2	11	2.2	3.5	60
3	2	11	2.7	3.2	273
-1	2	11	5.2	4.7	203
-2	2	11	5.3	5.7	333
-3	2	11	4.1	4.0	281
-4	2	11	2.4	2.6	192
-5	2	11	3.4	3.0	135
-7	2	11	5.4	4.2	357
-8	2	11	2.4	2.2	49
-9	2	11	2.4	2.0	27
-10	2	11	4.0	4.5	320
-11	2	11	2.2	1.4	234
-12	2	11	2.1	2.1	175
-13	2	11	5.3	2.0	232
-15	2	11	2.8	2.3	242
-16	2	11	1.9	2.6	316
0	2	12	1.8	1.9	169
1	2	12	2.4	1.9	184
-1	2	12	2.8	2.7	313
-2	2	12	5.0	4.8	128
-3	2	12	5.1	5.9	255
-5	2	12	4.8	4.7	49
-6	2	12	2.1	2.7	193
-7	2	12	4.3	5.4	210
-8	2	12	2.1	2.8	50
-9	2	12	2.9	4.1	266
-10	2	12	2.0	2.6	285
-11	2	12	4.3	5.0	142
-14	2	12	3.2	3.7	232
-15	2	12	2.8	3.4	325
0	2	13	1.6	2.0	28
-2	2	13	4.0	5.0	224
-3	2	13	6.0	7.1	318
-4	2	13	1.7	2.9	7
-5	2	13	1.7	2.3	51
-6	2	13	1.7	1.1	126
-8	2	13	4.1	4.2	1
-9	2	13	1.6	3.0	238
-10	2	13	1.5	1.5	154
-11	2	13	3.5	3.8	151
-12	2	13	1.2	1.5	63
1	3	0	8.5	7.2	306
2	3	0	9.3	6.2	175
3	3	0	6.3	7.0	71
4	3	0	4.7	6.1	47
5	3	0	31.7	34.2	40
6	3	0	47.2	47.1	283
7	3	0	27.3	25.2	158
8	3	0	6.3	4.8	16
9	3	0	4.2	5.4	203
10	3	0	11.3	9.5	19
11	3	0	3.7	3.0	333
12	3	0	9.2	8.2	30
13	3	0	3.9	3.2	52
14	3	0	2.2	1.6	32
15	3	0	2.1	2.1	[illegible]
16	3	0	2.7	3.3	158
17	3	0	2.3	2.1	289
18	3	0	4.8	4.6	43
0	3	1	7.1	5.0	338
1	3	1	9.5	14.8	134
2	3	1	7.7	11.2	245
3	3	1	35.7	36.6	358
4	3	1	16.0	13.9	160
5	3	1	48.2	49.3	21
6	3	1	51.3	53.7	53
7	3	1	29.7	28.9	258
8	3	1	3.2	2.6	126
9	3	1	3.9	3.8	302
10	3	1	4.7	3.6	23
11	3	1	5.4	6.3	194
12	3	1	2.3	2.5	264
13	3	1	9.0	8.7	47
14	3	1	3.1	3.6	159
15	3	1	2.0	1.7	97
16	3	1	3.2	4.3	346
-1	3	1	20.6	19.7	274
-2	3	1	55.7	53.5	41
-3	3	1	22.2	21.1	346
-4	3	1	18.5	19.0	17
-5	3	1	14.2	13.2	142
-6	3	1	26.2	25.2	222
-7	3	1	9.4	11.0	35
-8	3	1	24.0	23.2	236
-9	3	1	9.0	7.6	132
-10	3	1	16.2	15.8	246
-11	3	1	4.2	5.1	43
-12	3	1	7.0	6.1	168
-13	3	1	8.5	8.7	149
-14	3	1	2.5	2.6	300
-15	3	1	3.1	3.3	188
-17	3	1	3.1	3.2	280
-18	3	1	3.9	4.2	198
-19	3	1	4.6	6.7	48
0	3	2	26.2	23.5	48
1	3	2	43.0	43.7	80
2	3	2	20.2	22.1	238
3	3	2	15.8	18.9	184
4	3	2	11.5	11.9	226
5	3	2	11.0	7.7	320
6	3	2	8.1	8.3	335
7	3	2	18.2	17.5	305
8	3	2	8.2	9.0	45
9	3	2	10.5	11.3	331
10	3	2	8.1	8.8	67
11	3	2	2.2	2.5	188
12	3	2	5.1	3.8	258
13	3	2	4.4	4.3	320
14	3	2	2.1	1.7	113
15	3	2	2.7	3.1	333
16	3	2	1.6	1.9	136
17	3	2	3.6	3.7	46
-1	3	2	12.7	10.5	4
-2	3	2	10.9	12.2	144
-3	3	2	7.5	9.0	120
-4	3	2	12.8	15.0	332
-5	3	2	16.2	12.7	264
-6	3	2	24.5	25.0	268
-7	3	2	71.4	75.8	42
-8	3	2	11.0	11.6	38
-9	3	2	8.1	8.8	47
-10	3	2	13.5	13.0	301
-11	3	2	3.6	1.7	20
-12	3	2	2.2	2.8	173
-13	3	2	5.5	7.3	228
-16	3	2	2.1	2.8	130
-17	3	2	2.6	2.3	110
-18	3	2	2.2	2.1	190
-19	3	2	2.3	2.3	26
0	3	3	16.5	13.0	266
1	3	3	29.4	30.0	9
2	3	3	13.3	13.0	254
3	3	3	20.2	17.0	4
4	3	3	25.7	26.6	147
5	3	3	39.5	40.2	44
6	3	3	21.0	17.4	304
7	3	3	7.8	8.5	195
8	3	3	8.5	7.6	344
9	3	3	6.1	5.7	195
11	3	3	6.8	5.9	228
12	3	3	3.9	3.5	169
14	3	3	1.9	0.7	194
16	3	3	1.3	3.1	188
-1	3	3	9.4	10.1	80
-2	3	3	5.6	3.9	188
-3	3	3	15.0	8.5	188
-4	3	3	5.2	4.7	106
-5	3	3	12.2	13.8	3
-6	3	3	29.0	30.7	27
-7	3	3	40.8	42.2	352
-8	3	3	26.2	26.9	298
-9	3	3	17.7	15.9	79
-10	3	3	12.4	11.9	239
-11	3	3	5.5	5.3	158
-12	3	3	4.4	4.1	40
-13	3	3	2.2	3.3	159
-14	3	3	6.4	6.9	21
-15	3	3	3.8	3.7	130
-17	3	3	4.3	4.0	148
-18	3	3	6.8	5.8	207
-19	3	3	2.8	2.7	39
0	3	4	8.3	7.4	317
1	3	4	21.7	19.5	152
2	3	4	21.4	18.0	257
3	3	4	4.3	4.5	9
4	3	4	24.1	26.1	235
5	3	4	18.1	16.8	38
6	3	4	18.1	14.9	28
7	3	4	14.1	11.4	332
8	3	4	7.5	8.7	211
9	3	4	4.5	4.7	43
10	3	4	4.5	3.8	328
11	3	4	8.4	8.5	207
12	3	4	3.0	3.6	240
13	3	4	2.8	2.4	343
14	3	4	2.4	2.1	143
-1	3	4	14.4	14.3	157
-2	3	4	4.0	4.1	291
-3	3	4	8.2	7.3	138
-4	3	4	7.4	10.9	204
-5	3	4	23.1	26.9	151
-6	3	4	10.6	12.9	225
-7	3	4	24.1	20.9	342
-8	3	4	15.4	12.3	74
-9	3	4	4.2	3.9	167
-10	3	4	16.8	15.4	353
-11	3	4	13.4	11.5	30
-12	3	4	6.6	5.6	2
-13	3	4	7.8	7.9	62
-14	3	4	7.5	8.3	338
-15	3	4	2.2	1.7	304
-17	3	4	1.9	3.0	313
-18	3	4	2.3	2.1	131
-19	3	4	6.2	6.2	43
0	3	5	22.4	23.7	347
1	3	5	24.4	27.7	67
2	3	5	7.8	6.8	306
3	3	5	9.0	7.2	189
4	3	5	9.4	10.1	169
5	3	5	5.0	4.9	141
6	3	5	9.5	10.1	178
7	3	5	9.1	8.1	213
8	3	5	4.5	5.5	177
9	3	5	3.2	3.4	166
10	3	5	4.5	3.7	149
11	3	5	4.8	5.6	117
12	3	5	6.2	7.5	227
14	3	5	2.4	2.2	252
-1	3	5	14.7	14.6	241
-2	3	5	14.3	15.5	310
-3	3	5	9.5	10.1	234
-4	3	5	18.1	15.6	341
-5	3	5	12.0	13.5	235
-6	3	5	9.4	9.8	284
-7	3	5	44.4	43.6	59
-8	3	5	16.0	17.1	209
-9	3	5	7.8	4.9	180
-10	3	5	23.2	24.2	286
-11	3	5	6.8	5.9	153
-13	3	5	2.3	2.1	243
-14	3	5	3.2	3.6	64
-16	3	5	3.6	4.1	81
-17	3	5	1.9	1.7	14
-19	3	5	4.7	4.7	325
0	3	6	14.9	13.5	269
1	3	6	5.0	6.0	117
2	3	6	20.1	21.8	304
3	3	6	9.1	8.2	246
4	3	6	15.5	13.6	197
5	3	6	14.4	15.5	83
7	3	6	6.8	6.2	170
8	3	6	4.5	4.2	280
9	3	6	2.2	2.1	101
10	3	6	4.7	4.3	353
12	3	6	1.7	2.2	326
-1	3	6	15.7	16.6	153
-2	3	6	7.5	8.3	188
-3	3	6	4.7	6.4	142
-4	3	6	6.1	7.6	181
-5	3	6	11.8	10.4	7
-6	3	6	4.1	3.6	261
-7	3	6	6.6	6.4	223
-8	3	6	14.0	13.0	201
-9	3	6	24.0	22.6	158
-10	3	6	11.8	11.4	181
-11	3	6	5.4	5.1	270
-12	3	6	12.1	12.3	62
-13	3	6	6.4	6.2	303
-14	3	6	4.5	4.7	11
-15	3	6	4.5	5.2	290
-17	3	6	3.2	2.9	286
-19	3	6	4.5	4.5	346
0	3	7	4.5	6.1	152
1	3	7	2.1	2.3	115
2	3	7	6.4	8.2	185
3	3	7	4.9	5.4	137
4	3	7	14.8	13.9	213
5	3	7	9.8	11.0	150
7	3	7	4.5	3.9	137
8	3	7	3.0	4.1	217
9	3	7	5.7	6.2	96
10	3	7	4.9	6.2	8
11	3	7	3.9	3.8	140
12	3	7	1.8	2.5	301
-1	3	7	4.5	1.6	317
-2	3	7	6.2	5.3	303
-3	3	7	9.8	9.5	325
-4	3	7	5.5	6.0	71
-5	3	7	7.4	7.3	235
-6	3	7	2.0	2.8	50
-7	3	7	17.0	17.9	316
-8	3	7	20.8	20.2	120
-9	3	7	15.8	14.4	215
-10	3	7	8.8	8.9	336
-12	3	7	2.3	2.7	301
-13	3	7	3.2	3.2	173
-15	3	7	4.6	6.4	227
-16	3	7	1.9	2.1	112
-17	3	7	1.8	1.8	278
-18	3	7	2.6	2.9	137
-19	3	7	3.0	2.3	20
0	3	8	10.1	9.6	341
1	3	8	5.9	4.8	115
2	3	8	9.3	8.1	2
3	3	8	5.5	5.9	252
4	3	8	5.5	5.4	118
5	3	8	3.7	6.8	356
6	3	8	2.2	2.0	138
7	3	8	3.6	4.4	343
10	3	8	2.1	2.7	39
11	3	8	2.6	3.3	71
-1	3	8	12.1	12.5	209
-4	3	8	5.2	6.8	105
-5	3	8	6.8	7.3	194
-6	3	8	7.5	8.0	131
-7	3	8	10.0	10.4	106
-8	3	8	14.6	13.3	187
-9	3	8	14.1	12.1	232
-10	3	8	4.5	4.4	297
-11	3	8	5.1	5.7	177
-12	3	8	7.5	8.0	104
-14	3	8	5.7	5.8	154
-15	3	8	5.7	5.5	315
-16	3	8	2.6	2.9	109
-17	3	8	4.4	4.5	318
0	3	9	6.0	6.2	143
1	3	9	3.9	4.5	114
2	3	9	5.5	5.4	357
3	3	9	5.4	6.1	273
4	3	9	4.3	3.7	227
5	3	9	2.0	1.3	68
6	3	9	9.5	5.4	79
7	3	9	1.8	2.0	159
-1	3	9	5.1	4.9	159
-2	3	9	9.8	10.1	81
-3	3	9	3.9	5.0	308
-4	3	9	5.0	5.5	72
-5	3	9	11.2	12.3	318
-7	3	9	5.0	4.4	269
-8	3	9	5.1	5.4	136
-9	3	9	5.1	4.6	132
-10	3	9	3.2	3.7	239
-11	3	9	8.7	8.0	354
-12	3	9	4.9	5.2	82
-13	3	9	2.1	3.3	244
-14	3	9	2.0	1.8	232
-15	3	9	1.9	2.7	225
-16	3	9	1.7	2.4	118
0	3	10	3.1	3.5	123
1	3	10	4.3	3.6	326
2	3	10	3.6	3.1	68
3	3	10	4.0	4.2	342
5	3	10	2.5	3.3	301
6	3	10	2.2	2.6	16
7	3	10	1.3	2.4	311
-1	3	10	5.0	4.7	5
-3	3	10	5.5	5.2	12
-6	3	10	2.3	3.0	111
-7	3	10	7.3	4.1	355
-8	3	10	9.2	9.5	124
-9	3	10	7.0	8.1	219
-10	3	10	10.7	11.0	343
-11	3	10	3.7	3.6	147
-13	3	10	4.4	5.0	152
-14	3	10	3.7	3.6	146
-15	3	10	1.7	1.6	198
1	3	11	2.7	3.1	130
2	3	11	1.8	3.2	351
3	3	11	2.3	3.2	249
4	3	11	2.9	2.3	124
5	3	11	1.7	1.6	313
-1	3	11	2.0	2.8	170
-2	3	11	2.1	2.0	132
-3	3	11	3.0	2.3	39
-4	3	11	2.1	2.4	125
-5	3	11	3.0	3.7	222
-6	3	11	3.0	3.0	53
-7	3	11	3.0	2.9	317
-8	3	11	3.0	2.9	331
-9	3	11	5.8	6.7	318
-10	3	11	6.3	6.0	9
-11	3	11	2.7	3.6	45
-13	3	11	1.7	1.9	107
0	3	12	1.6	1.0	119
2	3	12	3.4	3.6	31
3	3	12	2.0	2.9	314
-1	3	12	1.7	2.1	183
-2	3	12	4.7	4.5	14
-3	3	12	2.6	2.9	236
-4	3	12	2.6	2.5	259
-5	3	12	2.6	2.3	309
-6	3	12	1.9	1.5	175
-7	3	12	1.8	1.7	115
-9	3	12	3.1	3.3	111
-10	3	12	4.6	6.2	306
-11	3	12	4.0	4.7	40
-14	3	12	1.6	1.9	236
-2	3	13	3.0	1.8	245
-3	3	13	1.3	1.1	237
-9	3	13	3.0	3.3	268
-10	3	13	5.3	5.9	339
-11	3	13	2.3	2.8	84
1	4	0	16.5	22.3	149
2	4	0	1.5	3.6	221
3	4	0	33.3	37.4	161
4	4	0	31.4	34.2	262
5	4	0	14.5	14.2	170
6	4	0	13.0	11.4	160
7	4	0	10.1	10.2	106
8	4	0	10.1	9.1	121
9	4	0	8.5	7.4	203
10	4	0	6.2	6.3	120
11	4	0	3.3	3.1	277
12	4	0	7.9	6.8	73
13	4	0	5.3	7.2	278
14	4	0	3.3	2.9	127
15	4	0	2.2	1.7	347
17	4	0	1.6	0.2	191
18	4	0	2.7	2.3	282
0	4	1	13.1	17.0	153
1	4	1	7.3	9.0	187
2	4	1	14.2	12.9	163
3	4	1	6.4	7.2	220
4	4	1	12.7	13.8	163
5	4	1	15.8	14.6	116
6	4	1	19.5	22.7	358
7	4	1	26.7	27.9	137
8	4	1	10.6	9.3	275
9	4	1	14.4	12.9	132
10	4	1	5.1	4.1	256
11	4	1	8.2	7.6	345
12	4	1	10.2	11.0	19
13	4	1	4.1	4.5	85
-1	4	1	19.8	21.3	236
-2	4	1	14.1	15.4	308
-3	4	1	10.0	8.4	216
-4	4	1	21.2	18.8	152
-5	4	1	32.3	32.1	251
-6	4	1	19.1	19.0	168
-7	4	1	8.2	7.3	189
-8	4	1	7.7	7.9	151
-9	4	1	14.0	12.4	217
-10	4	1	8.9	9.5	164
-11	4	1	4.6	4.7	125
-12	4	1	3.4	3.2	199
-13	4	1	11.0	11.3	124
-14	4	1	3.3	3.9	222
-15	4	1	3.2	2.5	99
-16	4	1	2.0	2.4	16
-18	4	1	4.4	4.1	255
0	4	2	33.3	33.9	103
1	4	2	21.3	21.0	182
2	4	2	16.6	13.8	21
3	4	2	10.1	11.2	200
4	4	2	10.7	9.4	74
5	4	2	18.9	20.3	299
6	4	2	10.9	9.1	276
7	4	2	14.4	11.8	350
8	4	2	3.0	3.7	349
9	4	2	6.4	4.1	351
10	4	2	6.6	5.3	2

Measured and calculated values of the structure factors.
The phase angles are in degrees.

Table 2-11 (cont.)

H	K	L	Fo	Fc	α
11	4	2	4.1	4.1	309
12	4	2	9.9	10.3	342
16	4	2	2.2	2.3	69
17	4	2	4.0	4.8	55
-1	4	2	12.7	16.5	7
-2	4	2	15.6	15.3	330
-3	4	2	5.0	6.7	47
-4	4	2	12.1	9.7	226
-5	4	2	17.4	18.8	131
-6	4	2	3.8	3.7	240
-7	4	2	24.1	25.8	167
-8	4	2	12.5	11.6	215
-9	4	2	15.0	15.5	159
-10	4	2	3.0	3.6	260
-11	4	2	3.2	4.1	228
-12	4	2	2.3	3.5	82
-13	4	2	12.9	14.6	171
-14	4	2	8.6	10.0	222
-16	4	2	2.1	2.4	150
-17	4	2	1.9	1.9	319
-18	4	2	2.2	1.6	245
0	4	3	11.2	9.4	301
1	4	3	15.8	12.1	130
2	4	3	21.1	23.7	47
3	4	3	19.7	17.1	125
4	4	3	19.6	19.8	1
5	4	3	9.5	9.5	241
6	4	3	6.0	3.5	308
7	4	3	6.0	5.6	350
8	4	3	6.3	4.2	327
9	4	3	10.2	8.6	20
10	4	3	2.4	2.5	32
11	4	3	6.3	6.0	24
12	4	3	5.7	5.6	21
13	4	3	3.1	2.7	315
14	4	3	1.9	3.1	35
15	4	3	1.6	1.4	217
-1	4	3	4.8	2.2	1
-2	4	3	4.4	4.0	266
-3	4	3	7.8	7.8	116
-4	4	3	29.8	28.5	326
-5	4	3	15.9	16.3	35
-6	4	3	21.2	20.8	27
-7	4	3	3.0	2.8	158
-8	4	3	6.2	6.0	307
-9	4	3	16.3	17.3	221
-10	4	3	4.8	5.2	241
-11	4	3	2.2	2.4	303
-12	4	3	8.8	9.1	177
-13	4	3	5.3	5.0	270
-14	4	3	7.6	7.0	182
-16	4	3	4.8	4.3	188
-17	4	3	1.9	2.2	198
-19	4	3	1.1	1.2	167
0	4	4	25.8	25.3	327
1	4	4	6.5	6.1	307
2	4	4	12.8	14.2	336
3	4	4	15.0	14.3	206
4	4	4	4.7	4.8	237
5	4	4	7.1	6.4	3
6	4	4	26.1	26.9	346
7	4	4	6.0	5.4	79
8	4	4	12.6	12.8	344
11	4	4	7.4	7.4	243
12	4	4	7.2	6.0	46
13	4	4	2.0	2.7	89
14	4	4	2.9	2.9	50
15	4	4	1.8	2.1	144
-1	4	4	9.2	6.2	349
-2	4	4	11.4	10.0	323
-3	4	4	29.0	25.6	69
-4	4	4	4.7	5.7	195
-5	4	4	15.7	13.9	339
-6	4	4	9.3	7.1	31
-7	4	4	14.8	12.9	14
-8	4	4	8.9	9.2	35
-9	4	4	10.3	9.6	282
-10	4	4	17.8	17.7	109
-11	4	4	5.6	6.1	26
-12	4	4	9.4	10.2	132
-13	4	4	2.4	2.8	52
-14	4	4	8.3	7.5	235
-15	4	4	2.3	2.7	327
-16	4	4	2.1	1.9	182
-17	4	4	2.7	3.1	283
-18	4	4	3.3	3.9	209
0	4	5	20.2	19.8	133
1	4	5	10.0	10.3	32
2	4	5	12.4	13.9	66
3	4	5	12.8	10.1	160
4	4	5	11.8	12.6	68
5	4	5	3.1	3.4	179
6	4	5	8.3	9.0	332
7	4	5	4.1	4.1	13
8	4	5	6.4	5.8	283
9	4	5	5.3	6.2	88
10	4	5	2.3	2.5	270
11	4	5	4.4	4.0	185
12	4	5	3.9	4.7	311
13	4	5	1.7	2.2	225
14	4	5	1.8	2.0	334
-1	4	5	32.2	31.7	350
-2	4	5	17.3	18.0	251
-3	4	5	1.7	2.8	199
-4	4	5	6.6	7.5	179
-5	4	5	8.8	8.2	289
-6	4	5	10.1	9.1	62
-7	4	5	25.4	27.5	323
-8	4	5	13.2	13.7	48
-9	4	5	14.2	14.5	343
-10	4	5	10.9	10.4	323
-11	4	5	11.6	11.6	353
-12	4	5	3.4	2.7	281
-13	4	5	3.4	3.1	137
-14	4	5	8.6	9.2	259
-15	4	5	10.0	10.2	27
-16	4	5	3.0	3.1	93
-17	4	5	1.9	1.5	12
-19	4	5	1.2	1.6	82
0	4	6	9.6	9.4	300
1	4	6	11.0	10.8	276
2	4	6	4.8	3.9	29
3	4	6	5.8	5.8	330
4	4	6	13.7	14.0	59
5	4	6	8.5	7.3	340
6	4	6	7.2	6.2	175
7	4	6	3.4	3.7	262
8	4	6	7.8	7.3	203
9	4	6	2.3	2.8	178
10	4	6	5.2	5.0	270
11	4	6	1.9	1.8	168
-1	4	6	13.7	14.4	294
-2	4	6	8.0	7.7	182
-3	4	6	15.7	14.1	33
-4	4	6	4.8	5.5	321
-5	4	6	13.4	14.7	32
-6	4	6	4.0	3.3	286
-7	4	6	3.6	2.6	134
-8	4	6	7.5	7.0	264
-9	4	6	2.2	2.2	247
-10	4	6	5.1	5.7	12
-11	4	6	11.6	12.2	24
-12	4	6	3.4	4.1	131
-13	4	6	9.9	9.7	9
-15	4	6	6.4	6.2	31
-16	4	6	2.9	3.2	154
-17	4	6	1.9	2.0	31
-18	4	6	2.2	2.5	67
-19	4	6	2.2	2.1	302
0	4	7	4.4	5.1	221
1	4	7	6.0	5.6	60
2	4	7	2.3	1.8	89
3	4	7	7.1	6.4	253
4	4	7	8.3	10.5	172
5	4	7	5.9	6.1	153
7	4	7	2.3	2.7	151
9	4	7	3.5	4.0	33
10	4	7	4.1	4.3	203
11	4	7	5.4	5.8	249
-1	4	7	11.7	13.2	76
-2	4	7	17.9	17.0	167
-3	4	7	15.0	15.4	70
-4	4	7	11.7	12.2	196
-5	4	7	8.6	8.1	290
-6	4	7	3.1	3.5	93
-7	4	7	8.3	7.5	259
-8	4	7	9.1	7.3	66
-9	4	7	8.0	7.2	293
-10	4	7	10.8	9.9	100
-11	4	7	13.8	12.0	347
-13	4	7	12.1	12.8	330
-14	4	7	9.2	10.0	271
-15	4	7	2.2	2.6	1
-16	4	7	2.8	3.0	32
-17	4	7	1.8	1.3	284
-18	4	7	1.4	2.0	26
0	4	8	5.8	5.7	167
1	4	8	12.8	13.4	333
2	4	8	2.4	2.3	147
3	4	8	2.4	2.9	242
4	4	8	7.5	7.3	144
5	4	8	4.0	5.4	186
6	4	8	3.2	3.4	147
9	4	8	2.9	3.3	69
10	4	8	2.8	3.4	124
-1	4	8	13.8	14.5	304
-2	4	8	21.6	19.6	231
-3	4	8	9.5	7.7	161
-4	4	8	4.6	2.4	205
-5	4	8	4.0	2.7	349
-6	4	8	4.6	5.1	137
-7	4	8	12.6	11.3	354
-8	4	8	12.8	13.7	117
-9	4	8	5.8	7.0	191
-10	4	8	4.2	4.2	261
-11	4	8	3.4	3.4	123
-13	4	8	3.2	4.2	140
-14	4	8	3.1	4.2	307
-15	4	8	8.0	8.1	59
-16	4	8	1.9	2.1	71
-17	4	8	2.3	1.9	208
-18	4	8	1.7	2.2	334
0	4	9	2.4	3.1	220
1	4	9	3.4	2.7	185
2	4	9	5.8	4.8	221
3	4	9	6.9	7.3	225
4	4	9	6.3	6.1	94
5	4	9	4.7	4.3	35
7	4	9	3.0	3.3	20
8	4	9	1.5	2.9	218
8	4	9	2.1	2.9	208
9	4	9	1.5	2.0	30
-1	4	9	3.4	3.3	170
-2	4	9	9.0	8.8	157
-3	4	9	11.7	10.9	66
-4	4	9	2.4	1.8	171
-5	4	9	3.4	4.1	199
-7	4	9	12.2	12.2	189
-8	4	9	4.2	4.0	159
-9	4	9	5.9	5.6	197
-10	4	9	3.4	2.6	97
-11	4	9	2.3	2.6	45
-12	4	9	2.3	2.3	217
-13	4	9	3.5	3.5	42
-14	4	9	5.0	4.9	291
-15	4	9	2.7	2.9	170
-16	4	9	1.7	0.8	212
-17	4	9	3.1	3.0	254
3	4	10	4.1	4.0	282
4	4	10	2.7	3.1	194
5	4	10	1.7	2.0	117
-1	4	10	2.3	2.4	108
-2	4	10	2.3	1.5	199
-3	4	10	2.3	1.2	291
-4	4	10	4.7	5.1	306
-6	4	10	5.8	6.0	256
-7	4	10	4.1	4.9	187
-8	4	10	4.7	4.2	52
-9	4	10	5.2	5.9	158
-10	4	10	3.2	2.1	212
-11	4	10	3.1	2.7	173
-12	4	10	3.7	3.1	225
-13	4	10	2.0	2.7	224
-14	4	10	5.2	5.2	217
-15	4	10	5.7	7.0	123
-16	4	10	3.7	3.2	161
0	4	11	2.8	3.3	175
1	4	11	2.7	2.5	12
2	4	11	1.8	2.3	345
-1	4	11	2.1	2.5	175
-2	4	11	6.4	6.8	226
-3	4	11	8.9	10.2	98
-4	4	11	5.3	5.7	15
-5	4	11	2.2	2.5	53
-8	4	11	4.3	5.3	112
-9	4	11	10.1	10.0	221
-10	4	11	2.0	3.6	303
-11	4	11	1.9	1.6	176
-13	4	11	1.7	3.1	165
-14	4	11	1.5	1.0	172
-15	4	11	2.2	2.2	107
-1	4	12	1.6	2.0	182
-2	4	12	2.5	2.9	55
-3	4	12	4.0	3.8	40
-4	4	12	1.8	2.7	328
-5	4	12	3.7	3.9	252
-6	4	12	1.9	2.2	4
-7	4	12	2.6	2.3	191
-9	4	12	2.5	2.7	345
-11	4	12	2.2	2.1	9
-12	4	12	1.5	1.0	287
-13	4	12	3.1	3.2	129
-7	4	13	1.4	0.8	114
-8	4	13	3.7	3.9	41
-9	4	13	1.2	1.2	136
1	5	0	54.3	56.6	250
2	5	0	17.2	16.2	2
3	5	0	10.7	13.9	295
4	5	0	5.0	2.1	233
5	5	0	4.0	4.5	268
6	5	0	17.5	19.5	149
7	5	0	5.8	6.4	22
8	5	0	11.4	12.0	174
9	5	0	5.7	6.1	221
10	5	0	9.7	9.5	179
11	5	0	9.7	11.0	197
12	5	0	12.0	11.9	154
13	5	0	2.3	2.4	115
14	5	0	3.2	2.1	214
16	5	0	3.1	3.3	90
17	5	0	1.4	1.3	176
0	5	1	41.0	35.7	57
1	5	1	14.2	11.7	349
2	5	1	21.9	22.0	290
3	5	1	17.8	18.1	152
4	5	1	13.9	12.5	354
5	5	1	8.1	8.0	141
6	5	1	6.4	6.2	55
7	5	1	1.9	3.2	166
8	5	1	13.3	13.6	127
9	5	1	6.7	7.4	87
10	5	1	14.7	11.7	177
11	5	1	4.8	3.9	114
12	5	1	11.9	11.6	138
13	5	1	6.9	6.9	238
15	5	1	5.1	5.4	265
17	5	1	1.9	2.0	251
-1	5	1	47.1	45.3	143
-2	5	1	28.9	27.4	253
-3	5	1	12.0	12.5	211
-4	5	1	7.8	7.8	223
-5	5	1	6.6	8.2	159
-6	5	1	4.6	5.8	229
-7	5	1	6.5	6.9	152
-8	5	1	15.4	13.8	91
-9	5	1	4.2	3.7	304
-10	5	1	8.1	7.0	97
-11	5	1	8.4	9.3	288
-12	5	1	10.7	11.5	251
-13	5	1	11.9	11.6	45
-14	5	1	2.3	2.4	60
-15	5	1	3.0	3.3	5
-16	5	1	2.7	2.7	277
-17	5	1	1.6	2.2	79
0	5	2	35.2	32.2	214
1	5	2	26.7	25.2	171
2	5	2	10.9	12.3	145
3	5	2	18.3	17.7	164
4	5	2	13.8	13.6	160
5	5	2	17.0	16.5	179
6	5	2	11.9	11.4	105
7	5	2	7.5	8.3	172
8	5	2	3.8	3.7	17
9	5	2	10.6	11.7	182
10	5	2	2.3	2.3	261
11	5	2	4.1	4.7	117
12	5	2	6.6	5.4	74
14	5	2	2.0	2.8	277
16	5	2	1.3	2.1	35
-1	5	2	28.9	29.6	128
-2	5	2	9.0	9.0	20
-3	5	2	15.3	16.7	287
-4	5	2	21.0	22.2	188
-5	5	2	6.5	5.5	169
-6	5	2	10.4	11.9	214
-7	5	2	19.5	19.1	74
-8	5	2	1.9	1.5	318
-9	5	2	3.6	3.3	332
-10	5	2	3.8	3.2	289
-11	5	2	5.2	4.9	97
-12	5	2	4.1	5.9	318
-13	5	2	14.8	14.6	133
-14	5	2	4.6	4.6	145
-15	5	2	3.1	2.7	82
-16	5	2	2.8	2.8	307
0	5	3	21.5	24.2	131
1	5	3	26.8	26.2	202
2	5	3	2.7	2.6	26
3	5	3	3.4	4.1	94
4	5	3	6.6	5.4	227
5	5	3	10.1	10.1	251
6	5	3	7.8	5.5	175
7	5	3	10.8	12.4	48
8	5	3	5.6	5.5	264
9	5	3	2.3	2.6	345
10	5	3	9.0	8.4	335
11	5	3	2.3	2.8	240
12	5	3	3.2	2.2	339
13	5	3	2.1	2.7	224
15	5	3	2.0	2.2	267
-1	5	3	53.6	55.0	71
-2	5	3	20.5	21.8	220
-3	5	3	12.9	11.0	89
-4	5	3	14.9	17.1	211
-5	5	3	5.9	6.8	263
-6	5	3	3.8	3.4	136
-7	5	3	16.8	16.7	159
-8	5	3	7.1	6.5	197
-9	5	3	11.1	11.1	167
-10	5	3	2.2	3.3	46
-11	5	3	5.7	4.8	147
-12	5	3	8.6	8.2	260
-13	5	3	11.7	12.4	174
-14	5	3	5.3	6.0	153
-15	5	3	3.2	2.6	3
-16	5	3	5.8	5.5	228
-17	5	3	3.1	2.4	25
0	5	4	17.9	17.0	110
1	5	4	8.4	8.6	129
2	5	4	16.6	16.1	266
3	5	4	3.8	3.8	113
4	5	4	5.7	4.3	272
6	5	4	2.2	2.5	318
7	5	4	4.6	4.3	20
9	5	4	6.6	8.6	14
10	5	4	3.3	4.1	311
11	5	4	7.5	8.6	32
12	5	4	2.9	2.5	71
13	5	4	2.6	2.3	85
14	5	4	1.5	1.0	67
-1	5	4	30.7	31.4	178
-2	5	4	28.6	27.2	271
-3	5	4	9.9	8.7	298
-4	5	4	7.4	6.9	293
-5	5	4	8.6	7.2	49
-6	5	4	4.0	3.0	257
-7	5	4	8.6	9.5	124
-8	5	4	9.3	7.6	165
-9	5	4	11.1	8.5	191
-10	5	4	9.5	8.8	178
-11	5	4	5.2	6.1	269
-12	5	4	12.8	13.5	210
-13	5	4	15.3	16.3	70
-14	5	4	4.7	4.7	130
-15	5	4	3.9	4.0	265
-16	5	4	2.0	2.4	172
-17	5	4	1.8	1.2	165
-18	5	4	1.4	1.6	137
0	5	5	5.3	5.5	245
1	5	5	8.9	7.8	340
2	5	5	6.4	7.4	313
3	5	5	3.0	2.5	81
4	5	5	4.4	5.1	277
6	5	5	5.7	6.4	261
7	5	5	7.9	7.4	342
8	5	5	8.3	8.1	341
9	5	5	3.3	3.1	315
10	5	5	3.9	4.1	325
11	5	5	2.0	0.5	156
-1	5	5	25.5	23.2	73
-2	5	5	21.3	20.2	322
-3	5	5	11.2	9.3	356
-4	5	5	13.3	11.5	161
-5	5	5	9.8	9.4	56
-6	5	5	3.4	3.1	98
-7	5	5	10.4	12.0	47
-8	5	5	6.0	5.1	180
-9	5	5	9.4	10.3	269
-10	5	5	9.5	10.7	165
-11	5	5	6.7	6.7	178
-12	5	5	4.1	4.7	155
-13	5	5	11.2	11.6	140
-14	5	5	7.7	8.0	192
-15	5	5	6.2	7.0	290
-16	5	5	4.5	4.2	274
-17	5	5	1.8	2.5	241
-18	5	5	1.4	1.2	200
0	5	6	5.9	6.2	179
1	5	6	2.2	2.1	195
2	5	6	2.2	2.1	232
3	5	6	11.6	12.9	40
4	5	6	2.3	2.5	97
5	5	6	4.7	4.2	269
6	5	6	2.4	2.3	53
7	5	6	5.3	5.4	57
9	5	6	3.1	2.3	71
10	5	6	4.5	4.0	334
11	5	6	3.1	4.5	70
12	5	6	2.0	2.3	288
-1	5	6	17.3	17.5	14
-2	5	6	15.4	12.2	276
-3	5	6	12.2	10.6	291
-4	5	6	9.3	8.8	255
-5	5	6	17.8	17.7	314
-6	5	6	3.6	3.5	49
-7	5	6	6.5	5.7	0
-8	5	6	12.4	13.0	264
-9	5	6	2.3	2.5	13
-10	5	6	6.6	6.4	356
-11	5	6	5.3	4.8	163
-12	5	6	4.1	4.4	260
-13	5	6	13.8	14.2	167
-14	5	6	4.0	5.1	45
-15	5	6	4.3	5.0	322
-16	5	6	1.9	1.2	199
-18	5	6	1.3	1.5	86
0	5	7	3.9	4.8	75
1	5	7	8.0	7.5	128
2	5	7	4.7	5.0	307
3	5	7	4.0	5.4	1
4	5	7	2.4	1.3	307
5	5	7	5.3	5.2	356
6	5	7	7.4	7.5	252
8	5	7	4.2	4.3	199
10	5	7	3.8	3.5	256
11	5	7	1.3	1.2	356
-1	5	7	7.1	5.4	194
-2	5	7	15.7	14.4	326
-3	5	7	14.5	14.4	14
-4	5	7	3.1	3.3	61
-5	5	7	10.2	9.8	42
-6	5	7	5.0	5.6	43
-8	5	7	5.2	5.4	21
-9	5	7	6.7	7.4	244
-10	5	7	10.4	10.5	25
-11	5	7	6.3	7.2	292
-13	5	7	6.1	6.8	56
-15	5	7	9.5	9.9	263
-16	5	7	1.9	2.7	113
-17	5	7	2.7	3.0	240
-18	5	7	1.7	1.7	111
0	5	8	3.4	3.8	42
1	5	8	4.1	3.9	30
3	5	8	2.3	2.7	66
4	5	8	2.3	3.0	37
5	5	8	6.0	6.7	91
6	5	8	4.3	4.5	185
7	5	8	2.0	1.4	243
-2	5	8	11.3	10.0	342
-3	5	8	10.5	10.2	221
-4	5	8	11.8	10.8	196
-5	5	8	4.7	6.3	227
-6	5	8	5.3	5.2	40
-7	5	8	6.7	6.3	289
-8	5	8	2.4	3.2	325
-9	5	8	8.3	9.1	298
-10	5	8	2.4	3.1	42
-11	5	8	3.3	3.7	321
-12	5	8	6.1	6.9	20
-14	5	8	2.9	3.4	299
-15	5	8	9.3	9.5	325
-17	5	8	2.0	3.0	112
0	5	9	5.3	4.8	21
1	5	9	4.6	5.5	239
3	5	9	2.2	3.2	342
4	5	9	2.1	2.1	79
6	5	9	1.8	2.9	111
7	5	9	2.7	3.1	202
-1	5	9	4.1	3.9	286
-3	5	9	2.4	3.5	176
-4	5	9	3.4	3.8	177
-5	5	9	3.4	4.1	46
-6	5	9	5.3	5.2	130
-7	5	9	4.1	4.3	82
-8	5	9	2.4	1.4	197
-9	5	9	2.3	3.3	70
-10	5	9	7.0	7.9	44
-11	5	9	2.2	2.3	162
-12	5	9	4.4	4.4	11
-14	5	9	9.6	10.0	360
-15	5	9	6.9	7.2	328
-16	5	9	1.5	2.0	147
0	5	10	2.2	2.0	33
1	5	10	4.7	5.5	153
4	5	10	3.0	3.7	88
5	5	10	2.2	2.5	95
-1	5	10	2.2	2.4	256
-2	5	10	6.0	6.3	10
-3	5	10	2.3	3.2	294
-4	5	10	5.2	6.1	144
-5	5	10	5.2	4.1	239
-6	5	10	4.0	3.9	134
-7	5	10	3.2	2.9	213
-8	5	10	2.2	1.9	199
-9	5	10	2.2	1.9	215
-10	5	10	3.1	4.1	34
-12	5	10	2.0	1.2	24
-13	5	10	1.8	1.9	32
-14	5	10	2.3	3.4	335
-15	5	10	3.5	3.4	268
-16	5	10	2.8	2.9	111
0	5	11	1.9	2.1	9
3	5	11	2.0	3.1	237
4	5	11	1.5	1.3	295
-1	5	11	3.9	4.1	231
-3	5	11	5.0	5.3	191
-4	5	11	5.5	5.3	137
-7	5	11	2.1	1.4	320
-8	5	11	2.0	1.3	97
-11	5	11	1.8	2.1	162
-12	5	11	2.4	2.3	76
-14	5	11	1.8	1.8	229
-2	5	12	2.2	2.2	131
-6	5	12	2.4	1.8	238
1	6	0	7.3	6.4	187
2	6	0	4.4	5.6	86
3	6	0	12.4	11.9	185
4	6	0	12.4	13.9	326
6	6	0	9.7	9.7	113
7	6	0	13.6	14.9	194
8	6	0	12.8	11.9	8
9	6	0	5.9	5.8	310
10	6	0	5.0	5.0	285
11	6	0	5.9	5.5	176
12	6	0	13.8	14.4	353
13	6	0	3.5	4.2	347
14	6	0	4.6	4.6	228
15	6	0	1.7	1.8	91
16	6	0	1.4	1.9	167
0	6	1	36.0	39.2	340
1	6	1	19.5	18.4	270
2	6	1	22.0	20.4	3
3	6	1	9.1	7.3	245
4	6	1	5.3	3.2	223
5	6	1	14.3	11.8	12
5	6	1	11.8	11.8	12
6	6	1	12.9	14.3	351
7	6	1	13.2	14.1	165
8	6	1	6.9	6.2	114
9	6	1	4.5	3.7	161
10	6	1	3.5	4.6	181
11	6	1	11.4	13.0	189
12	6	1	5.8	6.0	104
14	6	1	2.5	2.6	257
15	6	1	1.5	1.2	165
-1	6	1	12.0	13.0	70
-2	6	1	11.2	12.3	213
-3	6	1	2.8	5.8	243
-4	6	1	9.8	8.3	347
-5	6	1	6.0	4.8	15
-6	6	1	6.3	6.2	307
-7	6	1	5.0	4.7	214
-8	6	1	7.3	8.8	204
-9	6	1	5.1	6.2	296
-10	6	1	2.0	1.5	75
-11	6	1	5.1	5.3	21
-12	6	1	11.0	12.2	3
-13	6	1	2.0	1.2	221
-14	6	1	3.9	3.9	177
-16	6	1	3.0	2.8	193
0	6	2	23.4	22.2	5
1	6	2	11.9	13.2	20
2	6	2	9.0	9.1	346
3	6	2	6.9	6.9	83
4	6	2	5.2	5.1	115
5	6	2	8.1	8.1	183
6	6	2	17.3	18.4	4
7	6	2	3.2	4.5	66
8	6	2	10.5	9.0	152
9	6	2	2.9	2.7	216
10	6	2	10.6	11.2	197
11	6	2	4.6	5.0	139
12	6	2	5.5	5.3	348
13	6	2	1.8	1.4	128
15	6	2	1.9	1.9	233
-1	6	2	15.2	15.2	200
-2	6	2	13.1	12.7	95
-3	6	2	32.7	32.3	187
-4	6	2	13.2	15.0	141
-5	6	2	6.1	6.4	104
-6	6	2	3.7	3.6	323
-7	6	2	4.4	2.8	211
-8	6	2	4.4	2.3	197
-9	6	2	6.9	10.3	343
-10	6	2	11.1	10.7	244
-11	6	2	5.4	6.0	8
-12	6	2	6.2	6.1	300
-13	6	2	10.1	10.8	5
-14	6	2	5.6	5.1	270
-15	6	2	2.6	2.0	79
0	6	3	6.2	4.5	309
1	6	3	5.5	7.6	131
2	6	3	3.6	4.1	157
3	6	3	1.6	2.1	173
4	6	3	11.7	12.5	241
5	6	3	9.9	9.6	354
6	6	3	4.7	5.8	113
7	6	3	14.4	14.6	173
8	6	3	9.2	10.0	56
9	6	3	9.8	8.5	185
10	6	3	4.1	4.7	141
11	6	3	11.3	13.2	184
12	6	3	5.3	6.0	26
13	6	3	3.7	4.0	12
14	6	3	2.9	2.9	264
15	6	3	1.9	2.3	52
-1	6	3	21.8	19.6	12
-2	6	3	5.5	5.1	200
-3	6	3	15.4	12.2	144
-4	6	3	11.4	13.2	308
-5	6	3	12.8	14.4	36
-6	6	3	12.9	12.0	173
-7	6	3	5.9	7.4	39
-8	6	3	7.5	9.1	323
-9	6	3	8.6	10.7	19
-10	6	3	2.0	1.8	264
-11	6	3	2.0	1.9	142
-12	6	3	13.9	13.6	8
-13	6	3	2.0	2.7	233
-14	6	3	4.4	4.9	164
-15	6	3	1.8	1.5	83
-16	6	3	3.7	4.2	167
-17	6	3	1.4	1.6	29
0	6	4	13.0	11.9	352
1	6	4	3.9	4.8	203
2	6	4	3.3	3.8	189
3	6	4	7.7	6.2	92
4	6	4	10.1	12.7	185
5	6	4	9.0	9.0	351
6	6	4	15.0	15.5	352
7	6	4	4.6	5.2	140
9	6	4	4.1	4.6	51
10	6	4	4.0	3.8	295
11	6	4	5.6	5.6	198
12	6	4	1.7	2.3	102
13	6	4	2.0	1.7	30
14	6	4	2.3	2.5	280
-1	6	4	25.3	24.8	109
-2	6	4	29.1	25.0	182
-3	6	4	6.0	4.8	315
-4	6	4	7.8	9.1	72
-5	6	4	8.3	9.2	108
-6	6	4	18.7	18.8	187
-7	6	4	6.9	6.5	183
-8	6	4	4.6	4.6	144
-9	6	4	8.7	8.2	340
-10	6	4	8.8	7.5	52
-11	6	4	6.9	6.2	9
-12	6	4	5.1	4.6	354
-13	6	4	2.0	3.0	229
-14	6	4	3.9	4.4	164
-16	6	4	1.6	1.5	197
-17	6	4	3.1	3.2	2
-18	6	4	1.6	2.1	75
0	6	5	8.0	7.2	100
1	6	5	8.4	6.8	285
2	6	5	10.1	8.7	290
3	6	5	10.9	11.1	33
4	6	5	13.5	14.0	180
5	6	5	15.8	15.8	179
6	6	5	10.6	9.7	314
7	6	5	9.3	9.2	41
8	6	5	2.0	1.4	111
9	6	5	4.4	4.1	357
10	6	5	2.6	2.2	188
11	6	5	1.7	2.3	169
12	6	5	2.0	2.1	356
13	6	5	2.1	2.1	89
-1	6	5	19.9	17.7	199
-2	6	5	10.9	8.9	176
-3	6	5	13.3	13.7	197
-4	6	5	3.4	3.4	59
-5	6	5	10.1	10.0	22
-6	6	5	1.8	2.5	290
-7	6	5	14.7	14.1	345
-8	6	5	8.6	9.1	156
-9	6	5	5.3	4.2	347
-10	6	5	7.7	7.7	144
-11	6	5	4.7	4.1	25
-12	6	5	2.9	2.7	104
-13	6	5	5.0	4.7	33
-14	6	5	5.5	5.2	136
-16	6	5	1.6	2.3	53
-17	6	5	1.3	0.7	331
-18	6	5	1.3	1.3	85
0	6	6	3.3	4.4	49
1	6	6	3.4	3.6	140
2	6	6	8.2	7.2	116
3	6	6	4.1	4.9	23
4	6	6	9.0	7.5	213
5	6	6	5.9	5.0	326
6	6	6	2.9	2.8	286
7	6	6	4.0	4.6	328
8	6	6	4.3	3.7	16
9	6	6	3.1	3.4	10
11	6	6	4.1	5.1	196
-1	6	6	12.5	12.8	358
-2	6	6	12.9	12.5	169
-3	6	6	8.9	7.8	200
-4	6	6	9.9	10.9	8
-5	6	6	4.2	4.1	305
-6	6	6	7.5	6.1	84
-7	6	6	6.8	6.6	291
-8	6	6	11.2	11.7	190
-9	6	6	4.1	4.4	153
-10	6	6	7.2	7.1	167
-11	6	6	6.2	6.2	204
-12	6	6	2.1	2.3	59
-13	6	6	7.0	6.5	185
-14	6	6	4.7	4.1	171
-16	6	6	1.6	1.2	197
-17	6	6	3.4	2.9	16
0	6	7	7.9	8.2	36
1	6	7	9.0	8.7	260
2	6	7	6.6	6.1	184
4	6	7	15.3	14.7	170
5	6	7	6.8	6.4	267
6	6	7	8.1	7.9	360
7	6	7	1.8	2.2	266
8	6	7	2.4	2.9	33
10	6	7	2.5	3.1	8
-1	6	7	8.1	7.4	167
-2	6	7	10.3	9.9	205
-3	6	7	16.0	14.5	327
-4	6	7	6.7	6.3	14
-5	6	7	7.0	6.1	51
-6	6	7	14.8	16.7	233
-7	6	7	5.8	5.6	13
-9	6	7	5.9	5.8	340
-10	6	7	4.2	4.4	81
-11	6	7	8.0	6.3	187
-12	6	7	4.5	2.7	148
-13	6	7	7.0	7.0	189
-14	6	7	3.6	3.6	129
-15	6	7	4.1	4.2	186
-16	6	7	1.5	1.5	335
-17	6	7	3.4	3.4	3
0	6	8	9.3	8.0	108
1	6	8	2.1	2.8	218
2	6	8	4.6	4.4	305
3	6	8	13.0	12.4	6
4	6	8	4.4	4.8	114
5	6	8	9.4	9.3	184
6	6	8	6.3	6.7	265
7	6	8	3.2	3.8	24
9	6	8	3.2	3.4	350
-1	6	8	5.1	6.2	182
-2	6	8	3.6	3.8	262
-3	6	8	5.1	4.4	19
-4	6	8	9.1	8.8	15
-5	6	8	4.7	5.5	341
-6	6	8	4.2	3.9	4
-7	6	8	11.7	12.3	319
-8	6	8	8.6	7.9	163
-9	6	8	4.1	4.1	205
-10	6	8	3.5	3.7	93
-11	6	8	5.3	4.6	244
-12	6	8	3.9	3.0	206
-14	6	8	4.8	4.9	184
-15	6	8	2.6	2.9	227
-16	6	8	3.2	3.4	320
0	6	9	2.0	1.4	31
1	6	9	2.0	3.2	227
2	6	9	2.7	3.1	102
3	6	9	4.3	5.2	21
6	6	9	2.0	2.1	207
7	6	9	1.6	2.0	299
-1	6	9	6.1	6.6	256
-2	6	9	5.0	5.1	170
-3	6	9	4.6	3.5	233
-4	6	9	7.5	8.4	[illegible]
-5	6	9	4.1	1.0	212
-6	6	9	4.1	3.7	350
-7	6	9	13.6	3.8	236
-9	6	9	5.7	5.5	42
-10	6	9	4.8	5.2	340
-11	6	9	3.3	3.0	295
-13	6	9	3.3	3.1	122
-14	6	9	2.6	3.6	275
-15	6	9	2.6	2.6	57
-16	6	9	2.2	2.4	292
0	6	10	4.0	4.6	37
1	6	10	3.5	3.1	323
3	6	10	4.5	5.0	20
4	6	10	5.0	6.8	143
5	6	10	2.7	2.8	220
-1	6	10	2.6	3.4	112
-2	6	10	3.8	3.9	252
-3	6	10	8.2	8.6	23
-5	6	10	4.7	5.1	115
-6	6	10	8.4	9.4	240
-7	6	10	2.7	3.6	94
-8	6	10	2.6	3.0	28
-10	6	10	2.5	2.4	23
-11	6	10	3.4	3.4	181
-12	6	10	2.2	2.6	323
-13	6	10	1.4	0.9	216
-14	6	10	2.8	3.1	334
-15	6	10	2.1	2.0	211
0	6	11	2.5	2.9	71
1	6	11	3.0	2.7	176
2	6	11	2.0	2.5	267
3	6	11	4.7	6.3	360
-1	6	11	2.2	2.1	225
-2	6	11	4.8	4.5	310
-3	6	11	2.3	3.2	51
-5	6	11	2.4	2.7	174
-7	6	11	2.9	2.5	350
-8	6	11	7.3	7.8	176
-9	6	11	5.0	4.9	111
-10	6	11	4.3	4.4	14
-11	6	11	3.4	2.9	263
-12	6	11	1.3	1.5	347
-13	6	11	2.3	2.4	44
-3	6	12	2.1	3.1	78
-4	6	12	1.8	2.6	351
-5	6	12	3.4	3.0	145
-7	6	12	4.9	4.8	200
-8	6	12	6.6	6.8	136
-9	6	12	5.7	6.1	10
-10	6	12	4.9	5.8	354
1	7	0	24.8	22.4	275
2	7	0	19.0	17.2	189
3	7	0	4.5	3.2	126
4	7	0	18.4	19.8	204
5	7	0	8.7	9.7	80
6	7	0	10.5	9.8	301
7	7	0	23.0	21.3	290
8	7	0	6.9	6.1	248
9	7	0	4.8	5.0	318
10	7	0	1.8	2.3	333
11	7	0	1.8	2.4	285
12	7	0	3.6	3.9	86
13	7	0	1.7	1.5	321
15	7	0	1.8	1.9	224
16	7	0	2.7	3.3	151
0	7	1	13.2	12.6	91
1	7	1	8.5	7.9	1
2	7	1	12.4	10.7	231
3	7	1	8.5	9.4	203
4	7	1	13.4	13.1	247
5	7	1	23.6	25.7	103
6	7	1	26.8	26.2	117
7	7	1	22.0	21.6	239
8	7	1	8.5	8.7	18
9	7	1	1.8	2.0	183
10	7	1	9.5	9.8	42
11	7	1	4.5	4.2	312
12	7	1	1.7	1.7	193
13	7	1	1.6	1.7	309
14	7	1	2.5	2.9	203
15	7	1	1.1	0.7	137
-1	7	1	13.2	13.0	153
-2	7	1	17.8	15.9	283
-3	7	1	8.8	6.7	337
-4	7	1	8.3	9.9	288
-5	7	1	12.3	11.2	190
-6	7	1	26.9	30.5	287
-7	7	1	23.8	25.8	23
-8	7	1	20.4	19.9	136
-9	7	1	5.9	5.2	180
-10	7	1	8.2	8.3	256
-11	7	1	6.9	5.6	158
-12	7	1	3.7	3.6	319
-13	7	1	1.7	1.2	122
-14	7	1	2.3	2.2	119
-16	7	1	3.4	3.7	342
0	7	2	17.2	18.4	247
1	7	2	9.2	7.9	306
2	7	2	6.9	5.1	327
3	7	2	7.8	7.5	181
4	7	2	14.1	13.9	261
5	7	2	19.5	20.4	357
6	7	2	13.6	13.3	92
7	7	2	25.3	24.6	169
8	7	2	18.1	19.2	273
9	7	2	1.8	0.9	262
10	7	2	3.2	3.4	90
11	7	2	3.1	2.9	333
12	7	2	2.4	2.3	119
13	7	2	1.5	1.2	209
-1	7	2	12.9	12.9	154
-2	7	2	6.9	6.8	302
-3	7	2	8.1	9.4	59
-4	7	2	3.0	3.2	309
-5	7	2	19.7	20.0	292
-6	7	2	15.0	15.0	246
-7	7	2	42.0	43.4	98
-8	7	2	11.6	11.1	176
-9	7	2	11.6	10.8	254
-10	7	2	12.9	11.2	182
-11	7	2	4.1	3.8	64
-12	7	2	6.4	5.1	26
-13	7	2	3.5	3.0	84
-15	7	2	1.5	1.0	138
-16	7	2	2.1	2.3	352
-17	7	2	1.3	2.3	205
0	7	3	11.3	10.0	100
1	7	3	12.3	12.7	318
2	7	3	6.1	5.7	203
3	7	3	10.9	11.9	82
4	7	3	9.5	10.0	232
5	7	3	19.6	18.3	106
6	7	3	3.5	2.8	37
7	7	3	4.1	4.1	96
8	7	3	5.2	4.4	249
9	7	3	1.9	1.4	284
10	7	3	3.6	3.7	235
11	7	3	2.9	3.5	112
12	7	3	2.2	2.2	254
13	7	3	1.3	0.9	28
14	7	3	1.7	1.9	344
-1	7	3	11.8	14.7	92
-2	7	3	8.4	7.1	347
-3	7	3	9.7	10.3	200
-4	7	3	6.1	5.2	325
-5	7	3	21.0	19.8	224
-6	7	3	21.5	20.6	155
-7	7	3	16.8	15.5	77
-8	7	3	19.1	20.6	278
-9	7	3	11.5	11.7	288
-10	7	3	6.3	6.9	233
-11	7	3	4.1	3.6	354
-12	7	3	5.2	6.6	279
-13	7	3	1.8	1.9	206
-14	7	3	4.7	4.2	94
-15	7	3	1.5	1.3	271
-16	7	3	1.3	1.0	23
0	7	4	10.2	10.1	92
1	7	4	12.7	12.0	14
2	7	4	6.3	6.1	291
3	7	4	8.9	9.0	291
4	7	4	22.1	21.4	272
5	7	4	15.0	16.2	23
6	7	4	10.2	9.6	152
7	7	4	8.5	8.4	176
9	7	4	3.6	4.2	174
10	7	4	2.9	3.1	122
11	7	4	1.5	1.7	149
12	7	4	1.9	1.7	51
13	7	4	1.0	2.0	356
-1	7	4	4.0	3.2	86
-2	7	4	9.1	9.8	272
-3	7	4	1.4	1.3	164
-4	7	4	9.7	7.5	227
-5	7	4	11.4	11.2	143
-6	7	4	7.1	7.6	307
-7	7	4	16.7	15.4	72
-8	7	4	17.8	18.5	129
-9	7	4	10.2	11.0	248
-10	7	4	6.1	5.9	359
-11	7	4	4.9	5.1	244
-12	7	4	4.9	4.8	15
-13	7	4	1.5	1.9	230
-14	7	4	2.4	3.3	127
-15	7	4	2.6	2.7	287
-16	7	4	1.3	1.5	77
0	7	5	4.3	2.0	143
1	7	5	4.1	2.7	308
2	7	5	7.7	7.7	27
3	7	5	5.0	5.8	137
4	7	5	6.8	6.1	235
5	7	5	17.3	16.2	353
6	7	5	12.3	13.5	40
7	7	5	9.8	11.2	145
8	7	5	4.7	5.1	251
9	7	5	1.7	1.9	225
10	7	5	2.6	3.1	133
11	7	5	2.6	2.5	30
12	7	5	1.0	0.3	273
-1	7	5	6.0	3.7	175
-2	7	5	5.3	4.1	22
-3	7	5	6.6	5.7	104
-4	7	5	2.3	2.5	104
-5	7	5	5.5	5.4	269
-6	7	5	17.0	18.0	174
-7	7	5	31.9	34.0	94
-8	7	5	9.8	11.0	277
-9	7	5	23.1	24.0	284
-10	7	5	5.9	5.2	133
-11	7	5	11.1	12.1	353
-12	7	5	6.8	6.8	29
-13	7	5	1.7	0.6	274
-15	7	5	2.6	3.2	219
-17	7	5	2.9	3.1	195
0	7	6	6.4	6.8	94
1	7	6	11.0	10.7	316
2	7	6	1.8	1.7	32
3	7	6	5.5	4.6	334
4	7	6	8.3	7.6	278
5	7	6	6.1	5.7	91
7	7	6	6.2	7.0	92
9	7	6	2.0	1.7	310
10	7	6	1.8	1.4	236
11	7	6	2.2	2.2	123
-1	7	6	4.6	3.7	9
-2	7	6	3.0	3.1	323
-3	7	6	4.6	4.0	175
-4	7	6	3.5	3.9	279
-5	7	6	9.3	8.5	219
-6	7	6	17.1	18.5	118
-7	7	6	15.0	15.9	15
-8	7	6	17.1	16.5	295
-9	7	6	5.7	6.8	273
-10	7	6	10.5	11.5	93
-11	7	6	5.8	4.7	55
-12	7	6	3.1	3.2	19
-13	7	6	1.7	2.8	120
-14	7	6	3.5	4.4	78
-15	7	6	2.0	2.1	240
-16	7	6	1.7	1.2	330
0	7	7	7.6	7.0	104
1	7	7	5.2	6.0	65
2	7	7	7.2	6.4	94
3	7	7	1.8	2.7	175
3	7	7	8.8	2.7	175
5	7	7	10.0	11.3	350
6	7	7	1.7	1.0	252
7	7	7	1.5	1.3	146
8	7	7	1.4	1.5	38
9	7	7	1.6	1.9	321
-1	7	7	2.6	2.2	313
-2	7	7	2.6	1.8	169
-3	7	7	8.0	8.0	243
-4	7	7	3.2	3.5	4
-5	7	7	5.2	4.9	130
-6	7	7	6.4	6.9	12
-7	7	7	3.7	4.0	103
-8	7	7	3.2	2.6	133
-9	7	7	9.3	8.4	273
-10	7	7	4.5	5.4	13
-12	7	7	2.4	2.5	18
-13	7	7	1.6	1.9	134
-14	7	7	1.5	1.6	26
-15	7	7	1.3	1.0	312
-16	7	7	1.0	1.1	332
0	7	8	1.8	1.5	114
1	7	8	4.4	4.5	246
2	7	8	3.1	3.4	57
3	7	8	4.2	4.4	186
4	7	8	7.9	8.4	190
5	7	8	4.1	3.9	317
6	7	8	5.3	5.8	4
8	7	8	0.9	1.6	323
-2	7	8	2.6	2.5	290
-3	7	8	4.9	5.7	88
-4	7	8	4.1	4.6	316
-5	7	8	3.7	4.6	54
-6	7	8	6.4	5.8	195
-7	7	8	12.2	13.8	62
-8	7	8	17.2	18.1	305
-9	7	8	15.5	15.0	266
-10	7	8	10.6	11.5	96
-11	7	8	3.4	2.9	45
-12	7	8	4.3	4.7	85
-13	7	8	4.2	4.8	175
-15	7	8	2.6	2.9	206
0	7	9	1.7	2.1	144
1	7	9	2.3	2.2	188
2	7	9	3.2	2.5	95
3	7	9	1.5	2.4	229
4	7	9	4.4	4.8	284
5	7	9	1.2	0.9	123
6	7	9	1.7	2.3	298
-1	7	9	2.5	2.6	205
-3	7	9	4.7	4.7	191
-4	7	9	4.0	4.2	318
-5	7	9	3.6	3.4	237
-6	7	9	5.9	7.0	72
-7	7	9	10.4	11.8	341
-8	7	9	8.7	9.8	275
-9	7	9	5.6	5.9	174
-10	7	9	9.1	10.2	106
-11	7	9	1.5	1.5	83
-12	7	9	2.0	2.1	216
-13	7	9	1.3	1.4	182
-14	7	9	2.0	1.7	186
-15	7	9	2.0	2.2	256
0	7	10	1.4	1.1	214
1	7	10	1.4	0.7	169
2	7	10	3.6	4.1	88
3	7	10	5.1	4.9	159
4	7	10	2.3	2.8	205
-1	7	10	2.2	1.8	262
-2	7	10	3.1	2.9	191
-3	7	10	4.5	4.9	264
-4	7	10	3.2	3.5	350
-5	7	10	3.6	3.8	85
-6	7	10	1.6	2.0	357
-7	7	10	1.6	1.8	270
-9	7	10	4.8	4.7	279
-10	7	10	3.2	3.2	83
-11	7	10	2.7	1.9	166
-12	7	10	1.7	1.6	223
-13	7	10	1.8	2.3	165
1	7	11	0.9	1.4	282
-1	7	11	1.2	1.1	145
-2	7	11	1.2	1.5	256
-4	7	11	1.9	1.9	359
-5	7	11	1.3	0.9	113
-6	7	11	1.9	2.5	228
-7	7	11	2.6	2.2	347
-8	7	11	6.5	8.0	297
-9	7	11	5.8	7.1	211
-10	7	11	8.6	9.4	134
-11	7	11	1.0	0.7	165
1	8	0	4.3	3.4	63
2	8	0	6.0	4.9	299
3	8	0	13.4	12.3	217
4	8	0	8.6	7.7	125
5	8	0	9.3	9.4	3
6	8	0	31.7	30.6	309
7	8	0	9.3	9.7	127
8	8	0	8.1	7.4	31
9	8	0	3.7	3.5	66
10	8	0	2.9	2.3	63
11	8	0	2.3	2.1	277
12	8	0	5.4	5.2	39
14	8	0	1.2	1.5	47
15	8	0	0.9	1.6	280
0	8	1	2.0	2.1	85
1	8	1	7.3	5.6	42
2	8	1	12.2	11.0	18
3	8	1	12.9	13.9	171
4	8	1	14.7	14.3	143
5	8	1	29.5	30.5	90
6	8	1	36.0	38.4	309
7	8	1	17.4	16.0	270
8	8	1	4.7	5.0	192
9	8	1	3.3	3.4	45
10	8	1	3.7	3.4	269
13	8	1	3.2	3.0	195
14	8	1	1.5	1.3	158
-1	8	1	8.2	8.2	327
-2	8	1	16.5	17.5	22
-3	8	1	3.1	3.0	9
-4	8	1	13.0	14.8	20
-5	8	1	17.0	17.0	130
-6	8	1	9.3	9.4	37
-7	8	1	16.9	16.5	302
-8	8	1	8.0	8.4	52
-9	8	1	2.9	2.3	128
-10	8	1	6.1	6.1	249
-11	8	1	7.0	7.2	322
-12	8	1	2.2	1.7	59
-13	8	1	1.5	2.6	274
-14	8	1	1.3	2.2	176
0	8	2	10.2	10.6	182
1	8	2	16.6	16.9	256
2	8	2	5.1	3.2	328
3	8	2	3.7	4.4	265
4	8	2	4.9	5.3	286
5	8	2	19.4	19.1	142
6	8	2	8.6	8.5	64
7	8	2	3.7	3.3	246
8	8	2	2.9	2.3	49
9	8	2	3.7	3.3	152
10	8	2	2.8	2.4	19
11	8	2	4.3	3.8	285
12	8	2	3.4	3.2	40
13	8	2	2.0	1.8	163
-1	8	2	4.2	3.3	325
-2	8	2	6.3	6.2	15
-3	8	2	6.0	4.0	211
-4	8	2	3.4	3.3	142
-5	8	2	15.0	16.4	44
-6	8	2	45.5	48.0	292
-7	8	2	6.8	6.2	285
-8	8	2	22.8	23.8	119
-9	8	2	5.7	4.3	66
-10	8	2	6.5	6.9	111
-11	8	2	6.0	5.0	72
-12	8	2	4.6	4.1	111
-13	8	2	1.5	1.5	260
-14	8	2	1.3	1.6	63
-15	8	2	3.6	3.9	349
-16	8	2	3.4	4.2	318
0	8	3	5.0	3.2	129
1	8	3	5.0	5.8	123
2	8	3	5.0	5.7	68
3	8	3	11.8	12.7	222
4	8	3	21.5	22.0	138
5	8	3	9.1	8.3	166
6	8	3	13.7	14.1	285
7	8	3	5.6	5.0	187
8	8	3	1.7	1.5	264
10	8	3	2.2	2.6	227
11	8	3	4.4	4.5	220
-1	8	3	5.6	6.6	48
-2	8	3	6.3	6.4	198
-3	8	3	4.3	3.5	267
-4	8	3	8.5	7.3	40
-5	8	3	16.3	16.9	350
-6	8	3	17.4	18.0	311
-7	8	3	17.9	18.3	113
-8	8	3	12.5	11.9	135
-9	8	3	2.3	1.9	5
-10	8	3	8.2	6.9	180
-11	8	3	5.0	5.3	328
-12	8	3	3.2	2.5	176
-13	8	3	1.5	1.5	307
-15	8	3	2.1	2.3	296
0	8	4	10.4	9.7	167
1	8	4	7.4	5.7	333
2	8	4	2.1	1.9	322
3	8	4	8.8	8.2	241
4	8	4	19.8	20.0	177
5	8	4	24.9	26.3	109
6	8	4	10.2	11.8	344
7	8	4	6.0	5.4	262
8	8	4	3.9	3.6	212
9	8	4	3.1	2.3	46
11	8	4	2.1	1.5	164
12	8	4	1.6	0.7	53
-1	8	4	7.3	6.7	287
-2	8	4	6.5	7.2	181
-3	8	4	5.6	6.1	242
-4	8	4	3.2	2.1	135
-5	8	4	7.8	7.6	161
-6	8	4	6.9	6.6	217
-7	8	4	16.2	17.1	305
-8	8	4	12.8	11.7	115
-9	8	4	9.6	8.8	179
-10	8	4	9.5	9.0	238
-11	8	4	5.2	4.9	345
-12	8	4	5.3	5.4	93
-13	8	4	3.0	2.9	211
-14	8	4	4.4	4.7	109
-15	8	4	2.4	2.3	47
-16	8	4	2.3	1.8	297
0	8	5	8.9	9.1	148
1	8	5	4.5	4.5	270
2	8	5	4.3	3.2	208
3	8	5	7.8	7.2	318
4	8	5	6.9	6.3	238
5	8	5	5.8	4.7	204
6	8	5	3.7	4.4	83
7	8	5	3.2	3.0	26
8	8	5	5.2	5.2	186
9	8	5	1.4	1.6	164
10	8	5	2.1	1.7	145
11	8	5	2.2	2.2	293
-1	8	5	7.8	6.1	70
-2	8	5	5.8	6.2	108
-3	8	5	4.3	3.8	169
-4	8	5	5.8	5.6	133
-5	8	5	3.9	4.3	202
-6	8	5	20.5	20.7	285
-7	8	5	11.9	11.3	122
-8	8	5	27.6	28.7	115
-9	8	5	7.5	7.5	245
-11	8	5	5.2	5.4	330
-12	8	5	5.5	4.8	222
-13	8	5	5.3	2.8	222
-15	8	5	4.5	1.8	331
-16	8	5	3.3	2.6	285
2	8	6	8.4	8.5	17
3	8	6	8.9	10.2	309
4	8	6	8.3	9.2	108
5	8	6	4.8	4.6	134
6	8	6	2.2	3.1	310
7	8	6	1.4	1.3	196
8	8	6	2.3	2.6	112
9	8	6	1.1	1.5	39
10	8	6	2.5	2.9	100
-1	8	6	2.9	3.4	324
-2	8	6	6.5	5.9	205
-3	8	6	3.3	2.7	229
-4	8	6	1.6	1.3	281
-5	8	6	1.6	2.5	99
-6	8	6	6.2	6.1	226
-7	8	6	18.7	19.2	126
-8	8	6	11.5	12.2	187
-9	8	6	7.1	7.2	311
-10	8	6	4.4	4.4	245
-11	8	6	5.1	5.1	315
-12	8	6	2.1	2.4	180
-13	8	6	1.4	2.1	195
-14	8	6	2.6	1.8	70
-15	8	6	2.1	2.0	174
1	8	7	4.7	5.5	29
2	8	7	7.0	6.9	1
3	8	7	10.3	11.0	318
4	8	7	6.4	6.9	218
5	8	7	7.4	7.9	124
6	8	7	4.5	4.4	30
7	8	7	1.2	0.7	50
8	8	7	2.2	2.7	221
-1	8	7	1.7	1.9	304
-2	8	7	2.4	2.9	213
-4	8	7	4.7	4.8	52
-5	8	7	5.6	5.0	171
-6	8	7	3.3	3.1	163
-7	8	7	5.8	5.7	296
-8	8	7	10.7	10.8	109
-9	8	7	6.1	7.3	189
-10	8	7	8.7	9.7	296
-11	8	7	7.7	7.0	28
-12	8	7	2.0	1.9	71
-13	8	7	2.6	2.8	194
-14	8	7	2.0	1.9	100
-15	8	7	1.3	1.1	211
0	8	8	1.6	1.3	58
1	8	8	3.5	3.6	39
3	8	8	4.4	4.7	11
4	8	8	1.3	1.7	286
5	8	8	3.9	4.2	228
6	8	8	2.4	2.4	359
-1	8	8	1.6	1.7	66
-2	8	8	1.6	2.8	68
-3	8	8	2.3	2.6	300
-5	8	8	4.6	4.9	220
-6	8	8	5.2	5.9	319
-7	8	8	9.6	9.8	87
-8	8	8	10.9	11.0	126
-9	8	8	11.3	12.2	283
-10	8	8	6.4	6.6	332
-11	8	8	4.5	4.7	360
-12	8	8	1.9	1.5	217
-13	8	8	2.0	1.7	147
-14	8	8	2.0	1.7	27
0	8	9	2.0	2.1	205
1	8	9	1.3	1.0	146
2	8	9	1.9	2.1	18
3	8	9	6.2	7.2	317
4	8	9	2.2	2.9	35
-1	8	9	1.5	1.4	62
-2	8	9	1.5	0.9	223
-3	8	9	1.5	1.5	57
-4	8	9	1.5	1.9	331
-5	8	9	3.0	3.4	100
-6	8	9	2.1	2.1	19
-7	8	9	3.7	4.1	124
-8	8	9	4.6	5.1	257
-9	8	9	8.3	8.7	330
-10	8	9	3.3	3.5	355
-11	8	9	1.2	1.5	37
-12	8	9	1.9	2.3	104
1	8	10	3.7	3.4	136
2	8	10	1.9	1.8	90
-1	8	10	2.7	3.5	249
-2	8	10	1.3	0.9	79
-3	8	10	1.3	1.5	8
-4	8	10	2.3	3.0	12
-5	8	10	1.3	1.6	230
-7	8	10	2.9	3.7	341
-8	8	10	3.3	3.2	104
-9	8	10	1.7	2.0	337
-10	8	10	6.1	6.7	310
-11	8	10	5.5	6.7	50
-2	8	11	1.6	1.3	122
-3	8	11	2.7	2.9	326
-4	8	11	0.9	1.6	261
-5	8	11	1.3	1.5	234
-6	8	11	2.7	3.1	25
-3	8	11	2.7	2.9	326
-8	8	11	2.4	2.6	184

Table 2-12 Aflatoxin G_1 benzene solvate

Final fractional atomic coordinates and their estimated standard deviations in Å

Atom	x/a	y/b	z/c	σ(x)	σ(y)	σ(z)
C(1)	0.4083	0.5555	0.0193	0.008	0.009	0.008
O(2)	0.4849	0.4914	0.0710	0.006	0.007	0.007
C(3)	0.4826	0.3684	0.1301	0.008	0.011	0.009
C(4)	0.4122	0.2816	0.0445	0.008	0.011	0.009
C(5)	0.3221	0.3501	0.0041	0.007	0.009	0.007
C(6)	0.3246	0.4815	0.0098	0.008	0.010	0.008
C(7)	0.2427	0.5571	-0.0114	0.008	0.009	0.008
O(8)	0.1637	0.4918	-0.0392	0.005	0.006	0.006
C(9)	0.1596	0.3595	-0.0520	0.007	0.009	0.007
C(10)	0.2377	0.2855	-0.0336	0.008	0.009	0.008
C(11)	0.2248	0.1507	-0.0536	0.007	0.009	0.008
C(12)	0.1388	0.0926	-0.0860	0.008	0.010	0.009
C(13)	0.0687	0.1757	-0.0971	0.007	0.010	0.008
C(14)	0.0761	0.3066	-0.0830	0.008	0.010	0.008
C(15)	-0.0164	0.3623	-0.1093	0.008	0.010	0.009
C(16)	-0.0760	0.2422	-0.1325	0.009	0.012	0.010
O(17)	-0.0168	0.1335	-0.1238	0.006	0.007	0.007
O(18)	-0.1396	0.2494	-0.2519	0.008	0.010	0.009
C(19)	-0.1207	0.3543	-0.3146	0.011	0.014	0.013
C(20)	-0.0543	0.4227	-0.2414	0.009	0.013	0.011
O(21)	0.4093	0.6636	-0.0153	0.007	0.008	0.008
O(22)	0.2375	0.6709	0.0009	0.006	0.007	0.007
O(23)	0.2988	0.0838	-0.0455	0.006	0.007	0.007
C(24)	0.2921	-0.0591	-0.0498	0.010	0.010	0.011
C(25)	0.1546	0.2654	0.2550	0.013	0.019	0.013
C(26)	0.2295	0.1843	0.2807	0.014	0.014	0.014
C(27)	0.3134	0.2376	0.3213	0.012	0.016	0.013
C(28)	0.3260	0.3649	0.3467	0.013	0.018	0.013
C(29)	0.2522	0.4483	0.3269	0.015	0.015	0.014
C(30)	0.1665	0.3908	0.2752	0.014	0.016	0.014

Table 2-12 (cont.)

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1')	0.1199	0.0275	0.6061	0.009	0.011	0.010
O(2')	0.0410	0.0838	0.5682	0.006	0.007	0.007
C(3')	0.0392	0.2122	0.5088	0.010	0.011	0.011
C(4')	0.1134	0.2966	0.5936	0.009	0.011	0.009
C(5')	0.2032	0.2292	0.6245	0.008	0.010	0.008
C(6')	0.2020	0.0969	0.6168	0.009	0.010	0.008
C(7')	0.2817	0.0245	0.6280	0.010	0.010	0.009
O(8')	0.3591	0.0945	0.6435	0.006	0.007	0.006
C(9')	0.3640	0.2221	0.6627	0.009	0.011	0.009
C(10')	0.2873	0.2961	0.6548	0.009	0.010	0.009
C(11')	0.2998	0.4297	0.6764	0.010	0.011	0.010
C(12')	0.3855	0.4851	0.7024	0.011	0.012	0.010
C(13')	0.4551	0.4043	0.7024	0.010	0.012	0.009
C(14')	0.4475	0.2787	0.6833	0.009	0.010	0.009
C(15')	0.5360	0.2209	0.6817	0.009	0.012	0.011
C(16')	0.5959	0.3478	0.7175	0.010	0.013	0.012
O(17')	0.5417	0.4520	0.7248	0.007	0.008	0.008
O(18')	0.6291	0.3661	0.6145	0.009	0.011	0.010
C(19')	0.5934	0.2756	0.5238	0.010	0.013	0.012
C(20')	0.5411	0.1953	0.5540	0.009	0.011	0.010
O(21')	0.1202	-0.0838	0.6449	0.007	0.008	0.008
O(22')	0.2912	-0.0897	0.6229	0.008	0.009	0.009
O(23')	0.2266	0.4967	0.6776	0.007	0.008	0.007
C(24')	0.2315	0.6373	0.6739	0.014	0.013	0.013

Table 2-13 Aflatoxin G_1 benzene solvate

Anisotropic temperature factors

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	0.047	0.046	0.052	-0.010	0.048	-0.015
O(2)	0.039	0.064	0.072	-0.015	0.036	-0.012
C(3)	0.050	0.064	0.063	-0.011	0.030	-0.002
C(4)	0.040	0.063	0.062	-0.014	0.027	0.009
C(5)	0.036	0.042	0.045	-0.005	0.037	-0.004
C(6)	0.040	0.051	0.056	-0.009	0.045	-0.001
C(7)	0.037	0.044	0.054	0.004	0.037	-0.002
O(8)	0.043	0.053	0.058	-0.004	0.048	-0.003
C(9)	0.045	0.040	0.041	-0.002	0.046	0
C(10)	0.043	0.048	0.047	0.006	0.033	-0.001
C(11)	0.045	0.043	0.049	0.001	0.038	-0.007
C(12)	0.052	0.048	0.055	-0.004	0.039	-0.013
C(13)	0.036	0.065	0.046	0	0.030	-0.017
C(14)	0.039	0.058	0.052	0.007	0.042	-0.009
C(15)	0.040	0.069	0.064	-0.002	0.048	-0.001
C(16)	0.045	0.076	0.066	0.005	0.042	-0.010
O(17)	0.046	0.053	0.078	0.004	0.051	-0.026
O(18)	0.063	0.094	0.105	0.003	0.022	-0.020
C(19)	0.068	0.089	0.095	0.052	0.049	0.022
C(20)	0.052	0.074	0.080	0.023	0.035	0.022
O(21)	0.053	0.065	0.082	0.001	0.051	-0.023
O(22)	0.048	0.050	0.085	0.006	0.055	0.006
O(23)	0.053	0.043	0.077	-0.002	0.048	0.010
C(24)	0.072	0.043	0.068	-0.008	0.029	0.018
C(25)	0.092	0.118	0.071	0.013	0.034	-0.024
C(26)	0.102	0.083	0.080	0.031	0.071	0.003
C(27)	0.080	0.092	0.081	0.007	0.052	0.011
C(28)	0.088	0.107	0.078	0.022	0.054	-0.006
C(29)	0.111	0.109	0.078	0.048	0.071	0.052
C(30)	0.107	0.102	0.088	0.057	0.075	0.072

Table 2-13 (cont.)

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1')	0.058	0.054	0.056	-0.014	0.034	-0.010
O(2')	0.054	0.066	0.078	-0.005	0.036	-0.001
C(3')	0.061	0.057	0.069	0.002	0.022	-0.003
C(4')	0.048	0.058	0.060	-0.013	0.041	0.006
C(5')	0.060	0.051	0.040	-0.002	0.041	0
C(6')	0.056	0.040	0.048	-0.001	0.039	0.001
C(7')	0.066	0.048	0.054	0.006	0.052	-0.001
O(8')	0.057	0.050	0.060	0.016	0.054	0.016
C(9')	0.060	0.058	0.048	0.004	0.056	-0.005
C(10')	0.055	0.054	0.044	0.002	0.041	-0.007
C(11')	0.067	0.047	0.058	-0.009	0.056	-0.009
C(12')	0.078	0.057	0.062	-0.013	0.054	-0.024
C(13')	0.062	0.079	0.053	-0.017	0.047	-0.022
C(14')	0.060	0.060	0.049	0.009	0.045	-0.004
C(15')	0.050	0.083	0.071	0.017	0.047	0.012
C(16')	0.060	0.095	0.081	-0.008	0.051	-0.033
O(17')	0.076	0.087	0.078	-0.027	0.065	-0.054
O(18')	0.083	0.117	0.124	-0.027	0.119	-0.065
C(19')	0.056	0.088	0.081	-0.023	0.048	-0.011
C(20')	0.053	0.082	0.059	0.009	0.049	0.002
O(21')	0.078	0.056	0.072	-0.005	0.051	-0.027
O(22')	0.082	0.064	0.096	0.002	0.090	0.008
O(23')	0.077	0.049	0.079	-0.006	0.070	0.011
C(24')	0.113	0.037	0.091	0.014	0.064	0.010

Table 2-14 Aflatoxin G_1 benzene solvate

Bond lengths and their estimated standard deviations

	Molecule 1		Molecule 1'		Average
C(1)-O(2)	1.331	± 0.010	1.312	± 0.011	1.322
C(1)-C(6)	1.502	0.011	1.454	0.013	1.478
C(1)-O(21)	1.196	0.012	1.240	0.013	1.218
O(2)-C(3)	1.448	0.012	1.490	0.013	1.469
C(3)-C(4)	1.498	0.013	1.512	0.013	1.505
C(4)-C(5)	1.519	0.011	1.514	0.012	1.516
C(5)-C(6)	1.377	0.013	1.388	0.014	1.382
C(5)-C(10)	1.424	0.011	1.436	0.012	1.430
C(6)-C(7)	1.464	0.011	1.436	0.013	1.450
C(7)-O(8)	1.362	0.010	1.384	0.011	1.373
C(7)-O(22)	1.205	0.011	1.209	0.013	1.207
O(8)-C(9)	1.392	0.011	1.351	0.013	1.371
C(9)-C(10)	1.409	0.011	1.412	0.013	1.411
C(9)-C(14)	1.362	0.011	1.392	0.012	1.377
C(10)-C(11)	1.432	0.013	1.421	0.015	1.427
C(11)-C(12)	1.418	0.011	1.409	0.014	1.413
C(12)-C(13)	1.379	0.012	1.384	0.015	1.381
C(13)-C(14)	1.380	0.014	1.331	0.016	1.355
C(13)-O(17)	1.354	0.009	1.396	0.012	1.375
C(14)-C(15)	1.505	0.011	1.524	0.013	1.515
C(15)-C(16)	1.539	0.014	1.600	0.017	1.569
C(15)-C(20)	1.508	0.014	1.453	0.013	1.481
C(16)-O(17)	1.454	0.012	1.403	0.014	1.428
C(16)-O(18)	1.359	0.012	1.408	0.014	1.383
O(18)-C(19)	1.380	0.017	1.353	0.015	1.366
C(19)-C(20)	1.300	0.016	1.295	0.015	1.298
C(11)-O(23)	1.337	0.010	1.354	0.012	1.346
O(23)-C(24)	1.499	0.012	1.476	0.015	1.487

Table 2-15 Aflatoxin G_1 benzene solvate

Bond angles and their estimated standard deviations

	Molecule 1	Molecule 1'	Average
O(2)C(1)O(21)	120.4±0.7°	115.2±0.8°	117.8°
C(6)C(1)O(21)	124.8 0.7	122.8 0.8	123.8
C(6)C(1)O(2)	114.9 0.7	121.6 0.9	118.3
C(1)O(2)C(3)	118.9 0.6	114.9 0.7	116.9
O(2)C(3)C(4)	112.6 0.7	111.1 0.8	111.9
C(3)C(4)C(5)	108.7 0.8	109.7 0.8	109.2
C(4)C(5)C(6)	116.7 0.7	117.3 0.8	117.0
C(6)C(5)C(10)	119.8 0.7	119.5 0.8	119.7
C(4)C(5)C(10)	123.5 0.8	123.1 0.9	123.3
C(5)C(6)C(7)	121.5 0.7	122.0 0.8	121.7
C(1)C(6)C(5)	122.0 0.7	119.8 0.8	120.9
C(1)C(6)C(7)	116.0 0.8	118.1 0.9	117.1
C(6)C(7)O(22)	127.3 0.7	129.6 0.9	128.4
O(8)C(7)O(22)	115.5 0.7	114.3 0.8	114.9
C(6)C(7)O(8)	117.0 0.8	116.1 0.8	116.6
C(7)O(8)C(9)	122.0 0.6	123.0 0.7	122.5
C(8)C(9)C(10)	121.6 0.7	122.0 0.8	121.8
O(8)C(9)C(14)	116.0 0.7	116.6 0.8	116.3
C(10)C(9)C(14)	122.5 0.8	121.3 0.9	121.9
C(9)C(10)C(5)	117.8 0.8	116.8 0.9	117.3
C(9)C(10)C(11)	116.6 0.7	117.5 0.8	117.0
C(5)C(10)C(11)	125.7 0.7	125.7 0.8	125.7
C(10)C(11)C(12)	122.4 0.8	120.6 1.0	121.5
C(10)C(11)O(23)	115.5 0.7	116.1 0.8	115.8
C(12)C(11)O(23)	122.0 0.7	123.2 0.9	122.6
C(11)C(12)C(13)	114.9 0.9	117.0 1.0	115.9
C(12)C(13)C(14)	125.6 0.7	125.1 0.9	125.3
C(12)C(13)O(17)	121.5 0.9	120.6 1.0	121.1
C(14)C(13)O(17)	112.9 0.7	114.3 0.9	113.2
C(13)C(14)C(9)	118.0 0.7	118.4 0.9	118.2
C(15)C(14)C(9)	133.1 0.9	130.4 0.9	131.8
C(13)C(14)C(15)	108.9 0.7	111.1 0.8	110.0
C(14)C(15)C(16)	102.3 0.8	97.7 0.9	100.0
C(14)C(15)C(20)	113.2 0.7	115.4 0.8	114.3
C(16)C(15)C(20)	99.2 0.7	100.0 0.8	99.6
C(15)C(16)O(17)	106.7 0.8	109.9 0.8	108.3
C(15)C(16)O(18)	109.1 0.9	105.2 0.9	107.2
O(17)C(16)O(18)	110.9 0.6	109.6 1.0	110.3
C(13)O(17)C(16)	109.2 0.7	106.8 0.9	108.0
C(16)O(18)C(19)	108.2 0.9	108.6 0.9	108.4
O(18)C(19)C(20)	113.2 1.0	114.3 1.0	113.8
C(19)C(20)C(15)	109.9 1.0	111.7 1.0	110.8
C(11)O(23)C(24)	118.0 0.6	117.7 0.8	117.8

Table 2-16

Bond lengths and angles of the benzene molecule
and their estimated standard deviations

C(25)-C(26)	1.404	± 0.020	C(28)-C(29)	1.412	± 0.020
C(26)-C(27)	1.365	0.018	C(29)-C(30)	1.412	0.019
C(27)-C(28)	1.363	0.024	C(30)-C(25)	1.334	0.024

C(30)C(25)C(26)	120.0	± 1.3°	C(27)C(28)C(29)	121.2	± 1.2°
C(25)C(26)C(27)	118.5	1.4	C(28)C(29)C(30)	115.2	1.4
C(26)C(27)C(28)	121.7	1.2	C(29)C(30)C(25)	123.3	1.3

Intramolecular non-bonded distances
of the benzene molecule

C(25)...C(27)	2.38	C(26)...C(30)	2.37
C(25)...C(28)	2.75	C(27)...C(29)	2.42
C(25)...C(29)	2.41	C(27)...C(30)	2.72
C(26)...C(28)	2.38	C(28)...C(30)	2.38
C(26)...C(29)	2.81		

Bond lengths and angles in aflatoxin G_1 benzene solvate.

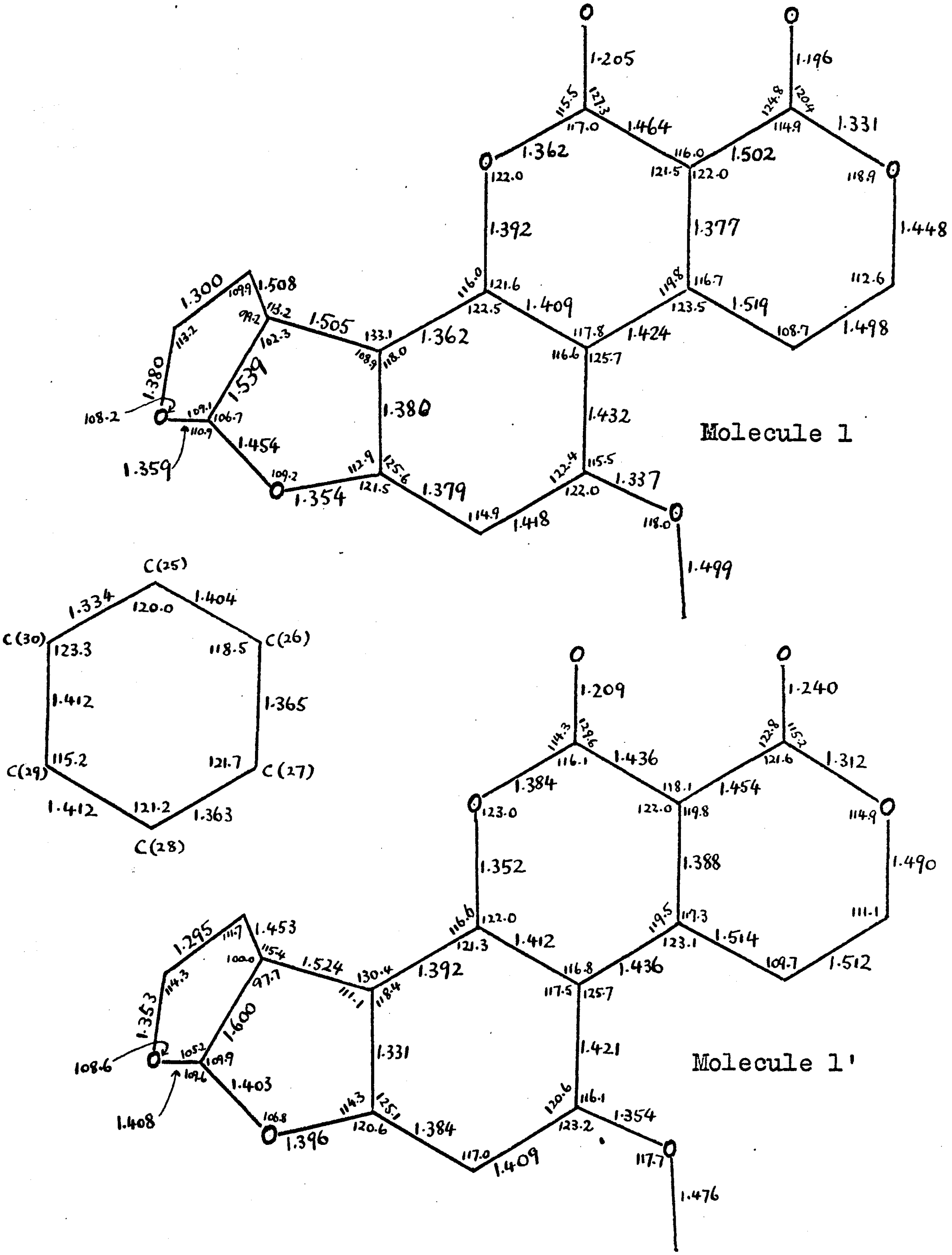


Table 2-17 Aflatoxin G_1 benzene solvate

Intramolecular non-bonded distances $\leqslant 3.5$A

	Mol. 1	Mol. 1'
C(1)...C(3)	2.39	2.36
C(1)...C(7)	2.52	2.47
C(1)...C(5)	2.52	2.46
C(1)...C(4)	2.88	2.82
C(1)...O(22)	2.89	2.91
O(2)...O(21)	2.19	2.15
O(2)...C(6)	2.39	2.42
O(2)...C(4)	2.45	2.48
O(2)...C(5)	2.84	2.86
C(3)...C(5)	2.45	2.47
C(3)...C(6)	2.67	2.72
C(3)...O(21)	3.49	3.49
C(4)...C(6)	2.47	2.48
C(4)...C(10)	2.59	2.59
C(4)...O(23)	2.71	2.71
C(4)...C(11)	3.10	3.10
C(5)...C(9)	2.43	2.43
C(5)...C(7)	2.48	2.47
C(5)...C(11)	2.54	2.54
C(5)...O(8)	2.80	2.78
C(5)...O(23)	2.84	2.86
C(6)...O(22)	2.39	2.39
C(6)...O(21)	2.40	2.37
C(6)...O(8)	2.41	2.39
C(6)...C(10)	2.42	2.44
C(6)...C(9)	2.77	2.76
C(7)...C(9)	2.41	2.40
C(7)...C(10)	2.85	2.86
C(7)...O(21)	2.86	2.85
O(8)...O(22)	2.17	2.18
O(8)...C(14)	2.33	2.33
O(8)...C(10)	2.44	2.42
O(8)...C(15)	3.00	2.99
O(8)...C(20)	3.49	3.49
C(9) ...C(13)	2.35	2.34
C(9) ...C(11)	2.42	2.42
C(9) ...C(15)	2.63	2.65
C(9) ...C(12)	2.82	2.79
C(9) ...C(20)	3.38	3.39
C(9) ...O(22)	3.46	3.44
C(10)...O(23)	2.34	2.36
C(10)...C(14)	2.43	2.44
C(10)...C(12)	2.50	2.46
C(10)...C(13)	2.77	2.76
C(11)...C(13)	2.36	2.38
C(11)...C(24)	2.43	2.42
C(11)...C(14)	2.78	2.79
C(12)...O(17)	2.38	2.41
C(12)...O(23)	2.41	2.43
C(12)...C(14)	2.45	2.41
C(12)...C(24)	2.81	2.83
C(13)...C(16)	2.29	2.25
C(13)...C(15)	2.35	2.36
C(13)...O(18)	3.24	3.22
C(13)...C(20)	3.30	3.28
C(14)...O(17)	2.28	2.29
C(14)...C(16)	2.37	2.35
C(14)...C(20)	2.52	2.52
C(14)...C(19)	3.32	3.32
C(14)...O(18)	3.34	3.32
C(15)...C(19)	2.30	2.28
C(15)...O(18)	2.36	2.39
C(15)...O(17)	2.40	2.46
C(16)...C(19)	2.22	2.24
C(16)...C(20)	2.32	2.34
O(17)...O(18)	2.32	2.30
O(17)...C(19)	3.18	3.18
O(17)...C(20)	3.27	3.27
O(18)...C(20)	2.24	2.22

Table 2-18 Aflatoxin G_1 benzene solvate
Intermolecular distances ≤4Å

O(22)...C(24)XI	3.06
C(4) ...O(21)X	3.18
C(3) ...O(21)X	3.24
O(21)...C(24)XI	3.39
C(16)...O(22)IV	3.40
O(2) ...C(24)IX	3.63
O(2) ...O(23)IX	3.64
O(8)....O(17)II	3.67
O(18)...O(22)IV	3.67
C(15)...O(17)II	3.74
O(8) ...C(16)II	3.75
O(2) ...C(4)IX	3.85
C(1) ...C(4)IX	3.93
O(2) ...O(21)X	3.95
C(7) ...C(16)II	3.99

O(22')...C(24')XII	3.12
C(19')...O(22')VI	3.14
O(18')...O(22')VI	3.28
C(3')...O(21')VII	3.30
O(21')...C(24')XII	3.37
O(8')...O(18')V	3.75
C(7')...O(18')V	3.78
O(17')...C(20')VI	3.86
O(2')...C(4')VIII	3.90
C(4')...O(21')VII	3.94
C(7')...C(19')V	3.95
O(8')...C(19')V	4.00

C(30)...O(17)II	3.49
C(25)...C(14)	3.52
C(25)...C(9)	3.53
C(30)...C(9)	3.57
C(29)...O(18)II	3.58
C(30)...O(8)	3.59
C(26)...C(10)	3.65
C(26)...C(11)	3.66
C(27)...C(10)	3.70
C(27)...C(5)	3.72
C(28)...C(5)	3.74
C(25)...C(13)	3.76
C(30)...O(18)II	3.78
C(25)...C(10)	3.81
C(29)...O(8)	3.81

C(30)...C(14)	3.81
C(26)...C(20)IV	3.81
C(29)...C(7)	3.83
C(27)...C(4)	3.87
C(28)...C(6)	3.88
C(26)...C(9)	3.90
C(25)...C(20)IV	3.90
C(26)...C(12)	3.91
C(26)...C(19)IV	3.93
C(28)...C(3)	3.95
C(27)...C(11)	3.98

C(27)...C(9')	3.56
C(28)...C(9')	3.63
C(28)...C(14')	3.66
C(29)...C(11')	3.66
C(27)...C(20')	3.67
C(27)...O(8')	3.68
C(28)...C(10')	3.70
C(28)...C(13')	3.77
C(29)...C(10')	3.80
C(28)...C(20')	3.83
C(28)...C(11')	3.83
C(25)...C(3')	3.84
C(27)...C(14')	3.85
C(27)...C(10')	3.86
C(25)...C(5')	3.88
C(27)...O(17')V	3.89
C(28)...C(12')	3.90
C(26)...O(8')	3.93
C(26)...C(5')	3.95
C(26)...C(6')	3.96
C(26)...O(18')V	3.96
C(29)...C(12')	3.97
C(30)...C(4')	3.98
C(25)...C(4')	3.99
C(26)...C(7')	3.99

C(12)...C(1')I	3.36
C(7) ...O(23')I	3.39
C(12)...O(21')I	3.41
O(2) ...O(8')VI	3.44
C(1) ...C(12')I	3.45
C(6) ...O(23')I	3.45

Table 2-18 (cont.)

C(7) ...C(24')I	3.50
O(21)...C(15')VI	3.52
C(11)...C(5')I	3.53
O(21)...C(12')I	3.53
O(23)...C(6')I	3.54
C(24)...O(21')I	3.54
C(1) ...C(15')VI	3.55
C(11)...C(6')I	3.55
O(23)...C(7')I	3.55
O(8) ...O(23')I	3.56
O(22)...C(24')I	3.57
C(6) ...C(11')I	3.58
C(12)...O(2')I	3.58
C(5) ...C(11')I	3.59
C(19)...O(22')II	3.59
C(24)...C(7')I	3.59
C(24)...O(22')I	3.60
C(24)...C(16')V	3.61
O(21)...C(24')I	3.63
C(10)...C(5')I	3.65
O(18)...O(18')III	3.65
C(24)...O(17')V	3.65
C(13)...O(2')I	3.67
C(19)...C(1')II	3.68
C(19)...O(21')II	3.68
C(3) ...O(22')VI	3.71
C(12)...C(6')I	3.71
O(2) ...C(15')VI	3.72
C(3) ...O(8')VI	3.72
C(5) ...O(23')I	3.72
C(5) ...C(10')I	3.73
O(23)...C(5')I	3.74
C(9) ...O(23')I	3.75
C(10)...C(10')I	3.75
C(9) ...C(14')I	3.76
C(9) ...O(18')III	3.76
C(11)...C(1')I	3.77
C(14)...C(4')I	3.78
O(21)...C(14')VI	3.78
C(6) ...C(12')I	3.80
O(22)...C(16')VI	3.80
C(1) ...C(11')I	3.81
C(10)...O(23')I	3.81
O(17)...O(2')I	3.81
O(21)...C(16')VI	3.81
O(2) ...C(12')I	3.82
C(24)...C(6')I	3.82
O(23)...O(17')V	3.83
O(23)...O(8')I	3.83
C(6) ...C(24')I	3.84
C(20)...C(4')I	3.89
C(10)...C(4')I	3.90
C(13)...C(4')I	3.90
C(12)...C(5')I	3.91
O(2) ...C(9')VI	3.92
C(13)...C(1')I	3.92
O(23)...C(10')I	3.92
C(1) ...O(23')I	3.93
C(10)...C(11')I	3.93
O(2) ...C(14')VI	3.94
C(20)...C(1')II	3.94
O(22)...O(23')I	3.94
O(8) ...C(24')I	3.95
C(11)...C(10')I	3.95
C(19)...C(3')I	3.95
C(19)...C(7')II	3.95
O(23)...C(9')I	3.95
C(24)...C(1')I	3.95
O(23)...C(1')I	3.96
C(1) ...C(24')I	3.97
C(11)...C(4')I	3.98

The Roman numerals refer to the following equivalent positions:

I: x,y,-1+z

II: -x,½+y,-z

III: -1+x,y,-1+z

IV: -x,-½+y,-z

V: 1-x,-½+y,1-z

VI: 1-x,½+y,1-z

VII: -x,½+y,1-z

VIII: -x,-½+y,1-z

IX: 1-x,½+y,-z

X: 1-x,-½+y,-z

XI: x,1+y,z

XII: x,-1+y,z

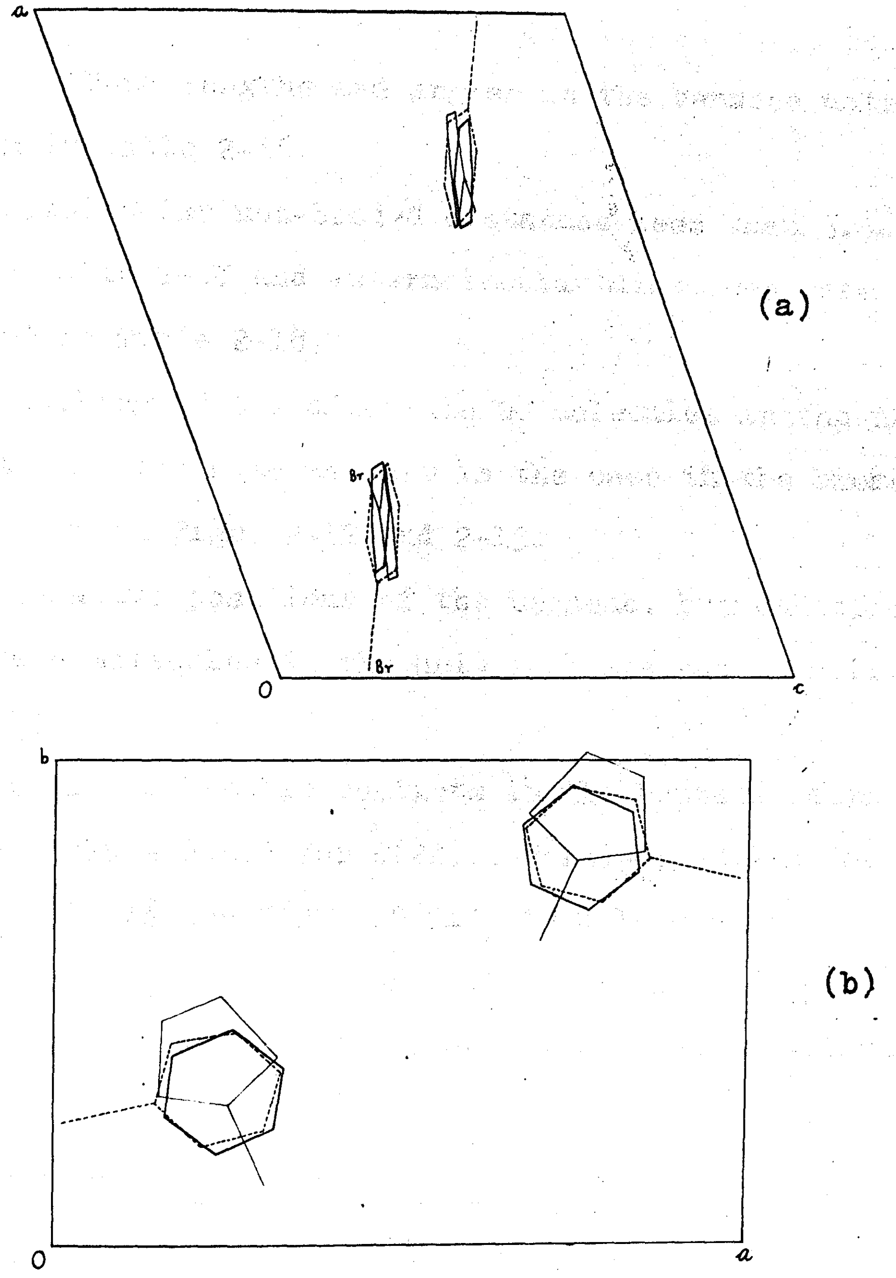


Fig.2-16. Relative positions of the benzene, bromothiophen, and bromobenzene solvent molecules in the unit cell. (a) along the b-axis, (b) along the c-axis.

Tables 2-12 and 2-13. The bond lengths and bond angles and their estimated standard deviation are listed in Tables 2-14 and 2-15. Bond lengths and angles in the benzene molecule are given in Table 2-16.

Intramolecular non-bonded distances less than 3.5Å are listed in Table 2-17 and intermolecular distances less than 4Å are listed in Table 2-18.

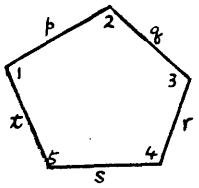
The packing of the aflatoxin G_1 molecules in the unit cell is essentially the same as that in the case in the bromobenzene solvate shown in Figs. 2-12 and 2-13.

The relative positions of the benzene, bromothiophen and bromobenzene molecules in the unit cell are shown in Fig. 2-16(a) and 2-16(b)

Close intermolecular contacts in the benzene solvate structure include 3.06Å for 0(22)...C(24)$_{XI}$; 3.18Å for C(4) ...0(21)$_{X}$; 3.24Å for C(3)...0(21)$_{X}$ in molecule 1; 3.12Å for 0(22')...C(24')$_{XII}$; 3.14Å for C(19')...0(22')$_{VI}$; 3.28Å for 0(18')...0(22')$_{VI}$; and 3.30Å for C(3')...0(21')$_{VII}$ in molecule 1'.

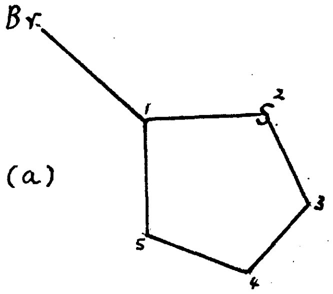
2.7. AFLATOXIN G_1- BROMOTHIOPHEN SOLVATE.

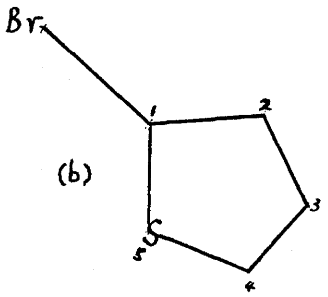
Using the coordinates of the bromine atom in the bromothiophen solvate together with the coordinates of the forty-eight atoms of the aflatoxin G_1 molecules in the bromobenzene solvate, a set of structure factors was calculated. The R factor was 0.368. The calculated phase constants were used to compute an Fo Fourier synthesis. The thiophen molecule was revealed in the electron density map. However, the peak heights at the



2 and 5 positions were comparable and higher than the other three peaks.

Also, since it was not possible to locate the peaks corresponding to the Br-S vectors in the three-dimensional Patterson function (section 2.3(D)) it was thought that there is a random rotation of the ring about the Br-C bond and the bromothiophen molecule are present as (a) and (b):





The refinement proceeded with this assumption.

With the coordinates of all the fifty-four atoms, the second set of structure factors calculated had an R factor of 0.296.

A second Fo Fourier synthesis was computed and the improved coordinates gave an R value of 0.261 for the third set of structure factors.

The third Fo Fourier and an Fc Fourier syntheses were then computed. Back-shift corrections were applied to the atomic coordinates and individual temperature factors were also adjusted.

The improved atomic parameters used in the fourth S.F. calculation gave an R factor of 0.230.

The bond length calculation at this stage showed that in the bromothiophen molecule, the bonds *q* and *r* were 1.65 and 1.77Å respectively while bonds *p* and *t* were only 1.52 and 1.48Å respectively. The first least squares cycle was accordingly calculated with the S atom placed at the position (3). The R factor went up by 5% to 0.280 and the temperature factor for the sulphur atom became very high (correspondingto B = 9.6Å^2) while the temperature factor of the carbon atom at positions *2* became almost zero. The temperature factor for the carbon atom at position *5* was reduced only slightly.

Therefore the second least squares cycle was calculated by placing the S atom at position *2*. The R factor after another least squares was 0.158. However bond length calculation showed that the bonds *p*, *q* and *s*, *t* had all lengthened while the bond *r* became shorter (they were 1.67, 1.63, 1.49, 1.65 and 1.55Å for bonds *p*, *q*, *r*, *s*, and *t* respectively). At the same time, the carbon atom at position *5* had a significantly lower temperature factor than the other four atoms while the estimated standard deviation of its coordinates were intermediate between the values for the C and S atoms. This suggested that the bromothiophen is mostly likely to be disordered.

Subsequent refinement was calculated assuming the disordered structure. Four more least squares cycles were calculated. The thermal parameters of all the fifty-four atoms were refined anisotropically in the last two cycles.

The weights, $\sqrt{w}$, applied in the least squares refinement, were calculated according to an equation suggested by Cruickshank et al. (1961) as follows:-

$$\sqrt{w} = \frac{1}{(p_1 + |F| + p_2|F|^2 + p_3|F|^3)^{\frac{1}{2}}}$$

where $p_1 = 2 \times F\ \mathrm{min} = 2.0$

$p_2 = 2/F\ \ \mathrm{max} = 0.0058$

$p_3 = 0$

The final structure factor calculation has an R factor of 0.103.

The final bond lengths in the bromothiophen molecule and the estimated standard deviation of the atomic coordinate indicated that the molecule must be disordered. The split may not be equal with 50% S and C at either of the positions 2 and 5, but this must be close to the true ratio.

The course of the analysis is summarized in Table 2-19.

The observed and calculated structure factors for the 3019 reflections are listed in Table 2-20.

The final atomic coordinates and their estimated standard deviations and the anisotropic thermal parameters are listed in Tables 2-21 and 2-22.

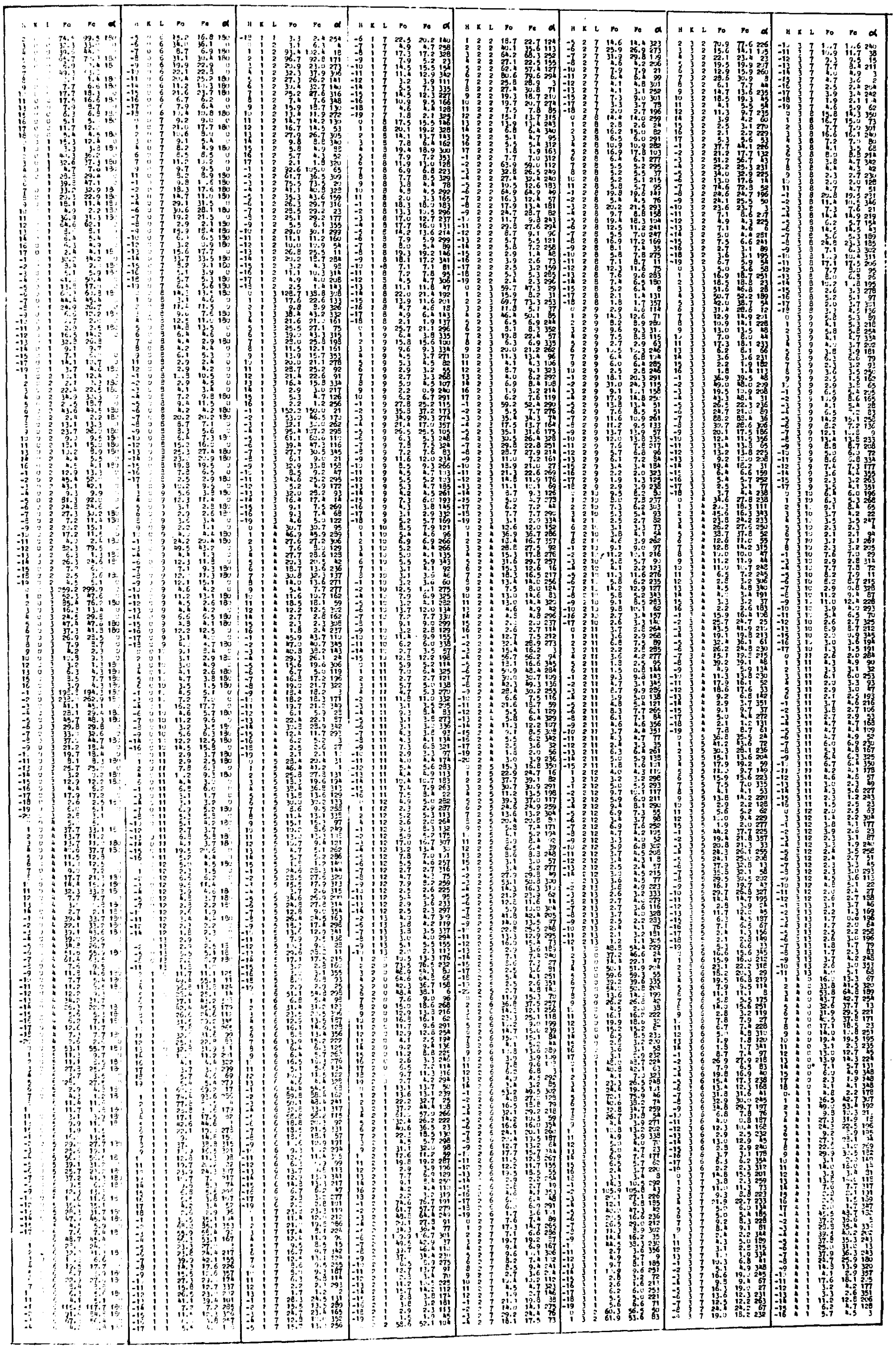
The bond lengths and bond angles with their estimated standard deviations are listed in Tables 2-23 and 2-24. Bond lengths and angles in the bromothiophen molecule are given in

Table 2-19

<u>Course of Analysis</u>: Bromothiophen Solvate

1.	1st S.F. calculation.	1 Br, 34C and 14 0 atoms Individual isotropic temperature factors	R = 0.368
2.	1st Fo Fourier synthesis		
3.	2nd S.F.	1 Br, 40 C, 14 0, 2 atoms ½(S+C)	R = 0.296
4.	2nd Fo Fourier synthesis		
5.	3rd S.F. Calculation		R = 0.261
6.	3rd Fo and 1st Fc synthesis		
7.	4th S.F. calculation		R = 0.230
8.	1st S.F.L.S.	$C_4H_3B_rS$ as 3-bromothiophen	R = 0.280
9.	2nd S.F.L.S.	$C_4H_3B_rS$ as 2-bromothiophen	R = 0.183
10.	3rd S.F.L.S.		R = 0.158
11.	4th S.F.L.S.	$C_4H_3B_rS$ disordered	R = 0.193
12.	5th S.F.L.S.	$C_4H_3B_rS$ disordered	R = 0.148
13.	6th S.F.L.S.	Thermal parameters refined anisotropically	R = 0.140
14.	7th S.F.L.S.		R = 0.113
15.	Final S.F. calculation		R = 0.103

Table 2-20 Aflatoxin G_1 bromothiophen solvate.



Measured and calculated values of structure factors. The phase angles are in degrees.

Table 2-20 (cont.)

H	K	L	Fo	Fc	α
-17	4	1	2.5	2.2	122
-18	4	1	2.[illegible]	4.2	297
0	4	2	18.7	14.9	30
1	4	2	68.5	65.7	193
2	4	2	17.0	17.3	60
3	4	2	22.2	21.7	341
4	4	2	15.3	19.9	197
5	4	2	15.8	10.6	[illegible]
6	4	2	28.8	30.4	21
7	4	2	18.6	[illegible]	308
8	4	2	18.6	20.8	182
9	4	2	12.5	12.1	33
10	4	2	11.5	15.0	11
11	4	2	4.6	4.9	168
12	4	2	6.5	4.8	50
13	4	2	9.4	7.6	7
14	4	2	4.2	4.6	192
15	4	2	2.6	1.7	[illegible]
16	4	2	2.2	2.0	58
17	4	2	3.9	4.7	66
-1	4	2	[illegible]	21.8	334
-2	4	2	33.2	27.0	160
-3	4	2	25.1	27.2	59
-4	4	2	31.0	36.3	358
-5	4	2	44.1	44.1	196
-6	4	2	16.8	17.5	211
-7	4	2	9.0	9.7	37
-8	4	2	28.8	27.6	236
-9	4	2	30.7	32.9	197
-10	4	2	16.5	14.6	32
-11	4	2	11.1	9.9	353
-12	4	2	[illegible]	12.2	289
-13	4	2	13.5	[illegible]	161
-14	4	2	[illegible]	[illegible]	230
-15	4	2	5.0	4.2	221
-16	4	2	4.1	3.8	[illegible]
-17	4	2	4.5	4.9	345
-18	4	2	[illegible]	3.4	[illegible]
-19	4	2	1.9	2.0	149
0	4	3	[illegible]	32.3	176
1	4	3	[illegible]	27.2	127
2	4	3	[illegible]	40.8	23

Table 2-21 Aflatoxin G_1 bromothiophen solvate

Final fractional atomic coordinates and their estimated standard deviations in Å

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	0.4109	0.5665	0.0199	0.011	0.016	0.013
O(2)	0.4883	0.5018	0.0679	0.009	0.013	0.011
C(3)	0.4847	0.3839	0.1302	0.013	0.016	0.014
C(4)	0.4110	0.2983	0.0431	0.011	0.014	0.012
C(5)	0.3229	0.3675	0.0051	0.010	0.015	0.012
C(6)	0.3268	0.5017	0.0107	0.010	0.016	0.012
C(7)	0.2497	0.5766	-0.0036	0.012	0.014	0.015
O(8)	0.1687	0.5153	-0.0312	0.007	0.009	0.008
C(9)	0.1630	0.3827	-0.0438	0.012	0.015	0.012
C(10)	0.2398	0.3089	-0.0276	0.010	0.013	0.011
C(11)	0.2230	0.1724	-0.0473	0.010	0.013	0.010
C(12)	0.1389	0.1222	-0.0728	0.010	0.016	0.011
C(13)	0.0701	0.2033	-0.0832	0.012	0.015	0.012
C(14)	0.0785	0.3358	-0.0690	0.010	0.014	0.012
C(15)	-0.0097	0.3928	-0.0962	0.012	0.016	0.016
C(16)	-0.0714	0.2752	-0.1176	0.014	0.019	0.017
O(17)	-0.0148	0.1651	-0.1072	0.008	0.011	0.010
O(18)	-0.1322	0.2878	-0.2423	0.013	0.017	0.017
C(19)	-0.1091	0.3880	-0.3066	0.022	0.026	0.024
C(20)	-0.0450	0.4592	-0.2220	0.013	0.021	0.019
O(21)	0.4142	0.6751	-0.0136	0.009	0.012	0.012
O(22)	0.2447	0.6876	0.0063	0.010	0.011	0.011
O(23)	0.2958	0.1028	-0.0413	0.008	0.010	0.009
C(24)	0.2866	-0.0357	-0.0459	0.014	0.015	0.015
C(25)	0.2471	0.2893	0.3049	0.015	0.019	0.015
S/C(26)	0.3174	0.4147	0.3498	0.007	0.008	0.007
C(27)	0.2326	0.5168	0.3256	0.022	0.025	0.021
C(28)	0.1507	0.4652	0.2943	0.023	0.029	0.023
S/C(29)	0.1454	0.3054	0.2757	0.010	0.013	0.010
Br	0.2978	0.1250	0.3068	0.002	0	0.002

Table 2-21 (cont.)

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1')	0.1120	0.0372	0.6167	0.013	0.017	0.014
O(2')	0.0353	0.0968	0.5824	0.009	0.012	0.010
C(3')	0.0343	0.2184	0.5207	0.016	0.020	0.019
C(4')	0.1099	0.2989	0.6030	0.012	0.016	0.012
C(5')	0.1974	0.2316	0.6279	0.011	0.014	0.011
C(6')	0.1924	0.1032	0.6199	0.011	0.016	0.011
C(7')	0.2715	0.0251	0.6258	0.012	0.015	0.013
O(8')	0.3488	0.0897	0.6429	0.008	0.010	0.009
C(9')	0.3572	0.2187	0.6640	0.012	0.014	0.011
C(10')	0.2805	0.2936	0.6558	0.011	0.014	0.011
C(11')	0.2961	0.4282	0.6805	0.013	0.017	0.014
C(12')	0.3809	0.4797	0.7047	0.013	0.017	0.015
C(13')	0.4504	0.3955	0.7016	0.012	0.016	0.013
C(14')	0.4391	0.2688	0.6819	0.012	0.017	0.013
C(15')	0.5263	0.2155	0.6781	0.013	0.017	0.015
C(16')	0.5882	0.3274	0.7225	0.018	0.023	0.018
O(17')	0.5345	0.4357	0.7223	0.009	0.012	0.011
O(18')	0.6325	0.3472	0.6317	0.012	0.017	0.015
C(19')	0.5899	0.2693	0.5238	0.016	0.024	0.020
C(20')	0.5312	0.1931	0.5428	0.013	0.018	0.015
O(21')	0.1107	-0.0755	0.6566	0.010	0.013	0.011
O(22')	0.2732	-0.0863	0.6126	0.011	0.013	0.012
O(23')	0.2256	0.4965	0.6855	0.011	0.012	0.011
C(24')	0.2308	0.6342	0.6728	0.018	0.020	0.016

Table 2-22 Aflatoxin G_1 bromothiophen solvate

Anisotropic temperature factors

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	0.036	0.060	0.062	0.007	0.044	-0.006
O(2)	0.042	0.065	0.090	-0.018	0.048	-0.012
C(3)	0.050	0.044	0.062	-0.007	0.036	0.002
C(4)	0.040	0.045	0.058	-0.010	0.036	0.010
C(5)	0.038	0.042	0.054	0	0.039	0.009
C(6)	0.034	0.051	0.052	-0.003	0.034	-0.008
C(7)	0.046	0.025	0.081	0.004	0.053	-0.004
O(8)	0.035	0.038	0.066	0.002	0.046	0.007
C(9)	0.035	0.051	0.057	-0.005	0.033	-0.001
C(10)	0.043	0.030	0.055	0.001	0.048	0.002
C(11)	0.042	0.038	0.047	0.011	0.027	-0.005
C(12)	0.044	0.040	0.053	0.009	0.034	-0.010
C(13)	0.044	0.054	0.049	0.003	0.041	-0.021
C(14)	0.035	0.040	0.061	0.009	0.044	0.015
C(15)	0.038	0.058	0.094	0.009	0.054	-0.006
C(16)	0.049	0.052	0.078	0.006	0.040	-0.008
O(17)	0.045	0.050	0.075	0.011	0.046	-0.017
O(18)	0.075	0.083	0.128	0.032	-0.022	-0.028
C(19)	0.089	0.064	0.131	0.036	0.033	0.032
C(20)	0.044	0.084	0.110	0.058	0.038	0.010
O(21)	0.049	0.058	0.099	0.006	0.053	-0.009
O(22)	0.057	0.048	0.090	0.008	0.058	-0.005
O(23)	0.040	0.045	0.073	0.006	0.036	0.014
C(24)	0.061	0.039	0.075	0.012	0.050	0.026
C(25)	0.083	0.062	0.058	0.006	0.055	0.027
S/C(26)	0.081	0.071	0.068	0.011	0.067	0.003
C(27)	0.114	0.087	0.092	0.031	0.075	0.028
C(28)	0.119	0.100	0.105	0.070	0.092	0.046
S/C(29)	0.120	0.129	0.076	0.022	0.042	0.037
Br	0.091	0.084	0.077	-0.002	0.039	0.031

Table 2-22 (cont.)

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1')	0.051	0.056	0.059	-0.007	0.025	-0.013
O(2')	0.052	0.065	0.081	-0.004	0.046	-0.014
C(3')	0.055	0.065	0.089	0.003	0.026	0.007
C(4')	0.041	0.067	0.050	-0.024	0.015	-0.004
C(5')	0.050	0.041	0.043	-0.007	0.037	-0.002
C(6')	0.051	0.052	0.050	-0.001	0.042	-0.004
C(7')	0.057	0.037	0.061	-0.007	0.050	0.009
O(8')	0.050	0.045	0.062	0.004	0.050	0
C(9')	0.052	0.037	0.047	-0.008	0.045	-0.010
C(10')	0.056	0.037	0.043	-0.004	0.043	-0.017
C(11')	0.059	0.051	0.060	-0.001	0.060	0
C(12')	0.055	0.045	0.074	-0.012	0.059	-0.022
C(13')	0.054	0.054	0.060	0.006	0.050	-0.026
C(14')	0.049	0.052	0.064	-0.008	0.048	-0.008
C(15')	0.044	0.055	0.074	-0.005	0.027	-0.010
C(16')	0.073	0.079	0.076	-0.034	0.037	-0.009
O(17')	0.053	0.065	0.081	-0.019	0.047	-0.031
O(18')	0.073	0.099	0.137	-0.017	0.105	-0.046
C(19')	0.054	0.103	0.099	-0.021	0.058	-0.001
C(20')	0.046	0.079	0.072	-0.009	0.056	0.010
O(21')	0.063	0.070	0.076	-0.008	0.049	-0.041
O(22')	0.075	0.048	0.103	-0.024	0.086	-0.002
O(23')	0.069	0.054	0.075	-0.001	0.064	0.002
C(24')	0.091	0.043	0.080	-0.017	0.050	0.035

Table 2-23 Aflatoxin G_1 bromothiophen solvate

Bond lengths and their estimated standard deviations

	Molecule 1		Molecule 1'		Average
C(1)-O(2)	1.352	±0.016	1.309	±0.017	1.330
C(1)-C(6)	1.464	0.016	1.434	0.018	1.449
C(1)-O(21)	1.206	0.020	1.266	0.021	1.236
O(2)-C(3)	1.424	0.020	1.444	0.023	1.438
C(3)-C(4)	1.542	0.018	1.509	0.021	1.526
C(4)-C(5)	1.507	0.016	1.496	0.017	1.502
C(5)-C(6)	1.415	0.021	1.355	0.021	1.385
C(5)-C(10)	1.388	0.015	1.408	0.016	1.398
C(6)-C(7)	1.415	0.017	1.476	0.018	1.446
C(7)-O(8)	1.376	0.014	1.356	0.015	1.366
C(7)-O(22)	1.180	0.018	1.183	0.020	1.182
O(8)-C(9)	1.403	0.017	1.377	0.017	1.390
C(9)-C(10)	1.401	0.016	1.422	0.017	1.411
C(9)-C(14)	1.364	0.015	1.349	0.017	1.356
C(10)-C(11)	1.466	0.018	1.449	0.021	1.458
C(11)-C(12)	1.371	0.015	1.387	0.019	1.379
C(12)-C(13)	1.356	0.018	1.416	0.020	1.386
C(13)-C(14)	1.405	0.021	1.354	0.023	1.380
C(13)-O(17)	1.341	0.014	1.341	0.016	1.341
C(14)-C(15)	1.456	0.016	1.495	0.018	1.476
C(15)-C(16)	1.546	0.023	1.510	0.026	1.528
C(15)-C(20)	1.484	0.023	1.515	0.020	1.500
C(16)-O(17)	1.445	0.020	1.419	0.024	1.432
C(16)-O(18)	1.405	0.021	1.389	0.022	1.397
O(18)-C(19)	1.376	0.030	1.420	0.025	1.398
C(19)-C(20)	1.361	0.028	1.287	0.025	1.324
C(11)-O(23)	1.344	0.014	1.338	0.017	1.341
O(23)-C(24)	1.465	0.018	1.462	0.023	1.463

Table 2-24 Aflatoxin G$_1$ bromothiophen solvate

Bond angles and their estimated standard deviations

	Molecule 1	Molecule 1'	Average
O(2)C(1)O(21)	118.5±1.1°	115.9±1.2°	117.2°
C(6)C(1)O(21)	122.9 1.1	123.6 1.2	123.3
C(6)C(1)O(2)	118.6 1.3	120.2 1.4	119.4
C(1)O(2)C(3)	117.4 1.0	115.8 1.1	116.6
O(2)C(3)C(4)	110.5 1.0	109.2 1.3	109.8
C(3)C(4)C(5)	109.9 1.1	110.7 1.3	110.3
C(4)C(5)C(6)	116.5 1.0	115.4 1.1	116.0
C(6)C(5)C(10)	118.7 1.0	120.5 1.1	119.6
C(4)C(5)C(10)	124.7 1.3	124.0 1.3	124.4
C(5)C(6)C(7)	121.8 1.0	121.5 1.1	121.6
C(1)C(6)C(5)	119.7 1.1	121.2 1.2	120.5
C(1)C(6)C(7)	118.3 1.3	117.1 1.4	117.7
C(6)C(7)O(22)	128.4 1.2	126.7 1.2	127.6
O(8)C(7)O(22)	113.7 1.1	117.5 1.1	115.6
C(6)C(7)O(8)	117.9 1.2	115.7 1.2	116.8
C(7)O(8)C(9)	121.0 0.9	123.7 1.0	122.3
O(8)C(9)C(10)	120.8 1.0	120.0 1.0	120.4
O(8)C(9)C(14)	114.1 1.1	116.6 1.1	115.4
C(10)C(9)C(14)	125.0 1.3	123.1 1.3	124.1
C(9)C(10)C(5)	119.6 1.2	118.0 1.2	118.8
C(9)C(10)C(11)	114.6 1.0	116.0 1.1	115.3
C(5)C(10)C(11)	125.9 1.0	126.0 1.1	125.9
C(10)C(11)C(12)	121.8 1.1	121.1 1.3	121.4
C(10)C(11)O(23)	114.5 0.9	115.9 1.1	115.2
C(12)C(11)O(23)	123.7 1.2	122.9 1.5	123.3
C(11)C(12)C(13)	118.0 1.4	117.0 1.4	117.5
C(12)C(13)C(14)	124.8 1.1	123.8 1.2	124.3
C(12)C(13)O(17)	123.2 1.3	121.9 1.4	122.5
C(14)C(13)O(17)	112.0 1.1	114.3 1.2	113.2
C(13)C(14)C(9)	115.8 1.1	118.7 1.2	117.2
C(15)C(14)C(9)	134.4 1.4	133.8 1.5	134.1
C(13)C(14)C(15)	109.6 1.0	107.3 1.1	108.5
C(14)C(15)C(16)	102.5 1.2	101.8 1.3	102.1
C(14)C(15)C(20)	116.4 1.2	114.0 1.1	115.2
C(16)C(15)C(20)	101.5 1.2	102.2 1.2	101.8
C(15)C(16)O(17)	106.7 1.0	107.6 1.3	107.2
C(15)C(16)O(18)	106.2 1.4	107.7 1.5	107.0
O(17)C(16)O(18)	111.8 1.4	107.9 1.6	109.9
C(13)O(17)C(16)	109.1 1.1	107.7 1.3	108.4
C(16)O(18)C(19)	110.8 1.5	106.8 1.4	108.8
O(18)C(19)C(20)	109.6 1.9	113.9 1.6	111.8
C(19)C(20)C(15)	110.7 1.8	108.3 1.4	109.5
C(11)O(23)C(24)	117.8 0.9	117.3 1.2	117.6

Table 2-25

Bond lengths and angles of the bromothiophen molecule and their estimated standard deviations

C(25)-S/C(26)	1.695	±0.019	C(28)-S/C(29)	1.694	±0.019
C(27)-S/C(26)	1.670	0.024	C(25)-S/C(29)	1.541	0.017
C(27)-C(28)	1.341	0.032	C(25)-Br	1.903	0.018

S/C(26)C(25)Br	118.1	±0.8°	S/C(26)C(27)C(28)	116.0	±2.1°
S/C(29)C(25)Br	120.5	1.1	S/C(29)C(28)C(27)	116.1	2.0
S/C(29)C(25)S/C(26)	121.2	1.2	C(28)S/C(29)C(25)	94.3	1.2
C(25)S/C(26)C(27)	92.1	1.0			

Intramolecular non-bonded distances of the bromothiophen molecule

C(25)...C(27)	2.42	C(27)...S/C(29)	2.58
C(25)...C(28)	2.37	Br ... S/C(29)	2.99
S/C(26)...C(28)	2.56	Br ... S/C(26)	3.09
S/C(26)...S/C(29)	2.82		

Bond lengths and angles in
aflatoxin G_1 bromothiophen solvate.

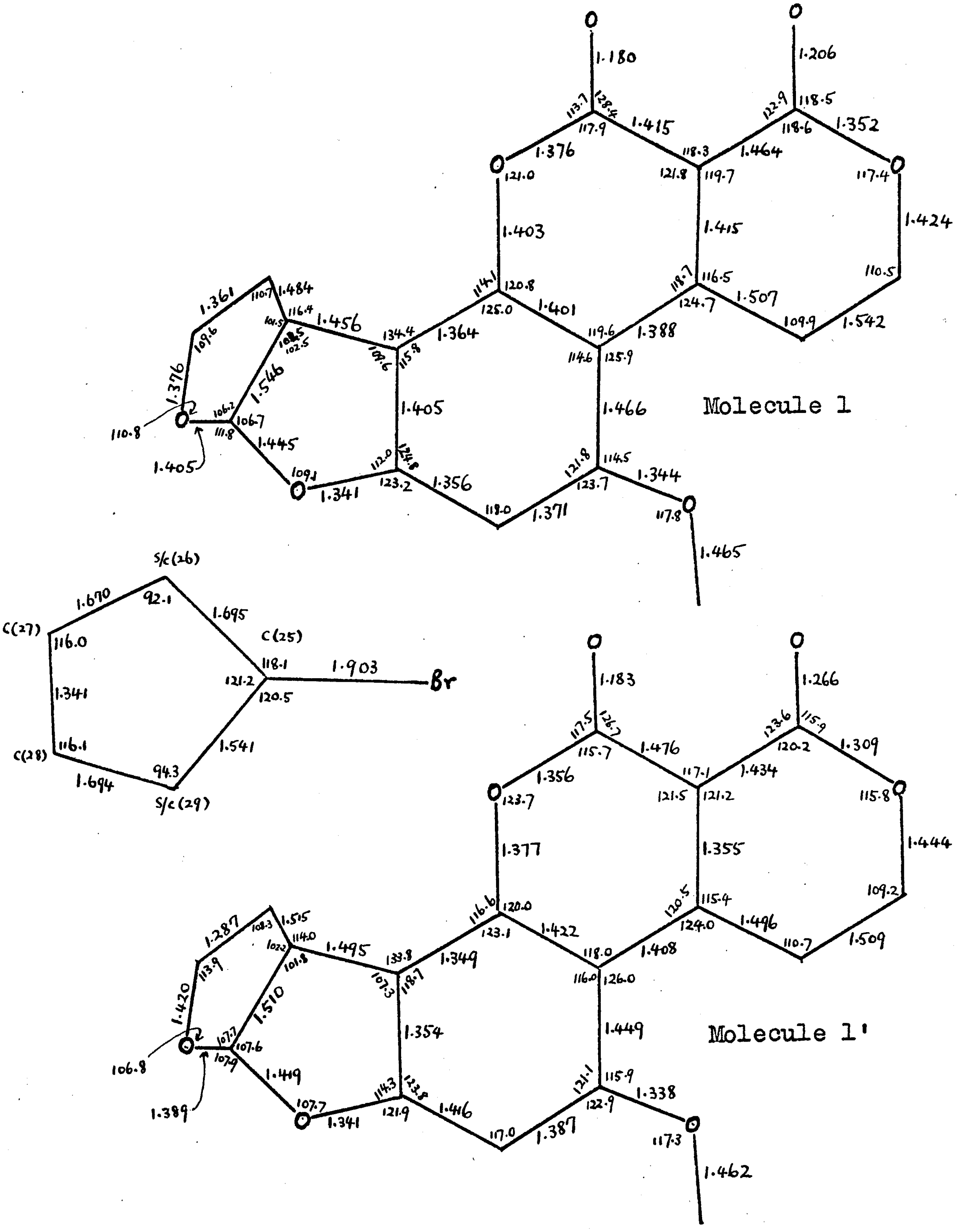


Table 2-26 Aflatoxin G_1 bromothiophen solvate

Intramolecular non-bonded distances ≤3.5A

	Mol. 1	Mol. 1'		Mol. 1	Mol. 1'
C(1)...C(3)	2.37	2.33	C(9) ...C(11)	2.41	2.43
C(1)...C(7)	2.47	2.48	C(9) ...C(15)	2.60	2.62
C(1)...C(5)	2.49	2.43	C(9) ...C(12)	2.77	2.79
C(1)...C(4)	2.84	2.76	C(9) ...C(20)	3.35	3.40
C(1)...O(22)	2.87	2.86	C(9) ...O(22)	3.44	3.45
O(2)...O(21)	2.20	2.18	C(10)...O(23)	2.36	2.36
O(2)...C(6)	2.42	2.39	C(10)...C(14)	2.45	2.44
O(2)...C(4)	2.44	2.41	C(10)...C(12)	2.48	2.47
C(3)...C(5)	2.50	2.47	C(10)...C(13)	2.78	2.78
C(3)...C(6)	2.72	2.68	C(11)...C(13)	2.34	2.39
C(3)...O(21)	3.47	3.48	C(11)...C(24)	2.41	2.39
C(4)...C(6)	2.49	2.41	C(11)...C(14)	2.80	2.80
C(4)...C(10)	2.56	2.57	C(12)...O(17)	2.37	2.41
C(4)...O(23)	2.71	2.73	C(12)...O(23)	2.39	2.39
C(4)...C(11)	3.11	3.10	C(12)...C(14)	2.45	2.44
C(5)...C(9)	2.41	2.43	C(12)...C(24)	2.80	2.80
C(5)...C(7)	2.47	2.47	C(13)...C(16)	2.27	2.23
C(5)...C(11)	2.54	2.55	C(13)...C(15)	2.34	2.30
C(5)...O(8)	2.80	2.77	C(13)...O(18)	3.24	3.22
C(5)...O(23)	2.84	2.86	C(13)...C(20)	3.34	3.24
C(6)...O(22)	2.34	2.38	C(14)...O(17)	2.28	2.26
C(6)...O(21)	2.35	2.38	C(14)...C(16)	2.34	2.33
C(6)...O(8)	2.39	2.40	C(14)...C(20)	2.50	2.53
C(6)...C(10)	2.41	2.40	C(14)...O(18)	3.31	3.36
C(6)...C(9)	2.76	2.77	C(14)...C(19)	3.32	3.33
C(7)...C(9)	2.42	2.41	C(15)...C(19)	2.34	2.28
C(7)...O(21)	2.82	2.86	C(15)...O(18)	2.36	2.34
C(7)...C(10)	2.83	2.85	C(15)...O(17)	2.40	2.36
O(8)...O(22)	2.14	2.17	C(16)...C(19)	2.29	2.26
O(8)...C(14)	2.32	2.32	C(16)...C(20)	2.35	2.35
O(8)...C(10)	2.44	2.42	O(17)...O(18)	2.36	2.27
O(8)...C(15)	2.97	3.01	O(17)...C(19)	3.23	3.11
O(8)...C(20)	3.41	3.54	O(17)...C(20)	3.32	3.21
C(9)...C(13)	2.35	2.33	O(18)...C(20)	2.24	2.27

Table 2-27 Aflatoxin G_1 bromothiophen solvate

Intermolecular distances ≤ 4Å

O(22)...C(24)XI	3.08
C(4) ...O(21)X	3.15
C(3) ...O(21)X	3.20
C(16)...O(22)IV	3.43
C(15)...O(17)II	3.57
O(8) ...O(17)II	3.59
O(21)...C(24)XI	3.60
O(2) ...C(24)IX	3.64
O(2) ...O(23)IX	3.65
O(18)...O(22)IV	3.69
O(8) ...C(16)II	3.74
O(2) ...C(4)IX	3.86
O(2) ...O(21)X	3.89
C(1) ...C(4)IX	3.93
C(12)...C(15)IV	3.96

O(22')...C(24')XII	3.13
C(3') ...O(21')VII	3.30
C(19')...O(22')VI	3.34
O(18')...O(22')VI	3.49
O(21')...C(24')XII	3.57
O(17')...C(20')VI	3.86
C(4') ...O(21')VII	3.97

C(28)...O(17)II	3.23
C(27)...O(18)II	3.25
C(28)...O(18)II	3.44
S/C(29)...C(14)	3.58
C(25)...C(10)	3.59
S/C(29)...C(9)	3.66
C(28)...O(8)	3.68
C(27)...O(8)	3.70
Br . . .C(11)	3.70
C(27)...C(7)	3.73
C(25)...C(9)	3.74
Br . . .O(23)	3.78
C(28)...C(16)II	3.80
S/C(26)...C(5)	3.81
C(28)...C(9)	3.84
S/C(26)...C(6)	3.85
C(27)...O(17)II	3.85
S/C(29)...C(13)	3.87
Br . . .C(19)IV	3.88
C(25)...C(5)	3.89
C(25)...C(11)	3.93
C(27)...C(16)II	3.93
S/C(29)...C(20)IV	3.94
C(27)...O(22)	3.97
Br . . .C(10)	3.97

Br . . .O(18')V	3.12
Br . . .O(17')V	3.40
Br . . .O(8')	3.51
Br . . .C(16')V	3.67
C(25)...C(10')	3.69
S/C(26)...C(11')	3.72
S/C(29)...C(3')	3.72
S/C(26)...C(20')VI	3.73
S/C(26)...C(12')	3.74
S/C(29)...C(5')	3.74
S/C(26)...C(13')	3.76
S/C(26)...C(10')	3.77
S/C(29)...C(4')	3.77
Br . . .C(7')	3.77
C(27)...C(11')	3.79
C(25)...C(9')	3.83
C(28)...O(2')VII	3.83
Br . . .C(9')	3.84
Br . . .C(20')	3.84
C(27)...C(19')VI	3.84
S/C(26)...C(14')	3.85
C(25)...C(5')	3.88
S/C(26)...C(9')	3.88
C(27)...O(23')	3.96
C(27)...C(24')	3.98

C(7) ...O(23')I	3.39
C(12)...C(1')I	3.39
C(6) ...O(23')I	3.40
C(1) ...C(12')I	3.44
O(8) ...O(23')I	3.47
C(5) ...C(11')I	3.48
C(7) ...C(24')I	3.49
C(11)...C(5')I	3.49
C(1) ...C(15')VI	3.50
O(21)...C(15')VI	3.50
O(2) ...O(8')VI	3.51
C(12)...O(21')I	3.52

Table 2-27 (cont.)

C(19)...O(22')II	3.54
O(23)...C(6')I	3.54
C(6) ...C(11')I	3.56
O(21)...C(16')VI	3.56
C(24)...C(7')I	3.56
C(11)...C(6')I	3.58
O(18)...O(18')III	3.58
C(24)...O(21')I	3.58
C(9) ...O(23')I	3.59
O(21)...C(12')I	3.59
C(5) ...O(23')I	3.61
C(12)...O(2')I	3.61
O(22)...C(16')VI	3.61
O(22)...C(24')I	3.61
O(23)...C(7')I	3.62
O(2) ...C(15')VI	3.63
C(10)...O(23')I	3.64
C(13)...O(2')I	3.68
C(6) ...C(12')I	3.69
C(10)...C(5')I	3.69
C(24)...O(22')I	3.69
C(3) ...O(8')VI	3.70
C(10)...C(10')I	3.70
C(12)...C(6')I	3.70
C(19)...C(1')II	3.71
O(23)...C(5')I	3.71
C(5) ...C(10')I	3.73
O(21)...C(24')I	3.75
C(24)...C(16')V	3.75
C(10)...C(11')I	3.77
C(14)...C(4')I	3.77
O(17)...O(2')I	3.77
O(21)...C(14')VI	3.77
C(24)...C(6')I	3.77
C(6) ...C(24')I	3.78
C(9) ...C(4')I	3.78
O(23)...O(8')I	3.78
C(24)...O(17')V	3.78
C(13)...C(4')I	3.79
O(2) ...C(12')I	3.80
C(19)...C(3')I	3.80
O(23)...C(10')I	3.80
C(11)...C(1')I	3.81
C(12)...C(5')I	3.82
C(19)...O(21')II	3.82
O(23)...C(9')I	3.82
O(2) ...C(14')VI	3.83
C(11)...C(10')I	3.83
C(5) ...C(12')I	3.84
O(8) ...C(24')I	3.85
C(1) ...C(11')I	3.86
C(12)...C(4')I	3.89
C(11)...C(4')I	3.90
C(19)...C(7')II	3.90
O(2) ...C(9')VI	3.91
C(10)...C(4')I	3.91
C(20)...C(4')I	3.91
C(1) ...C(16')VI	3.92
C(24)...C(1')I	3.92
C(19)...O(18')III	3.93
C(13)...C(1')I	3.94
O(22)...O(23')I	3.96
C(19)...C(6')II	3.96
C(1) ...O(23')I	3.97
C(3) ...O(22')VI	3.98
C(19)...C(4')I	3.98
C(1) ...C(14')VI	3.99

The Roman numerals refer to the following equivalent positions:

I: $x, y, -1+z$
II: $-x, \frac{1}{2}+y, -z$
III: $-1+x, y, -1+z$
IV: $-x, -\frac{1}{2}+y, -z$
V: $1-x, -\frac{1}{2}+y, 1-z$
VI: $1-x, \frac{1}{2}+y, 1-z$
VII: $-x, \frac{1}{2}+y, 1-z$
IX: $1-x, \frac{1}{2}+y, -z$
X: $1-x, -\frac{1}{2}+y, -z$
XI: $x, 1+y, z$
XII: $x, -1+y, z$

Table 2-25.

Intramolecular non-bonded distances less than 3.5Å are listed in Table 2-26 and intermolecular distances less than 4Å are given in Table 2-27.

The packing of the molecules in the unit cell is essentially the same as in the case of the bromobenzene solvate shown in Figs. 2-12 and 2-13, and the relative positions of the benzene, bromobenzene and bromothiophen molecules are shown in Fig. 2-16 (a) and (b).

Close intermolecular contacts in the bromothiophen solvate structure include 3.08Å for O(22)...$C(24)_{XI}$; 3.15Å for C(4) ...$O(21)_{X}$; 3.20Å for C(3)...$O(21)_{X}$ in molecule 1; and 3.13Å for O(22')...$C(24')_{XII}$; 3.30Å for C(3')...$O(21')_{VII}$; 3.34Å for C(19')...$O(22')_{VI}$ in molecule 1'.

A very close intermolecular contact involving the Br atom is of value 3.12Å for Br...$O(18')_{V}$; other close contact involving the bromothiophen molecule are 3.23Å for C(28)...$O(17)_{II}$; and 3.25Å for C(27)...$O(18)_{II}$.

2.8. DISCUSSION OF THE RESULTS.

All three isomorphous solvates of aflatoxin G_1 have been refined to the same extent. No attempts have been made to locate the hydrogen atoms. As there are two molecules in an asymmetric unit, the results of this structural analysis yield a total of six measures of the molecular dimensions. Average bond lengths and angles and their estimated standard deviations over the six independent values have been calculated using the equations

$$U\text{av.} = \sum_{i=1}^{6} \frac{U_i}{\sigma_i^2} \bigg/ \sum_{i=1}^{6} \frac{1}{\sigma_i^2} \quad \text{, and} \quad \sigma\text{av.} = \left(\sum_{i=1}^{6} \frac{1}{\sigma_i^2}\right)^{-\frac{1}{2}},$$

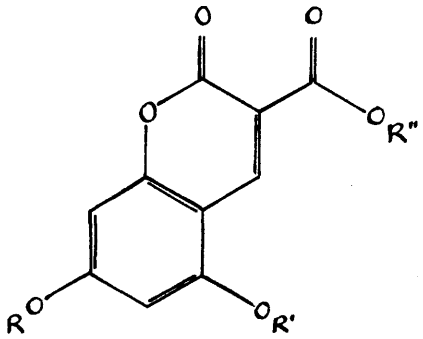
where U_i are the individual observations and σ_i their estimated standard deviations.

The estimated standard deviations thus calculated vary from 0.005Å to 0.008Å while those for the angles vary from 0.4° to 0.5°. These are given with the bond lengths and angles in Table 2-28. They are also shown in Fig. 2-15(a).

Only five bond lengths out of the six sets of twenty-eight bond-lengths have values which when compared with the average values, have differences greater than three times their own estimated standard deviations. They are: (i) 1.502Å for C(1) - C(6) in molecule "1" of the benzene solvate, (ii) 1.350Å for C(9) - C(10); (iii) 1.534Å for C(10) - C(11); (iv) 1.333Å for C(11) - C(12); and (v) 1.451Å for C(16) - O(18) all in molecule "1" of the bromobenzene solvate. Only eight bond angles out of the six sets of forty-three bond angles have values which when compared with the average values, have differences greater than three times their own estimated standard deviations. They are: (i) 120.4° for O(2) - C(1) - O(21); (ii) 114.9° for C(6) - C(1) - O(2); (iii) 118.9° for C(1) - O(2) - C(3) in molecule "1" of the benzene solvate; (iv) 121.6° for C(6) - C(1) - O(2); and (v) 130.4° for C(15) - C(14) - C(9) in molecule "1" of the benzene solvate;

(vi) 128.8° for C(12) - C(11) - O(23); (vii) 121.0° for C(11) - C(12) - C(13); and (viii) 106.0° for O(17) - C(16) - O(18) in molecule "1" of the bromobenzene solvates.

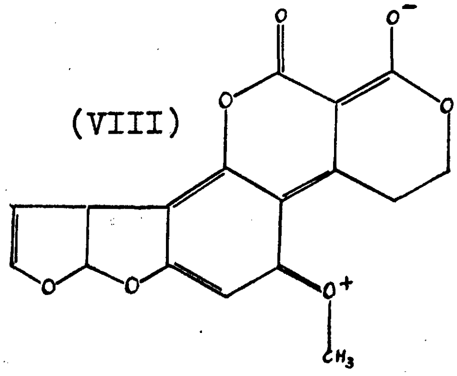
Examination of the bond lengths shows that they are influenced by resonance and the π-electron delocalised system extends over the grouping, (VII),



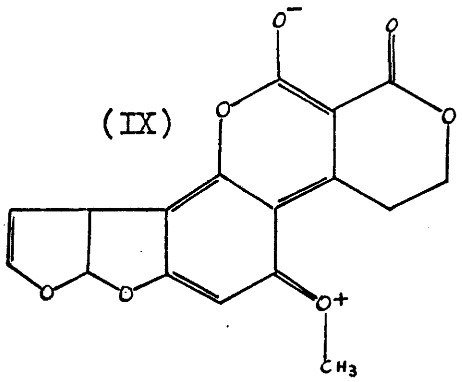
(VII)

and that contributions from the resonance structures such as (VIII) to (XI) are important.

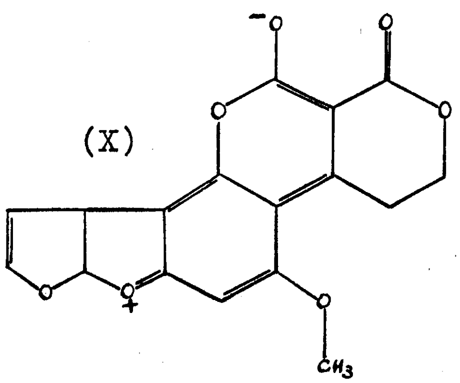
(VIII)



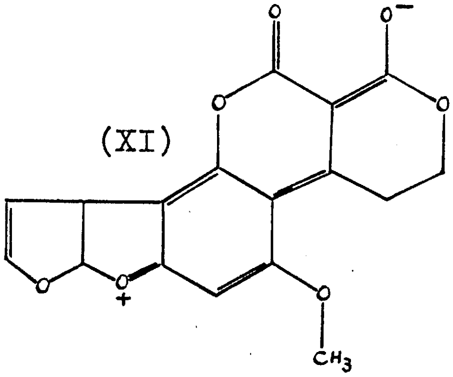
(IX)



(X)

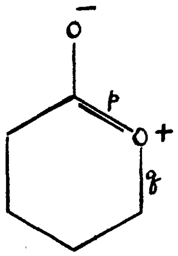


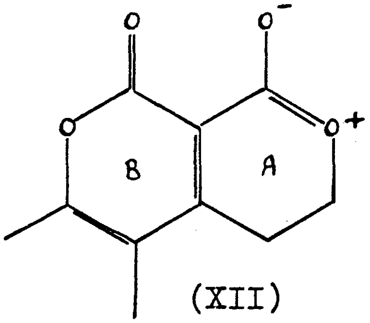
(XI)



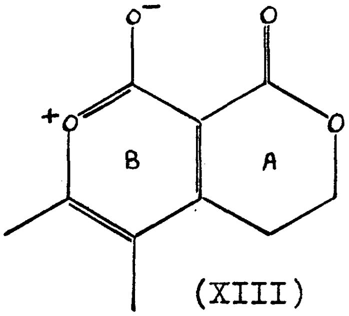
Thus the value of 1.362Å for O(17 - C(13) bond is significantly shorter than 1.434Å for O(17) - C(16); and the value of 1.344Å for the C(11) - O(23) bond indicates that, there is some double bond character.

The resonance of the δ-lactone systems giving rise to structures (XII) and (XIII) may also contribute to the bonding with





(XII)



(XIII)

a short C - O bond p and a long C - O bond q. This type of resonance is common in both γ- and δ-lactones as well as in esters. This effect seems to be present in ring A because the average bond length for C(1) - O(2) or 1.329Å is significantly shorter than 1.455Å for O(2) - C(3). However, in ring B, the value of 1.373Å for C(7) - O(8) does not differ significantly from the value of 1.387Å for O(8) - C(9).

The bond lengths and angles are best compared with the results obtained in the crystal structure analysis of the dihydro-aflatoxin B_1 carried out by van Soest and Peerdeman (1964). A set of bond lengths and angles have been kindly supplied by Professor Peerdeman and they are shown in Fig. 2-

15(b). Their results were obtained by least squares refinement (including anisotropic temperature factors) of 1267 reflections with $\sin\theta/\lambda > 0.4$; hydrogen atoms were not yet included. The R factor was 0.087 and the standard deviation in the coordinates were about 0.005Å. The estimated standard deviations of the bond lengths are therefore about 0.007Å. The comparison of the two sets of results shows that they agree extremely well in most cases. There are three bond lengths where the differences between the two results are just over the significant test limits. They are the bonds C(10) - C(11), O(23) - C(24) and C(16) - O(17).

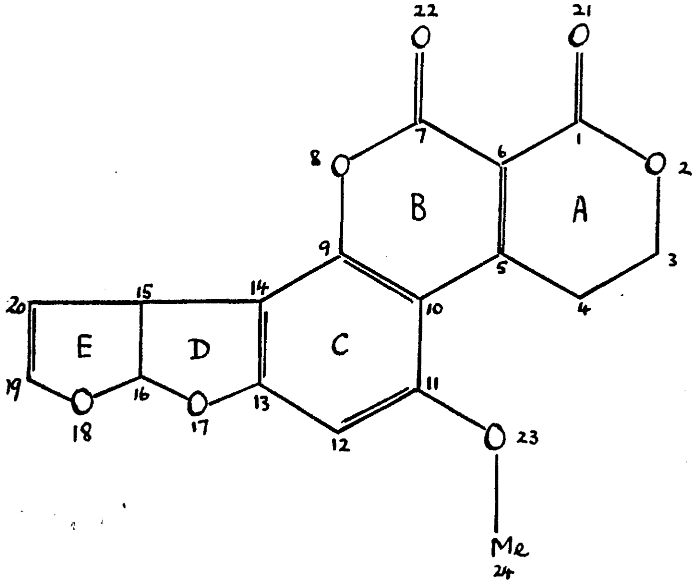
The average value of 1.344Å for C(11) - O(23) also compares very well with the values of 1.361Å in salicylic acid (Cochran, 1953) and 1.36Å in phloroglucinol (Hassel and Viervoll, 1947) where resonance also occur.

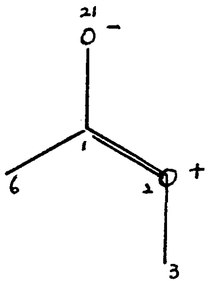
The average values of 1.288Å and 1.202Å for the sp^2-carbon-oxygen double bond also compares very satisfactorily with an averaged value of 1.212Å inparabamic acid (Davies and Blum, 1955) and 1.222Å in p-benzoquinone (Trotter, 1960).

The six bond lengths in the benzene molecule (Table 2-16) do not differ significantly from one another. The average value is 1.382Å compared with 1.397Å in benzene (Stoicheff, 1954). The angles range from 118.5° to 123.3°, the difference is within experimental errors.

The six C - C bond lengths in the bromobenzene molecule do not differ significantly from one another. The average value of 1.361Å is shorter than the value of 1.397Å in benzene. The C-Br distance of 1.946Å is longer than the value of 1.86Å in bromobenzene (Yuzawa and Yamaha, 1953). Value of 1.94Å has been reported in p-bromobenzoate-glaucarubin (Kartha and Haas, 1964); 1.931Å in 4-bromoestradiol (Norton et al. 1964); and 1.91Å in 5-bromogriseofulvin (Brown and Sim, 1963). In the disordered bromothiophen molecule, the C-Br distance is 1.903Å.

Mean plane calculations (Schomaker et al., 1959) through various parts of the molecule showed that the molecule





containing rings B, C, and D are roughly planar. Ring E is planar and is cis-fused to ring D, the dihedral angle is about 63°. A mean plane calculation through the atoms C(6), O(21), C(1), O(2) and C(3) showed that these atoms are not planar because O(2) and C(3) deviate significantly from the plane. This is unexpected as the hybridization of the δ-lactone would impart planarity.

The results of the mean plane calculations are summarized in Table 2-29. It is seen that some of the atoms, notably C(5), C(15), O(22) and O(23), in mean plane (II) through the fifteen atoms also deviates significantly from the plane. However, much better planes can be fitted to individual members of each of the rings B, C, and D.

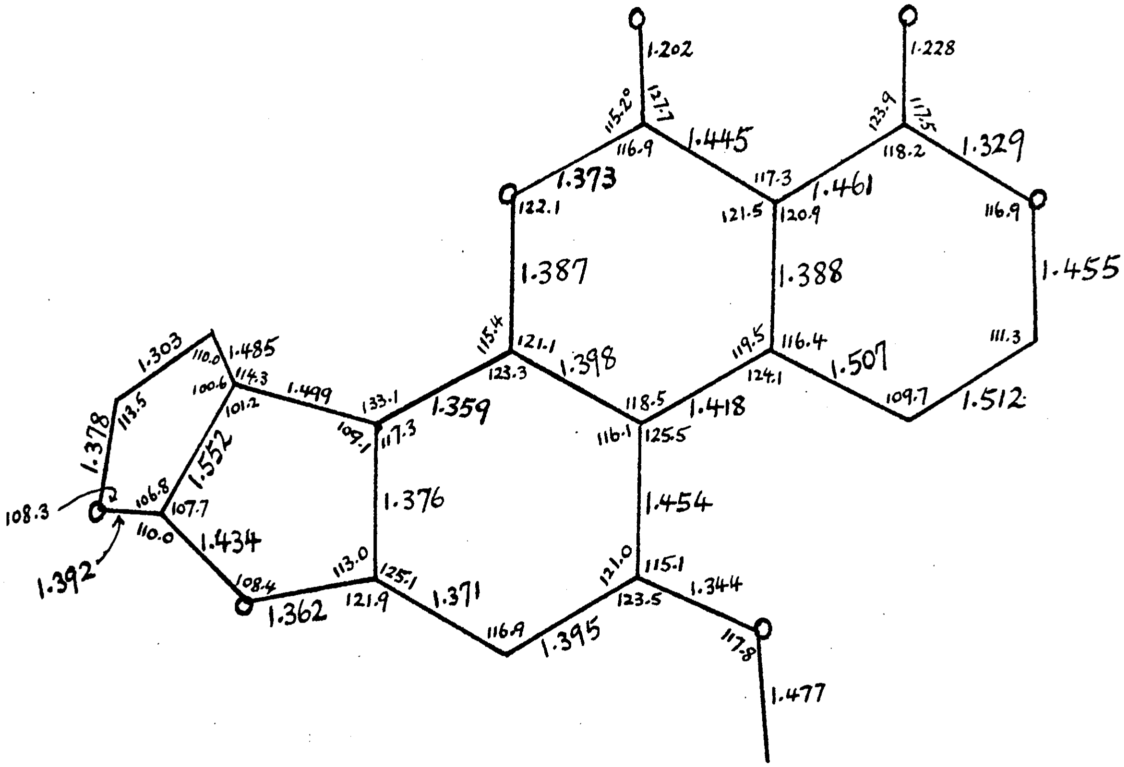
Table 2-28 Aflatoxin G_1

Average bond lengths and angles
and their estimated standard deviations

C(1)-O(2)	1.329	±	0.006
C(1)-C(6)	1.461		0.006
C(1)-O(21)	1.228		0.007
O(2)-C(3)	1.455		0.007
C(3)-C(4)	1.512		0.007
C(4)-C(5)	1.507		0.006
C(5)-C(6)	1.388		0.007
C(5)-C(10)	1.418		0.006
C(6)-C(7)	1.445		0.006
C(7)-O(8)	1.373		0.005
C(7)-O(22)	1.202		0.006
O(8)-C(9)	1.387		0.006
C(9)-C(10)	1.398		0.006
C(9)-C(14)	1.359		0.006
C(10)-C(11)	1.454		0.007
C(11)-C(12)	1.395		0.006
C(12)-C(13)	1.371		0.007
C(13)-C(14)	1.376		0.008
C(13)-O(17)	1.362		0.005
C(14)-C(15)	1.499		0.006
C(15)-C(16)	1.552		0.008
C(15)-C(20)	1.485		0.007
C(16)-O(17)	1.434		0.007
C(16)-O(18)	1.392		0.007
O(18)-C(19)	1.378		0.008
C(19)-C(20)	1.303		0.008
C(11)-O(23)	1.344		0.006
O(23)-C(24)	1.477		0.007

O(2)C(1)O(21)	117.5	±	0.4°
C(6)C(1)O(21)	123.9		0.4
C(6)C(1)O(2)	118.2		0.4
C(1)O(2)C(3)	116.9		0.3
O(2)C(3)C(4)	111.3		0.4
C(3)C(4)C(5)	109.7		0.4
C(4)C(5)C(6)	116.4		0.4
C(6)C(5)C(10)	119.5		0.4
C(4)C(5)C(10)	124.1		0.4
C(5)C(6)C(7)	121.5		0.4
C(1)C(6)C(5)	120.9		0.4
C(1)C(6)C(7)	117.3		0.4
C(6)C(7)O(22)	127.7		0.4
O(8)C(7)O(22)	115.2		0.4
C(6)C(7)O(8)	116.9		0.4
C(7)O(8)C(9)	122.1		0.3
O(8)C(9)C(10)	121.1		0.4
O(8)C(9)C(14)	115.4		0.4
C(10)C(9)C(14)	123.3		0.4
C(9)C(10)C(5)	118.5		0.4
C(9)C(10)C(11)	116.1		0.4
C(5)C(10)C(11)	125.5		0.4
C(10)C(11)C(12)	121.0		0.4
C(10)C(11)O(23)	115.1		0.4
C(12)C(11)O(23)	123.5		0.4
C(11)C(12)C(13)	116.9		0.5
C(12)C(13)C(14)	125.1		0.4
C(12)C(13)O(17)	121.9		0.5
C(14)C(13)O(17)	113.0		0.4
C(13)C(14)C(9)	117.3		0.4
C(15)C(14)C(9)	133.1		0.5
C(13)C(14)C(15)	109.1		0.4
C(14)C(15)C(16)	101.2		0.4
C(14)C(15)C(20)	114.3		0.4
C(16)C(15)C(20)	100.6		0.4
C(15)C(16)O(17)	107.7		0.4
C(15)C(16)O(18)	106.8		0.5
O(17)C(16)O(18)	110.0		0.4
C(13)O(17)C(16)	108.4		0.4
C(16)O(18)C(19)	108.3		0.5
O(18)C(19)C(20)	113.5		0.5
C(19)C(20)C(15)	110.0		0.5
C(11)O(23)C(24)	117.8		0.4

Fig.2-15(a) Average values of bond lengths and angles in the three isomorphous solvates of aflatoxin G_1.



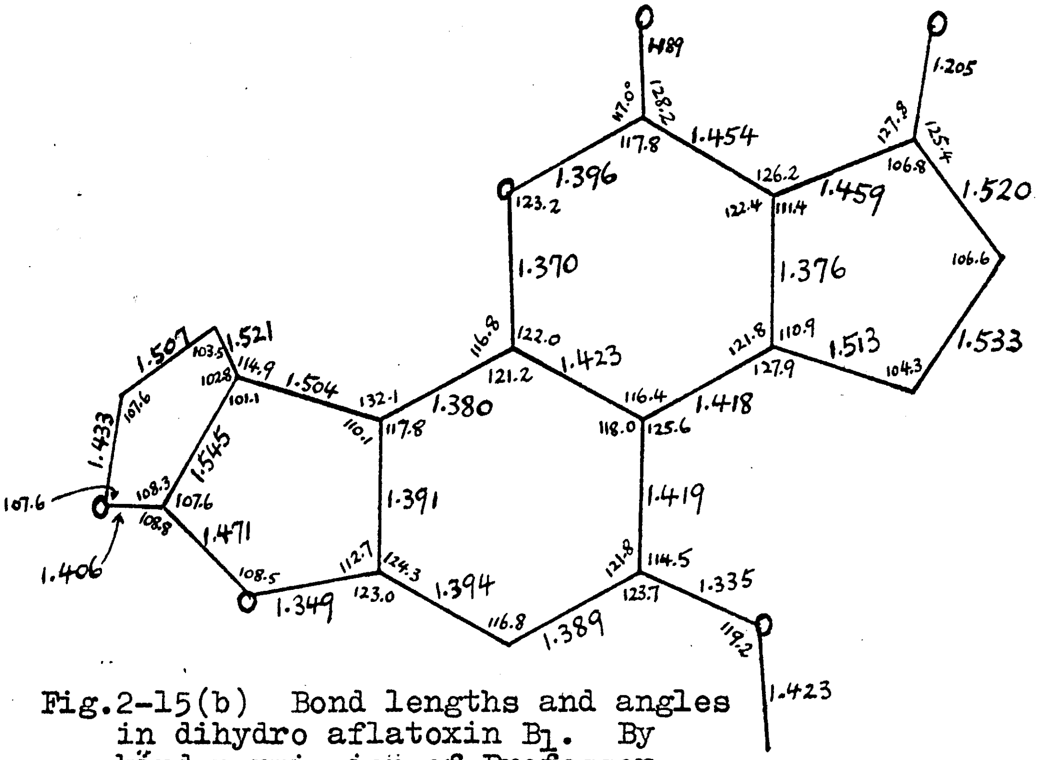


Fig.2-15(b) Bond lengths and angles in dihydro aflatoxin B_1. By kind permission of Professor A.F. Peerdeman.

Table 2-29

Mean plane (I) through atoms C(6), O(21), C(1), O(2) and C(3).

Equation of plane

Bromobenzene solvate	Mol.1	$0.2936X - 0.3757Y - 0.8790Z + 0.6117 = 0$
	Mol.1'	$0.2735X - 0.3057Y - 0.9119Z + 6.1975 = 0$
Benzene solvate	Mol.1	$0.2855X - 0.3689Y - 0.8845Z + 0.5181 = 0$
	Mol.1'	$0.2366X - 0.3458Y - 0.9080Z + 5.8892 = 0$
Bromothiophen solvate	Mol.1	$0.2580X - 0.3747Y - 0.8905Z + 0.7840 = 0$
	Mol.1'	$0.1889X - 0.3622Y - 0.9128Z + 6.0430 = 0$

Deviation in Å from plane (I)

	Bromobenzene solvate		Benzene solvate		Bromothiophen solvate	
Atom	Mol.1	Mol.1'	Mol.1	Mol.1'	Mol.1	Mol.1'
C(6)	0.018	-0.018	0.018	-0.029	0.023	-0.028
O(21)	-0.043	0.049	-0.049	0.033	-0.063	0.041
C(1)	0.007	-0.012	0.012	0.026	0.016	0.017
O(2)	0.071	-0.082	0.078	-0.084	0.101	-0.092
C(3)	-0.053	0.062	-0.060	0.054	-0.077	0.063
C(4)	0.868	-0.806	0.831	-0.845	0.840	-0.849
C(5)	0.597	-0.521	0.572	-0.582	0.593	-0.583

Table 2-29 (cont.)

Mean plane (II) through atoms C(5), C(6), C(7), O(8), C(9), C(10), C(11), C(12), C(13), C(14), C(15), C(16), O(17), O(22), and O(23).

Equation of plane

Bromobenzene solvate	Mol.1	$0.2769X + 0.0970Y - 0.9560Z - 1.6991 = 0$
	Mol.1'	$0.0916X + 0.1203Y - 0.9885Z + 6.4941 = 0$
Benzene solvate	Mol.1	$0.2054X + 0.1019Y - 0.9734Z - 1.4660 = 0$
	Mol.1'	$0.1150X + 0.1235Y - 0.9857Z + 6.0783 = 0$
Bromothiophen solvate	Mol.1	$0.1851X + 0.0929Y - 0.9783Z - 1.3191 = 0$
	Mol.1'	$0.1100X + 0.1384Y - 0.9843Z + 6.0565 = 0$

Deviation in Å from plane (II)

	Bromobenzene solvate		Benzene solvate		Bromothiophen solvate	
Atom	Mol.1	Mol.1'	Mol.1	Mol.1'	Mol.1	Mol.1'
C(5)	-0.065	0.184	-0.095	0.120	-0.074	0.107
C(6)	0.024	0.097	-0.008	0.029	0.009	-0.004
C(7)	0.017	-0.040	0.037	-0.039	0.011	-0.044
O(8)	0.022	-0.008	0.011	0.026	0.012	0.004
C(9)	0.010	-0.014	-0.004	-0.003	0.000	-0.017
C(10)	-0.018	0.050	-0.029	0.038	-0.022	0.047
C(11)	-0.009	-0.017	0.002	0.004	0.008	0.009
C(12)	-0.020	-0.095	0.012	-0.045	-0.013	-0.025
C(13)	0.005	-0.033	-0.007	-0.023	-0.022	0.005
C(14)	0.016	-0.032	0.004	0.003	-0.021	0.010
C(15)	0.173	0.082	0.049	0.105	0.071	0.123
C(16)	-0.110	0.078	-0.021	-0.002	0.006	-0.076
O(17)	-0.047	-0.018	-0.040	-0.042	-0.049	-0.010
O(22)	-0.066	-0.161	0.009	-0.116	-0.001	-0.064
O(23)	0.069	-0.074	0.082	-0.055	0.086	-0.066

Table 2-29 (cont.)

Mean plane (III) through atoms C(15), C(16), O(18), C(19) and C(20).

Equation of plane

Bromobenzene solvate	Mol.1	$0.7514X - 0.6010Y - 0.2723Z + 1.7826 = 0$
	Mol.1'	$-0.7500X + 0.5387Y - 0.3837Z + 5.6693 = 0$
Benzene solvate	Mol.1	$0.7860X - 0.5221Y - 0.3311Z + 1.4693 = 0$
	Mol.1'	$-0.6881X + 0.5800Y - 0.4360Z + 5.7992 = 0$
Bromothiophen solvate	Mol.1	$0.7874X - 0.5256Y - 0.3220Z + 1.7297 = 0$
	Mol.1'	$-0.6223X + 0.6772Y - 0.3926Z + 4.9118 = 0$

Deviation in Å from plane (III)

	Bromobenzene solvate		Benzene solvate		Bromothiophen solvate	
Atom	Mol.1	Mol.1'	Mol.1	Mol.1'	Mol.1	Mol.1'
C(15)	-0.035	-0.011	-0.026	-0.025	0.010	-0.044
C(16)	0.042	0.012	0.039	0.025	0.025	0.060
O(18)	-0.036	-0.008	-0.037	-0.018	-0.053	-0.053
C(19)	0.013	-0.001	0.018	-0.001	0.062	0.022
C(20)	0.015	0.008	0.007	0.018	-0.043	0.016

Table 2-29 (cont.)

Equation of plane (IV) through the atoms of the bromobenzene molecule:

$$0.3477X + 0.2775Y - 0.8956Z + 1.1620 = 0$$

Atom	Deviation in Å from plane (IV)
C(25)	0.014
C(26)	-0.024
C(27)	-0.005
C(28)	0.026
C(29)	0.005
C(30)	-0.033
Br	0.016

Equation of plane (V) through the atoms of the benzene molecule:

$$0.3140X + 0.1412Y - 0.9388Z + 1.6100 = 0$$

Atom	Deviation in Å from plane (V)
C(25)	0.000
C(26)	-0.027
C(27)	0.026
C(28)	0.000
C(29)	-0.026
C(30)	0.026

Equation of plane (VI) through the atoms of the bromothiophen molecule:

$$0.2409X + 0.1152Y - 0.9637Z + 2.0312 = 0$$

Atom	Deviation in Å from plane (VI)
C(25)	0.031
S/C(26)	-0.035
C(27)	0.029
C(28)	-0.007
S/C(29)	-0.020
Br	0.003

PART III

The Stereochemistry of Isocolumbin: X-ray Analysis of 1-p-iodophenyl,3-phenyl Pyrazoline Adduct of Isocolumbin Monoacetone Solvate.

3.1. INTRODUCTION

Columbin is one of the three neutral bitter principles isolated from the root of Jatrorrhiza palmata Miers (columbo root). The other two being chasmanthin, $C_{20}H_{22}O_7$, and the isomeric palmarin.

In the presence of alkali, columbin is easily isomerized to isocolumbin.

The chemistry of columbin and isocolumbin has been studied since the 1930's by Wessely et al. (1935), and also by Feist et al. (1935).

After extensive re-analysis, Barton and Elad (1956) have described in detail the functioning groups of this diterpene and proposed the constitution (I) for columbin. In the same year, Cava and Soboczenski (1956) also described the nature of columbin.

In 1959, Cava, Weinstein and Malhotra proposed the stereochemistry of columbin, as shown in (II).

The stereochemistry of columbin, as shown in (III), proposed by Overton, Weir and Wylie (1961) differs fromm (II) in that the ring fusion of A and B is cis and not trans. They also indicated that the change from columbin to isocolumbin involves a change of ring fusion from cis to trans at rings B and C.

Weir (1962) on the basis of chemical, spectroscopic, and optical rotatory dispersion investigations, has defined the absolute configuration of columbin as (IV) and isocolumbin as (V). (III) and (V) are mirror images of one another.

In view of the uncertainty, it was decided to carry out x-ray analysis in order to establish the stereochemistry.

The heavy atom derivative for x-ray study was prepared and kindly supplied Dr. K. Overton and Mr. D. Meville of this Chemistry Department. They have devised an ingenious way of forming an adduct at the double bond, 2:3-, so that the heavy atom is remote from the diterpene. In this way, the conformation and stereochemistry should not be affected.

The crystals employed were recrystallized from acetone. During the x-ray analysis, it was found that the adduct formed a monoacetone solvate.

3.2. EXPERIMENTAL

Crystal data. 1-p-iodophenyl,3-phenyl pyrazoline adduct of isocolumbin monoactone solvate, $C_{33}H_{31}O_6N_2I.(CH_3)_2CO$. Molecular weight = 724.6. Tetragonal, a = 9.68, c = 70.79 Å. Volume of unit cell = 6626 $Å^3$. Number of asymmetric units per unit cell, Z = 8. Density(cal.)= 1.452 gm cm^{-3}. Absent spectra: h00 when h is odd, 0k0 when k is odd, 00ℓ when $\ell \neq 4n$. Space group: $P4_12_12$ (D_4^4) or its enantiomorph $P4_32_12$ (D_4^8). Absorption coefficient for x-rays (λ=1.542Å), μ=81.6 cm^{-1}. Total number of electrons per unit cell, F(000) = 2960. $\sum f^2$ "heavy" atom = 2809, $\sum f^2$ "light" atoms = 1843.

Intensity data. Rotation, Weissenberg and precession photographs were taken with Cu Kα radiation and Mo Kα radiation. The cell dimensions were obtained from precession photographs. Three-dimensional intensity data were recorded by taking zero layer and equi-inclination Weissenberg photographs about the 9.68Å axis. A pack of six films was used for each exposure. The intensities of the reflections in the layers 0kℓ, 1kℓ, ... , 7kℓ were estimated visually. These were reduced to structure amplitudes by applying the Lorentz, polarization and Tunell rotation factors.

Because of the tetragonal symmetry, the structure amplitudes in different layers were inter-related. For example, in the zero layer, $|F(01\ell)|$ are equal to $|F(10\ell)|$ in the first layer; and $|F(02\ell)|$ are equal to $|F(20\ell)|$. Therefore all the three-dimensional intensity data could be brought to the same scale and this was done, so that at various stages throughout the analysis, only one scale factor was used to ensure $\Sigma|Fo| = \Sigma|Fc|$.

From the estimated intensities of a total of 3539 reflections, 2068 independent structure amplitudes were obtained. During the least squares refinement, it was observed that the agreement between the $|Fo|$ and $|Fc|$ for the higher order reflections (when the ℓ index reaches about 60 or larger) was generally poor, and this had a marked effect on the tempeature factors. It was therefore decided to exclude these reflections from the later stage of the least squares refinement and as a result, only 1860 terms are listed in the final structure factor table (Table 3-3).

3.3. DETERMINATION OF THE STRUCTURE

The Patterson projection P(uw) was calculated and is shown in Fig. 3-1(a). All peaks of considerable weights appeared on the line z=0, 1/4, and 1/2. This suggested that the z-coordinate of the iodine atom is at a special position and it was not easy to obtain an unique solution for the iodine atom from the projection alone. Accordingly, the Harker sections P(U1/2W), P(UV1/2), and P(UV1/4) as well as the section P(UV0) of the three-dimensional Patterson function were computed. They are shown in Fig. 3-1(b) and Fig. 3-2 respectively.

As there are eight equivalent positions, even though there is only one heavy atom in an asymmetric unit, the number of peaks corresponding to the I-I vectors in the three-dimensional Patterson function is 28. However, because the z-coordinate of the iodine atom is nearly 0.250, it was possible to assign peaks corresponding to all the 28 I-I vectors in the four sections mentioned. These were marked A, B, ... , J in Figs. 3-1(b) and 3-2, and on Table 3-1 showing the coordinates of the vectors expected. These were also marked on Fig. 3-1(a), showing how some of these peaks overlapped.

The coordinates of the iodine atom thus found were:

$x = 0.077, \quad y = 0.260, \quad z = 0.249.$

[The final coordinates shown in Table 3-4 were refined using the equivalent position: 1/2+y, 1/2-x, 3/4+z and thus $x = 0.760$, $y = 0.423$, $z = -0.001$.]

Owing to the limitation of the Fourier program for the Deuce computer, it was necessary to transform the space group into another one of lower symmetry. The transformation was originally suggested by Dr. T.A. Hamor, formerly of this Chemistry Department, and has been applied in the structure determination of byssochlamic acid bis-p-bromophenyl-hydrazide (Paul et al. 1963), and chimonanthine dihydrobromide (Grant, 1962). This involves doubling the number of atoms in the asymmetric unit and including more than the basic non-equivalent set of $F(hk\ell)$ [ie. including $F(hk\ell)$ as well as $F(kh\ell)$ when $h \neq k$]. Under these conditions, and if the origin is shifted to (1/4,0,3/8), the space group becomes effectively

Table 3-1

The vectors to be expected in the space group $P4_12_12$ between symmetrically related atoms.

Atomic Positions	x y z	$-x$ $-y$ $\frac{1}{2}+z$	$\frac{1}{2}-x$ $\frac{1}{2}+y$ $\frac{1}{4}-z$	$\frac{1}{2}+x$ $\frac{1}{2}-y$ $\frac{3}{4}-z$	y x $-z$	$-y$ $-x$ $\frac{1}{2}-z$	$\frac{1}{2}-y$ $\frac{1}{2}+x$ $\frac{1}{4}+z$	$\frac{1}{2}+y$ $\frac{1}{2}-x$ $\frac{3}{4}+z$
x y z	origin peak	$-2x$ $-2y$ $\frac{1}{2}$ D	$\frac{1}{2}-2x$ $\frac{1}{2}$ E $\frac{1}{4}-2z$	$\frac{1}{2}$ H $\frac{1}{2}-2y$ $\frac{3}{4}-2z$	$y-x$ $x-y$ G $-2z$	$-y-x$ $-x-y$ $\frac{1}{2}-2z$ J	$\frac{1}{2}-y-x$ $\frac{1}{2}+x-y$ $\frac{1}{4}$ B	$\frac{1}{2}+y-x$ $\frac{1}{2}-x-y$ $\frac{3}{4}$ A
$-x$ $-y$ $\frac{1}{2}+z$	$2x$ $2y$ $-\frac{1}{2}$	origin peak	$\frac{1}{2}$ H $\frac{1}{2}+2y$ $-\frac{1}{4}-2z$	$\frac{1}{2}+2x$ $\frac{1}{2}$ E $\frac{1}{4}-2z$	$y+x$ $x+y$ J $-\frac{1}{2}-2z$	$-y+x$ $-x+y$ $-2z$ G	$\frac{1}{2}-y+x$ $\frac{1}{2}+x+y$ $-\frac{1}{4}$ A	$\frac{1}{2}+y+x$ $\frac{1}{2}-x+y$ $\frac{1}{4}$ B
$\frac{1}{2}-x$ $\frac{1}{2}+y$ $\frac{1}{4}-z$	$-\frac{1}{2}+2x$ $-\frac{1}{2}$ $-\frac{1}{2}+2z$	$-\frac{1}{2}$ $-\frac{1}{2}-2y$ $\frac{1}{4}+2z$	origin peak	$2x$ $-2y$ $\frac{1}{2}$ D	$-\frac{1}{2}+y+x$ $-\frac{1}{2}+x-y$ $-\frac{1}{4}$ B	$-\frac{1}{2}-y+x$ $-\frac{1}{2}-x-y$ $\frac{1}{4}$ A	$-y+x$ $x-y$ $2z$ G	$y+x$ $-x-y$ $\frac{1}{2}+2z$ J
$\frac{1}{2}+x$ $\frac{1}{2}-y$ $\frac{3}{4}-z$	$-\frac{1}{2}$ $-\frac{1}{2}+2y$ $-\frac{3}{4}+2z$	$-\frac{1}{2}-2x$ $-\frac{1}{2}$ $-\frac{1}{4}+2z$	$-2x$ $2y$ $-\frac{1}{2}$	origin peak	$-\frac{1}{2}+y-x$ $-\frac{1}{2}+x+y$ $-\frac{3}{4}$ A	$-\frac{1}{2}-y-x$ $-\frac{1}{2}-x+y$ $-\frac{1}{4}$ B	$-y-x$ $x+y$ $-\frac{1}{2}+2z$ J	$y-x$ $-x+y$ $2z$ G
y x $-z$	$x-y$ $y-x$ $2z$	$-x-y$ $-y-x$ $\frac{1}{2}+2z$	$\frac{1}{2}-x-y$ $\frac{1}{2}-x+y$ $\frac{1}{4}$	$\frac{1}{2}+x-y$ $\frac{1}{2}-y-x$ $\frac{3}{4}$	origin peak	$-2y$ C $-2x$ $\frac{1}{2}$	$\frac{1}{2}-2y$ $\frac{1}{2}$ F $\frac{1}{4}+2z$	$\frac{1}{2}$ $\frac{1}{2}-2x$ I $\frac{3}{4}+2z$
$-y$ $-x$ $\frac{1}{2}-z$	$x+y$ $y+x$ $-\frac{1}{2}+2z$	$-x+y$ $-y+x$ $2z$	$\frac{1}{2}-x+y$ $\frac{1}{2}+y+x$ $-\frac{1}{4}$	$\frac{1}{2}+x+y$ $\frac{1}{2}-y+x$ $\frac{1}{4}$	$2y$ $2x$ $-\frac{1}{2}$	origin peak	$\frac{1}{2}$ I $\frac{1}{2}+2x$ $-\frac{1}{4}+2z$	$\frac{1}{2}+2y$ $\frac{1}{2}$ F $\frac{1}{4}+2z$
$\frac{1}{2}-y$ $\frac{1}{2}+x$ $\frac{1}{4}+z$	$-\frac{1}{2}+x+y$ $-\frac{1}{2}+y-x$ $-\frac{1}{4}$	$-\frac{1}{2}-x+y$ $-\frac{1}{2}-y-x$ $\frac{1}{4}$	$-x+y$ $y-x$ $-2z$	$x+y$ $-y-x$ $\frac{1}{2}-2z$	$-\frac{1}{2}+2y$ $-\frac{1}{2}$ $-\frac{1}{4}-2z$	$-\frac{1}{2}$ $-\frac{1}{2}-2x$ $\frac{1}{4}-2z$	origin peak	$2y$ $-2x$ C $\frac{1}{2}$
$\frac{1}{2}+y$ $\frac{1}{2}-x$ $\frac{3}{4}+z$	$-\frac{1}{2}+x-y$ $-\frac{1}{2}+y+x$ $-\frac{3}{4}$	$-\frac{1}{2}-x-y$ $-\frac{1}{2}-y+x$ $-\frac{1}{4}$	$-x-y$ $y+x$ $-\frac{1}{2}-2z$	$x-y$ $-y+x$ $-2z$	$-\frac{1}{2}$ $-\frac{1}{2}+2x$ $-\frac{3}{4}-2z$	$-\frac{1}{2}-2y$ $-\frac{1}{2}$ $-\frac{1}{4}-2z$	$-2y$ $2x$ $-\frac{1}{2}$	origin peak

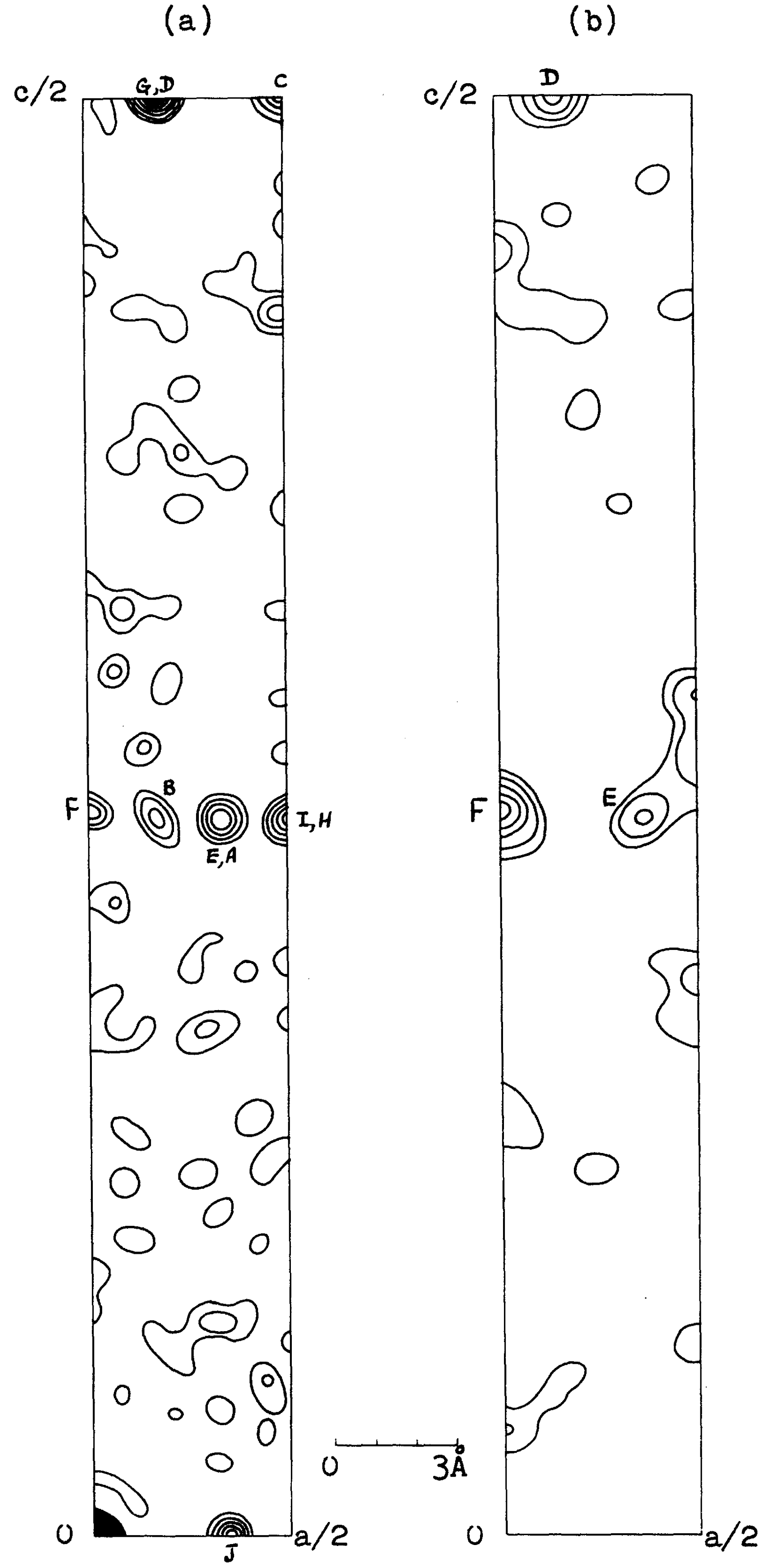
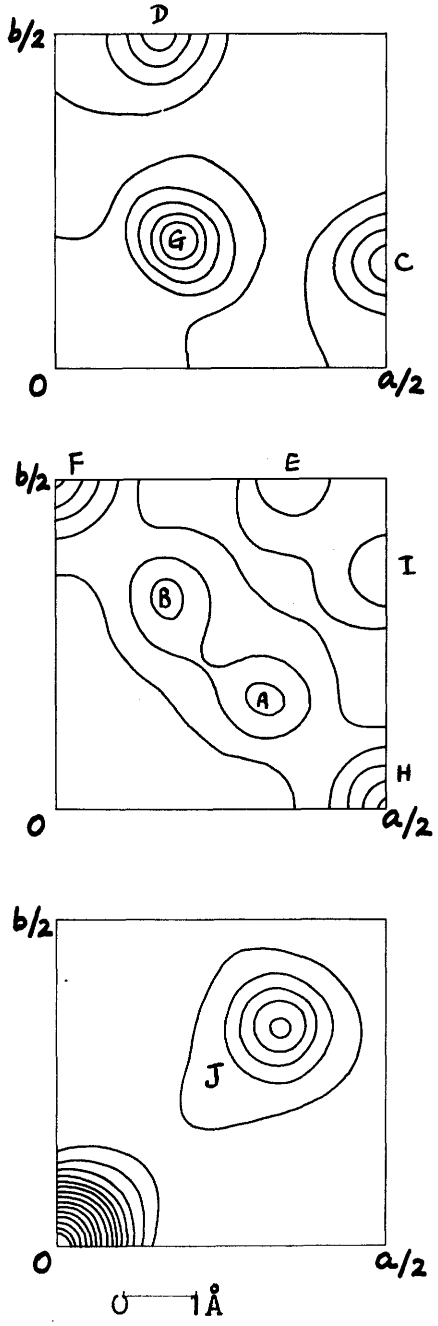


Fig. 3-1. (a) Patterson projection along the b-axis,
(b) Section at V=1/2 through the 3-dimensional Patterson synthesis. Contour scale arbitrary.
The peaks corresponding to the vectors given in Table 3-1 are marked.



(a) Section P(UV1/2)

(b) Section P(UV1/4)

(c) Section P(UV0)

Fig. 3-2. Three-dimensional Patterson synthesis. Contour scale arbitrary.

The peaks corresponding to the vectors given in Table 3-1 are marked.

$P2_12_12_1$.

This is shown algebraically as follows: after the transformation, the eight equivalent positions will become

i. −1/4+x, y, −3/8+z; v. −1/4+y, x, −3/8−z;
ii. −1/4−x, −y, 1/8+z; vi. −1/4+y, −x, 1/8−z;
iii. 1/4−x, 1/2+y, −1/8−z; vii. 1/4−y, 1/2+x, −1/8+z;
iv. 1/4+x, 1/2−y, 3/8−z; viii. 1/4+y, 1/2−x, 3/8+z.

Putting X_1=x−1/4, Y_1=y, Z_1=z−3/8, we have:
i. X_1, Y_1, Z_1; ii. 1/2−X_1, −Y_1, 1/2+Z_1; iii. −X_1, 1/2+Y_1, 1/2−Z_1; iv. 1/2+X_1, 1/2−Y_1, −Z_1.

Putting X_2=y−1/4, Y_2=x, Z_2=−z−3/8, we have:
v. X_2, Y_2, Z_2; vi. 1/2−X_2, −Y_2, 1/2+Z_2; vii. −X_2, 1/2+Y_2, 1/2−Z_2; viii. 1/2+X_2, 1/2−Y_2, −Z_2.

These are the equivalent positions for the space group $P2_12_12_1$.

Therefore the coordinates of the iodine atoms became

I_1: x = −0.173, y = 0.260, z = −0.126;
I_2: x = 0.010, y = 0.077, z = −0.624.

A set of structure factors was calculated with these coordinates. The discrepancy factor $R(=\Sigma||Fo|-|Fc||/\Sigma|Fo|)$ over all the reflections was 0.438.

With the exception of those terms where |Fo| was greater than three times |Fc|, the structure factors were put into an Fo Fourier synthesis. Sections of the three-dimensional electron-density distribution were drawn on glass sheets stacked in a metal frame. This revealed the atomic arrangement and therefore the stereochemistry of this isocolumbin adduct.

The coordinates of 30 carbon atoms, 6 oxygen atoms, and 2 nitrogen atoms (together with a set of symmetry-related coordinates of all these atoms) were calculated. The peaks for the two methyl carbon atoms and C(6) were not too distinct and they were not included at this stage. Together with the improved coordinates of the iodine atoms, these were put into structure factor calculation. The R factor dropped to 0.301.

All the structure factors were included in the second Fo Fourier synthesis, from which coordinates could be calculated for the three carbon atoms previously left out; together with a set of improved coordinates for the rest of the atoms. In addition, it was observed that in the space between two molecules, there were peaks which looked like an acetone molecule. But their presence was ignored at this stage because they might well be spurious peaks.

These improved coordinates were used to calculate another set of phase constants which were then used in the third Fo Fourier synthesis.

In the resulting electron-density distribution, the group of peaks resembling an acetone molecule appeared more clearly. By comparing the peak height, it was possible to determine which was the oxygen atom; this was confirmed by bond length and bond angle calculations.

With two I, fourteen O, four N, and seventy C atoms, and an isotropic temperature factor of $B=4.5\text{Å}^2$(equivalent to $U = 0.048\text{Å}^2$) for all the atoms, the fourth set of structure factors thus calculated had an R factor of 0.233.

3.4. LEAST SQUARES REFINEMENT

The least squares refinement was carried out in the KDF 9 computer using a program written by Mr. J.G.F. Smith and Professor D.W.J. Cruickshank. Isotropic block-diagonal refinement was employed throughout except in the last two cycles when the iodine atom was allowed to refine anisotropically. The weights, $\sqrt{w}$, were calculated according to an equation suggested by Cruickshank et al. (1961) as follow:

$$\sqrt{w} = \frac{1}{(\ p_1 + |F| + p_2 \times |F|^2 + p_3 \times |F|^3\ )^{1/2}}$$

where $p_1 = 2 \times F_{min} = 14.4$,

$p_2 = 2/F_{max} = 0.006$, and $p_3 = 0$.

Altogether, seven least squares cycles were carried out. The course of the analysis and refinement is summarized in Table 3-2. The final R factor over 1860 reflections is 0.095.

Table 3-2 Course of analysis

1. Patterson function, iodine atom located.
2. Space group transformed to $P2_12_12_1$.
3. 1st S.F. calculation 2 iodine atoms, B=4.4Å^2, R=0.438.
4. 1st Fo synthesis.
5. 2nd S.F. calculation, 2 I, 12 O, 4 N, and 60 C atoms, B=4.5Å^2, R=0.301.
6. 2nd Fo synthesis. An acetone solvent molecule observed.
7. 3rd S.F. calculation, 2 I, 12 O, 4 N, 66 C atoms, B=4.5Å^2, R=0.271.
8. 3rd Fo synthesis. Presence of acetone molecule confirmed.
9. 4th S.F. calculation, 2 I, 14 O, 4 N, 72 C atoms, B=4.5Å^2, R=0.233.
10. Space group transformed back to $P4_12_12$.
11. S.F.L.S. cycle 1 * R=0.246.
12. S.F.L.S. cycle 2* R=0.183.
13. S.F.L.S. cycle 3* R=0.172.
14. S.F.L.S. cycle 4* R=0.135.
15. S.F.L.S. cycle 5 R=0.134.
16. S.F.L.S. cycle 6** R=0.105.
17. S.F.L.S. cycle 7 R=0.098.
18. Final S.F. calculation, R=0.095.

Calculations in stages (1), (3) - (9) performed in the Deuce computer; (11) - (18) in the KDF 9 computer.

* Only 1882 terms out of the total 2068 observed S.F. were used in each cycle to fit in with a three hour time limit of computing at the time of refinement.

** (i) 226 high order reflections excluded; and (ii) the iodine atom refined anisotropically, from this cycle.

3.5. RESULTS AND DISCUSSION

The observed and calculated structure amplitudes together with the phase constants for 1860 reflections are listed in Table 3-3.

The final atomic coordinates, thier estimated standard deviations and the temperature factors are listed in Table 3-4.

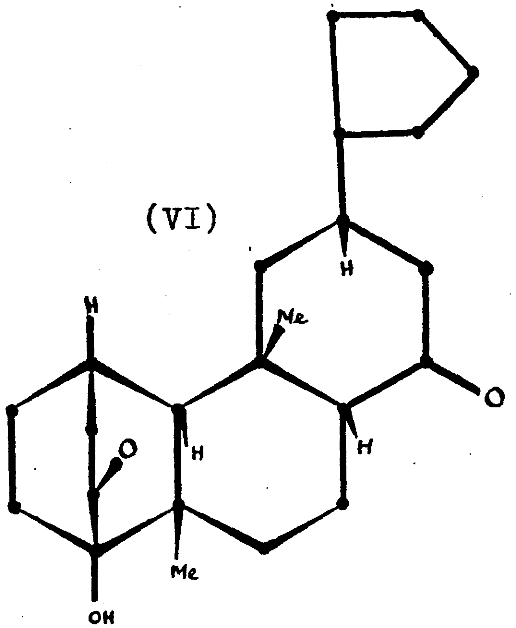
The bond lengths and angles with their estimated standard deviations are listed in Tables 3-5 and 3-6 respectively. Intramolecular non-bonded distances less than 3.5Å and intermolecular distances less than 4Å are listed in Tables 3-7 and 3-8 respectively.

The packing around half the unit cell as viewed down the b axis is shown in Fig. 3-3. Because of the tetragonal symmetry, this projection also shows the packing viewed down the a axis.

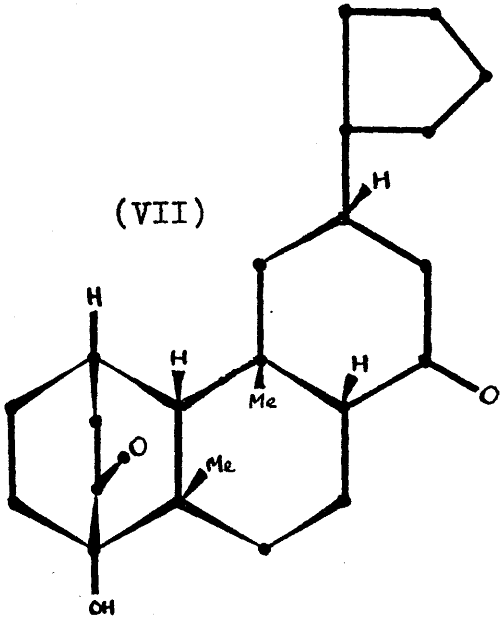
The atomic arrangement in both these views are drawn in a larger scale and shown in Fig. 3-4. This diagram is sufficient to depict the stereochemistry of the molecule.

Stereochemistry. The configuration of isocolumbin is therefore defined either as in (VI) or (VII).

(VI)



(VII)



The absolute configuration has not been established in the present analysis. According to the results of Weir (1962), the absolute configuration is (VI) (which is the same as (V)).

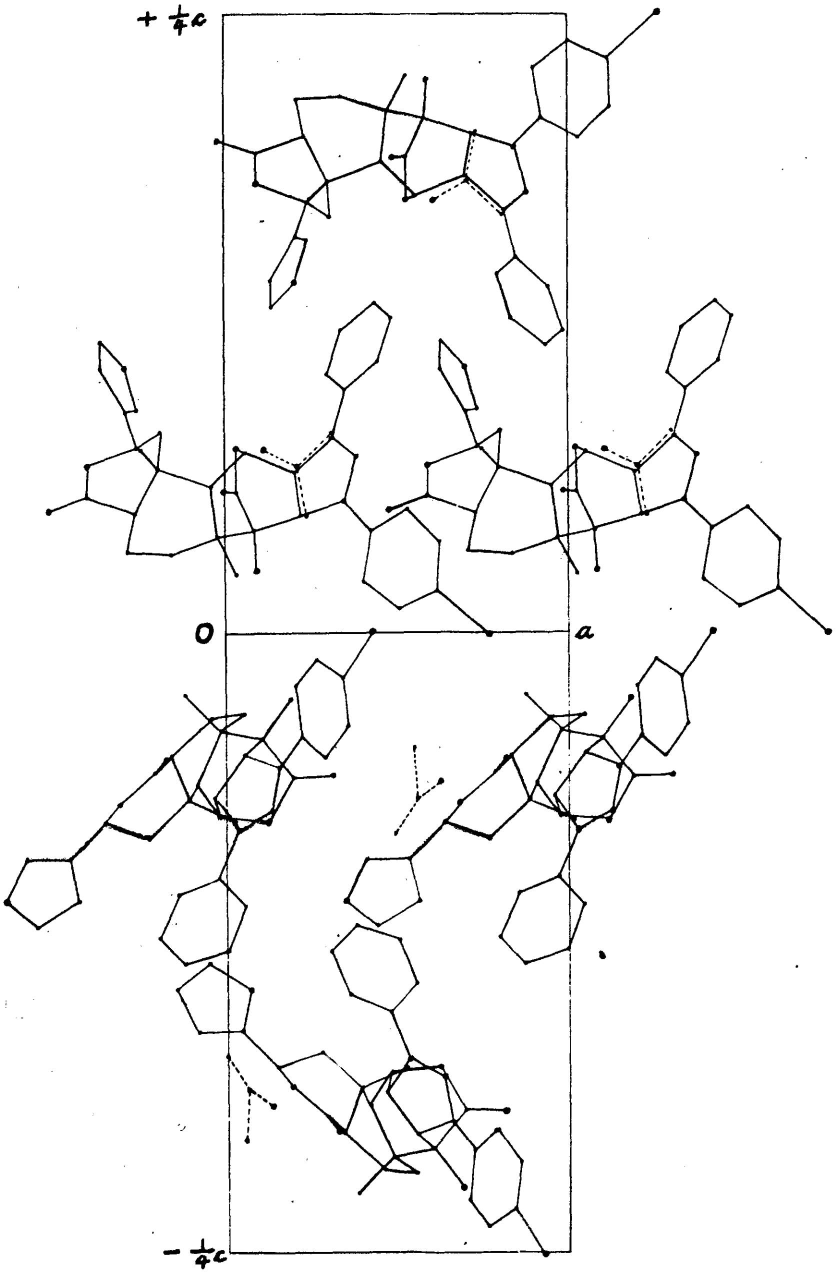


Fig. 3-3. The arrangement of the molecules in the crystal of the iodo-derivative of isocolumbin around half the unit cell as viewed along the b-axis.

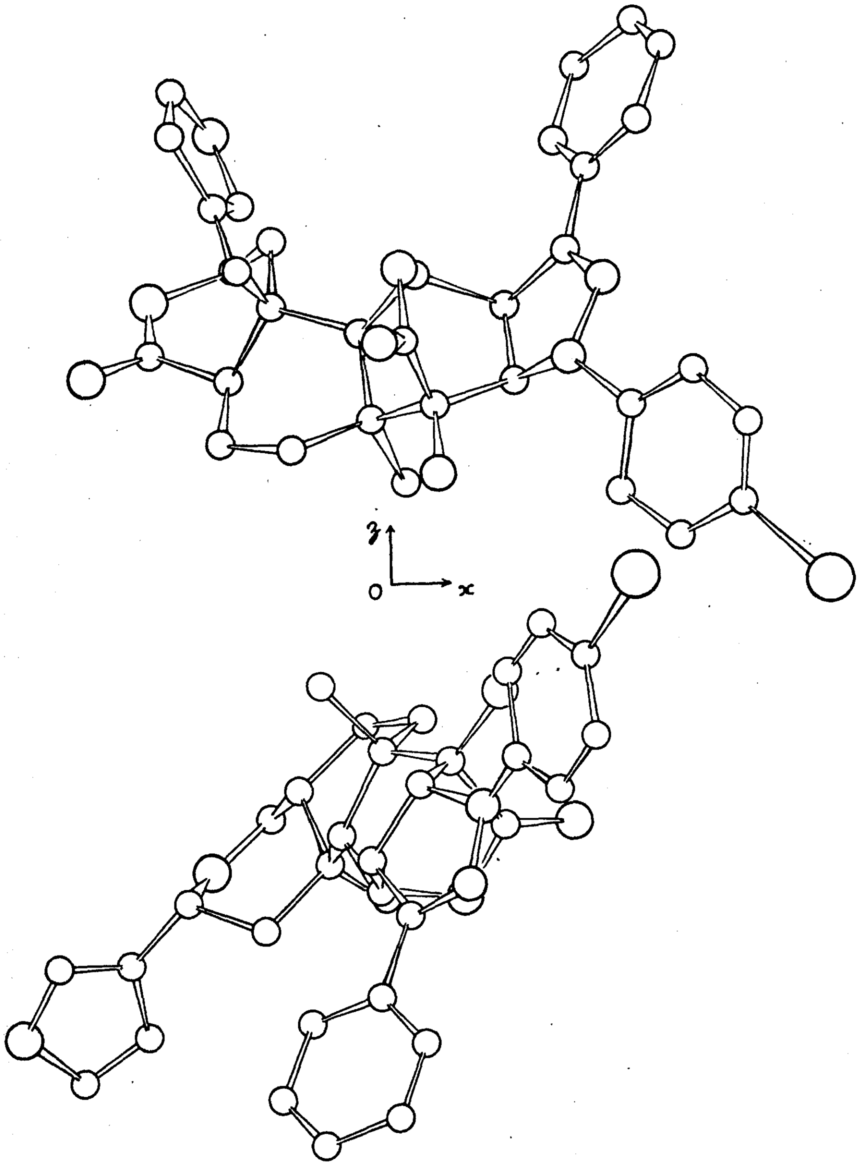


Fig. 3-4. Two perspective views (top, along the b-axis, bottom, along the a-axis) of the iodo-derivative of isocolumbin showing the configuration of the atoms in the molecule.

Table 3-3. Iodo-derivative of isocolumbin

H	K	L	Fo	Fc	α
0	0	8	238.6	225.2	180
0	0	12	155.7	172.5	0
0	0	16	462.9	526.2	0
0	0	20	145.3	154.6	0
0	0	24	283.5	266.0	0
0	0	28	317.1	343.8	0
0	0	32	67.4	76.8	0
0	0	36	166.6	181.4	0
0	0	40	288.7	307.4	0
0	0	44	143.8	147.2	0
0	0	48	88.2	82.0	0
0	0	52	93.2	105.4	0
0	0	56	117.8	140.9	0
0	0	60	26.3	34.4	0
0	0	64	51.9	51.5	0
0	0	68	28.8	29.6	0
0	1	1	18.4	8.1	225
0	1	2	25.1	15.6	180
0	1	3	18.1	12.5	315
0	1	4	129.0	139.4	90
0	1	5	22.2	14.9	225
0	1	6	194.5	201.7	180
0	1	7	33.0	16.5	135
0	1	8	93.4	101.7	90
0	1	9	131.2	145.5	225
0	1	10	338.8	369.2	180
0	1	11	22.3	19.9	315
0	1	12	37.4	32.4	90
0	1	13	208.0	212.5	225
0	1	14	83.6	85.3	180
0	1	15	96.8	68.0	315
0	1	16	74.9	77.4	270
0	1	17	13.1	16.5	45
0	1	18	257.3	228.6	180
0	1	19	83.4	87.9	315
0	1	20	13.4	5.6	90
0	1	21	40.9	37.3	225
0	1	22	205.1	193.6	180
0	1	23	83.1	78.5	315
0	1	24	50.5	56.8	90
0	1	25	39.3	44.7	225
0	1	26	97.3	82.6	180
0	1	27	73.8	86.2	315
0	1	28	14.7	21.1	90
0	1	29	64.1	61.6	225
0	1	30	11.4	9.7	180
0	1	31	112.3	126.2	315
0	1	32	77.0	69.2	90
0	1	33	49.1	36.8	225
0	1	34	67.2	76.5	180
0	1	35	49.1	49.8	315
0	1	36	79.1	74.3	270
0	1	37	75.2	72.4	225
0	1	38	21.0	25.8	180
0	1	39	28.3	25.6	135
0	1	40	28.2	27.3	90
0	1	41	28.0	25.3	45
0	1	42	56.4	52.7	180
0	1	43	65.7	62.7	315
0	1	44	11.8	18.8	90
0	1	46	73.6	66.5	180
0	1	47	55.4	52.9	315
0	1	48	19.5	18.9	90
0	1	49	48.7	36.0	45
0	1	51	46.3	50.4	315
0	1	52	31.0	24.8	270
0	1	53	30.2	30.8	45
0	1	54	28.4	33.3	180
0	1	55	23.2	21.3	315
0	1	56	17.8	16.6	270
0	1	57	52.7	57.2	225
0	1	58	69.8	80.3	180
0	1	59	43.8	38.5	315
0	1	62	17.8	40.3	180
0	1	63	46.2	48.6	315
0	1	64	52.4	44.4	90
0	2	0	36.0	30.3	180
0	2	1	96.7	100.8	315
0	2	2	138.1	127.8	90
0	2	3	29.2	30.2	225
0	2	4	90.9	96.6	180
0	2	5	115.6	114.3	315
0	2	6	145.5	159.0	270
0	2	7	191.4	202.1	225
0	2	8	48.4	60.2	180
0	2	9	230.7	258.5	315
0	2	10	153.5	163.8	270
0	2	11	30.8	24.5	225
0	2	12	123.6	121.1	180
0	2	13	46.6	41.9	315
0	2	14	115.9	113.3	90
0	2	15	137.0	145.4	225
0	2	16	30.4	42.5	180
0	2	17	47.5	51.7	315
0	2	18	99.1	96.1	270
0	2	19	168.1	164.4	225
0	2	20	146.3	130.0	180
0	2	21	209.9	223.1	315
0	2	22	173.9	168.0	270
0	2	23	116.8	108.3	225
0	2	24	36.0	33.9	180
0	2	25	25.7	31.4	315
0	2	26	51.6	63.1	270
0	2	27	36.6	35.1	225
0	2	28	17.9	8.3	0
0	2	30	76.0	73.4	90
0	2	31	134.1	151.0	225
0	2	32	33.4	27.0	180
0	2	33	78.6	84.0	315
0	2	34	49.3	39.9	90
0	2	35	92.9	91.6	225
0	2	36	73.6	76.2	180
0	2	37	94.4	95.7	315
0	2	38	22.7	28.7	270
0	2	39	60.1	54.0	225
0	2	40	50.4	43.6	180
0	2	42	34.2	34.2	270
0	2	43	48.4	47.7	225
0	2	44	26.1	25.4	180
0	2	47	73.6	74.9	225
0	2	48	33.7	36.4	180
0	2	49	65.0	71.3	315
0	2	51	59.0	59.0	225
0	2	52	22.0	24.5	0
0	2	54	53.0	49.1	270
0	2	55	44.5	42.0	225
0	2	56	18.1	26.2	180
0	2	59	70.4	59.8	225
0	2	60	58.2	53.0	180
0	2	61	41.1	47.4	315
0	3	1	258.8	281.2	45
0	3	2	20.3	17.1	0
0	3	3	100.1	110.1	135
0	3	4	113.7	120.2	270
0	3	5	185.0	208.9	45
0	3	6	171.0	164.5	180
0	3	7	153.7	174.4	135
0	3	8	14.4	19.3	90
0	3	9	277.8	261.5	45
0	3	10	39.3	41.7	0
0	3	11	219.0	229.4	135
0	3	12	42.0	28.7	270
0	3	13	224.5	253.4	45
0	3	14	60.4	59.1	180
0	3	15	73.4	87.4	135
0	3	16	47.2	40.1	90
0	3	17	146.5	162.7	45
0	3	18	68.8	61.0	180
0	3	19	84.7	92.7	135
0	3	20	23.0	18.6	270
0	3	21	128.2	119.2	45
0	3	22	117.7	117.7	180
0	3	23	228.4	237.4	135
0	3	24	24.6	21.1	270
0	3	25	198.2	206.1	45
0	3	26	39.4	36.5	0
0	3	27	242.7	248.9	135
0	3	28	30.1	20.5	90
0	3	29	89.1	106.0	45
0	3	30	20.0	21.5	0
0	3	31	57.4	60.9	135
0	3	32	118.3	109.6	90
0	3	33	109.7	118.3	45
0	3	34	53.6	59.5	180
0	3	35	122.0	134.8	135
0	3	36	45.1	45.8	90
0	3	37	105.6	107.7	45
0	3	38	34.5	31.6	180
0	3	39	153.8	157.7	135
0	3	40	7.9	13.5	270
0	3	41	122.8	121.6	45
0	3	42	64.3	64.4	180
0	3	43	109.9	114.1	135
0	3	44	25.7	29.0	270
0	3	45	91.9	96.4	45
0	3	46	86.9	78.0	180
0	3	47	13.6	25.8	135
0	3	48	41.7	36.3	90
0	3	49	68.4	58.8	45
0	3	50	35.0	32.7	0
0	3	51	71.6	66.4	135
0	3	53	88.2	85.2	45
0	3	54	58.3	52.6	0
0	3	55	90.4	49.6	135
0	3	56	39.6	33.5	270
0	3	57	60.3	66.9	45
0	3	58	17.7	28.1	180
0	3	60	44.4	41.7	270
0	3	62	28.9	28.6	180
0	3	63	48.1	52.6	135
0	4	0	139.9	126.2	0
0	4	1	29.5	18.0	135
0	4	2	68.4	59.2	270
0	4	3	45.1	53.9	225
0	4	4	81.3	75.0	0
0	4	5	121.7	121.9	315
0	4	6	66.8	66.3	270
0	4	7	88.9	90.7	225
0	4	8	18.4	20.0	0
0	4	9	35.7	36.3	315
0	4	10	97.3	97.1	90
0	4	11	84.5	81.1	225
0	4	12	73.9	63.1	0
0	4	13	74.6	79.8	315
0	4	14	76.1	70.2	90
0	4	15	67.6	53.1	45
0	4	16	78.4	82.8	0
0	4	17	146.0	144.5	315
0	4	18	17.5	12.0	90
0	4	19	25.8	1.4	225
0	4	20	144.7	129.4	0
0	4	21	24.2	23.8	315
0	4	22	27.4	17.2	270
0	4	23	94.4	85.1	225
0	4	24	152.1	146.0	0
0	4	25	28.7	31.2	135
0	4	26	40.4	40.3	90
0	4	27	79.3	75.2	225
0	4	28	20.1	24.3	180
0	4	29	48.0	38.4	315
0	4	30	17.3	18.0	90
0	4	31	109.8	106.5	225
0	4	32	41.1	39.9	0
0	4	33	84.1	92.2	315
0	4	34	77.9	67.5	90
0	4	36	57.5	65.6	0
0	4	37	18.2	6.3	135
0	4	38	35.7	41.1	90
0	4	39	13.0	17.3	45
0	4	40	45.2	35.0	0
0	4	41	17.8	17.6	315
0	4	42	20.9	17.6	90
0	4	44	19.0	7.5	180
0	4	45	76.9	80.4	315
0	4	46	21.3	7.7	270
0	4	47	55.2	57.7	225
0	4	48	33.2	31.8	0
0	4	50	27.2	32.9	90
0	4	52	14.2	7.1	0
0	4	53	55.2	59.7	315
0	4	54	29.2	37.6	90
0	5	1	25.0	21.7	225
0	5	2	160.9	159.5	0
0	5	3	46.7	44.6	315
0	5	4	24.3	23.0	270
0	5	5	28.5	22.7	45
0	5	6	83.7	100.1	0
0	5	7	18.4	12.1	135
0	5	8	38.9	31.8	270
0	5	9	45.2	44.4	45
0	5	10	61.6	62.4	0
0	5	11	88.2	88.0	315
0	5	12	72.1	70.2	90
0	5	13	44.5	38.6	225
0	5	14	96.4	104.5	0
0	5	15	34.6	32.1	315
0	5	16	62.1	55.7	270
0	5	17	28.4	30.4	225
0	5	18	88.0	90.7	0
0	5	19	25.8	28.3	315
0	5	20	97.0	101.9	270
0	5	21	25.1	24.8	225
0	5	22	48.7	56.7	0
0	5	23	11.9	3.4	315
0	5	24	18.3	3.6	90
0	5	25	12.8	10.3	225
0	5	26	84.8	77.6	0
0	5	27	23.6	20.1	135
0	5	30	90.1	86.1	0
0	5	31	46.0	53.8	315
0	5	32	55.1	63.1	270
0	5	33	35.1	38.3	225
0	5	34	84.3	73.9	0
0	5	36	59.0	73.6	270
0	5	37	14.9	25.4	45
0	5	38	34.0	40.6	0
0	5	39	14.3	12.9	315
0	5	41	14.0	6.5	225
0	5	42	59.9	64.4	0
0	5	43	11.4	15.5	135
0	5	44	22.7	15.0	90
0	5	46	61.3	65.1	0
0	5	48	29.2	41.2	270
0	5	50	63.5	65.5	0
0	5	51	32.8	27.2	315
0	5	52	18.2	31.1	270
0	5	53	16.8	20.4	225
0	5	54	46.3	43.2	0
0	5	55	35.1	35.5	315
0	5	56	7.9	14.8	270
0	5	58	36.5	32.7	0
0	5	59	24.7	29.9	315
0	6	0	84.2	81.5	180
0	6	1	36.9	30.3	315
0	6	2	45.3	40.2	90
0	6	4	157.9	166.2	180
0	6	5	42.3	34.2	315
0	6	6	47.0	44.5	90
0	6	7	12.5	11.2	225
0	6	8	148.2	149.3	180
0	6	9	28.6	18.0	135
0	6	10	10.6	5.7	90
0	6	11	83.0	78.2	225
0	6	12	150.8	157.1	180
0	6	15	38.8	51.4	225
0	6	16	112.1	113.5	180
0	6	17	73.5	77.7	315
0	6	18	12.4	2.1	90
0	6	19	19.2	22.1	225
0	6	20	109.2	121.3	180
0	6	21	20.5	22.8	315
0	6	22	9.1	9.6	270
0	6	23	52.2	62.2	225
0	6	24	117.8	125.2	180
0	6	25	43.1	47.3	315
0	6	26	33.2	27.8	270
0	6	27	17.3	18.2	225
0	6	28	121.5	131.6	180
0	6	29	17.7	19.3	315
0	6	30	16.9	10.6	90
0	6	32	88.2	90.8	180
0	6	33	32.0	34.9	315
0	6	34	33.8	38.5	270
0	6	35	18.8	23.3	225
0	6	36	75.1	71.3	180
0	6	38	43.5	43.4	270
0	6	39	70.1	65.1	225
0	6	40	41.1	41.5	180
0	6	42	16.3	18.6	270
0	6	43	60.0	66.1	45
0	6	44	111.3	115.9	180
0	6	45	20.6	13.2	315
0	6	48	55.5	63.3	180
0	6	51	59.6	65.6	225
0	6	52	47.0	48.9	180
0	6	55	26.0	27.2	225
0	6	56	19.2	29.7	180
0	6	57	17.1	20.3	315
0	6	58	19.5	16.0	90
0	6	59	13.0	12.2	225
0	6	60	19.1	21.8	180
0	6	64	33.5	30.3	180
0	7	1	70.3	60.3	45
0	7	2	21.1	21.7	0
0	7	5	18.5	13.7	45
0	7	7	51.3	51.0	135
0	7	9	13.2	11.1	225
0	7	10	35.2	33.0	0
0	7	11	61.1	57.9	135
0	7	12	18.1	14.2	270
0	7	14	41.8	40.1	0
0	7	15	63.2	55.4	135
0	7	16	15.5	19.5	90
0	7	17	25.8	26.8	45
0	7	18	35.2	33.2	0
0	7	19	21.7	16.5	135
0	7	20	25.2	29.8	90
0	7	21	16.9	19.9	225
0	7	22	25.0	30.5	0
0	7	23	9.7	9.1	135
0	7	24	18.4	20.3	90
0	7	25	9.3	3.3	45
0	7	27	7.4	5.4	135
0	7	28	21.3	0.9	270
0	7	29	7.9	9.6	225
0	7	30	24.9	22.6	0
0	7	31	10.4	10.7	315
0	7	32	48.3	49.4	0
0	7	39	79.3	91.8	135
0	7	40	27.2	21.8	270
0	8	0	24.5	29.3	180
0	8	1	24.5	22.8	135
0	8	2	20.0	23.3	90
0	8	3	14.1	11.1	225
0	8	5	69.3	57.9	135
0	8	7	21.9	25.1	45
0	8	8	26.9	21.0	0
0	8	9	28.3	29.0	135
0	8	10	34.8	34.9	270
0	8	11	84.8	84.7	45
0	8	12	51.4	53.9	0
0	8	13	35.5	29.6	135
0	8	14	20.2	19.6	90
0	8	15	81.8	79.2	45
0	8	17	22.2	23.4	135
0	8	19	37.4	31.6	45
0	8	20	20.4	26.0	180
0	8	21	22.3	12.4	135
0	8	22	30.3	28.3	270
0	8	23	24.1	28.0	45
0	8	24	30.4	26.0	0
0	9	1	53.5	50.0	225
0	9	3	59.7	51.2	315
0	9	5	39.9	39.6	225
0	9	7	36.7	34.4	315
0	9	9	53.4	41.3	225
0	9	13	14.4	7.6	225
1	1	0	126.7	107.9	0
1	1	1	190.9	223.2	90
1	1	2	54.4	43.6	0
1	1	3	145.5	159.5	90
1	1	4	136.5	121.4	0
1	1	5	56.3	32.8	90
1	1	6	282.7	256.9	0
1	1	7	24.4	7.1	90
1	1	8	145.4	130.9	180
1	1	9	236.8	221.9	90
1	1	10	344.1	345.9	0
1	1	11	280.4	275.6	90
1	1	12	11.4	11.9	180
1	1	13	40.2	26.2	270
1	1	14	47.8	44.7	0
1	1	15	218.1	203.9	90
1	1	16	111.4	102.1	180
1	1	17	246.6	243.8	90
1	1	18	200.2	179.9	0
1	1	19	226.7	219.6	90
1	1	20	97.5	76.5	180
1	1	21	142.1	160.4	90
1	1	22	138.9	128.8	0
1	1	23	130.9	125.3	90
1	1	24	33.5	29.2	180
1	1	25	63.6	58.9	90
1	1	26	126.9	120.2	0
1	1	27	67.5	67.6	90
1	1	28	45.1	52.7	0
1	1	29	66.0	55.8	90
1	1	30	167.0	159.9	0
1	1	31	141.1	136.0	90
1	1	32	9.3	5.5	180
1	1	33	117.1	133.5	90
1	1	34	77.3	84.3	0
1	1	35	76.9	78.6	90
1	1	36	28.6	26.2	180
1	1	37	151.6	168.5	90
1	1	38	37.8	47.7	0
1	1	39	68.8	58.2	90
1	1	40	54.4	46.9	180
1	1	41	156.5	157.4	90
1	1	42	31.2	37.6	0
1	1	43	31.5	33.6	90
1	1	44	11.6	13.3	0
1	1	45	89.4	87.2	90
1	1	46	61.9	66.9	0
1	1	47	54.5	50.6	90
1	1	49	37.6	41.0	90
1	1	50	56.8	62.4	0
1	1	51	35.6	41.1	90
1	1	53	56.1	60.2	90
1	1	54	29.8	43.7	0
1	1	55	39.1	48.8	90
1	1	57	26.0	30.5	90
1	1	58	53.6	45.6	0
1	1	59	27.9	27.1	90
1	1	60	42.0	29.8	180
1	1	61	14.2	12.2	90
1	1	62	14.1	22.7	0
1	1	63	8.9	6.0	90
1	1	65	38.6	37.7	90
1	1	66	49.0	52.8	0
1	1	69	40.3	38.8	90
1	1	70	13.8	5.6	0
1	1	73	39.1	41.2	90
1	2	0	256.0	279.8	180
1	2	1	148.8	145.6	240
1	2	2	285.4	293.5	347
1	2	3	161.3	166.1	159
1	2	4	168.7	170.4	165
1	2	5	184.2	182.5	336
1	2	6	228.2	229.8	310
1	2	7	191.0	195.4	224
1	2	8	178.3	187.4	138
1	2	9	259.4	250.1	306
1	2	10	127.4	127.2	49
1	2	11	51.8	43.7	209
1	2	12	74.5	66.3	239
1	2	13	96.4	91.8	312
1	2	14	159.2	158.8	20
1	2	15	60.6	55.9	265
1	2	16	90.6	89.0	191
1	2	17	89.1	85.6	305
1	2	18	130.2	117.4	359
1	2	19	81.6	83.0	248
1	2	20	143.7	133.8	162
1	2	21	112.3	113.1	353
1	2	22	181.5	158.0	355
1	2	23	119.3	124.3	202
1	2	24	128.7	123.0	167
1	2	25	88.4	91.3	351
1	2	26	142.5	138.9	7
1	2	27	68.3	62.8	229
1	2	28	65.9	66.6	139
1	2	29	89.5	90.3	288
1	2	30	79.7	79.3	353
1	2	31	122.8	120.3	223
1	2	32	82.1	82.6	136
1	2	33	145.3	135.9	308
1	2	34	38.1	41.8	51
1	2	35	64.1	58.4	248
1	2	36	83.1	83.9	167
1	2	37	112.0	106.4	343
1	2	38	54.4	53.7	39
1	2	39	36.8	37.6	201
1	2	40	58.5	53.5	198
1	2	41	69.7	65.6	331
1	2	42	94.7	97.3	15
1	2	43	42.6	45.3	215
1	2	44	65.7	67.9	142
1	2	45	54.0	56.1	304
1	2	46	87.0	86.0	344
1	2	47	43.5	46.6	258
1	2	48	51.7	53.3	176
1	2	49	29.8	30.1	13
1	2	50	52.9	48.6	40
1	2	51	36.4	37.4	259
1	2	52	32.2	30.9	163
1	2	53	54.5	54.0	333
1	2	54	66.3	70.1	21
1	2	55	51.0	54.2	251
1	2	56	46.4	46.7	242
1	2	57	66.8	68.0	316
1	2	58	21.8	28.9	333
1	2	60	34.0	39.0	143
1	3	0	37.8	41.2	0
1	3	1	86.2	84.8	327
1	3	2	72.1	68.7	4
1	3	3	118.4	119.7	322
1	3	4	71.7	68.5	44
1	3	5	123.1	122.1	338
1	3	6	101.5	96.6	332
1	3	7	107.4	112.4	311
1	3	8	85.8	85.3	197
1	3	9	99.7	94.8	275
1	3	10	75.2	80.7	18
1	3	11	113.6	118.2	202
1	3	12	51.5	44.4	35
1	3	13	69.8	70.2	330
1	3	14	140.0	135.4	321
1	3	15	136.8	141.9	201
1	3	16	44.2	44.8	338
1	3	17	167.2	157.0	332
1	3	18	58.3	57.5	298
1	3	19	32.4	33.7	220
1	3	20	58.9	53.6	10
1	3	21	101.2	92.9	326
1	3	22	113.4	105.7	3
1	3	23	81.0	84.6	195
1	3	24	84.2	73.5	313
1	3	25	64.8	67.2	205
1	3	26	70.3	70.1	44
1	3	27	61.6	55.2	211
1	3	28	71.6	64.5	291
1	3	29	38.3	39.9	274
1	3	30	31.6	38.6	320
1	3	31	79.8	72.0	237
1	3	32	26.6	26.4	221
1	3	33	96.5	101.6	304
1	3	34	47.1	49.1	333
1	3	35	103.6	110.6	193
1	3	37	55.2	52.2	296
1	3	38	48.8	53.9	24
1	3	39	33.8	35.5	243
1	3	40	66.1	63.6	322
1	3	41	31.9	33.6	320
1	3	42	62.1	51.9	313
1	3	43	24.4	21.5	182
1	3	44	50.4	44.7	262
1	3	45	39.7	39.1	294
1	3	47	29.2	25.7	177
1	3	48	10.6	14.0	205
1	3	49	56.1	46.8	278
1	3	50	16.5	29.2	342
1	3	51	51.6	48.8	175
1	3	52	55.4	48.7	4
1	3	53	29.2	32.1	296
1	3	54	35.7	37.2	292
1	3	56	14.5	18.6	347
1	3	57	40.6	38.8	321
1	3	58	33.8	35.1	338
1	3	61	33.7	31.2	12
1	3	62	45.5	40.4	359
1	4	0	94.4	103.0	180
1	4	1	74.2	77.6	139
1	4	2	102.7	116.7	187
1	4	4	166.0	167.8	204
1	4	5	55.4	57.2	154
1	4	6	127.8	130.5	143
1	4	7	37.8	41.3	348
1	4	8	96.9	102.7	189
1	4	9	129.1	112.1	156
1	4	10	132.3	143.5	170
1	4	11	53.1	56.8	47
1	4	12	78.3	81.6	179
1	4	13	114.4	124.5	120
1	4	14	109.4	108.1	168
1	4	15	104.7	100.8	30
1	4	16	148.9	146.7	190
1	4	17	108.9	113.8	169
1	4	18	82.9	90.7	195
1	4	19	115.4	114.9	25
1	4	20	121.6	124.6	202
1	4	21	58.6	52.3	197
1	4	22	120.3	114.7	160
1	4	23	80.0	81.4	23
1	4	24	140.0	148.1	172
1	4	25	30.0	34.8	179
1	4	26	56.5	54.9	156
1	4	27	55.1	52.6	53
1	4	28	87.3	90.7	206
1	4	29	54.1	54.6	150
1	4	30	68.9	61.1	202
1	4	31	64.8	58.1	67
1	4	32	85.5	91.3	187
1	4	33	60.2	63.6	170
1	4	34	88.2	88.7	197
1	4	35	44.0	49.2	45
1	4	36	64.8	67.7	170
1	4	37	48.6	51.6	222
1	4	38	72.5	67.5	157
1	4	39	63.0	56.9	78
1	4	40	80.0	75.7	195
1	4	41	21.0	22.5	19
1	4	42	77.4	71.5	153
1	4	43	42.6	42.4	22
1	4	44	67.4	68.4	196
1	4	45	30.0	17.7	187
1	4	46	47.9	47.8	146
1	4	47	26.1	26.7	9
1	4	48	25.2	23.3	199
1	4	49	42.0	43.9	150
1	4	50	50.5	48.6	203
1	4	51	19.5	10.8	111
1	4	52	49.3	44.0	205
1	4	54	28.3	27.6	146
1	4	55	15.0	16.1	16
1	4	56	35.4	37.8	223
1	4	59	26.8	25.0	16
1	4	60	34.6	36.8	171
1	4	61	22.9	22.5	190
1	4	62	30.0	21.6	150
1	4	63	13.9	13.1	48
1	4	64	40.1	37.9	188
1	4	65	42.9	47.0	171
1	4	68	32.2	31.6	200
1	5	0	39.7	43.4	180
1	5	1	41.6	49.2	177
1	5	2	105.3	104.9	349
1	5	3	92.8	92.6	26
1	5	4	46.6	52.8	119
1	5	5	75.2	74.0	165
1	5	6	94.0	92.5	358
1	5	7	79.8	74.7	16
1	5	8	49.3	43.8	143
1	5	9	85.4	89.0	169
1	5	10	70.2	69.2	24
1	5	11	57.4	58.6	352
1	5	12	65.7	75.6	316
1	5	13	93.8	103.3	173
1	5	14	144.8	157.5	7
1	5	15	50.7	51.3	0
1	5	16	44.4	42.1	146
1	5	17	54.9	51.7	221
1	5	18	110.1	109.4	5
1	5	19	37.3	40.3	41
1	5	20	65.1	70.8	139
1	5	21	93.6	96.5	187
1	5	22	81.1	78.3	4
1	5	23	34.3	40.7	351
1	5	24	36.9	35.0	219
1	5	25	55.0	56.9	166
1	5	26	49.3	51.2	3
1	5	27	72.4	63.6	347
1	5	28	28.9	24.8	353
1	5	29	44.0	41.2	151
1	5	30	89.3	85.9	15
1	5	31	55.3	54.7	20
1	5	32	35.0	33.4	124
1	5	33	73.4	69.2	167
1	5	34	66.0	67.4	21
1	5	35	25.3	26.0	358
1	5	36	42.1	37.3	117
1	5	37	84.1	80.7	180
1	5	38	55.0	57.6	20
1	5	39	32.4	37.0	32
1	5	40	44.0	37.4	220
1	5	41	42.7	44.6	141
1	5	42	102.8	99.1	14
1	5	43	25.9	23.0	9
1	5	44	45.4	49.0	64
1	5	45	46.9	41.6	174
1	5	46	39.2	37.7	28
1	5	47	41.6	40.6	54
1	5	48	32.9	32.0	174
1	5	49	37.2	28.5	210
1	5	50	21.8	13.2	37
1	5	51	29.1	25.1	138
1	5	52	20.5	25.2	143
1	5	53	35.4	36.7	152
1	5	54	29.3	25.2	354
1	5	56	16.2	8.2	125
1	5	57	34.0	30.4	118
1	5	58	44.4	40.3	359
1	5	61	34.4	32.8	165
1	5	62	31.1	35.1	39
1	6	0	20.9	18.6	180
1	6	1	58.7	57.8	99
1	6	2	66.8	72.1	37
1	6	3	38.0	39.4	77
1	6	4	35.9	35.3	276
1	6	5	58.0	56.7	32
1	6	6	59.6	66.1	338
1	6	7	22.6	22.0	102
1	6	8	24.3	20.6	73
1	6	9	23.0	23.1	160
1	6	10	87.0	92.6	317
1	6	11	15.2	17.5	132
1	6	12	22.3	25.9	63
1	6	13	95.1	92.8	118
1	6	14	53.2	51.4	350
1	6	15	61.6	52.6	26
1	6	16	12.8	13.1	253
1	6	17	61.6	59.9	75
1	6	18	44.7	41.8	23
1	6	19	80.6	71.2	44
1	6	20	19.0	23.8	111
1	6	22	35.2	42.6	351
1	6	23	52.8	52.0	79
1	6	24	11.4	12.5	115
1	6	25	17.5	17.5	220
1	6	26	55.0	59.6	315
1	6	27	23.0	23.3	21
1	6	28	12.3	11.3	89
1	6	29	42.2	46.3	36
1	6	30	51.2	51.1	352
1	6	31	64.6	65.7	15
1	6	32	36.7	44.8	347
1	6	33	46.4	52.7	44
1	6	34	51.0	53.4	10
1	6	35	52.7	54.4	70
1	6	36	25.8	33.2	137
1	6	37	27.5	31.1	123
1	6	38	42.9	39.7	321
1	6	39	49.7	53.6	55
1	6	40	17.7	19.5	119
1	6	41	40.2	42.8	35
1	6	42	39.2	29.8	335
1	7	0	15.3	19.2	0
1	7	1	49.7	47.3	247
1	7	2	28.5	27.7	175
1	7	3	49.6	50.9	279
1	7	4	37.8	45.4	357
1	7	5	80.2	80.5	286
1	7	6	46.2	48.0	154
1	7	7	83.2	90.4	260
1	7	8	41.2	46.7	328
1	7	9	65.8	71.7	251
1	7	10	58.3	50.3	207
1	7	11	38.9	45.5	268
1	7	13	50.8	54.5	290
1	7	14	50.3	39.4	172
1	7	15	35.1	29.2	304
1	7	16	27.4	19.7	95
1	7	17	53.1	50.6	252
1	7	18	26.1	17.2	196
1	7	19	36.0	37.6	300
1	7	20	22.3	21.5	46
1	7	21	54.1	45.2	247
1	7	22	57.7	65.2	182
1	7	23	55.6	54.3	270
1	7	24	19.5	23.4	329
1	7	25	66.4	68.8	262
1	7	26	38.5	32.1	199
1	7	27	44.3	46.3	261
1	7	28	39.9	34.8	2
1	7	29	76.0	79.6	290
1	7	30	19.3	21.0	261
1	7	32	27.5	28.5	83
1	7	33	47.1	42.2	244
1	7	34	44.1	42.1	171
1	7	35	39.9	41.7	265
1	7	36	46.9	41.7	35
1	7	37	28.2	23.3	182
1	7	38	42.1	37.3	223
1	7	39	20.0	11.1	337
1	7	41	57.7	58.5	239
1	7	42	16.7	23.0	267
1	7	43	24.4	29.4	303
1	7	44	16.5	12.5	292
1	7	45	66.1	61.9	262
1	7	47	29.2	29.0	260
1	7	48	27.7	25.5	309
1	7	49	25.1	24.4	286
1	7	50	20.2	12.8	163
1	7	51	32.6	30.7	265
1	7	52	23.2	31.4	18
1	7	53	23.5	27.5	310
1	7	54	30.6	25.6	178
1	8	1	46.0	44.8	164
1	8	2	48.0	54.3	148
1	8	3	46.0	44.6	22
1	8	4	24.0	32.6	77
1	8	5	19.6	19.5	163
1	8	6	27.6	34.6	162
1	8	7	31.1	27.1	67
1	8	8	32.4	35.6	359
1	8	9	46.2	49.8	164
1	8	10	45.0	44.0	249
1	8	11	45.0	40.7	100
1	8	13	48.3	51.1	139
1	8	14	31.3	26.3	190
1	8	15	42.1	46.9	24
1	8	16	14.0	25.1	232
1	8	18	42.2	36.5	178
1	8	19	46.3	42.1	358
1	9	3	20.0	22.8	14
1	9	4	35.8	33.5	231
1	9	5	8.9	17.2	23
1	9	6	21.9	20.7	108
1	9	8	20.5	13.4	209
1	9	9	43.4	40.7	173
1	9	11	17.8	11.1	38
1	9	12	21.8	16.8	115
1	9	13	44.6	30.1	164
1	9	15	8.9	11.5	50
1	9	17	14.0	7.7	267
1	9	18	8.9	10.9	168
2	2	0	446.0	441.7	180
2	2	1	141.7	138.6	90
2	2	2	35.3	19.9	180
2	2	3	58.7	53.5	90
2	2	4	327.6	309.7	180
2	2	5	25.3	31.3	90
2	2	6	88.2	73.9	0
2	2	7	175.8	179.0	90
2	2	8	57.2	44.3	0
2	2	9	137.2	134.0	90
2	2	10	28.8	26.3	180
2	2	11	155.7	142.4	90
2	2	12	209.5	192.9	180
2	2	13	201.7	184.3	90
2	2	14	174.7	140.6	180
2	2	15	44.1	34.1	90
2	2	16	175.2	161.0	180
2	2	17	96.5	90.0	90
2	2	18	30.6	23.2	180
2	2	19	106.9	99.9	90
2	2	20	205.0	199.2	180
2	2	21	65.0	64.5	90
2	2	22	47.5	39.3	180
2	2	23	129.2	118.5	90
2	2	24	119.4	96.9	180
2	2	25	63.1	50.3	90
2	2	26	14.0	15.3	180
2	2	27	232.1	218.2	90
2	2	28	77.8	65.5	180
2	2	29	113.6	100.3	90
2	2	30	97.4	89.2	180
2	2	31	48.2	45.6	90
2	2	32	128.3	115.7	180
2	2	33	77.2	77.1	90
2	2	35	28.5	27.6	90
2	2	36	119.1	114.2	180
2	2	37	7.6	8.1	90
2	2	38	36.5	26.0	180
2	2	39	12.4	19.0	90
2	2	40	88.2	86.9	180
2	2	41	56.7	54.2	90
2	2	42	32.4	35.5	180
2	2	43	89.0	87.1	90
2	2	44	96.8	84.1	180
2	2	45	54.4	51.8	90
2	2	46	42.2	43.0	180
2	2	47	92.4	87.6	90
2	2	49	54.3	46.7	90
2	2	50	16.2	15.1	180
2	2	52	38.9	42.5	180
2	2	54	31.6	33.3	0
2	2	55	43.0	41.6	90
2	2	57	34.4	32.4	90
2	2	58	9.8	15.9	0
2	2	59	45.4	42.7	90
2	2	60	57.2	58.2	180
2	2	64	42.9	41.0	180
2	2	65	32.6	31.7	90
2	2	66	27.6	25.9	180
2	3	0	109.6	106.9	0
2	3	1	120.5	106.8	181
2	3	2	48.6	46.5	83
2	3	3	90.7	99.8	308
2	3	4	91.4	85.9	344
2	3	5	88.5	88.8	219
2	3	6	71.3	67.3	137
2	3	7	91.1	80.1	261
2	3	8	148.0	142.0	15
2	3	9	57.7	51.3	250
2	3	10	55.1	50.8	336
2	3	11	27.0	18.8	243
2	3	12	169.4	155.7	342
2	3	13	119.2	100.4	142
2	3	14	100.5	80.3	80
2	3	15	110.1	119.7	30
2	3	16	126.4	130.1	337
2	3	17	49.8	44.9	155
2	3	18	90.7	77.6	94
2	3	19	40.5	37.8	278
2	3	20	145.8	134.5	35
2	3	21	71.3	61.7	230
2	3	22	87.8	74.3	329
2	3	23	87.8	79.2	342
2	3	24	137.5	113.8	23
2	3	25	23.0	20.9	242
2	3	26	44.3	41.5	328
2	3	27	51.8	44.0	53
2	3	28	105.1	95.1	343
2	3	29	31.1	22.1	155
2	3	30	64.8	62.6	120
2	3	31	45.8	48.4	174
2	3	32	92.5	90.1	7
2	3	33	53.6	48.7	251
2	3	34	22.7	17.2	152
2	3	35	49.9	47.1	250
2	3	36	110.6	105.1	27
2	3	37	48.4	46.7	244
2	3	38	33.1	26.6	133
2	3	39	46.3	50.3	354
2	3	40	76.3	68.7	4
2	3	41	21.1	13.4	184
2	3	42	58.3	53.7	97
2	3	43	55.1	51.1	28
2	3	44	76.3	69.0	348
2	3	45	28.7	29.0	241
2	3	46	13.7	10.5	127
2	3	47	14.2	17.6	9
2	3	48	54.4	46.2	66
2	3	49	77.7	67.8	235
2	3	52	9.6	12.7	201
2	3	54	9.4	19.6	100
2	3	55	46.7	44.7	76
2	3	56	50.9	45.6	355
2	3	57	31.1	30.0	94
2	3	58	48.6	48.2	77
2	3	59	71.3	68.3	25
2	4	0	118.3	122.5	0
2	4	1	130.6	132.2	207
2	4	2	50.2	46.5	208
2	4	3	92.4	90.3	357
2	4	4	113.9	117.2	11
2	4	5	132.3	132.5	170
2	4	6	40.5	45.4	302
2	4	7	93.7	96.4	27
2	4	9	109.8	101.0	161
2	4	10	90.7	77.0	19
2	4	11	153.0	149.5	2
2	4	12	86.6	96.4	317
2	4	13	53.8	55.9	128
2	4	14	72.6	60.7	296
2	4	15	115.5	115.7	21
2	4	16	108.0	102.0	3
2	4	17	56.1	48.9	148
2	4	18	47.0	50.3	253
2	4	19	73.8	70.7	29
2	4	20	77.0	84.5	351
2	4	21	131.5	129.9	169
2	4	22	101.8	97.2	112
2	4	23	69.4	64.5	33
2	4	24	111.9	108.7	322
2	4	25	85.0	81.3	165
2	4	26	45.4	42.8	4
2	4	27	125.2	132.0	353
2	4	28	92.4	87.7	134
2	4	30	22.6	24.1	343
2	4	31	99.7	98.5	13
2	4	32	93.8	93.3	343
2	4	33	26.0	23.2	163
2	4	34	40.5	34.4	342
2	4	35	64.2	55.5	2
2	4	36	30.8	23.7	10
2	4	37	81.1	76.2	157
2	4	39	65.4	64.8	2
2	4	40	51.2	48.4	24
2	4	41	40.5	33.6	161
2	4	42	25.3	20.8	10
2	4	43	64.1	60.3	6
2	4	44	60.4	59.7	8
2	4	45	43.1	45.0	217
2	4	46	14.8	15.7	322
2	4	47	30.9	18.8	6
2	4	48	53.3	49.4	339
2	4	49	49.4	49.5	199
2	4	51	67.0	72.5	24
2	4	52	29.7	33.8	307
2	4	53	25.4	29.0	83
2	4	54	31.1	36.5	242
2	4	55	62.3	58.0	6
2	4	56	62.3	64.7	339
2	4	57	15.1	7.8	100
2	4	58	9.9	14.0	327
2	5	0	42.2	49.0	180
2	5	1	59.5	65.7	262
2	5	2	80.0	83.6	160
2	5	3	77.9	93.2	293
2	5	4	62.3	63.1	184
2	5	5	82.4	88.9	249
2	5	6	20.3	15.7	94
2	5	7	79.1	85.0	265
2	5	8	36.0	37.6	181
2	5	9	73.6	70.9	294
2	5	10	73.3	76.4	152
2	5	11	71.9	78.2	326
2	5	12	54.2	59.2	175
2	5	13	116.0	123.4	241
2	5	14	100.7	111.4	182
2	5	15	65.8	58.7	277
2	5	16	68.2	80.1	137
2	5	17	39.5	39.4	165
2	5	18	51.1	51.3	271
2	5	19	74.6	69.6	295
2	5	20	96.4	101.2	192
2	5	21	69.6	69.6	245
2	5	22	52.4	49.6	173
2	5	23	66.5	69.0	270
2	5	24	64.6	70.4	177
2	5	25	72.9	76.5	245
2	5	26	49.2	49.8	210
2	5	27	82.0	83.8	324
2	5	28	10.4	12.3	197
2	5	29	46.7	44.4	252
2	5	30	24.7	27.3	21
2	5	31	68.1	61.7	315
2	5	32	37.4	33.1	186
2	5	33	64.7	58.9	267
2	5	34	48.8	41.6	187
2	5	35	38.3	30.7	271
2	5	36	39.1	34.4	157
2	5	37	60.9	61.7	204
2	5	38	41.5	52.8	143
2	5	39	58.4	56.5	284
2	5	40	50.7	53.2	160
2	5	41	27.9	30.9	223
2	5	42	18.1	23.6	246
2	5	43	59.6	60.3	287
2	5	44	37.6	36.3	225
2	5	45	19.0	11.0	299
2	5	46	30.3	29.7	264
2	5	47	45.4	44.8	270
2	5	48	34.0	32.5	211
2	5	49	62.7	65.0	268
2	5	50	56.8	52.7	177
2	5	51	55.8	58.4	288
2	5	52	23.4	21.8	168
2	5	53	51.9	50.2	236
2	5	54	37.1	35.7	197
2	5	55	31.1	29.6	268
2	5	56	30.3	29.3	97
2	6	0	37.9	44.4	0
2	6	1	34.1	26.8	18
2	6	2	41.7	41.4	195
2	6	3	66.5	65.9	104
2	6	4	68.1	57.3	38
2	6	5	39.2	42.0	0
2	6	6	11.0	7.6	304
2	6	7	79.1	71.1	119
2	6	8	37.1	33.3	27
2	6	9	50.0	49.2	97
2	6	10	14.8	17.8	198
2	6	11	81.5	79.5	168
2	6	12	37.4	33.4	297
2	6	13	56.4	46.1	80
2	6	14	57.7	54.3	217
2	6	15	70.8	65.0	142
2	6	16	34.7	27.7	80
2	6	17	24.3	13.2	51
2	6	18	22.7	20.4	185
2	6	19	64.9	64.2	167
2	6	20	10.5	14.9	126
2	6	21	20.2	22.2	59
2	6	22	19.5	23.1	198
2	6	23	32.4	29.7	162
2	6	24	18.3	20.2	335
2	6	25	42.9	41.9	30
2	6	26	8.9	8.7	125
2	6	27	25.3	24.6	161
2	6	28	34.3	26.3	28
2	6	29	24.3	21.2	64
2	6	30	38.2	39.1	233
2	6	31	58.8	60.8	111
2	6	32	68.1	68.3	21
2	6	33	35.3	30.6	83
2	6	35	61.5	56.6	139
2	6	37	42.6	31.2	90
2	6	39	38.7	41.0	207
2	6	40	18.4	11.5	10
2	6	41	29.8	23.5	99
2	6	42	38.3	34.0	223
2	6	43	26.7	20.2	130
2	6	44	26.8	28.2	340
2	6	46	19.2	13.4	212
2	6	47	32.9	32.4	123
2	7	0	55.7	58.3	0
2	7	1	40.9	39.1	80
2	7	2	66.3	68.0	166
2	7	3	17.5	18.7	74
2	7	4	46.7	48.3	22
2	7	5	28.9	21.3	15
2	7	6	42.2	40.2	162
2	7	7	48.7	49.7	120
2	7	8	37.6	34.0	19
2	7	9	36.6	36.9	102
2	7	10	56.2	56.3	240
2	7	11	51.0	59.6	130
2	7	12	27.8	34.2	326
2	7	13	61.7	51.3	81
2	7	14	61.6	57.8	203
2	7	15	29.2	30.0	29
2	7	17	61.6	57.1	37
2	7	18	118.1	114.3	167
2	7	19	18.2	19.9	82
2	7	20	32.4	21.0	94
2	7	21	16.5	19.5	333
2	7	22	55.7	54.8	163
2	7	23	31.8	34.2	110
2	7	24	42.5	40.6	18
2	7	25	19.2	18.4	159
2	7	26	19.3	18.7	183
2	7	27	13.0	26.2	354
2	7	28	23.5	26.5	359
2	7	29	66.5	61.9	41
2	7	30	59.5	59.2	153
2	7	31	17.3	21.5	28
2	7	32	34.8	35.7	48
2	7	33	42.2	38.7	43
2	7	34	34.7	33.2	154
2	7	35	57.2	59.0	105
2	7	36	15.9	12.2	355
2	7	37	21.4	20.4	331
2	7	38	51.5	48.2	121
2	7	39	22.5	22.0	126
2	7	40	14.6	7.7	345
2	7	41	32.4	27.5	44
2	7	42	45.6	48.6	172
2	7	43	14.6	21.8	44
2	7	44	39.6	36.8	20
2	7	45	32.7	34.8	45
2	7	46	35.2	40.7	177
2	7	47	26.9	24.9	106
2	7	48	26.2	23.0	42
2	7	49	26.6	29.3	91
2	7	50	27.3	30.5	165
2	7	51	10.4	9.2	102
2	7	52	9.2	13.7	90
2	7	53	16.0	19.4	48
2	7	54	18.8	21.0	163
2	8	1	43.5	45.2	288
2	8	2	26.7	30.9	10
2	8	3	53.3	53.4	297
2	8	4	40.7	41.1	13
2	8	5	77.2	73.7	4
2	8	9	21.9	29.8	205
2	8	10	23.9	24.8	32
2	8	11	15.4	28.4	199
2	8	12	21.9	25.2	330
2	8	14	38.0	30.3	28
2	8	15	22.0	23.2	336
2	8	16	30.8	36.9	344
2	8	17	9.8	12.9	14
2	8	19	56.2	46.6	328
2	8	20	39.5	37.3	8
2	8	23	58.4	54.1	239
2	8	24	57.6	56.7	351
2	8	25	35.0	40.4	278
2	8	26	44.2	43.8	3
2	8	27	15.6	9.3	151
2	8	28	34.9	33.1	15
2	8	29	22.1	17.5	320
2	8	30	28.7	27.6	37
2	8	31	38.1	34.9	293
2	8	32	45.0	42.7	338
2	8	33	9.8	11.4	147
2	8	34	21.1	26.6	56
2	8	35	23.8	21.6	307
2	8	36	21.7	15.7	8
2	8	38	21.5	18.0	10
2	8	40	45.0	37.9	12
2	8	42	14.7	17.2	7
2	8	43	18.5	17.3	306
2	8	44	34.4	32.6	342
2	8	45	14.3	18.1	291
2	8	46	20.0	25.7	37
2	8	47	19.8	20.3	299
3	3	0	41.6	41.9	180
3	3	1	90.8	76.5	270
3	3	2	188.1	218.0	180
3	3	3	151.2	145.0	270
3	3	4	34.7	32.1	0
3	3	5	48.7	49.1	270
3	3	6	163.2	178.6	180
3	3	7	130.5	134.3	270
3	3	8	36.6	35.3	180
3	3	9	51.9	50.5	90
3	3	10	236.0	250.6	180
3	3	11	51.9	62.1	90
3	3	12	12.7	17.9	0
3	3	13	27.9	22.6	270
3	3	14	202.0	197.6	180
3	3	15	28.7	26.4	90
3	3	16	59.3	55.1	180
3	3	17	112.9	99.4	90
3	3	18	275.0	262.2	180
3	3	19	68.0	65.4	270
3	3	20	44.7	36.6	0
3	3	21	97.8	78.4	270
3	3	22	200.7	206.6	180
3	3	23	29.5	24.4	270
3	3	24	15.0	15.2	180
3	3	25	29.5	31.3	90
3	3	26	194.1	189.6	180
3	3	27	15.6	13.4	90
3	3	28	65.5	61.3	0
3	3	30	157.0	148.3	180
3	3	31	55.1	48.7	270
3	3	32	23.5	22.1	180
3	3	33	35.0	36.7	270
3	3	34	143.0	131.5	180
3	3	35	72.5	62.1	270
3	3	36	23.7	27.9	180
3	3	37	66.7	61.2	270
3	3	38	98.6	104.8	180
3	3	39	57.2	52.5	90
3	3	40	48.3	45.0	180
3	3	41	73.3	67.3	270
3	3	42	77.0	64.3	180
3	3	45	30.3	31.9	270
3	3	46	122.5	124.1	180
3	3	47	91.9	78.2	270
3	3	49	18.5	11.9	270
3	3	50	73.8	69.3	180
3	3	51	36.4	35.3	270
3	3	52	39.5	31.5	0
3	3	54	65.1	57.3	180
3	3	56	25.8	25.4	180
3	3	57	27.8	46.5	270
3	3	58	40.8	48.2	180
3	3	60	21.4	17.1	0
3	4	0	34.9	38.4	0
3	4	1	37.2	41.0	250
3	4	2	52.9	56.0	138
3	4	3	29.0	30.8	30
3	4	4	103.6	103.1	348
3	4	5	53.8	55.1	98
3	4	6	71.4	79.7	91
3	4	7	38.9	36.4	28
3	4	8	105.4	108.4	350
3	4	9	120.2	124.8	120
3	4	10	40.5	31.3	65
3	4	11	78.0	85.0	104
3	4	12	65.5	57.9	8
3	4	13	65.4	53.1	103
3	4	14	37.9	20.8	57
3	4	15	35.5	29.2	110
3	4	16	27.2	18.0	19
3	4	17	23.8	18.2	173
3	4	18	25.5	20.2	195
3	4	19	39.9	29.7	117
3	4	20	70.5	71.1	341
3	4	21	94.1	87.0	149
3	4	22	30.5	25.2	279
3	4	23	22.0	24.2	112
3	4	24	27.8	22.8	61
3	4	25	56.0	51.2	107
3	4	26	34.9	25.5	43
3	4	27	16.3	13.4	73
3	4	28	35.7	31.0	5
3	4	29	35.3	30.0	112
3	4	30	20.9	21.3	299
3	4	31	37.5	23.6	79
3	4	32	38.2	29.3	23
3	4	33	59.7	54.7	132
3	4	34	20.3	20.4	35
3	4	35	26.0	20.6	254
3	4	36	58.4	50.9	29
3	4	37	97.3	86.9	158
3	4	38	16.3	20.6	136
3	4	39	40.5	25.4	148
3	4	40	67.1	67.6	350
3	4	41	49.9	41.1	157
3	4	42	14.4	11.9	87
3	4	43	37.2	23.7	135
3	4	44	36.4	35.3	8
3	4	45	46.3	44.3	71
3	4	46	20.8	24.0	267
3	4	47	22.7	27.0	66
3	4	48	9.3	17.7	339
3	4	49	45.7	41.4	152
3	4	50	28.3	20.7	350
3	4	51	28.6	32.7	131
3	4	52	21.4	27.4	50
3	4	53	39.5	31.7	137
3	5	0	16.2	20.3	180
3	5	1	102.7	114.3	11
3	5	2	10.9	9.4	280
3	5	4	13.0	12.2	355
3	5	5	32.9	33.7	36
3	5	6	47.3	50.5	297
3	5	7	63.1	65.7	102
3	5	8	13.7	14.7	239
3	5	9	66.2	60.8	39
3	5	10	60.2	61.0	2
3	5	11	66.5	71.9	107
3	5	12	56.9	56.4	155
3	5	13	97.8	100.8	55
3	5	14	44.4	38.1	18
3	5	15	41.9	37.3	123
3	5	16	31.8	28.9	181
3	5	17	53.1	48.3	67
3	5	18	20.3	19.5	106
3	5	19	28.6	32.0	148
3	5	20	39.8	37.9	7
3	5	21	66.3	53.4	3
3	5	22	44.4	26.5	297
3	5	23	41.9	44.3	116
3	5	24	16.6	18.9	206
3	5	25	83.2	74.6	43
3	5	26	24.3	20.8	320
3	5	27	29.1	23.4	103
3	5	28	37.4	33.8	152
3	5	29	86.4	72.6	29
3	5	30	43.9	33.0	50
3	5	31	21.0	13.6	37
3	5	32	20.3	12.5	60
3	5	33	71.4	62.7	38
3	5	34	26.4	24.9	29
3	5	35	46.7	35.7	82
3	5	36	19.7	24.7	124
3	5	37	92.3	81.0	33
3	5	38	20.2	21.0	325
3	5	39	15.5	21.0	105
3	5	40	41.9	26.3	131
3	5	41	90.7	80.3	34
3	5	42	20.4	23.8	8
3	5	43	28.3	20.6	41
3	5	44	25.3	22.2	92
3	5	45	44.5	37.2	45
3	5	46	17.2	13.6	315
3	5	47	41.8	34.3	33
3	5	49	22.8	19.5	91
3	5	50	9.6	7.1	276
3	5	51	38.4	31.3	189
3	5	52	38.0	27.4	148
3	5	53	15.3	30.4	71
3	5	54	15.4	18.8	96
3	6	0	49.5	45.7	0
3	6	1	90.8	85.8	310
3	6	2	57.0	57.2	357
3	6	3	101.3	93.6	247
3	6	4	60.9	60.5	53
3	6	5	86.1	81.6	311
3	6	6	26.4	26.7	360
3	6	7	81.3	77.3	217
3	6	8	35.3	36.9	290
3	6	9	85.3	80.1	316
3	6	10	51.2	49.9	290
3	6	11	97.7	95.6	212
3	6	12	64.8	58.3	339
3	6	13	95.3	90.8	301
3	6	14	21.1	17.1	214
3	6	15	50.4	52.8	209
3	6	16	39.9	33.3	44
3	6	17	89.4	89.7	309
3	6	18	14.6	16.0	30
3	6	19	100.6	97.7	216
3	6	20	32.1	33.1	57
3	6	21	66.9	66.0	333
3	6	22	42.0	39.1	337
3	6	23	106.1	98.7	224
3	6	24	53.4	57.9	346
3	6	25	64.3	57.6	266
3	6	26	14.0	13.3	273
3	6	27	58.6	53.3	232
3	6	28	49.7	52.7	30
3	6	29	79.8	80.5	312
3	6	30	42.9	49.1	71
3	6	31	59.3	60.5	262
3	6	32	20.8	17.9	11
3	6	33	65.7	68.1	321
3	6	34	18.0	19.6	29
3	6	35	55.4	42.9	217
3	6	36	[illegible]	[illegible]	345
3	6	37	29.1	33.2	338
3	6	38	41.5	43.1	310
3	6	39	25.2	23.7	208
3	6	40	45.4	39.8	1
3	6	41	26.7	25.6	310
3	6	42	17.1	15.0	116
3	6	43	56.7	49.8	260
3	6	45	47.9	42.5	340
3	6	46	14.4	10.0	5
3	6	47	56.6	54.8	217
3	6	49	37.2	33.4	305
3	6	50	33.5	30.5	340
3	6	51	26.5	27.4	234
3	6	52	13.6	15.3	322
3	6	53	48.7	42.7	274
3	6	55	30.9	28.4	228
3	6	56	18.3	18.5	348
3	6	57	30.8	36.1	275
3	7	1	17.7	19.2	21
3	7	2	44.3	42.9	169
3	7	3	11.4	10.4	80
3	7	4	19.7	17.9	108
3	7	5	23.5	24.1	92
3	7	6	14.0	20.9	162
3	7	7	21.0	22.0	178
3	7	8	33.1	30.9	188
3	7	9	18.2	24.2	132
3	7	10	20.7	28.2	210
3	7	11	40.8	31.8	105
3	7	12	37.6	27.1	285
3	7	13	42.4	45.8	39
3	7	14	30.9	35.8	181
3	7	15	76.7	69.5	87
3	7	16	32.4	33.7	253
3	7	17	32.1	24.7	13
3	7	18	49.5	48.3	288
3	7	19	28.5	25.5	217
3	8	1	42.7	46.5	202
3	8	2	15.1	17.3	31
3	8	4	21.3	20.2	159
3	8	6	19.1	21.4	115
3	8	7	26.2	10.4	231
3	8	8	71.0	58.7	167
3	8	10	15.1	17.6	212
3	8	11	30.3	26.5	351
3	8	12	84.0	79.7	178
3	8	13	15.1	26.1	116
3	8	15	26.3	31.5	80
4	4	0	10.4	20.4	180
4	4	1	58.2	67.8	270
4	4	2	44.1	49.2	0
4	4	3	123.2	137.2	270
4	4	4	33.0	34.2	180
4	4	5	57.1	56.1	270
4	4	6	27.8	23.9	180
4	4	7	129.4	132.9	270
4	4	8	67.1	80.4	180
4	4	9	70.5	67.2	270
4	4	10	37.8	39.5	0
4	4	11	70.1	81.8	270
4	4	12	65.9	67.8	180
4	4	13	87.0	87.6	270
4	4	14	58.6	56.4	0
4	4	15	47.9	39.4	270
4	4	16	22.5	28.2	180
4	4	17	83.6	89.6	270
4	4	18	89.6	88.7	0
4	4	19	78.2	80.4	270
4	4	20	70.9	75.9	180
4	4	21	23.6	25.3	270
4	4	22	16.9	15.7	0
4	4	23	97.3	80.6	270
4	4	25	69.0	64.4	270
4	4	26	19.3	16.2	180
4	4	27	107.7	98.2	270
4	4	28	25.4	22.1	180
4	4	29	88.8	87.2	270
4	4	30	18.3	16.2	0
4	4	31	103.4	108.2	270
4	4	32	59.6	52.1	180
4	4	33	68.5	68.1	270
4	4	34	19.1	15.3	0
4	4	35	45.4	41.3	270
4	4	36	70.5	69.4	180
4	4	37	48.4	42.1	270
4	4	38	59.7	54.8	0
4	4	39	22.0	23.3	90
4	4	40	47.6	46.0	180
4	4	41	50.1	46.2	270
4	4	42	74.4	66.2	0
4	4	43	58.7	54.7	270
4	4	45	21.0	20.4	270
4	4	46	31.2	30.3	0
4	4	47	113.5	104.0	270
4	4	48	28.4	27.6	180
4	4	50	45.3	42.6	180
4	4	51	59.7	49.2	270
4	4	53	35.7	44.1	270
4	4	55	60.2	53.2	270
4	4	57	47.2	42.6	270
4	4	59	35.1	29.4	270
4	5	0	73.1	89.9	180
4	5	1	54.6	58.2	45
4	5	2	28.3	32.3	14
4	5	3	61.7	65.6	145
4	5	4	62.6	70.6	187
4	5	5	37.3	38.7	30
4	5	6	30.2	27.6	351
4	5	7	59.8	62.9	99
4	5	8	61.8	66.6	195
4	5	9	21.9	21.6	27
4	5	10	13.3	15.3	15
4	5	11	75.2	80.3	132
4	5	12	62.7	76.6	195
4	5	13	24.5	23.5	55
4	5	14	21.5	18.8	295
4	5	15	63.2	63.4	148
4	5	16	49.7	47.9	123
4	5	17	51.2	53.5	52
4	5	18	68.1	77.9	328
4	5	19	60.6	56.9	166
4	5	20	68.4	65.2	204
4	5	21	37.2	34.9	69
4	5	22	59.8	60.1	6
4	5	23	67.2	60.1	161
4	5	24	45.6	41.0	208
4	5	25	10.6	9.8	43
4	5	26	33.7	35.2	18
4	5	27	58.1	48.8	172
4	5	28	33.3	96.5	183
4	5	30	11.2	14.7	343
4	5	31	40.3	35.5	121
4	5	32	67.2	69.0	167
4	5	33	55.0	41.7	113
4	5	34	49.9	47.8	287
4	5	35	35.9	25.0	77
4	5	36	17.5	23.9	196
4	5	37	79.0	64.8	95
4	5	38	28.6	27.8	305
4	5	39	39.3	40.0	136
4	5	40	37.4	26.8	264
4	5	41	34.9	19.8	56
4	5	43	59.6	62.2	177
4	5	44	41.3	41.6	193
4	6	1	48.1	49.3	50
4	6	3	35.7	30.0	140
4	6	4	57.1	60.2	168
4	6	5	37.5	35.0	2
4	6	6	38.6	43.0	164
4	6	7	22.4	21.1	60
4	6	8	29.2	34.2	201
4	6	9	35.0	32.7	10
4	6	11	24.9	21.9	135
4	6	12	46.9	47.9	197
4	6	13	32.3	38.7	51
4	6	14	27.7	32.4	241
4	6	15	17.0	21.6	131
4	6	16	24.6	25.2	211
4	6	17	28.7	31.4	94
4	6	18	21.7	18.9	172
4	6	19	28.0	26.1	48
4	6	20	25.6	23.1	180
4	6	21	29.7	27.7	227
4	6	22	36.7	32.5	115
4	6	23	19.0	14.3	83
4	6	24	54.4	50.2	91
4	6	25	22.8	29.6	331
4	6	26	29.2	24.3	146
4	6	27	20.6	27.3	200
4	6	28	61.4	58.8	119
4	6	29	29.0	26.2	52
4	6	30	49.9	35.9	143
4	6	31	37.7	38.9	97
4	6	32	28.6	27.4	163
4	6	33	30.3	32.9	41
4	6	34	36.0	32.8	194
4	6	35	23.2	20.1	100
4	6	36	24.6	25.0	193
4	6	37	33.3	31.1	343
4	6	38	14.8	17.2	87
4	6	39	20.1	21.0	120
4	6	43	30.2	19.0	103
4	6	44	46.8	43.4	136
4	7	0	28.2	26.0	0
4	7	1	31.1	35.5	187
4	7	2	65.8	69.7	355
4	7	3	36.9	43.9	333
4	7	4	16.3	16.8	35
4	7	5	50.8	45.9	201
4	7	6	49.2	57.9	338
4	7	7	24.4	25.0	294
4	7	8	42.2	41.5	14
4	7	9	35.9	36.4	168
4	7	10	31.1	35.5	45
4	7	11	31.0	31.8	32
4	7	12	48.1	47.9	11
4	7	13	17.4	13.4	132
4	7	14	39.1	34.4	357
4	7	15	14.6	18.2	60
4	7	16	53.7	55.2	357
4	7	17	24.0	19.3	65
4	7	18	33.6	36.4	19
4	7	19	30.7	40.8	20
4	7	20	53.7	59.0	357
4	7	21	23.4	29.0	205
4	7	22	39.0	44.8	15
4	7	23	23.3	19.1	256
4	7	24	32.1	32.5	26
4	7	25	30.6	25.0	244
4	7	26	47.4	43.5	1
4	7	28	47.5	43.3	351
4	7	29	28.2	34.9	122
4	7	30	49.2	43.8	36
4	7	32	34.7	27.7	351
4	7	34	41.1	43.0	359
4	7	35	27.2	28.7	14
4	7	36	37.1	28.6	310
4	7	37	15.3	18.8	182
4	7	38	26.2	25.2	318
4	7	39	32.6	32.6	46
4	7	40	28.8	28.4	1
4	7	42	27.9	25.7	3
4	7	43	25.2	22.0	13
4	7	44	29.1	24.9	1
4	7	46	39.3	33.3	358
4	8	0	37.0	35.9	180
4	8	1	21.4	16.7	163
4	8	2	21.4	17.5	67
4	8	3	23.4	11.1	129
4	8	4	30.2	34.8	221
4	8	5	21.4	22.8	182
4	8	6	15.1	5.3	264
4	8	7	52.4	54.0	325
4	8	8	50.4	35.9	283
4	8	9	23.4	22.8	211
4	8	10	19.1	10.5	295
4	8	11	23.4	31.5	331
4	8	12	41.2	42.3	161
4	8	13	30.2	25.0	180
4	8	14	25.7	34.4	90
4	8	15	21.3	13.5	330
4	8	16	21.3	24.5	155
4	8	17	19.1	4.9	254
4	8	18	15.1	22.3	39
4	8	19	30.1	24.1	338
4	8	20	21.2	27.4	190
4	8	22	49.8	46.3	288
4	8	23	45.0	35.5	339
4	8	24	37.9	45.6	211
4	8	25	14.9	12.5	177
4	8	27	18.7	15.5	2
4	8	28	42.9	40.1	223
4	8	29	28.0	26.7	269
4	8	31	27.8	26.5	307
4	8	32	40.5	19.7	131
5	5	0	16.0	13.6	180
5	5	1	62.2	72.9	270
5	5	2	55.7	68.3	0
5	5	3	14.7	13.5	270
5	5	4	47.7	56.3	0
5	5	5	90.2	89.1	270
5	5	6	48.3	52.2	0
5	5	8	56.4	56.5	0
5	5	9	14.9	15.6	270
5	5	10	63.6	66.8	0
5	5	11	93.9	105.5	270
5	5	13	30.3	40.9	270
5	5	14	95.3	115.5	0
5	5	15	36.0	41.0	270
5	5	16	15.4	17.6	180
5	5	17	36.3	39.7	270
5	5	18	35.6	41.3	0
5	5	19	30.2	26.7	90
5	5	20	86.1	97.7	0
5	5	21	42.1	47.2	270
5	5	22	39.2	45.5	0
5	5	23	32.2	29.6	270
5	5	24	27.5	32.2	0
5	5	25	46.1	48.1	270
5	5	26	73.1	78.6	0
5	5	27	38.1	37.2	270
5	5	28	1.8	8.5	0
5	5	29	42.6	41.8	270
5	5	30	38.3	38.3	0
5	5	31	11.9	16.1	90
5	5	32	18.6	18.5	0
5	5	34	23.0	27.9	0
5	5	36	40.6	42.5	0
5	5	38	46.1	45.8	0
5	5	39	12.4	15.5	270
5	5	32	18.6	18.5	0
5	5	34	23.0	27.9	0
5	5	36	40.6	42.5	0
5	5	38	46.1	45.8	0
5	5	41	39.2	34.1	270
5	5	42	47.1	48.6	0
5	5	44	30.4	18.7	0
5	5	46	30.3	35.1	0
5	5	47	23.4	22.2	270
5	5	48	57.6	49.1	0
5	5	49	39.4	33.6	270
5	5	50	36.4	31.8	0
5	5	51	31.9	21.7	270
5	5	52	34.7	33.2	0
5	5	53	16.7	21.3	270
5	5	54	37.3	33.4	0
5	6	0	14.5	14.9	180
5	6	2	76.9	83.3	217
5	6	3	21.7	16.2	108
5	6	4	31.6	34.1	183
5	6	5	37.3	33.9	141
5	6	6	16.8	15.5	178
5	6	7	35.1	45.6	62
5	6	8	23.9	22.6	122
5	6	9	42.9	48.0	221
5	6	10	42.0	49.7	193
5	6	11	25.5	25.5	4
5	6	12	43.3	46.3	355
5	6	13	27.5	24.5	243
5	6	14	44.4	43.0	182
5	6	15	22.7	21.7	348
5	6	16	18.3	18.7	15
5	6	17	20.5	16.6	37
5	6	18	38.6	36.0	198
5	6	19	16.2	10.8	321
5	6	20	38.2	32.7	191
5	6	22	38.0	35.1	201
5	6	23	28.2	28.7	317
5	6	24	28.3	23.7	234
5	6	25	21.9	28.3	232
5	6	26	33.6	41.9	152
5	6	28	39.8	37.2	319
5	6	29	27.1	24.7	266
5	6	30	26.8	24.8	241
5	6	31	26.5	23.7	76
5	6	32	19.2	17.9	296
5	6	33	18.4	19.6	159
5	6	34	50.0	47.0	222
5	6	35	33.7	29.2	14
5	6	36	26.7	19.0	196
5	6	38	76.9	69.8	193
5	6	39	24.4	24.5	332
5	6	40	43.8	36.8	249
5	6	42	43.1	36.7	202
5	6	44	19.1	18.4	283
5	7	0	14.8	16.6	180
5	7	1	27.9	26.1	162
5	7	2	28.5	32.3	174
5	7	3	58.9	61.7	339
5	7	5	26.7	27.0	213
5	7	0	14.8	16.6	180
5	7	1	27.9	26.1	162
5	7	2	28.5	32.3	174
5	7	3	58.9	61.7	339
5	7	5	26.7	27.0	213
5	7	7	30.7	28.5	296
5	7	8	17.3	19.2	227
5	7	9	39.2	41.7	172
5	7	10	40.7	45.8	178
5	7	11	36.5	40.2	233
5	7	12	17.4	12.0	254
5	7	13	22.1	22.0	179
5	7	14	25.7	21.7	96
5	7	16	21.4	20.2	93
5	7	17	39.9	38.8	195
5	7	18	34.6	29.5	116
5	7	19	45.0	54.6	12
5	7	20	24.8	10.0	166
5	7	21	67.0	63.8	188
5	7	22	22.0	30.0	197
5	7	23	13.5	27.1	339
5	7	25	43.3	46.8	207
5	7	26	27.9	25.7	204
5	7	27	36.0	29.4	222
5	7	28	33.7	31.7	58
5	7	29	23.4	10.7	281
5	7	31	16.6	21.3	327
5	7	33	35.3	31.2	194
5	7	34	33.7	32.9	138
5	7	36	17.1	19.6	197
5	7	37	36.7	32.7	194
5	7	38	21.4	19.6	154
5	7	39	17.8	8.9	185
5	7	40	15.9	18.4	140
5	7	41	19.4	22.6	230
5	7	42	25.4	22.1	145
5	7	45	16.4	19.9	229
5	7	46	42.6	34.3	140
5	7	48	15.0	10.0	125
5	7	50	21.4	16.0	131
5	7	51	15.7	14.3	245
5	7	52	18.2	14.2	97
5	7	53	16.7	20.7	199
5	7	56	20.4	17.2	107
5	7	57	17.7	23.1	205
5	7	58	17.1	8.1	143
5	8	0	24.7	18.8	0
5	8	3	32.7	35.4	80
5	8	6	27.6	17.7	14
5	8	7	30.3	28.2	62
5	8	8	24.7	16.0	156
5	8	9	21.4	31.0	111
5	8	10	17.4	11.9	357
5	8	11	19.0	20.4	91
5	8	12	24.6	20.2	20
5	8	13	38.9	35.2	113
5	8	15	21.2	26.8	44
5	8	16	29.0	33.7	13
5	8	17	35.4	36.9	88
5	8	19	27.2	29.3	94
5	8	21	23.0	27.3	55
5	8	22	15.3	19.7	6
5	8	23	20.9	27.0	94
5	8	24	46.0	28.3	334
5	8	25	18.5	18.4	102
5	8	27	29.9	16.8	102
5	8	30	23.3	20.8	329
5	8	31	16.4	21.6	98
5	8	32	16.2	9.6	177
5	8	33	44.2	35.9	114
5	8	37	26.9	26.0	73
6	6	0	62.8	69.7	0
6	6	3	20.4	29.0	90
6	6	4	43.9	42.2	0
6	6	6	18.7	24.6	180
6	6	8	41.9	51.1	0
6	6	9	20.5	23.2	90
6	6	10	18.8	9.5	0
6	6	11	32.6	41.5	90
6	6	12	37.0	36.6	0
6	6	13	50.1	41.3	90
6	6	14	20.8	29.0	0
6	6	15	13.4	20.8	90
6	6	16	57.1	59.2	0
6	6	17	[illegible]	22.5	90
6	6	19	46.9	24.3	90
6	6	20	89.2	94.8	0
6	6	22	25.5	34.0	180
6	6	23	21.1	16.6	90
6	6	24	61.1	66.7	0
6	6	26	36.2	38.2	0
6	6	27	30.5	28.7	90
6	6	28	23.7	25.1	0
6	6	29	17.3	24.0	90
6	6	30	49.3	59.2	0
6	6	31	19.3	20.9	90
6	6	32	45.3	50.3	0
6	6	33	21.1	30.9	90
6	6	35	30.4	30.7	90
6	6	36	49.5	48.4	0
6	6	37	23.3	28.2	90
6	6	38	13.4	2.4	180
6	6	40	31.4	31.0	0
6	6	42	13.1	0.9	180
6	6	43	13.0	15.6	90
6	6	44	12.9	16.2	0
6	6	45	12.7	6.7	90
6	6	48	30.1	37.9	0
6	6	52	25.7	39.5	0
6	7	0	25.8	38.9	0
6	7	2	23.5	28.5	52
6	7	3	17.9	20.3	315
6	7	4	18.7	12.1	177
6	7	6	35.2	31.6	167
6	7	7	39.6	43.6	276
6	7	8	27.3	25.5	142
6	7	9	33.9	38.9	228
6	7	10	35.8	38.9	288
6	7	11	33.7	35.9	355
7	7	0	18.0	19.2	180
7	7	1	33.0	35.4	90
7	7	2	18.0	20.8	0
7	7	3	18.0	22.9	90

Measured and calculated values of structure factors.
The phase angles are in degrees.

Table 3-4

Final fractional atomic coordinates, their estimated standard deviations in Å, and isotropic temperature factors

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	U_{iso}
C(1)	0.0508	-0.0284	0.0735	0.016	0.016	0.014	0.030
C(2)	0.2057	-0.0434	0.0658	0.016	0.016	0.016	0.027
C(3)	0.2168	0.0449	0.0479	0.016	0.016	0.015	0.027
C(4)	0.0800	0.0956	0.0424	0.017	0.018	0.016	0.040
C(5)	-0.0309	-0.0218	0.0392	0.017	0.018	0.016	0.040
C(6)	-0.1697	0.0471	0.0325	0.018	0.017	0.016	0.035
C(7)	-0.2934	-0.0506	0.0339	0.016	0.017	0.015	0.035
C(8)	-0.2763	-0.1702	0.0491	0.016	0.017	0.015	0.030
C(9)	-0.2013	-0.1184	0.0668	0.016	0.017	0.016	0.029
C(10)	-0.0484	-0.0971	0.0599	0.017	0.018	0.016	0.041
C(11)	-0.1973	-0.2306	0.0823	0.016	0.016	0.014	0.029
C(12)	-0.2639	-0.3641	0.0753	0.020	0.020	0.018	0.048
C(13)	-0.2989	-0.4658	0.0903	0.017	0.017	0.016	0.039
C(14)	-0.3721	-0.4385	0.1074	0.025	0.024	0.023	0.082
C(15)	-0.3675	-0.5517	0.1181	0.029	0.028	0.026	0.084
C(16)	-0.2532	-0.5932	0.0906	0.022	0.022	0.021	0.061
C(17)	0.0272	0.1902	0.0579	0.018	0.018	0.016	0.038
C(18)	0.0270	-0.1275	0.0241	0.017	0.017	0.015	0.037
C(19)	-0.2676	0.0198	0.0755	0.020	0.020	0.018	0.052
C(20)	-0.4138	-0.2176	0.0557	0.018	0.018	0.017	0.034
O(21)	-0.4125	-0.3205	0.0682	0.012	0.013	0.011	0.046
O(22)	-0.5244	-0.1822	0.0501	0.014	0.015	0.013	0.058
O(23)	0.0835	0.1834	0.0261	0.013	0.013	0.011	0.048
O(24)	-0.0143	0.3085	0.0575	0.014	0.014	0.013	0.056
O(25)	0.0227	0.1169	0.0750	0.011	0.011	0.011	0.036
C(26)	0.3108	0.0232	0.0790	0.015	0.015	0.014	0.022
N(27)	0.3736	0.1269	0.0719	0.013	0.013	0.011	0.029
N(28)	0.3133	0.1502	0.0534	0.013	0.013	0.011	0.026
C(29)	0.4215	0.2105	0.0418	0.017	0.017	0.016	0.036
C(30)	0.3990	0.1977	0.0212	0.018	0.018	0.016	0.041

Table 3-4 (cont.)

Atom	x/a	y/b	z/c	σ(x)	σ(y)	σ(z)	U_{iso}
C(31)	0.5023	0.2575	0.0103	0.021	0.022	0.020	0.061
C(32)	0.6141	0.3334	0.0182	0.018	0.019	0.018	0.050
C(33)	0.6217	0.3508	0.0369	0.019	0.019	0.017	0.051
C(34)	0.5281	0.2846	0.0498	0.021	0.020	0.019	0.055
C(35)	0.3450	-0.0310	0.0986	0.016	0.016	0.014	0.025
C(36)	0.2929	-0.1508	0.1048	0.018	0.019	0.017	0.041
C(37)	0.3324	-0.2059	0.1228	0.020	0.020	0.018	0.046
C(38)	0.4324	-0.1315	0.1336	0.023	0.023	0.021	0.062
C(39)	0.4806	-0.0020	0.1276	0.022	0.021	0.018	0.054
C(40)	0.4367	0.0465	0.1098	0.018	0.018	0.016	0.040
O(41)	-0.3012	-0.6564	0.1075	0.017	0.018	0.016	0.085
C(42)	0.3026	0.4881	0.0806	0.022	0.023	0.021	0.062
C(43)	0.2300	0.5446	0.0464	0.026	0.027	0.024	0.080
C(44)	0.1984	0.5510	0.0673	0.021	0.021	0.019	0.051
O(45)	0.1048	0.6195	0.0742	0.017	0.016	0.015	0.080
I(46)	0.7602	0.4215	-0.0007	0.002	0.002	0.002	-

Anisotropic temperature factors of the iodine atom

U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
0.085	0.083	0.085	0.042	0.065	-0.017

Table 3-5

Bond lengths and their estimated standard deviations

Bond	Length	e.s.d.	Bond	Length	e.s.d.
C(1)-C(2)	1.600	0.022	C(15)-O(41)	1.415	0.032
C(1)-C(10)	1.510	0.023	C(16)-O(41)	1.417	0.027
C(1)-O(25)	1.436	0.020	C(17)-O(24)	1.213	0.022
C(2)-C(3)	1.531	0.022	C(17)-O(25)	1.402	0.020
C(2)-C(26)	1.524	0.021	C(20)-O(21)	1.332	0.021
C(3)-C(4)	1.464	0.023	C(20)-O(22)	1.192	0.023
C(3)-N(28)	1.437	0.020	C(26)-N(27)	1.275	0.020
C(4)-C(5)	1.579	0.024	C(26)-C(35)	1.521	0.020
C(4)-C(17)	1.516	0.024	N(27)-N(28)	1.451	0.017
C(4)-O(23)	1.438	0.021	N(28)-C(29)	1.455	0.021
C(5)-C(6)	1.573	0.025	C(29)-C(30)	1.478	0.023
C(5)-C(10)	1.648	0.023	C(29)-C(34)	1.379	0.027
C(5)-C(18)	1.581	0.023	C(30)-C(31)	1.387	0.027
C(6)-C(7)	1.529	0.024	C(31)-C(32)	1.421	0.028
C(7)-C(8)	1.590	0.022	C(32)-C(33)	1.333	0.025
C(8)-C(9)	1.533	0.022	C(33)-C(34)	1.440	0.027
C(8)-C(20)	1.483	0.024	C(35)-C(36)	1.337	0.024
C(9)-C(10)	1.571	0.024	C(35)-C(40)	1.405	0.023
C(9)-C(11)	1.542	0.022	C(36)-C(37)	1.432	0.030
C(9)-C(19)	1.604	0.026	C(37)-C(38)	1.429	0.030
C(11)-C(12)	1.524	0.025	C(38)-C(39)	1.403	0.031
C(12)-C(13)	1.487	0.023	C(39)-C(40)	1.408	0.025
C(12)-O(21)	1.581	0.023	C(44)-C(42)	1.505	0.030
C(13)-C(14)	1.423	0.029	C(44)-C(43)	1.514	0.031
C(13)-C(16)	1.310	0.028	C(44)-O(45)	1.224	0.026
C(14)-C(15)	1.333	0.036	C(32)-I(46)	2.125	0.018

Table 3-5 (cont.)

Bond lengths and numbering of the atoms in the iodo-derivative of isocolumbin

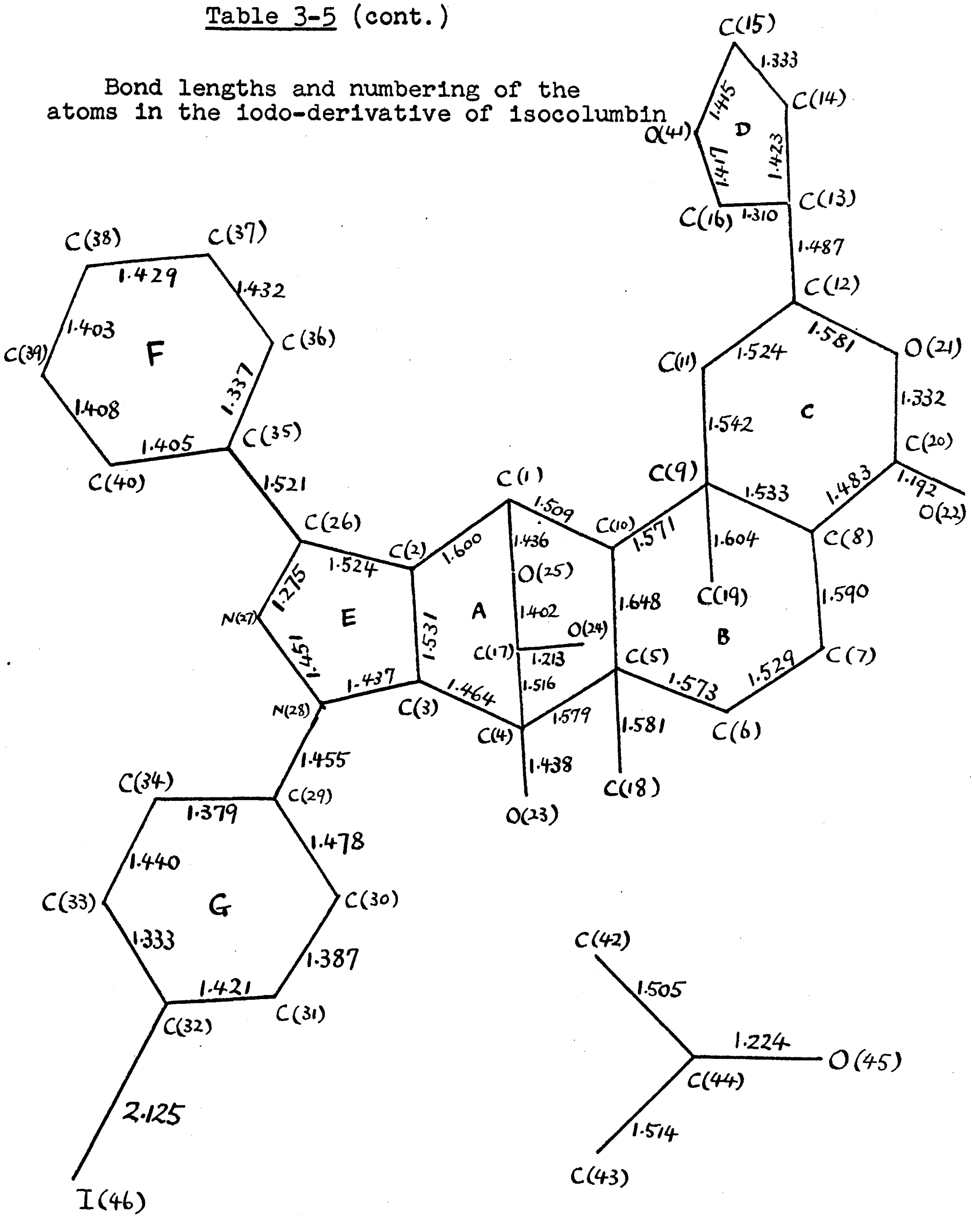


Table 3-6

Bond angles and their estimated standard deviations

C(10)C(1)O(25)	110.9	±1.3°	C(12)C(13)C(14)	126.6	±1.7°
C(2)C(1)O(25)	106.9	1.2	C(13)C(14)C(15)	108.3	2.1
C(2)C(1)C(10)	109.9	1.2	C(14)C(15)O(41)	107.5	2.1
C(3)C(2)C(26)	103.0	1.2	C(13)C(16)O(41)	108.0	1.8
C(1)C(2)C(3)	107.2	1.2	O(24)C(17)O(25)	119.3	1.5
C(1)C(2)C(26)	112.3	1.2	C(4)C(17)O(24)	131.5	1.6
C(4)C(3)N(28)	115.0	1.4	C(4)C(17)O(25)	109.1	1.4
C(2)C(3)N(28)	102.5	1.2	O(21)C(20)O(22)	116.5	1.6
C(2)C(3)C(4)	110.1	1.3	C(8)C(20)O(22)	127.6	1.6
C(3)C(4)O(23)	113.0	1.4	C(8)C(20)O(21)	115.6	1.5
C(3)C(4)C(17)	108.3	1.4	C(12)O(21)C(20)	114.9	1.3
C(3)C(4)C(5)	114.3	1.4	C(1)O(25)C(17)	115.3	1.2
C(17)C(4)O(23)	103.6	1.3	N(27)C(26)C(25)	121.8	1.3
C(5)C(4)O(23)	108.9	1.3	C(2)C(26)N(27)	114.2	1.2
C(5)C(4)C(17)	108.1	1.4	C(2)C(26)C(35)	124.0	1.3
C(4)C(5)C(18)	108.8	1.3	C(26)N(27)N(28)	106.6	1.2
C(4)C(5)C(6)	108.6	1.4	C(3)N(28)N(27)	113.3	1.1
C(4)C(5)C(10)	104.9	1.2	C(29)N(28)C(3)	126.8	1.2
C(6)C(5)C(18)	111.9	1.3	N(27)N(28)C(29)	106.6	1.2
C(6)C(5)C(10)	111.7	1.3	N(28)C(29)C(34)	120.8	1.5
C(10)C(5)C(18)	110.6	1.3	C(30)C(29)C(34)	124.1	1.6
C(5)C(6)C(7)	112.8	1.4	N(28)C(29)C(30)	114.8	1.4
C(6)C(7)C(8)	114.3	1.3	C(29)C(30)C(31)	113.9	1.6
C(7)C(8)C(9)	111.5	1.3	C(30)C(31)C(32)	123.1	1.7
C(7)C(8)C(20)	110.2	1.3	C(31)C(32)C(33)	119.7	1.7
C(9)C(8)C(20)	105.6	1.3	C(31)C(32)I(46)	122.5	1.4
C(8)C(9)C(10)	103.6	1.2	C(33)C(32)I(46)	117.8	1.3
C(10)C(9)C(11)	106.8	1.3	C(32)C(33)C(34)	122.7	1.8
C(10)C(9)C(19)	112.7	1.4	C(29)C(34)C(33)	116.0	1.6
C(8)C(9)C(11)	111.2	1.3	C(26)C(35)C(36)	121.0	1.4
C(8)C(9)C(19)	113.3	1.4	C(26)C(35)C(40)	117.8	1.4
C(11)C(9)C(19)	109.0	1.3	C(36)C(35)C(40)	121.2	1.5
C(1)C(10)C(9)	117.3	1.3	C(35)C(36)C(37)	120.8	1.7
C(5)C(10)C(9)	115.5	1.3	C(36)C(37)C(38)	118.0	1.8
C(1)C(10)C(5)	107.8	1.3	C(37)C(38)C(39)	120.8	1.9
C(9)C(11)C(12)	111.0	1.3	C(38)C(39)C(40)	118.0	1.8
C(11)C(12)C(13)	115.3	1.4	C(39)C(40)C(35)	121.0	1.6
C(13)C(12)O(21)	101.4	1.4	C(15)O(41)C(16)	106.7	1.8
C(11)C(12)O(21)	105.2	1.4	C(42)C(44)O(45)	117.7	1.8
C(14)C(13)C(16)	109.1	1.8	C(42)C(44)C(43)	117.2	1.8
C(12)C(13)C(16)	123.9	1.7	C(43)C(44)O(45)	124.3	1.9

Table 3-7

Intramolecular non-bonded distances $\leqslant 3.5$Å

C(1)...C(17)	2.40
C(1)...C(4)	2.52
C(1)...C(3)	2.52
C(1)...C(5)	2.55
C(1)...C(26)	2.59
C(1)...C(9)	2.63
C(1)...C(19)	3.12
C(1)...C(11)	3.16
C(1)...C(35)	3.36
C(1)...N(28)	3.38
C(1)...C(36)	3.44
C(1)...N(27)	3.47
C(2)...N(28)	2.32
C(2)...N(27)	2.35
C(2)...O(25)	2.44
C(2)...C(4)	2.46
C(2)...C(10)	2.55
C(2)...C(35)	2.69
C(2)...C(17)	2.90
C(2)...C(5)	2.97
C(2)...C(36)	3.07
C(3)...C(26)	2.39
C(3)...N(27)	2.41
C(3)...C(17)	2.42
C(3)...O(23)	2.42
C(3)...C(5)	2.56
C(3)...C(29)	2.59
C(3)...O(25)	2.77
C(3)...C(30)	2.98
C(3)...C(18)	3.00
C(3)...C(10)	3.03
C(3)...O(22)	3.33
C(3)...O(24)	3.46
C(4)...O(25)	2.38
C(4)...O(24)	2.49
C(4)...C(10)	2.56
C(4)...C(6)	2.56
C(4)...C(18)	2.57
C(4)...C(29)	3.49
C(4)...C(26)	3.49
C(5)...O(23)	2.46
C(5)...C(17)	2.51
C(5)...C(7)	2.58
C(5)...C(9)	2.72
C(5)...C(8)	2.86
C(5)...O(25)	2.91
C(5)...O(24)	3.45
C(5)...C(19)	3.46
C(6)...C(18)	2.61
C(6)...C(8)	2.62
C(6)...C(10)	2.66
C(6)...O(23)	2.81
C(6)...C(9)	2.93
C(6)...C(17)	2.97
C(6)...C(19)	3.20
C(6)...O(24)	3.43
C(7)...C(20)	2.52
C(7)...C(9)	2.58
C(7)...O(22)	2.82
C(7)...C(19)	3.03
C(7)...C(10)	3.04
C(7)...C(18)	3.26
C(8)...O(21)	2.38
C(8)...O(22)	2.40
C(8)...C(10)	2.44
C(8)...C(11)	2.54
C(8)...C(19)	2.62
C(8)...C(12)	2.64
C(8)...C(18)	3.45
C(9)...C(20)	2.40
C(9)...C(12)	2.53
C(9)...O(21)	2.83
C(9)...O(25)	3.20
C(9)...O(22)	3.40
C(10)...O(25)	2.43
C(10)...C(11)	2.50
C(10)...C(19)	2.64
C(10)...C(18)	2.66
C(10)...C(17)	2.88
C(10)...C(12)	3.49
C(11)...O(21)	2.47
C(11)...C(13)	2.54
C(11)...C(19)	2.56
C(11)...C(20)	2.82
C(11)...C(14)	3.17
C(12)...C(20)	2.46
C(12)...C(16)	2.47
C(12)...C(14)	2.60
C(13)...O(41)	2.21
C(13)...C(15)	2.23
C(13)...O(21)	2.38
C(14)...O(41)	2.22
C(14)...C(16)	2.23
C(14)...O(21)	3.03
C(15)...C(16)	2.27
C(16)...O(21)	3.44
C(17)...O(23)	2.32
C(17)...N(28)	2.81
C(18)...O(23)	3.06
C(19)...O(25)	2.96
C(19)...C(20)	3.04
O(21)...O(22)	2.15
O(23)...O(24)	2.70
O(23)...N(28)	2.97
O(23)...C(30)	3.08
O(23)...C(29)	3.46
O(24)...O(25)	2.26
O(24)...O(45)	3.43
O(25)...C(26)	2.94
O(25)...N(28)	3.21
O(25)...N(27)	3.40
C(26)...N(28)	2.19
C(26)...C(36)	2.49
C(26)...C(40)	2.51
C(26)...C(29)	3.37
N(27)...C(29)	2.33
N(27)...C(35)	2.45
N(27)...C(34)	2.65
N(27)...C(40)	2.86
N(28)...C(34)	2.46
N(28)...C(30)	2.47
C(29)...C(33)	2.39
C(29)...C(31)	2.40
C(29)...C(32)	2.77
C(30)...C(32)	2.47
C(30)...C(34)	2.52
C(30)...C(33)	2.84
C(31)...C(33)	2.38
C(31)...C(34)	2.82
C(31)...I(46)	3.06
C(32)...C(34)	2.43
C(33)...I(46)	3.06
C(35)...C(37)	2.41
C(35)...C(39)	2.45
C(35)...C(38)	2.79
C(36)...C(40)	2.39
C(36)...C(38)	2.45
C(36)...C(39)	2.82
C(37)...C(39)	2.46
C(37)...C(40)	2.80
C(38)...C(40)	2.41
C(42)...O(45)	2.34
C(42)...C(43)	2.58
C(43)...O(45)	2.42

Table 3-8

Intermolecular distances ≤ 4Å

O(22)...C(2)V	3.14	C(7) ...C(29)V	3.78
O(24)...C(44)	3.20	C(42)...N(28)	3.79
O(22)...C(26)V	3.27	C(39)...C(15)III	3.79
O(45)...C(10)II	3.28	C(14)...C(37)V	3.80
O(45)...C(11)II	3.31	C(43)...C(34)	3.83
O(22)...C(3)V	3.33	O(24)...C(33)V	3.83
O(24)...C(43)	3.38	C(7) ...C(34)V	3.84
O(24)...C(16)II	3.43	O(25)...C(38)III	3.84
O(45)...O(24)	3.43	C(39)...C(13)III	3.84
C(42)...O(21)I	3.44	C(40)...O(41)I	3.84
O(45)...C(1)II	3.45	O(45)...C(38)III	3.86
O(45)...C(2)II	3.46	C(19)...O(41)II	3.88
O(22)...N(27)V	3.51	O(22)...C(29)V	3.88
C(43)...O(22)I	3.56	C(42)...O(24)	3.88
O(45)...C(12)II	3.57	C(33)...O(21)I	3.89
C(6) ...C(33)V	3.58	C(42)...C(36)II	3.89
C(39)...C(16)III	3.58	C(19)...C(16)II	3.90
O(22)...I(46)VII	3.58	C(20)...C(26)V	3.91
O(22)...N(28)V	3.59	C(6) ...C(34)V	3.92
C(39)...O(41)III	3.60	C(44)...C(17)	3.92
C(42)...C(38)III	3.60	C(44)...O(22)I	3.92
O(45)...C(36)II	3.60	C(43)...N(28)	3.93
C(42)...N(27)	3.61	C(44)...C(2)II	3.93
C(6) ...C(32)V	3.61	C(7) ...C(30)V	3.93
C(38)...C(16)III	3.62	O(23)...O(23)IV	3.93
C(38)...O(41)III	3.63	O(45)...C(9)II	3.93
C(19)...N(27)V	3.63	C(42)...C(13)I	3.94
C(42)...C(34)	3.66	C(30)...C(31)IV	3.94
C(31)...C(31)IV	3.66	C(7) ...C(31)V	3.95
O(45)...C(39)III	3.70	O(22)...C(35)V	3.95
C(19)...C(34)V	3.71	C(44)...O(21)I	3.97
C(43)...C(29)	3.74	C(7) ...C(33)V	3.97
C(42)...C(14)I	3.74	C(11)...C(15)VI	3.97
C(40)...C(37)III	3.75	C(7) ...C(32)V	3.98
C(19)...C(40)V	3.76	C(14)...C(38)V	3.98
C(34)...C(16)I	3.77	C(39)...C(14)III	3.98

The Roman numerals refer to the following equivalent positions:

I: $1+x, 1+y, z$

II: $x, 1+y, z$

III: $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{4}-z$

IV: $y, x, -z$

V: $-1+x, y, z$

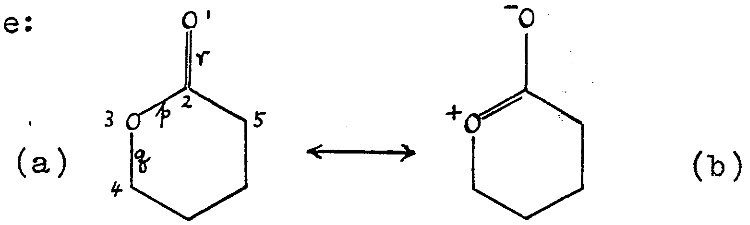
VI: $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{4}-z$

VII: $-1+y, -1+x, -z$

The hydrogen atoms at C(8), C(10) and C(12), the methyl carbon atoms at C(5) and C(9) are all axial while the hydrogen atom at C(1) and the hydroxyl oxygen atom at C(4) are equatorial.

In the 'cage' structure in ring A, the atoms C(1), C(2), C(3), C(4), C(5) and C(10) have the boat-configuration. It is cis-fused to ring B and also cis-fused to ring E.

In the δ-lactone system, hybridization between (a) and (b) takes place:



The consequence of this is the planarity of the five atoms (1), ..., (5), and the shortening of the bond p on account of the partial double bond character. Bond r will be lengthened because of the partial single bond character.

The mean plane through atoms C(1), O(25), C(17), O(24) and C(4) has equation:

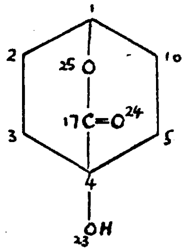
$$-0.9378X - 0.2626Y - 0.2272Z + 1.6344 = 0 \quad \ldots \quad (3.1)$$

The deviations of these atoms from the plane are +0.064, -0.075, -0.028, +0.055 and -0.017 Å respectively.

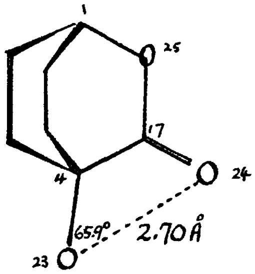
The deviations for the atoms C(1), O(25) and O(24) are more than three times the atomic estimated standard deviations and therefore the five atoms cannot strictly be described as planar.

The distances of the atoms C(2) and C(3) from the mean plane (3.1) are -1.18 and -1.22 Å respectively, while those of the atoms C(5) and C(10) are +1.34 and +1.36 Å respectively; indicating that both δ-lactone systems have the half-boat conformation.

The distance of the hydroxyl oxygen atom O(23) from the plane (3.1) is -0.007 Å and is therefore in the plane. The conformation of the 'cage' structure (c) is therefore as shown in (d).



(c)



(d)

The intramolecular distance between O(23) and O(24) is 2.70Å and that between O(23) and N(28) is 2.97Å. Both these distances are within the range for hydrogen bonding. Also there is no intermolecular distances between O(23) and other atoms that may suggest hydrogen bonding.

The angles O(24)...O(23)...C(4) and N(28)...O(23)...C(4) are only 65.9° and 55.1° respectively, indicating that the hydrogen atom would lie off the line joining either O(23) and O(24) or O(23) and N(28).

Ring B has also a boat-conformation. It is trans-fused to ring C.

In the δ-lactone system constituting ring C, hybridization between (a) and (b) also occurs and there is a long and a short C-O bond.

A mean plane through the five atoms C(8), C(20), O(22), O(21) and C(12) has equation:

$$0.0197X - 0.6950Y - 0.7188Z + 1.4215 = 0 \quad \ldots \quad (3.2)$$

The deviations of these atoms from the plane are +0.015, -0.001, -0.028, +0.028 and -0.014 Å respectively, all within the significant test limits.

The distances of the atoms C(9) and C(11) from the plane (3.2) are -1.22 and -1.25 Å respectively, indicating a half-boat conformation for ring C.

The furan ring D is planar. The equation of the mean plane through the five atoms is

$$-0.8533X - 0.2882Y - 0.4305Z - 1.0133 = 0 \quad \ldots \quad (3.3)$$

The deviations from the plane for the atoms C(13), C(14), C(15), O(41) and C(16) are +0.007, +0.017, -0.032, +0.035 and -0.032 Å respectively.

The pyrazole ring E is also planar. A mean plane fitted to the five atoms has an equation:

$$0.7199X - 0.5864Y - 0.3713Z + 0.0366 = 0 \quad \ldots \quad (3.4)$$

The deviations from the plane for the atoms C(2), C(3), N(28), N(27) and C(26) are -0.015, +0.032, -0.038, +0.028 and -0.007 Å respectively.

The equation of the mean plane through atoms C(26) and C(35) to C(40) of the benzene ring F is

$$0.7679X - 0.4845Y - 0.4191Z + 0.1954 = 0 \quad \ldots \quad (3.5)$$

The deviations of these atoms in order from the plane are +0.051, -0.023, -0.031, -0.013, +0.061, -0.010 and -0.036 Å respectively.

The equation of the mean plane through N(28), C(29) to C(34) of the benzene ring G and I(46) is

$$-0.5461X + 0.8370Y - 0.0361Z + 0.5883 = 0 \quad \ldots \quad (3.6)$$

The deviations of these atoms in order from the plane are +0.013, -0.041, +0.027, -0.007, -0.003, +0.050, -0.025 and -0.013 Å respectively.

The acetone solvent molecule is also planar within experimental errors. The equation through the four atoms is

$$-0.5462X - 0.8371Y - 0.0302Z + 5.7082 = 0 \quad \ldots(3.7)$$

the deviations of the atoms C(42), C(43), C(44) and O(45) are -0.016, -0.017, +0.054 and -0.021 Å respectively.

Molecular dimensions. With the exception of a few, most of the bond lengths and valency angles do not deviate significantly from the standard values.

The notably bad ones include 1.581Å for C(12)-O(21), 1.648Å for C(5)-C(10), 1.478Å for C(29)-C(30), and 1.464Å for C(3)-C(4).

The simple mean over the fifteen C(sp^3)-C(sp^3) single bonds is 1.558Å. The mean of the twelve C-C bonds in the two benzene rings is 1.396Å.

The value of 2.125A for C(32)-I(46) is longer than the value of 2.05Å (Tables of Interatomic Distances, 1958), though a value of 2.19Å has been reported (Mayer and Pratt, 1963).

The value of 1.437Å for C(3)-N(28) is shorter, though not significantly, than the values of 1.472±0.005Å (Tables of Interatomic Distances, 1958) and the very accurate value of 1.476±0.001Å obtained by Cruickshank et al. (1963).

The value of 1.455Å for N(28)-C(29) is not significantly longer than 1.426Å in acetanilide (Brown and Corbridge, 1954);

and 1.43Å in 5-iodo-2-phthalimidobenzoic acid monohydrate (Mayer and Pratt, 1963).

The value of 1.451Å for N(27)-N(28) compares well with the single N-N bond distance of 1.468Å in pyrazoline hydrochloride (Nardelli and Fava, 1962); 1.46Å in hydrazine (Giguère and Schomaker, 1943); 1.46Å, and 1.447Å in lithium hydrazinium sulphate (Brown, 1964; van den Hende and Boutin, 1964); 1.47Å in byssochlamic acid bis-p-bromophenyl-hydrazide (Paul et al. 1963).

The value of 1.275Å for C(26)-N(27) compares well with the normal carbon-nitrogen double bond length: 1.27Å in 2-(2-hydroxyethylamino)-2-thiazoline (Miller et al. 1964), 1.255Å in pyrazoline hydrochloride, 1.267Å in 2-amino 5-propyl 7-methyl s-triazolo[2,3-c] pyrimidine hydrochloride (Owston and Rowe, 1962), and a mean value of 1.269Å in oximes and amine oxides (Folting et al. 1964, and references cited there).

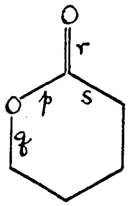
The dimensions of the furan ring are listed with results for furan itself (Bak et al. 1955), in 3,4-furandicarboxylic acid (Willians and Rundle, 1964), and in cedrelone iodoacetate (Grant et al. 1963) for comparison.

	Furan	3,4-furan dicarboxylic acid	Cedrelone iodoacetate	This structure
C-C	1.440±0.016	1.454±0.005	1.46±0.09	1.423±0.029
C=C	1.354±0.016	1.346±0.003	1.32±0.09 1.22±0.09	1.333±0.036 1.310±0.036
C-O	1.371±0.016	1.356±0.003	1.34±0.09	1.415±0.032 1.417±0.027
C-O-C	106.0±1.6°	107.2±0.3°	114°	106.7±1.8°
C=C-O	110.9±0.7°	110.8±0.2°	110° 100°	107.5±2.1° 108.0±1.8°
C-C=C	106.1±0.6°	105.7±0.2°	93° 108°	109.1±1.8° 108.3±2.1°

The acetone molecule, being a solvate, is associated with a high thermal vibration. Its C-C distances of 1.505 and 1.514Å and C=O distance of 1.224Å do not differ significantly from values of 1.55Å for the C-C bond and 1.22Å for the C=O bond in acetone measured by electron diffraction (Allen et al. 1952); and another set of values of 1.52Å for the C-C and 1.23Å for C=O also by electron diffraction method (Bauer, 1952).

The bond lengths in the δ-lactone system obtained in this analysis are not very good. In one case, the results of 1.402Å and 1.436Å are not significantly different and in another ring, the length of 1.581Å is anomalously long. However, they are listed with results from some other structures containing δ-lactone rings; namely, dihydro-β-erythroidine hydrobromide (Hanson, 1963), jacobine (Mathieson and Taylor, 1963), the two epimers of isoiridomyrmecin (McConnell et al. [1] 1962, [2] 1964), α-bromoisotutin (Craven, 1964), simarolide <u>m</u>-iodobenzoate (Brown, 1964), and epilimonol iodoacetate (Arnott et al. 1961).

Compound	p	q	r	s
Dihdro-β-erthroidine hydrobromide	1.33	1.43	1.21	1.54
Jacobine	1.36	1.44	1.19	1.52
	1.39	1.48	1.20	1.51
Isoiridomyrmecin [1]	1.361	1.488	1.180	1.506
[2]	1.365	1.428	1.317	1.502
α-bromoisotutin	1.35	1.44	1.12	1.56
Simarolide m-iodobenzoate	1.28	1.44	1.34	1.39
	1.41	1.63	1.26	1.43
Epilimonol iodoacetate	1.31	1.44	1.29	1.48
	1.33	1.46	1.27	1.52
This structure	1.402	1.436	1.213	1.516
	1.332	1.581	1.192	1.483



The molecules in the unit cell are held together by only van der Waals forces. The solvent molecule of acetone is probably important in these cohesive forces as 29 of the total of 72 shorter intermolecular distances involve the atoms of the acetone molecule.

There are a number of close intermolecular distances, all involving oxygen atoms. The carbonyl oxygen atom O(22) is 3.14Å from C(2); 3.27Å from C(26); and 3.33Å from C(3) in a neighbouring molecule (-1+x, y, z).

The carbonyl oxygen atom O(24) is 3.20Å from atom C(44) and 3.38Å from the atom C(43) of the acetone molecule.

The carbonyl oxygen atom O(45) of the acetone molecule is 3.28Å from atom C(10) and 3.31Å from atom C(11).

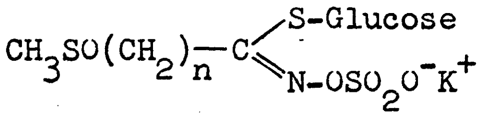
The only intermolecular distance less than 4Å involving the iodine atom is a value of 3.58Å for $O(22)\ldots I(46)_X$, just greater than the sum of the van der Waals radii for the oxygen and iodine atoms.

PART IV

The Structure and Absolute Configuration of N-(α-phenylethyl) N'-(ω-methylsulphinylpropyl) Thiourea.

4.1. INTRODUCTION

It is well-known that an asymmetric sulphur atom gives rise to optical activity and a knowledge of its absolute configuration is desirable. Schmid and Karrer (1948) assigned the structure $CH_3SOCH{=}CHCH_2CH_2NCS$ to sulphoraphen, a strongly laevorotatory mustard oil isolated from radish seed, and the first natural product in which optical activity was attributable solely to a sulphoxide group. This was followed by the isolation from cruciferous plants (Schultz et al. 1954; Kjaer et al. 1956,1958,1963; Procházka, 1959) of a homologous series of glucosides of the general formula (I).



$$CH_3SO(CH_2)_n{-}C\begin{matrix}\diagup \text{S-Glucose}\\ \diagdown N{-}OSO_2O^-K^+\end{matrix}$$

$$CH_3SO(CH_2)_nNCS$$

where n = 3-6, 8-10.

(I)

(II)

Upon enzymical hydrolysis, these gave a corresponding series of sulphoxide mustard oils (II).

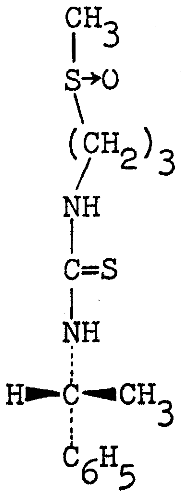
Identical configuration around the asymmetric sulphur atom in this homologous series of the naturally derived sulphoxide mustard oils was suggested by the fact reported by Kjaer et al. (1960) that the optical rotations at 589 mμ of the isothiocyanates measured, as well as of various thiourea derivatives were all negative and of comparable magnitude.

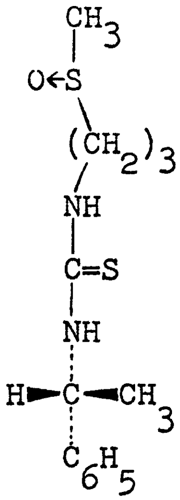
Schmid and Karrer (1948) proposed to use sulphoraphen as a configurational standard for sulphoxides, analogous to the

Fieser convention of calling the natural (+)-glyceraldehyde and its derivatives the D-series. This arbitrary sulphoxide reference standard was seperseded by the proposal of Synge and Wood (1956), which relates the sulphoxide configuration directly to that of a carbon atom, whose configuration is known.

The first absolute configuration around a sulphoxide group was established by Hine and Rogers (1956) in their x-ray structural analysis of (+)-S-methyl-L-cysteine sulphoxide. Their results established the (S)-configuration around the sulphoxide grouping.

The compound in the present work was prepared by Professor A. Kjaer by reacting iberin, $CH_3SO(CH_2)_2NCS$ with (R)-α-phenylethylamine to give the thiourea whose configuration is either (III) or (IV).


(III)


(IV)

Iberin itself was prepared by enzymic hydrolysis of glucoiberin from seeds of Iberis amara L. (Schultz and Gmelin, 1954).

4.2. EXPERIMENTAL

Crystal data N-(α-phenylethyl)N'-(ω-methylsulphinyl) thiourea, $C_{13}H_{20}N_2OS_2$. Molecular weight = 284.4, m.p. 148°, $[\alpha]_D^{21}$ -56 (MeOH, c=1.2). Orthorhombic, $\underline{a}$ = 16.48, $\underline{b}$ = 11.02, $\underline{c}$ = 8.54 Å. Volume of unit cell = 1550 $Å^3$. Number of asymmetric unit per unit cell = 4. Density(cal) = 1.218 gm cm^{-3} . Absent spectra: h00 when h=2n+1, 0k0 when k=2n+1, 00ℓ when ℓ=2n+1. Space group: $P2_12_12_1$,(D_2^4). Absorption coefficient for x-rays (λ=1.542Å),μ= 29.2 cm^{-1}. Total number of independent observed structure amplitudes = 1523.

Rotation, oscillation, and precession photographs were taken with copper-Kα (λ=1.542Å) and molybdenum-Kα (λ=0.7107Å) radiations. The cell dimensions were obtained from precession photographs. The space group was determined uniquely from the systematic halvings. Intensity data were obtained from equatorial and equi-inclination upper layer Weissenberg photographs taken from a crystal rotated about the $\underline{a}$ axis. The multiple-film technique was employed. The intensities were estimated visually by comparison with a calibrated step-wedge. Lorentz and polarization factors were applied. The rotation factor (Tunell, 1939) was applied to the upper layers. No correction for absorption was applied. The observed structure amplitudes were placed on the correct absolute scale by comparison with the calculated structure amplitudes at a later stage.

4.3. DETERMINATION OF THE STRUCTURE

A three-dimensional Patterson function

$$P(UVW) = \frac{8}{V}\sum_{0}^{\infty}\sum_{0}^{\infty}\sum_{0}^{\infty}|F(hk\ell)|^2 \cos 2\pi hU \cdot \cos 2\pi kV \cos 2\pi \ell W$$

was computed. For the space group $P2_12_12_1$, the vectors to be expected between the two sulphur atoms of coordinates (x_1,y_1,z_1) and (x_2,y_2,z_2) are:

Vectors	
S_1-S_1	$1/2-2x_1,-2y_1,1/2;\ -2x_1,1/2,1/2-2z_1;\ 1/2,1/2-2y_1,-2z_1;$
S_2-S_2	$1/2-2x_2,-2y_2,1/2;\ -2x_2,1/2,1/2-2z_2;\ 1/2,1/2-2y_2,-2z_2;$
S_1-S_2	$x_2-x_1,\ y_2-y_1,\ z_2-z_1;$ $-1/2+x_2+x,\ y_2+y_1,\ -1/2+z_2+z_1;$ $-1/2+x_2-x_1,\ -1/2+y_2+y_1,\ z_2+z_1;$ $x_2+x_1,\ -1/2+y_2-y_1,\ -1/2+z_2+z_1;$

The coordinates for S_1 and S_2 consistent with the above conditions were:

S_1: $x_1=0.153$, $y_1=0.211$, $z_1=0.177$;

S_2: $x_2=0.068$, $y_2=0.603$, $z_2=0.231$.

The three Harker sections are shown in Fig.4-1. The peaks corresponding to the S_1-S_1 vectors are marked A, B and C, and those corresponding to the S_2-S_2 vectore are marked F, E and D.

As the resolution of the Patterson map was rather good, attempts were made to find peaks corresponding to the vectors between sulphur and the lighter atoms. The coordinates of two plausible positions for two atoms were:

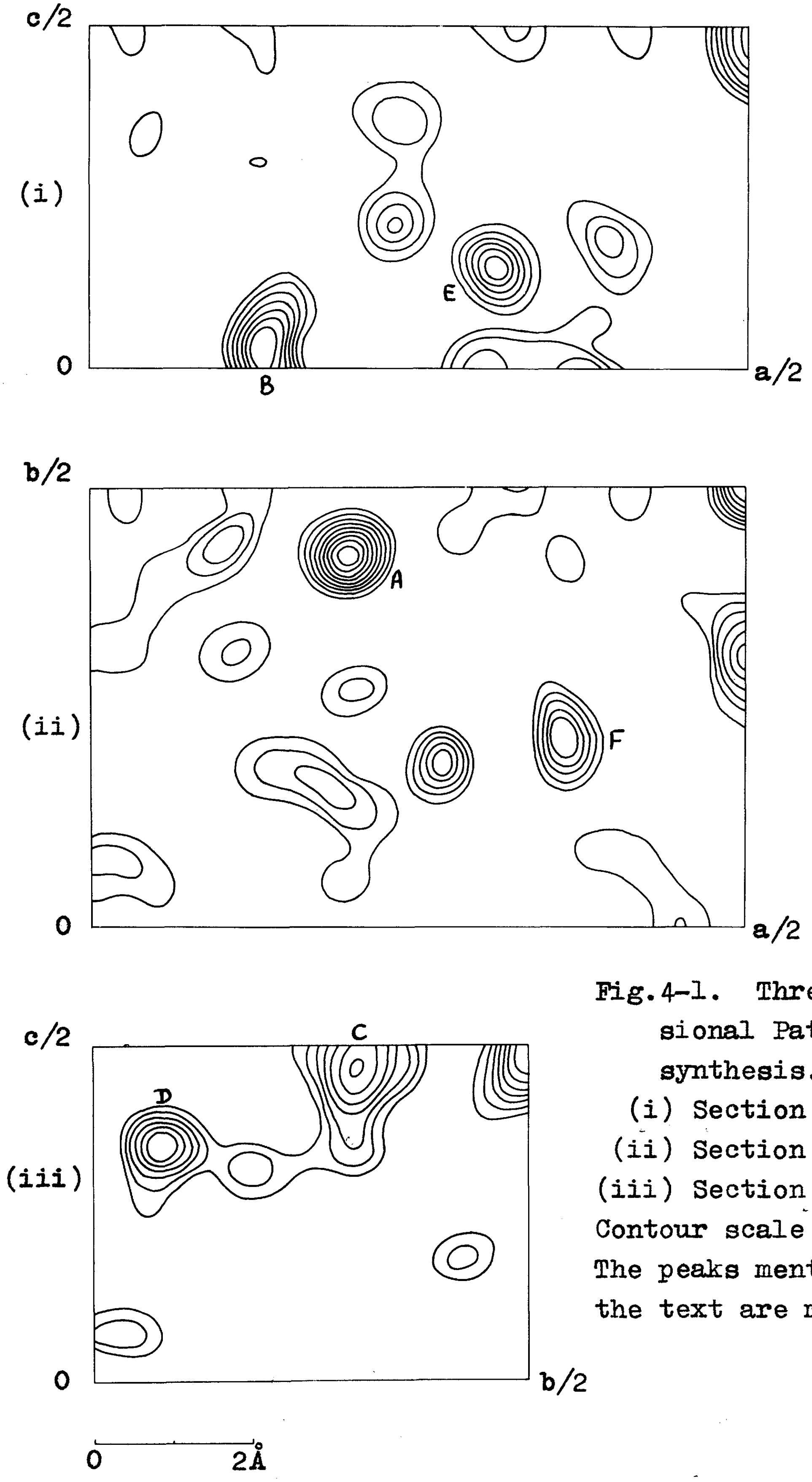


Fig.4-1. Three-dimensional Patterson synthesis.
(i) Section $P(U\frac{1}{2}W)$,
(ii) Section $P(UV\frac{1}{2})$,
(iii) Section $P(\frac{1}{2}VW)$.
Contour scale arbitrary. The peaks mentioned in the text are marked.

x_3=0.248, y_3=0.225, z_3=0.162;
x_4=0.116, y_4=0.721, z_4=0.114.

(It was later found that there was a mistake in z_4; its value should be about 1/2- 0.114).

These two atoms were put in as carbon atoms together with the two sulphur atoms in the calculation of structure factors. The discrepancy factor R $(=\Sigma\|Fo| - |Fc\|/\Sigma|Fo|)$ was 0.405.

The calculated phase constants were used to calculate a three-dimensional Fo Fourier synthesis. The arrangement of most of the atoms in the molecule was recognisable in the resulting electron-density distribution.

The peak height of the misplaced atom was only about 3 electrons Å^{-3}. The oxygen atom was distinguishable because it appeared as a higher peak. It was also distinguishable from the methyl carbon atom, C(1), because the oxygen atom was about 2.8Å from either of the two nitrogen atoms in a neighbouring molecule and was thus involved in hydrogen bonding.

The peaks corresponding to the two methyl carbon atoms were rather low and came up only to about 1.5 electrons Å^{-3}. They were therefore not included in the next cycle of structure factor calculation.

With two S atoms, one O atom, two N atoms and eleven C atoms, the R factor for the second set of structure factors was 0.300. The calculated phase constants were used in the second Fo Fourier synthesis. The two remaining carbon atoms showed up clearly in the resulting electron-density distribution.

4.4. REFINEMENT OF THE STRUCTURE

When the two methyl carbon atoms, C(1) and C(7), were included with the improved coordinates of the other sixteen atoms, the discrepancy R was 0.250. After another round of Fourier and structure factor calculation, R became 0.210.

The atomic parameters were then refined by the least squares method. The R factor after two cycle of refinement was 0.160. But it was noticed that two of the carbon atoms C(3) and C(4) in the middle of the chain had very high temperature factors and the bond length (=1.29Å) was too short for a C-C single bond. This was attributed to the very anisotropic thermal vibration of the atoms and incomplete refinement at this stage. It was expected that the position of these two carbon atoms would be adjusted during the course of refinement.

At this time, Professor G.A. Sim, with whom the work was started when he was in Glasgow, offered to carry on further least squares refinement on the IBM 7094 computer in the University of Illinois using tha program written by Gantzel, Sparks and Trueblood (1961).

During the several cycles of isotropic full matrix least squares refinement, the temperature factors for these two carbon atoms continued to get larger and had values corresponding to B = 9-10 Å^2, and the bond length did not improve appreciably. This suggusted that these two carbon atoms might be incorrectly placed.

Accordingly, a set of structure factors was calculated without these two carbon atoms, and the phase constants were used to calculate a Fo and (Fo-Fc) Fourier syntheses. The electron-density distributions from both Fourier syntheses revealed that C(4) had a very elongated peak and there were actually two peaks that could be assigned to C(3) at (-0.1579, 0.4766, 0.2365) and (-0.1808, 0.4468, 0.3247), about 1 Å apart. This strongly suggests that there is disorder in the structure.

The least squares refinement was then continued; by just assigning C(3) to the higher of the two peaks at (-0.1579, 0.4766, 0.2365) and all the atoms were allowed to refine anisotropically. The parameters of fourteen out of the eighteen atoms were refined at a time. After several cycles of refinement, while rotating the atoms to be refined, the refinement was stopped when the shifts of the positional parameters were of the order of the estimated standard deviations. The R factor of the final structure factor calculation was 0.114.

In the final (Fo-Fc) synthesis, there was no peak greater than 0.4 electron Å^{-3}. But it was not possible to locate the hydrogen atoms and confirm the hydrogen bonding.

4.5. RESULTS AND DISCUSSION

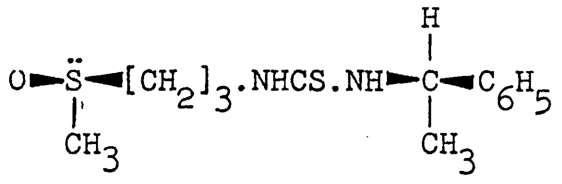
The observed and calculated structure amplitudes together with their calculated phase constants for the 1523 independent observed reflections are listed in Table 4-1.

The final atomic coordinates and their estimated standard deviations and the anisotropic temperature factors are listed in Tables 4-2 and 4-3.

The bond lengths and angles with their estimated standard deviations are listed in Table 4-4; while the intramolecular non-bonded distances less than 3.5 Å and the intermolecular distances less than 4 Å are listed in Table 4-5.

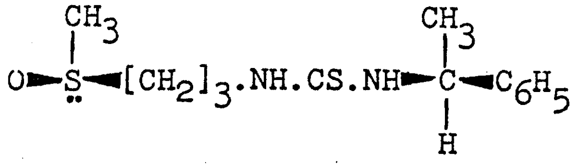
The molecular packing as viewed along the *c* axis is shown in Fig. 4-2, and the molecular packing as viewed along the *b* axis is shown in Fig. 4-3. The perspective projection of a molecule only as viewed along the *a* axis is shown in Fig. 4-4.

The absolute configuration. The result of the analysis defines the stereochemistry of the disubstituted thiourea to be either (V) or (VI); since the asymmetric carbon atom is known to possess the (R)-configuration, as indicated in (V), it follows that (V) with the (R)-configuration around the sulphoxide grouping, represents the correct absolute stereochemistry.



$$\mathrm{O}\blacktriangleright\underset{\mathrm{CH_3}}{\overset{\cdot\cdot}{\mathrm{S}}}\blacktriangleleft[\mathrm{CH_2}]_3.\mathrm{NHCS.NH}\blacktriangleright\underset{\mathrm{CH_3}}{\overset{\mathrm{H}}{\mathrm{C}}}\blacktriangleleft\mathrm{C_6H_5}$$

(V)



$$\mathrm{O}\blacktriangleright\underset{\cdot\cdot}{\overset{\mathrm{CH_3}}{\mathrm{S}}}\blacktriangleleft[\mathrm{CH_2}]_3.\mathrm{NH.CS.NH}\blacktriangleright\underset{\mathrm{H}}{\overset{\mathrm{CH_3}}{\mathrm{C}}}\blacktriangleleft\mathrm{C_6H_5}$$

(VI)

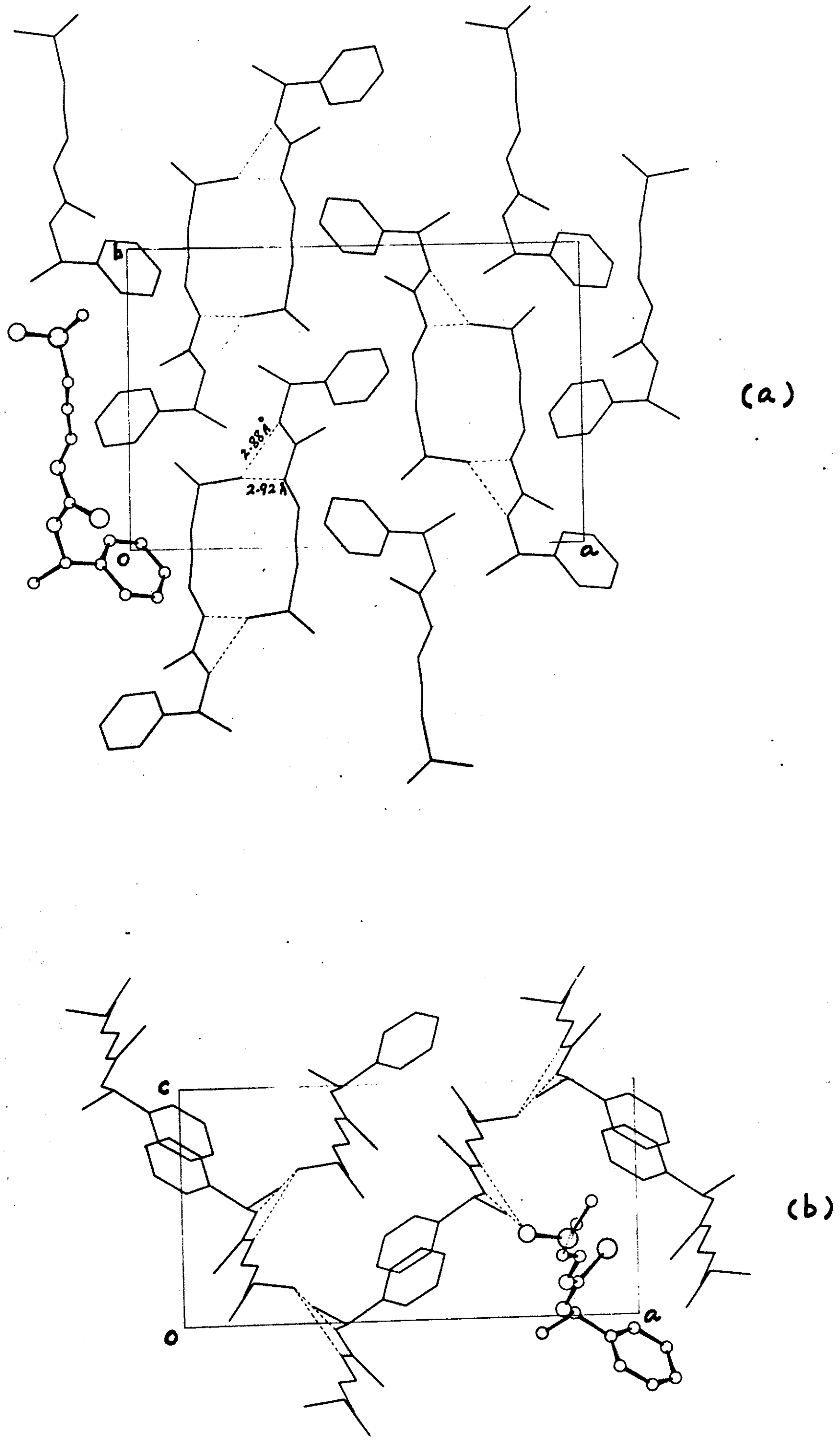


Fig.4-2. The arrangement of the molecules in the crystal of N-(α-phenylethyl)N'-(ω-methylsulphinyl) thiourea (a) as viewed along the b axis, (b) as viewed along the a axis. The broken lines denote possible H-bonds.

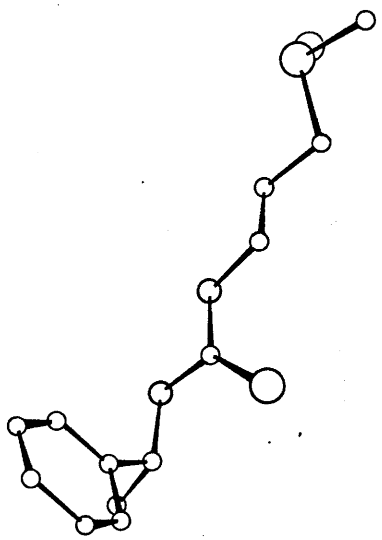


Fig.4-3(i) The arrangement of the atoms in a molecule of the disubstituted thiourea as viewed along the a axis.

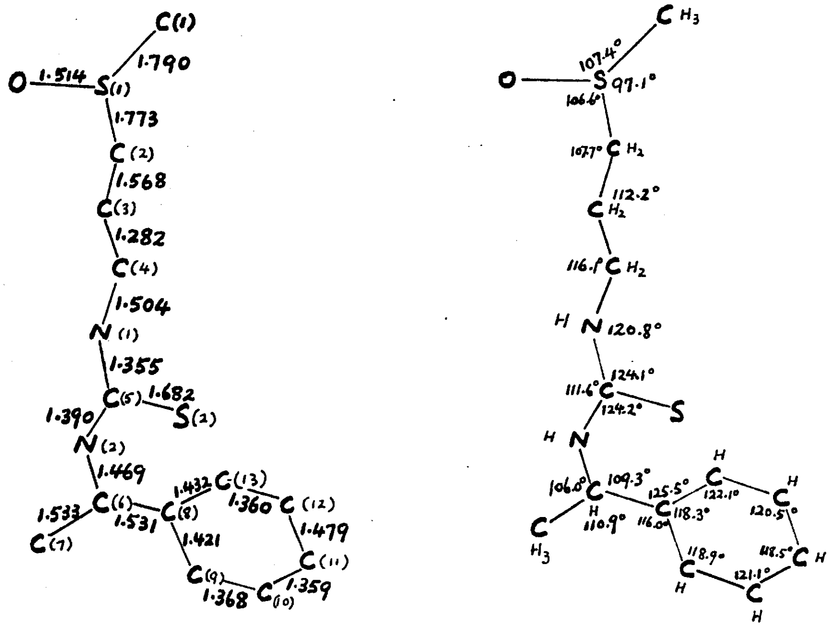
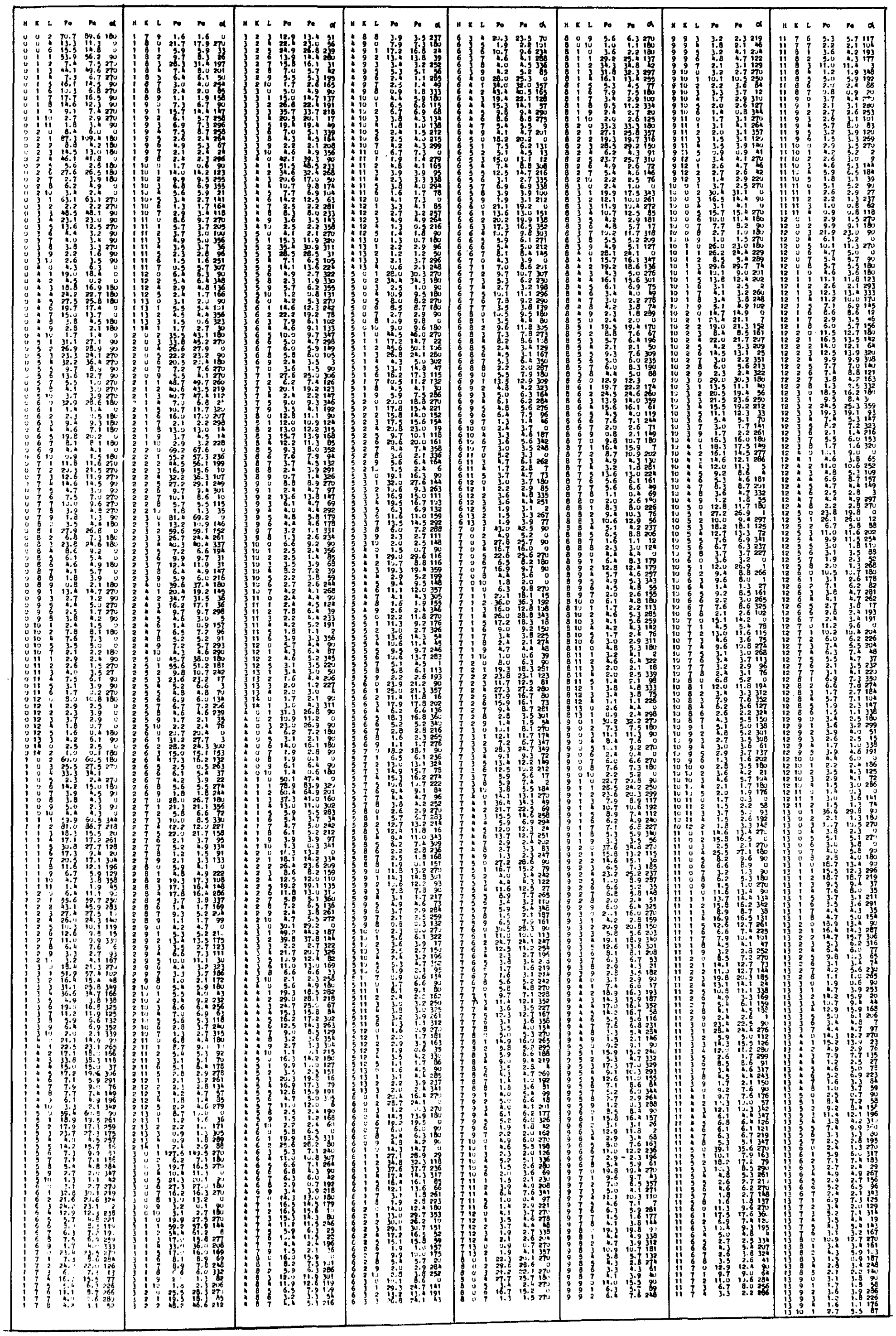


Fig.4-3(ii) Bond lengths and angles and the numbering of the atoms in the molecule of the disubstituted thiourea.

N-(α-phenylethyl) N'-(ω-methylsulphinylpropyl) thiourea



Measyred and calculated values of structure factors. The phase angles are in degrees.

Table 4-2

Final fractional atomic coordinates and their estimated standard deviations in Å

Atom	x/a	y/b	c/z	σ(x)	σ(y)	σ(z)
C(1)	-0.1004	0.7825	0.4785	0.012	0.011	0.013
C(2)	-0.1337	0.5574	0.3824	0.012	0.009	0.014
C(3)	-0.1665	0.4707	0.2513	0.014	0.012	0.020
C(4)	-0.1272	0.3703	0.2460	0.016	0.013	0.017
C(5)	-0.1326	0.1604	0.1395	0.009	0.011	0.009
C(6)	-0.1438	-0.0380	0.0073	0.011	0.011	0.013
C(7)	-0.2150	-0.1022	-0.0732	0.013	0.012	0.016
C(8)	-0.0670	-0.0455	-0.0932	0.011	0.009	0.011
C(9)	-0.0145	-0.1462	-0.0623	0.011	0.010	0.012
C(10)	0.0551	-0.1576	-0.1467	0.012	0.011	0.010
C(11)	0.0733	-0.0744	-0.2780	0.015	0.013	0.014
C(12)	0.0230	0.0213	-0.3034	0.014	0.014	0.015
C(13)	-0.0476	0.0327	-0.2201	0.013	0.012	0.011
N(1)	-0.1584	0.2770	0.1324	0.008	0.007	0.008
N(2)	-0.1681	0.0896	0.0235	0.007	0.007	0.007
O	-0.2433	0.7302	0.3483	0.007	0.007	0.007
S(1)	-0.1536	0.7086	0.3226	0.003	0.003	0.002
S(2)	-0.0680	0.1071	0.2755	0.003	0.003	0.003

Table 4-3

Anisotropic temperature factors

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	0.082	0.061	0.116	-0.048	-0.051	-0.024
C(2)	0.077	0.035	0.145	-0.014	-0.037	0.016
C(3)	0.100	0.085	0.288	-0.224	-0.164	0.080
C(4)	0.128	0.136	0.176	-0.223	-0.103	0.114
C(5)	0.032	0.093	0.055	-0.007	-0.006	0.004
C(6)	0.056	0.067	0.116	-0.008	-0.045	-0.019
C(7)	0.090	0.083	0.152	-0.065	-0.069	-0.059
C(8)	0.074	0.047	0.083	-0.033	-0.036	0.028
C(9)	0.072	0.063	0.097	-0.062	-0.044	0.034
C(10)	0.126	0.076	0.065	-0.045	-0.069	0.051
C(11)	0.107	0.093	0.115	-0.058	0.015	0.035
C(12)	0.118	0.101	0.121	-0.013	0.045	0.071
C(13)	0.117	0.079	0.068	0.053	0.019	0.048
N(1)	0.062	0.058	0.091	-0.052	-0.005	0.022
N(2)	0.065	0.051	0.068	-0.043	-0.033	0.021
O	0.062	0.070	0.099	0.017	0.022	0.043
S(1)	0.053	0.061	0.061	0.005	0.016	0.006
S(2)	0.051	0.092	0.072	-0.012	-0.026	0.006

Table 4-4

Bond lengths and angles
and their estimated siandard deviations

S(1)-C(1)	1.790	± 0.013	N(2)-C(6)	1.469	± 0.013
S(1)-C(2)	1.773	0.010	C(6)-C(7)	1.533	0.017
S(1)-O	1.514	0.007	C(6)-C(8)	1.531	0.016
C(2)-C(3)	1.568	0.021	C(8)-C(9)	1.432	0.014
C(3)-C(4)	1.282	0.019	C(8)-C(13)	1.421	0.015
C(4)-N(1)	1.504	0.017	C(9)-C(10)	1.360	0.016
N(1)-C(5)	1.355	0.013	C(10)-C(11)	1.479	0.018
C(5)-N(2)	1.390	0.012	C(11)-C(12)	1.359	0.020
C(5)-S(2)	1.682	0.010	C(12)-C(13)	1.368	0.019

C(1)S(1)O	107.4	± 0.5°	N(2)C(6)C(8)	109.3	±0.8°
C(2)S(1)O	106.6	0.5	N(2)C(6)C(7)	106.0	0.8
C(1)S(1)C(2)	97.1	0.6	C(7)C(6)C(8)	110.9	1.0
S(1)C(2)C(3)	107.7	0.9	C(6)C(8)C(9)	116.0	0.9
C(2)C(3)C(4)	112.2	1.4	C(6)C(8)C(13)	125.5	1.0
C(3)C(4)N(1)	116.1	1.4	C(9)C(8)C(13)	118.3	1.0
C(4)N(1)C(5)	120.8	0.9	C(8)C(9)C(10)	118.9	1.0
N(1)C(5)S(2)	124.1	0.7	C(9)C(10)C(11)	121.1	1.1
N(1)C(5)N(2)	111.6	0.8	C(10)C(11)C(12)	118.5	1.3
N(2)C(5)S(2)	124.2	0.8	C(11)C(12)C(13)	120.5	1.3
C(5)N(2)C(6)	119.3	0.8	C(12)C(13)C(8)	122.2	1.1

Table 4-5

Intramolecular non-bonded distances <3.5Å

C(1)...O	2.67	C(7)...C(9)	3.34
C(1)...C(2)	2.67	C(7)...C(13)	3.38
C(2)...C(4)	2.37	C(8)...C(10)	2.40
C(2)...O	2.64	C(8)...C(12)	2.44
C(3)...N(1)	2.37	C(8)...N(2)	2.45
C(3)...S(1)	2.70	C(8)...C(11)	2.82
C(3)...O	3.24	C(9)...C(13)	2.45
C(4)...C(5)	2.49	C(9)...C(11)	2.47
C(4)...S(2)	3.07	C(9)...C(12)	2.83
C(5)...C(6)	2.47	C(10)...C(12)	2.44
C(5)...C(8)	3.20	C(10)...C(13)	2.77
C(6)...C(9)	2.51	C(11)...C(13)	2.37
C(6)...C(13)	2.62	C(13)...N(2)	2.94
C(6)...S(2)	3.06	N(1)...N(2)	2.27
C(7)...N(2)	2.40	N(1)...S(2)	2.69
C(7)...C(8)	2.52	N(2)...S(2)	2.72

Intermolecular distances <4Å

O...N(2)V	2.88	C(10)...C(12)III	3.79
O...N(1)V	2.92	C(7)...S(2)II	3.80
O...C(5)V	3.44	C(1)...C(12)IV	3.81
C(2)...C(10)I	3.52	C(2)...C(11)I	3.81
O...C(11)VIII	3.53	O...C(10)VIII	3.83
C(5)...C(7)VI	3.57	C(9)...C(12)III	3.84
C(4)...C(9)I	3.58	C(1)...C(11)IV	3.87
C(10)...C(13)III	3.60	C(1)...C(13)IV	3.87
C(2)...S(2)I	3.63	S(1)...C(6)VII	3.88
C(4)...C(10)I	3.64	S(2)...C(9)I	3.90
C(2)...C(9)I	3.66	S(1)...S(2)I	3.91
C(11)...N(1)III	3.72	C(2)...C(8)I	3.93
C(7)...N(1)II	3.79	C(7)...N(2)II	3.95

The Roman numerals refer to the following equivalent positions:

I: $-x, \frac{1}{2}+y, \frac{1}{2}-z$

II: $-\frac{1}{2}-x, -y, -\frac{1}{2}+z$

III: $-x, -\frac{1}{2}+y, -\frac{1}{2}-z$

IV: $x, 1+y, 1+z$

V: $-\frac{1}{2}-x, 1-y, \frac{1}{2}+z$

VI: $-\frac{1}{2}-x, -y, \frac{1}{2}+z$

VII: $x, 1+y, z$

VIII: $-\frac{1}{2}+x, \frac{1}{2}-y, -z$.

From the results of Kjaer et al. (1958, 1960) on the basis of optical rotatory dispersion studies, the (R)-configuration can be assigned to all naturally derived sulphoxide mustard oils (II) as well as to the glucosides (I) whence they are derived.

Anderson (1964) by the optical rotatory dispersion comparison with a synthetic sulphoxide compound, (-)-N-methyl-N-[3-(methylsulphinyl)propyl] anibine, $CH_3SO(CH_2)_2N\begin{smallmatrix}\diagup CH_3\\ \diagdown C_6H_5\end{smallmatrix}$, of known configuration, (R)-, has tentatively assigned the (R)-configuration to this series of sulphoxide mustard oils (II).

Mislow et al. (1965) assigned the (S)-configuration to their synthetic sulphoxide compound, (+)-methyl-n-butyl sulphoxide, $CH_3CH_2CH_2CH_2\overset{\ddot{\downarrow}}{\underset{\underset{O}{\uparrow}}{S}}CH_3$, and by comparing with optical rotatory dispersion curves, have also proposed the (R)-configuration for the series of sulphoxide mustard oils (II). Both these conclusions are consistent with the result obtained in the present work.

Molecular dimension. The bond lengths and valency angles around the sulphur atom in the sulphoxide grouping is listed below together with the values in (+)-S-methyl-L-cysteine sulphoxide, [(+)MCS] (Hine, 1962), dimethyl sulphoxide, (Bastiansen and Viervoll, 1948), and diphenyl sulphoxide (Abrahams and Grenville-Wells, 1956) for comparison:

	[(+)MCS]	$(CH_3)_2SO$	$(C_6H_5)_2SO$	This structure
S-O	1.488±0.012	1.47±0.03	1.473±0.015	1.514±0.007
S-C(1)	1.814±0.017	1.82	1.760±0.015	1.790±0.013
S-C(2)	1.833±0.022			1.773±0.010
C(1)-S-O	107.5±0.7	107±5	106.2±0.7	107.4±0.5
C(2)-S-O	104.4±0.8			106.6±0.5
C(1)-S-C(2)	96.7±1.0	100±5	97.3±1.0	97.1±0.6

Athough the S-O bond is longer, while the S-C bonds are shorter than the results in [(+)MCS] and $(CH_3)_2SO$, none of the differences is significant.

Around the thioamide grouping, the bond lengths and valency angles are also listed and compared with the results in thiourea (Kunchur and Truter, 1958), in ethylenethiourea (Wheatley, 1953), and trimethylene thiourea (Dias and Truter, 1964):

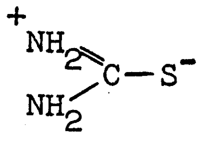
	Thiourea	Ethylene thiourea	Trimethylene thiourea	This structure
C-N	1.33±0.02	1.322±0.008	1.334±0.006	1.355±0.013 1.390±0.012
C-S	1.72±0.02	1.708±0.008	1.722±0.007	1.682±0.010
N-C-N	115.6±1.1	110.2±1.4	120.5±0.5	111.6±0.8
S-C-N	122.2±0.6	124.8	119.8±0.4	124.1±0.7 124.2±0.8

Other carbon-sulphur double bonds have values of 1.627Å in 4-methyl-1,2-dithia-4-cyclopentene-3-thione, (Jeffrey and Shiono, 1959); 1.65Å in 2-p-methoxyphenyl-3,4-dibenzyl-1,3,4-thiadiazolidine-5-thione, (Karle and Karle, 1965); and 1.647Å in 4,4-dihydroxythiobenzophenone monohydrate, (Manojlović and Edmunds, 1965).

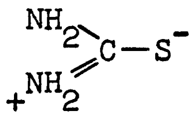
The difference in the C=S bond compared with trimethylenethiourea is just outside the significant test limit.

The difference in the two C-N bonds in the present work is not significant. Both these values are longer than that in the simple thiourea, though the differences are not significant. The longer of the two C-N bonds (1.390Å) is significantly longer than the values in the two cyclic compounds.

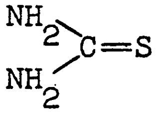
Kunchur and Truter (1958), after comparing the carbon-nitrogen and carbon-sulphur bond lengths with those in other molecules containing these bonds, suggested that structures (a) and (b) make a large contribution to the bonding while with some contribution from structure (c):



(a)



(b)



(c)

The results in the present work also suggest that (a), (b) and (c) all contribute to the bonding. The lengthening of the C-N bonds and the shortening of the C-S bond when compared with the values in the unsubstituted thiourea indicate that in the present structure, the contribution from (c) must be greater than in the case of the simple thiourea.

All the other four shorter C=S bonds mentioned have values greater than the sum of the double bond radii for C and S atoms (=1.607Å; Pauling, 1960), this would probably mean that the difference is again due to resonance.

In N-phenyl-N'-benzoylselenourea (Hope, 1965), the two C-N bonds are 1.40 and 1.32Å. The C-Se bond of 1.82Å is also intermediate between the sum of covalent bond radii of 1.94Å for the single bond and 1.74Å for the double bond.

The valency angles around the C(5) atom are in good agreement with the values in the simple thiourea.

The four atoms in the thioamide grouping are coplanar. The deviation of the central atom C(5) from the plane through the other three atoms is 0.0145Å. When a mean plane (Schomaker et al. 1959) was calculated through these four atoms N(1), N(2), S(2), and C(5), their deviations from the plane are -0.004, -0.004, -0.003 and +0.011Å respectively. The maximum deviation, in the case of C(5), is less than 1.5 times the estimated standard deviation.

The equation of the plane is

$$0.776X + 0.214Y - 0.606Z + 2.030 = 0 \quad \ldots \quad (4.1)$$

Around the asymmetric carbon atom, the values of 1.533Å for C(6)-C(7), 1.531Å for C(6)-C(8), and 1.469Å for C(6)-N(2) agree very well with the standard values (Tables of Interatomic Distances, 1958) of 1.541Å for $C(sp^3)$-$C(sp^3)$; 1.525Å for

$C(sp^3)$-C(aromatic); and 1.472Å for $C(sp^3)$-$N(sp^2)$. Although the hydrogen atom has not been located, the remaining three valency angles of 106.0°, 109.3°, and 110.9° are close to the normal tetrahedral angle of 109°28'.

The C-C bonds in the benzene ring vary from 1.359Å to 1.479Å, the difference being 4.5 times the estimated standard deviation. This must certainly reflect some experimental error in the data rather than indicating a real difference in the bond length. The mean value over the six bonds is 1.403Å, not significantly different from the value of 1.397Å in benzene. The valency angles, varying from 118.3° to 122.1° are all close to 120°.

The mean plane through the 7 atoms C(6), C(8), C(9), C(10), C(11), C(12), C(13) has equation

$$0.499X + 0.577Y + 0.647Z + 1.378 = 0 \quad \ldots \quad (4.2)$$

The deviations of these atoms from the plane are +0.005, -0.023, +0.015, -0.019, +0.027, -0.026, and +0.021Å respectively.

The value of 1.504A for the bond C(4)-N(1) does not differ significantly from the value of 1.469Å for C(6)-N(2), and the standard value of 1.472Å.

The C-C bond lengths in the middle of the molecule are 1.568A for C(2)-C(3) and 1.282Å for C(3)-C(4). The latter value is highly anomalous for a C-C single bond. There is certainly not a double bond in its place because a mass spectrometric examination of the compound revealed no significant peak at m/c=282, and the analytical results are in accord with a saturated chain [Found: C 54.85,54.8 ; H 7.1, 7.2; N 9.6,9.75. Calculated for $C_{13}H_{20}ON_2S_2$: C 54.9; H 7.1; N 9.85. For $C_{13}H_{18}ON_2S_2$: C 55.3; H 6.4; N 9.9 %].

As pointed out in section 4.4, the central atom C(3) occurred in the electron-density distributions is markedly elongated perpendicular to the chain direction, and correspondingly the least squares refinement assigned a very anisotropic thermal vibration to this atom and to a lesser extent, to the atom C(4). It is clear that there is some disorder associated with the position of this atom. A consideration of the effect of packing will indicate that the disorder is most likely.

As shown in Table 4-7, all intermolecular distances are greater than the sum of the van der Waals radii except the distances of 2.919Å for N(1)...O, and 2.884Å for N(2)...O. This strongly suggested that they are hydrogen bonded, although the hydrogen atoms have not been located to confirm this. These distances agree very well with other NH...O distances, e.g. 2.89, 2.81 and 2.83Å in (+)-S-methyl-L-cysteine sulphoxide (Hine, 1962); 2.84 and 2.85Å in thiourea dioxide (Sullivan and Hargreaves, 1962).

The two shortest C...O contacts are 3.44Å and 3.53Å, both greater than the sum of the van der Waals radii. It is therefore reasonable to say that the molecules in the unit cell are held together at both ends by hydrogen bonds (shown by dotted lines in Figs. 4-3 and 4-4) between the oxygen and nitrogen atoms, and only very weak van der Waals interaction. This leaves the middle atoms of the chain adequate space to vibrate and in the case of the atoms C(3), and to a lesser extent C(4), there is certain degree of disorder.

PART V

The Structure of 1,6-thiacyclodeca-cis-3,cis-8-diene Bis(mercuric chloride).

5.1. INTRODUCTION

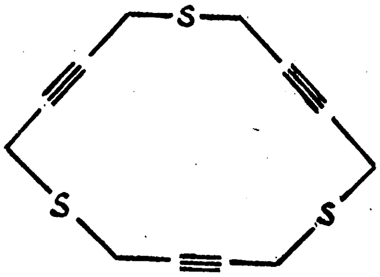
In their attempt to prepare a fifteen-membered trithia-compound, 1,6,11-trithiacyclopentadeca-3,8,13-triyne (I), Eglinton et al. (1964) obtained a ten-membered dithia-compound, 1,6-dithiacyclodeca-3,8-diyne (II). The result of their x-ray analysis showed that it has a chair-like conformation, as shown in (IIa).

Catalytic hydrogenation of (II) yielded the diene which has been shown by spectroscopic studies to be 1,6-dithiacyclodeca-cis-3,cis-8-diene. A solution of the diene and mercuric chloride in hot ethanol deposited a crystalline bis(mercuric chloride) complex.

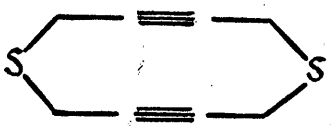
The obvious postulate for the structure of this complex is (III) with each mercury atom in three-fold coordination. The actual crystal structure determined by x-ray analysis is of the polymeric type (IV).

5.2. EXPERIMENTAL

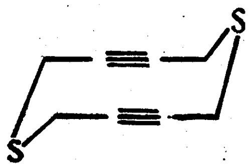
Crystal data. 1,6-dithiacyclodeca-cis-3,cis-8-diene bis(mercuric chloride), $C_8H_{12}S_2Hg_2Cl_4$. Molecular weight = 714.5. Monoclinic, $\underline{a}$=7.29, $\underline{b}$=17.01, $\underline{c}$=6.20 Å, β=92°43'. Volume of unit cell = 768.0 Å^3. Number of asymmetric units per unit cell = 2. Density(cal.)=3.083 gm cm^{-3}. The only absent spectra are 0k0 when k is odd. The space group is either $P2_1/m$ (C_{2h}^2) or



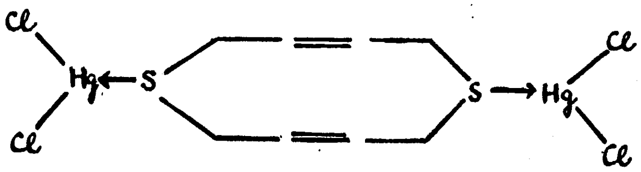
(I)



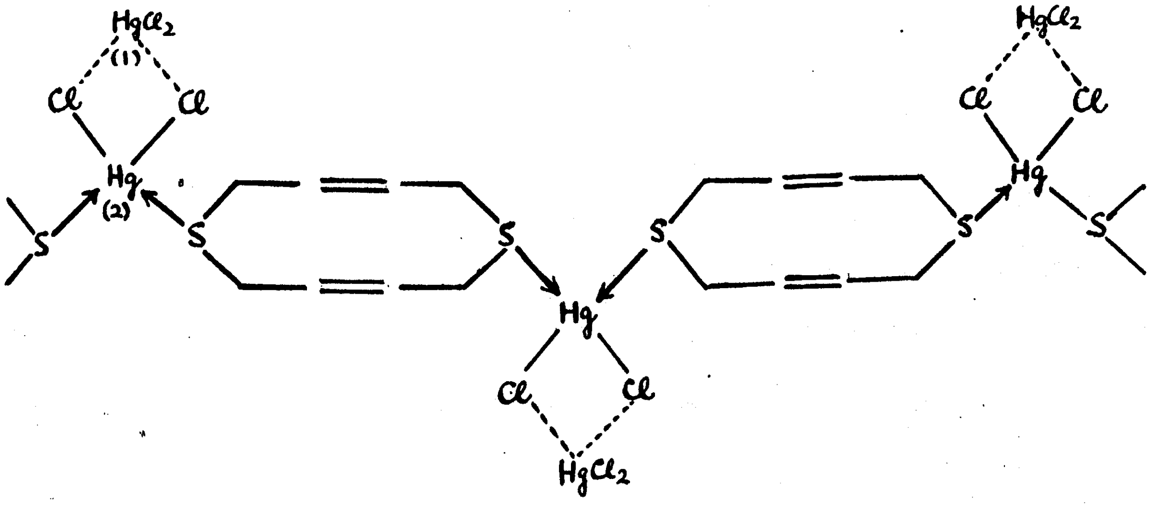
(II)



(IIa)



(III)



(IV)

$P2_1$ (C_2^2). Absortion ceofficient for x-rays ($\lambda = 1.542$ Å), $\mu = 466.3$ cm^{-1}. Total number of electrons per unit cell, F(000) = 620. Total number of independent observed structure amplitudes = 1158.

Intensity data. Equatorial and equi-inclination upper layer Weissenberg photographs were taken about the c axis. The intensities of hk0, hk1, ... , hk$\bar{1}$, ... , hk$\bar{4}$ were estimated visually by comparing with a calibrated step-wedge. Lorentz, polarization factors and the Tunell rotation factor for the upper-layers were applied. No corrections for absorption have been made. The observed structure amplitudes were put to the absolute scale by comparison with the calculated structure factors.

5.3. DETERMINATION OF THE STRUCTURE

It should be possible to distinguish between the space groups $P2_1$ and $P2_1/m$ from the Patterson function unless the Hg atoms are at special positions. Patterson projections P(vw) and P(uv) were calculated and shown in Fig. 5-1. These suggested that the y- coordinates of the two mercury atoms are at 1/4 and 3/4. The x- and z- coordinates were found from the Patterson projections. A better set of coordinates was obtained from the Harker section P(U½W) and they are listed as follows:

	x/a	y/b	z/c
Hg(1)	0.489	0.750	0.102
Hg(2)	0.134	0.250	0.409.

In Fig. 5-1(a), the Hg(1)-Hg(1) vector is marked A;

and the Hg(2)-Hg(2) vector is marked B;

the Hg(1)-Hg(2) vector due to $x_1 - x_2$, $y_1 - y_2$ (and $x_1 - x_2$, $-1/2+y_1+y_2$ if space group is $P2_1/m$) is marked C;

the Hg(1)-Hg(2) vector due to x_1+x_2, $-1/2+y_1-y_2$ (and x_1+x_2, y_1+y_2 if space group is $P2_1/m$) is marked D.

In Fig. 5-1(b), the Hg(1)-Hg(1), Hg(2)-Hg(2) vectors and the Hg(1)-Hg(2) vector due to y_1-y_2, z_1-z_2 all amalgamated into the very elongated peak E. The Hg(1)-Hg(2) vector due to $-1/2+y_1-y_2$, z_1+z_2 (and y_1+y_2, z_1+z_2 if space group is $P2_1/m$) is marked F.

The fact that the cross-vector peaks are higher suggests that they are due to overlap of vectors generated by the centre of symmetry in the space group $P2_1/m$. Also, with the above set of coordinates, both the Hg atoms would make no contribution to structure factors whenever k is odd if the space group is taken as $P2_1$. This is impossible because the magnitude of the Fo with k odd are comparable with those having k even. Therefore the space group must be $P2_1/m$.

Using the coordinates of the two Hg atoms and a temperature factor corresponding to $B = 2.5$ Å^2, a set of structure factors was calculated. This gave an R factor of 0.312.

The signs of the Fcal were used in the calculation of

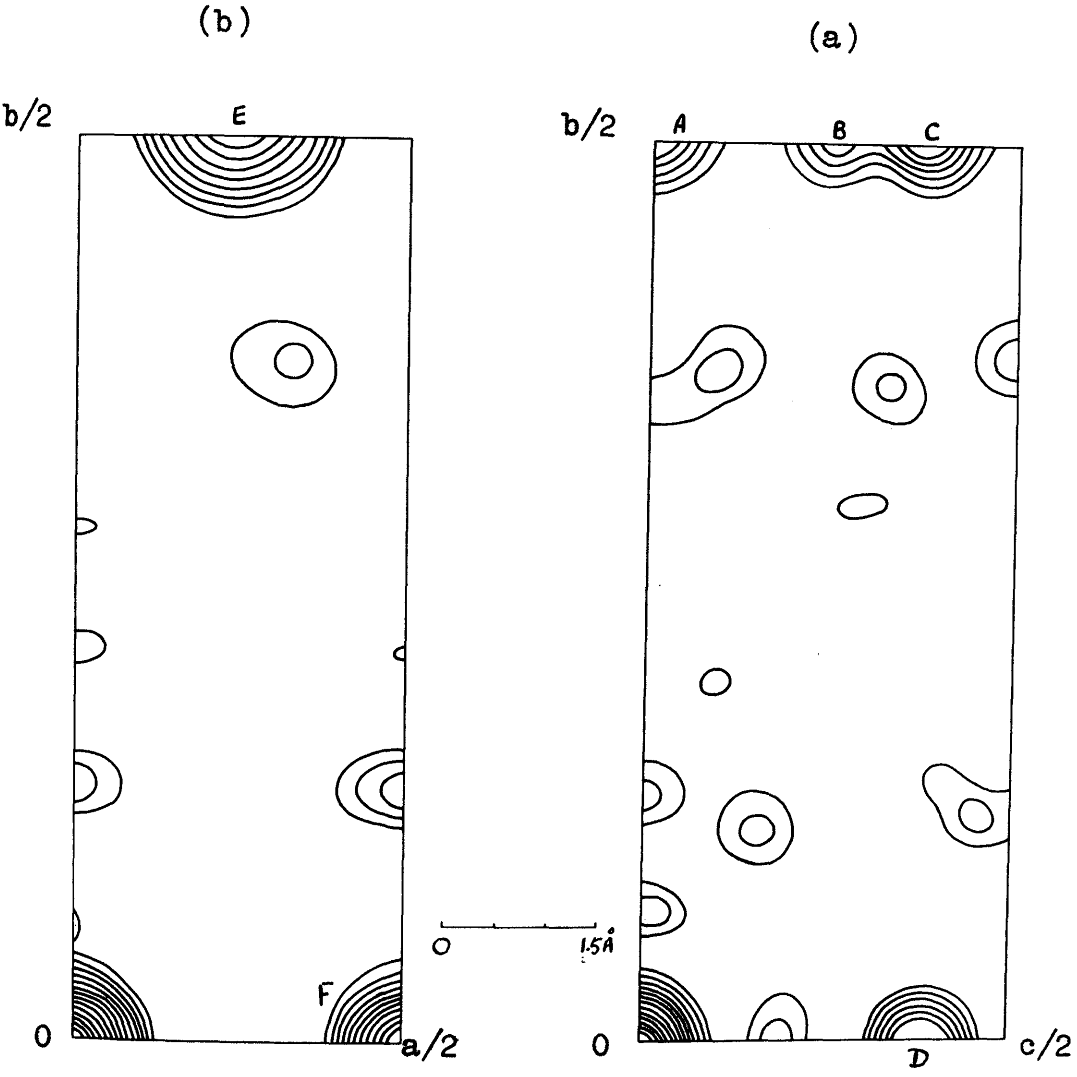


Fig.5-1. Patterson projections (a) along the c axis, (b) along the a axis. Contour scale arbitrary. The peaks mentioned in the text are marked.

a three-dimensional Fo Fourier synthesis. The electron-density distribution, as contoured sections drawn parallel to (001) on sheets of glass and stacked in a metal frame, had very strong diffraction effects and pronounced ripples appeared in the mirror planes at $y=1/4$ and $y=3/4$. This made the interpretation of the Fourier maps very difficult. To overcome that, a three-dimensional difference synthesis with (Fo-Fc) as coefficients was computed. By comparing corresponding sections of the two Fourier syntheses, the peaks which did not appear in the difference map must be due to diffraction effects and were ruled out. The electron-density distribution was then interpretable. Even the carbon atoms were recognisable at this stage, although their peaks were not so distinct. But only five peaks around the two mercury atoms could be picked up. They were all weighted as chlorine atoms and together with the improved coordinates of the mercury atoms, used to calculate another set of structure factors. The R factor fell to 0.233.

The main features of the structure were revealed in the second three-dimensional electron-density distribution. Part of the structure was seen to consist of polymeric chains running along the unique axis and part of the structure consists of a discrete molecule of mercuric chloride which is nearly, but not quite, linear. However only one chlorine and two sulphur atom peaks could be seen. The peaks for the carbon atoms were more distinct this time and they were included with the improved coordinates of the two Hg atoms, two Cl atoms, and one S atom in the calculation of another set of structure factors. This gave an R factor of 0.209.

Another three-dimensional Fo and (Fo-Fc) syntheses were computed. Although there were positive regions in the expected position of the missing chlorine atom, it was still not possible to derive a set of coordinates for it.

It was decided to refine the positional and thermal parameters of the other atoms by least squares procedure. After three cycles of least squares refinement, all the observed structure factors were put into another Fo and (Fo-Fc) syntheses. In the (Fo-Fc) maps, a rather pronounced peak appeared within bonding distance from the Hg(2) atom, although no corresponding peak in the Fo maps was present. The position of this peak was almost above the Hg(2) atom along the (001). Owing to the series termination effects, it is conceivable why it did not appear in the Fo synthesis. The stereochemistry of the Hg(2) atom could then be established as distorted tetrahedral.

Refinement of the structure was completed after six cycles of least squares calculations using a program written by Rollett (1961). The parameter shifts in the final cycle were all less than their estimated standard deviations. The R factor of the final set of structure factors is 0.136.

The weights, $\sqrt{w}$, used in the least squares refinement were as follows:

if $|Fo| \leqslant F^*$, $\sqrt{w} = |Fo| / F^*$;

if $|Fo| > F^*$, $\sqrt{w} = F^*/|Fo|$; where $F^* = 32.0$.

A remark about this last chlorine atom is that even in the final Fo synthesis, it still only appears as a rather low peak, comparable only to the peaks for the carbon atoms. However, in a Fourier synthesis with $(Fo-F^{Hg}_{cal})$ as coefficients, the peak height of this chlorine atom is comparable with the other chlorine atoms.

The course of analysis is summerized in Table 5-1.

<u>table 5-1</u> Course of analysis

1. Patterson function. Positions of 2 Hg atoms located.
2. 1st S.F. calculation. 2 Hg atoms, $B=2.5Å^2$, R=0.312.
3. 1st Fo and (Fo-Fc) syntheses.
4. 2nd S.F. calculation. 2 Hg and 3 Cl atoms R=0.233.
5. 2nd Fo and (Fo-Fc) syntheses.
6. 3rd S.F. calculation. 2Hg, 2Cl, 1S and 4C atoms, R=0.204.
7. 3 cycles of least squares refinement. R → 0.185.
8. 3rd Fo and (Fo-Fc) syntheses. Remaining Cl atom located.
9. 4th S.F. calculation. All atoms, coordianates as in (6), R=0.195.
10. 4th S.F.L.S. Cycle. Atomic parameters from (7) plus the last Cl atom, R=0.165.
11. 5th to 9th S.F.L.S. cycles. R=0.165 → 0.139.
12. Final S.F. calculation. R=0.136.
13. Fo and $(Fo-F^{Hg}_{cal})$ syntheses.

5.4. RESULTS AND DISCUSSION

The final fractional atomic coordinates and their estimated standard deviations are listed in Table 5-2. The anisotropic temperature factors are given in Table 5-3. The interatomic distances and valency angles are shown in Table 5-4. The observed and calculated structure factors of the 1158 independent reflections are listed in Table 5-5.

The structure of this mercuric chloride complex is of the polymeric type in which one mercury atom is tetrahedrally coordinated by two sulphur and two chlorine atoms while the other mercury atom is in an only slightly distorted mercuric chloride molecule. The arrangement of the molecules in the crystal as viewed in projections along the a and c axes is shown in Fig. 5-2 and Fig. 5-3. The packing within one layer of mercury atoms is shown in Fig. 5-4. The final electron-density distribution is shown in Fig. 5-5 as superimposed contour sections drawn parallel to (001).

The environment of the Hg(2) atom is not ideally tetrahedral; whereas the angle $SHg(2)S_{VI}$ is 109°, the angle Cl(2)Hg(2)Cl(3) is only 99°. On the other hand, the bond lengths for Hg(2)-Cl(2) and Hg(2)-Cl(3) of 2.50 and 2.51Å agree extremely well with the average value of 2.50Å for the tetrahedral $HgCl_4^{2-}$ anion in perloline tetrachloromercurate (II) (Jeffreys et al. 1963). The deviations of the angle from 109°28' can probably be ascribed to the involvement of the chlorine atoms in bonding with $Hg(1)_{IV}$; a value of 109°

for Cl(2)Hg(2)Cl(3) would necessarily involved unreasonably small values for the other angles in the ring Cl(2)Hg(2)Cl(3) $Hg(1)_{IV}$ (Fig. 5-4) (since the sum of the internal angles in a planar four-membered ring must equal 360°) and might bring the two non-bonded mercury atoms too close together. The Hg(2)-S distance of 2.53Å is in good agreement with values of 2.53Å in metacinnabarite where the Hg(II) atom is regularly tetrahedral (Aurivillius, 1950); an average value of 2.55Å in bis(ethylene diamine) copper(II) tetrathiocyanatomercurate(II) (Scouloudi, 1953). Values of 2.54 and 2.56Å have been reported in $K_2Hg(SCN)_4$ (Zvonkova, 1952), and in $CoHg(SCN)_4$ (Jeffery, 1963) respectively in which there are very small deviations from the regular tetrahedral angle.

The Hg(1) atom is in an only slightly distorted mercuric chloride molecule with the $Cl(1)Hg(1)Cl(1)_V$ angle of 168°. The Hg(1)-Cl(1) distance of 2.30Å is the same as the result in cyclohexane-1,4-dione mercuric chloride adduct where the Cl-Hg-Cl angle is 173° (Groth and Hassel, 1964). Values of 2.30 and 2.33Å in the discrete molecule of $HgCl_2$ (angle Cl-Hg-Cl is 172°) which exists with the $Cl\text{-}Hg\text{-}S(C_2H_5)_2^+$ and Cl^- ions in the structure of $2HgCl_2.(C_2H_5)_2S$ (Brändén, 1964). In CH_3HgCl (Gordy and Sheriden, 1954) and in $HgCl_2$ (Allen and Sutton, 1950), the Hg-Cl distance is 2.28Å.

The $Hg(1)\text{-}Cl(2)_I$ and $Hg(1)\text{-}Cl(3)_{II}$ distances of 3.02 and 2.93Å, respectively, are bridging distances comparable to other Cl-bridges found in $HgCl_2$ crystal and other $HgCl_2$ complexes. When the two other contacts $Hg(1)...Cl(2)_{II}$, 3.21Å, and $Hg(1)...Cl(3)_{III}$, 4.44Å, are taken into account, the coordination of the Hg(1) atom can be described as distorted octahedral, as in crystalline mercuric chloride (Braekken and Scholten, 1934; Grdenić, 1950). Alternatively, if the distance 4.44Å is considered too long even for a very weak bonding, Hg(1) can be described as five-coordinated with a square pyramidal configuration.

In the 1,6-dithiacyclodeca-cis-3,cis-8-diene, values of 1.84 and 1.87Å for $C(1)-S_{IX}$ and C(4)-S respectively are longer than the values of 1.82Å in dimethylsulphoxide (Bastiansen and Viervoll, 1948), and a mean of 1.82Å for S-C single bond (Abrahams, 1956). But with an estimated standard deviation as high as 0.05Å, the differences are within experimental errors. The values of 1.30Å (e.s.d. 0.06Å) for the C=C bond and 1.52 and 1.60Å (e.s.d. 0.06Å) for the $C(sp^2)-C(sp^3)$ bonds do not differ significantly from the accepted values of 1.33 and 1.51Å (Tables of Interatomic Distances, 1958).

The conformation of the ten-membered ring is of the distorted chair-type, as is also the case in 1,6-dithiacyclodeca-3,8-diyne (Eglinton et al. 1964).

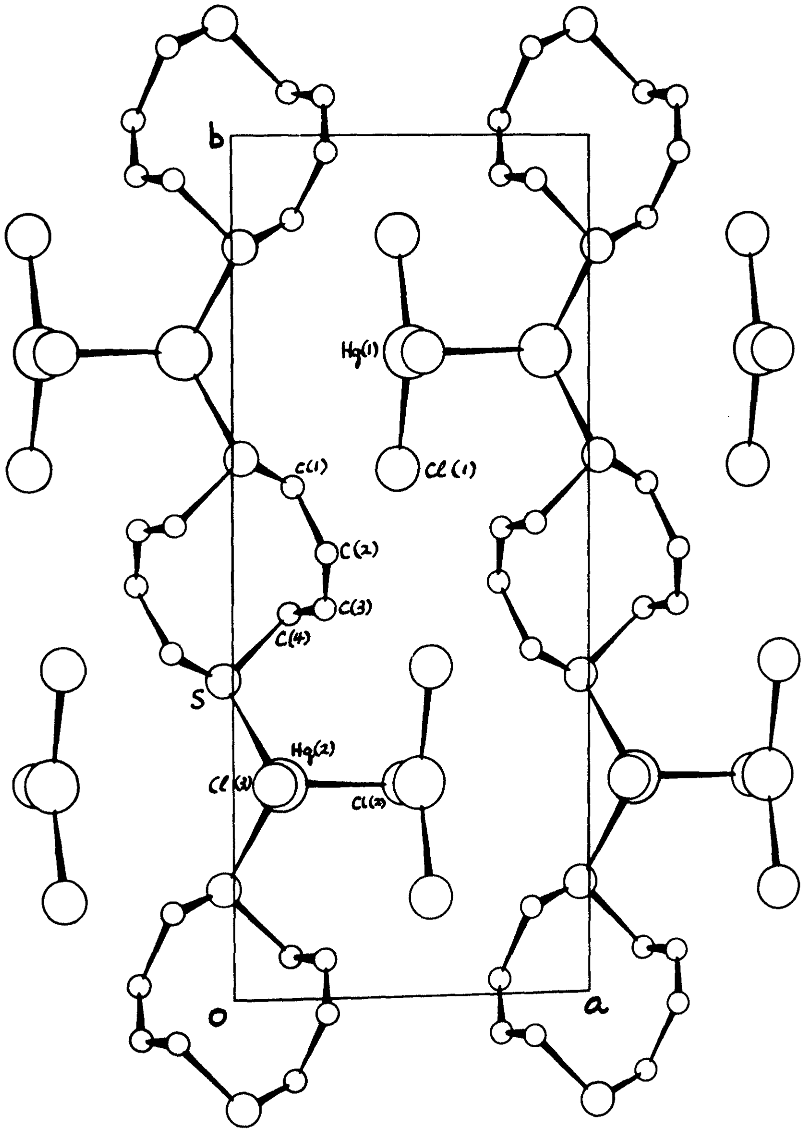


Fig.5-2. The packing of the molecules in the crystal as seen when viewed along the c-axis.

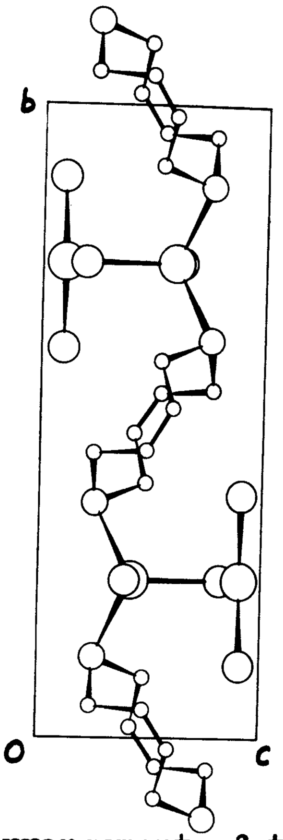


Fig. 5-3. The arrangement of the molecules in the crystal as seen when viewed along the a-axis.

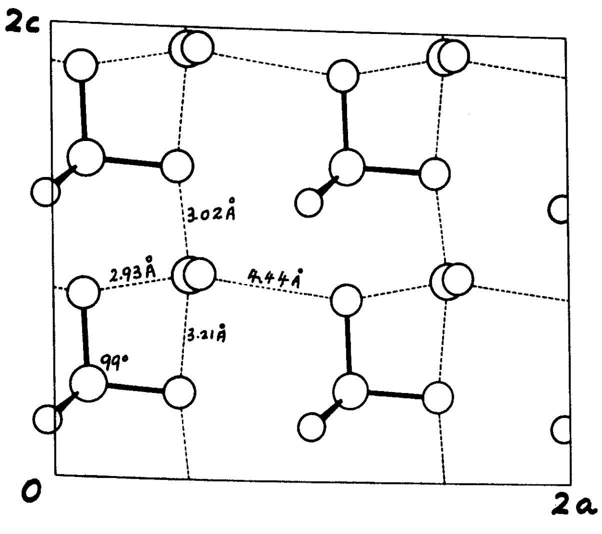


Fig. 5-4. The packing of the atoms within one layer of mercury atoms and the attached chlorine and sulphur atoms.

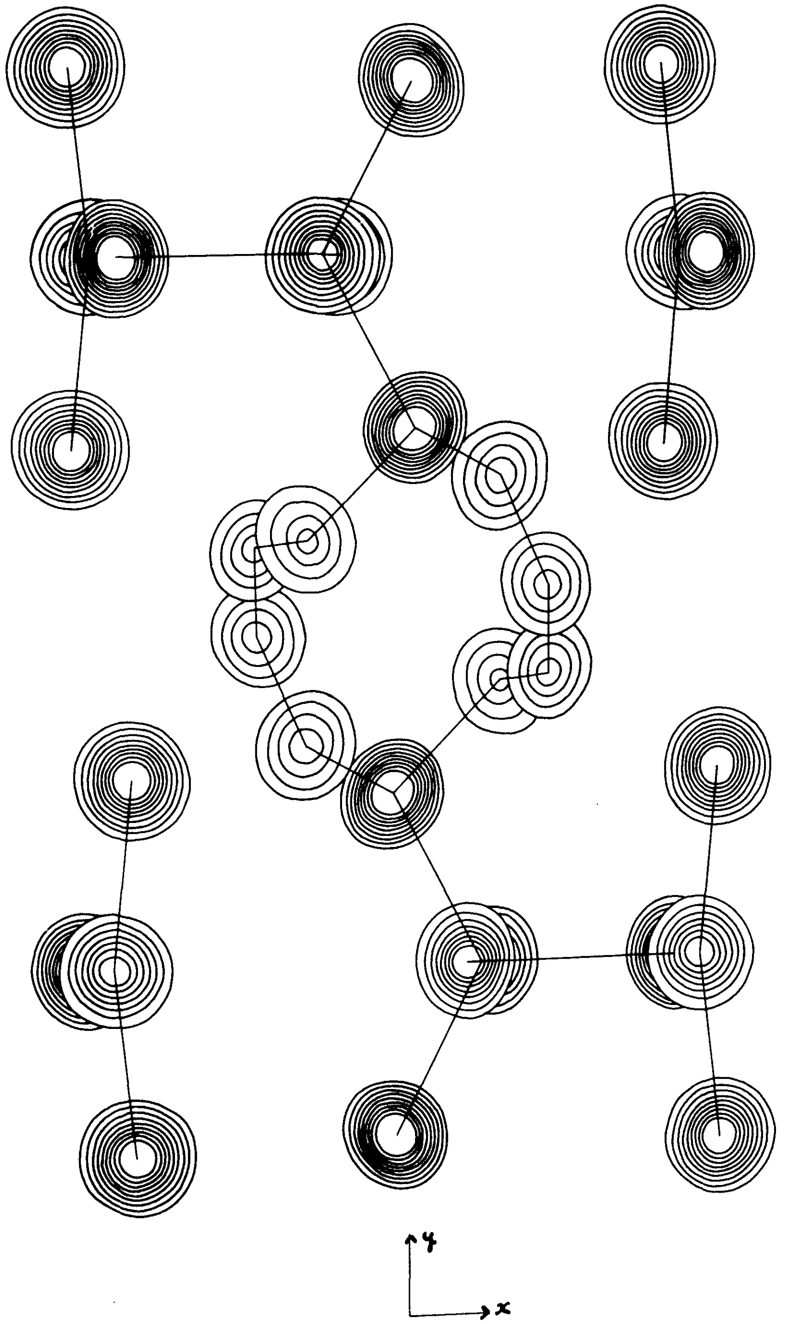


Fig.5-5. The final three-dimensional electron-density distribution shown by means of superimposed contour sections drawn parallel to (001). Contour interval 10 eÅ^{-3} around the Hg atoms, 2 eÅ^{-3} around the Cl and S atoms, and 1.5 eÅ^{-3} around the C atoms.

Table 5-2

Final fractional atomic coordinates and their estimated standard deviations in Å

Atom	x/a	y/b	z/c	σ(x)	σ(y)	σ(z)
Hg(1)	0.4874	0.7500	0.1002	0.002	0	0.003
Hg(2)	0.1296	0.2500	0.4068	0.002	0	0.003
Cl(1)	0.4538	0.6155	0.0959	0.011	0.011	0.014
Cl(2)	0.4704	0.2500	0.3813	0.014	0	0.017
Cl(3)	0.1141	0.2500	0.8108	0.016	0	0.022
S	-0.0248	0.3714	0.2462	0.009	0.009	0.012
C(1)	0.1642	0.5963	0.5308	0.036	0.043	0.050
C(2)	0.2548	0.5182	0.5858	0.039	0.039	0.054
C(3)	0.2510	0.4544	0.4704	0.036	0.041	0.053
C(4)	0.1554	0.4497	0.2340	0.038	0.038	0.054

Table 5-3

Anisotropic temperature factors

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Hg(1)	0.048	0.037	0.035	-	-0.004	-
Hg(2)	0.030	0.023	0.050	-	0	-
Cl(1)	0.043	0.034	0.035	-0.004	-0.008	-0.016
Cl(2)	0.031	0.032	0.020	-	-0.004	-
Cl(3)	0.033	0.059	0.038	-	0.014	-
S	0.028	0.023	0.029	-0.027	-0.016	0.014
C(1)	0.021	0.042	0.044	-0.012	0.041	0.002
C(2)	0.030	0.036	0.038	-0.031	-0.006	0
C(3)	0.019	0.034	0.059	-0.006	-0.001	0.004
C(4)	0.034	0.026	0.054	0.008	0.036	-0.010

Table 5-4

Interatomic distances and angles

C(4)-S	1.87±0.04Å	C(3)...C(1)IX	3.15Å
C(1)-S IX	1.84±0.05	C(4)...C(2)IX	3.29
C(1)-C(2)	1.52±0.06	C(2)...C(1)IX	3.67
C(2)-C(3)	1.30±0.06	C(3)...C(2)IX	3.72
C(3)-C(4)	1.60±0.07	C(3)...S IX	3.85
Hg(1)-Cl(1)	2.30±0.01	C(3)...C(3)VII	3.95
Hg(2)-Cl(2)	2.50±0.01	S ... Cl(1)VIII	3.70
Hg(2)-Cl(3)	2.51±0.02	C(1)...Cl(2)II	3.75
Hg(2)-S	2.53±0.01	Cl(1)...C(2)X	3.80
Hg(1)...Cl(3)II	2.93	Cl(1)...Cl(2)I	3.80
Hg(1)...Cl(2)I	3.02	Cl(3)...Cl(2)VI	3.81
Hg(1)...Cl(2)II	3.21	C(2) ...C(2)VII	3.83
C(1) ...Hg(2)III	3.41	C(1) ...Cl(3)III	3.88
C(3) ...Cl(1)VII	3.57	Cl(1)...Cl(3)II	3.91
C(4)... Hg(2)VI	3.57	Cl(1)...Cl(2)II	3.98
C(3) ...Hg(2)VI	3.60	Hg(2)...Hg(1)IV	4.04
C(2) ...Cl(1)VII	3.63	Hg(1)...Hg(2)I	4.30
C(3) ...C(2)VII	3.67	Hg(1)...S VIII	4.42
		Hg(1)...Cl(3)III	4.44

The Roman numerals refer to the following equivalent positions:

I: 1-x, $\frac{1}{2}$+y, -z
II: 1-x, $\frac{1}{2}$+y, 1-z
III: -x, $\frac{1}{2}$+y, 1-z
IV: 1-x, -$\frac{1}{2}$+y, 1-z
V: x, 1$\frac{1}{2}$-y, z
VI: x, $\frac{1}{2}$-y, z
VII: 1-x, 1-y, 1-z
VIII: -x, 1-y, -z
IX: -x, 1-y, 1-z
X: x, y, 1+z
XI: 1+x, y, z.

Table 5-4 (cont.)

Valency angles

C(1)	C(2)	C(3)	128°	S	Hg(2)	$\mathrm{S_{VI}}$	109°
C(2)	C(3)	C(4)	123	Cl(2)	$\mathrm{Hg(1)_{IV}}$	$\mathrm{Cl(3)_{XI}}$	86
C(3)	C(4)	S	106	Cl(3)	$\mathrm{Hg(1)_{IV}}$	$\mathrm{Cl(3)_{XI}}$	162
C(2)	C(1)	$\mathrm{S_{IX}}$	110	Cl(3)	$\mathrm{Hg(1)_{IV}}$	$\mathrm{Cl(2)_{X}}$	92
C(4)	S	$\mathrm{C(1)_{IX}}$	103	Cl(3)	$\mathrm{Hg(1)_{IV}}$	Cl(2)	76
Hg(2)	S	$\mathrm{C(1)_{IX}}$	102	$\mathrm{Cl(1)_{IV}}$	$\mathrm{Hg(1)_{IV}}$	Cl(3)	96
Hg(2)	S	C(4)	107	$\mathrm{Cl(1)_{IV}}$	$\mathrm{Hg(1)_{IV}}$	Cl(2)	91
Cl(1)	Hg(1)	$\mathrm{Cl(1)_{V}}$	168	Hg(2)	Cl(2)	$\mathrm{Hg(1)_{IV}}$	89
Cl(2)	Hg(2)	Cl(3)	99				
S	Hg(2)	Cl(2)	114				
S	Hg(2)	Cl(3)	111				

Table 5-5

1,6-dithiacyclodeca-cis-3,cis-8-diene bis(mercuric chloride).

Measured and calculated values of structure factors.

h	k	ℓ	$\lvert F_o \rvert$	F_c
0	1	4	87.0	112.9
0	2	2	37.4	-38.8
		3	11.0	5.9
		4	89.8	138.1
0	3	1	36.6	-41.8
		2	164.8	-226.8
		3	58.7	61.0
		4	55.3	-63.9
0	4	0	190.9	256.1
		1	58.3	-61.5
		2	76.6	92.0
		3	9.0	-4.8
		4	117.5	-140.9
0	5	2	212.0	237.8
		3	58.8	-57.2
		4	84.5	88.4
0	6	0	281.4	-318.5
		1	7.6	5.1
		2	72.6	-77.1
		3	27.2	28.3
		4	112.1	122.7
0	7	1	19.8	-16.9
		2	246.1	-242.4
		3	22.2	-23.1
		4	89.8	-93.8
0	8	0	281.7	318.0
		1	15.0	10.7
		2	42.5	43.7
		3	21.2	-14.7
		4	114.4	-108.6
0	9	1	36.4	-26.8
		2	197.9	204.9
		4	90.4	79.6
0	10	0	232.2	-218.2
		1	32.3	27.3
		2	38.9	-34.1
		3	10.3	5.7
		4	87.5	79.5
0	11	2	163.7	-141.6
		3	54.2	41.3
		4	55.7	-46.1
0	12	0	143.9	137.8
		1	39.7	-30.8
		2	72.5	64.9
		4	92.7	-99.2
0	13	1	24.6	18.3
		2	171.8	146.5
		3	57.6	-43.9
		4	59.4	60.3
0	14	0	187.1	-172.1
		1	14.4	9.6
		2	60.7	-54.5
		3	21.7	15.7
		4	72.7	81.7
0	15	1	17.5	8.0
		2	136.2	-126.5
		3	10.2	-10.1
		4	57.9	-61.4
0	16	0	158.5	153.8
		4	46.3	-53.6
0	17	1	27.3	-22.8
		2	94.7	96.2
		4	37.6	38.7
0	18	0	104.4	-97.9
		1	28.6	27.7
		2	23.0	-21.2
		4	31.0	41.5
0	19	1	10.3	9.6
		2	66.8	-72.0
		3	21.7	27.4
0	20	0	79.2	74.8
		1	22.5	-21.8
		2	28.3	32.2
0	21	2	46.2	67.0
1	0	0	18.6	-35.2
		2	85.6	-15.9
		3	70.9	-60.5
		4	117.6	116.0
		-1	126.9	-163.4
		-2	136.7	-138.1
		-3	128.8	104.6
		-4	78.5	78.5
1	1	0	72.6	-105.8
		1	48.8	-64.7
		2	81.8	-95.7
		3	177.7	-164.8
		4	13.8	1.2
		-1	212.9	256.3
		-2	65.4	62.5
		-3	170.2	144.2
		-4	75.7	65.4
1	2	0	44.0	55.3
		1	183.6	206.2
		2	82.9	-82.5
		3	73.6	70.6
		4	76.6	-72.2
		-1	94.6	117.0
		-2	103.9	113.6
		-3	123.6	-122.3
		-4	16.0	-11.8
1	3	0	93.0	129.3
		1	43.3	-50.3
		2	42.9	50.9
		3	131.7	155.3
		4	25.0	-24.2
		-1	101.7	-124.1
		-2	22.6	15.4
		-3	113.6	-142.7
		-4	53.1	-55.6
1	4	0	37.6	-49.5
		1	108.9	-164.7
		2	101.7	112.0
		3	42.4	-41.6
		4	32.7	32.4
		-1	44.1	-69.1
		-2	54.1	-59.6
		-3	77.6	82.7
		-4	27.2	-29.3
1	5	0	105.9	-136.9
		1	26.7	24.8
		2	37.8	-45.9
		3	127.9	-148.3
		-1	101.6	140.6
		-2	9.8	15.5
		-3	117.8	148.3
		-4	28.2	35.3
1	6	0	43.3	50.8
		1	136.6	215.3
		2	54.1	-64.5
		3	94.3	99.4
		4	68.9	-70.1
		-1	68.5	104.1
		-2	68.1	86.4
		-3	75.6	-95.0
		-4	11.8	-19.3
1	7	0	58.5	61.4
		1	48.2	46.1
		2	77.2	81.2
		3	143.1	161.0
		4	13.1	-12.0
		-1	136.9	-191.6
		-2	51.2	-50.3
		-3	108.9	-121.9
		-4	59.2	-63.9
1	8	0	55.7	-33.9
		1	161.3	-188.2
		2	16.8	8.5
		3	56.9	-53.5
		4	93.1	82.9
		-1	108.5	-121.6
		-2	108.4	-99.1
		-3	112.4	98.4
		-4	50.0	51.8
1	9	0	81.1	-79.7
		1	40.4	-38.4
		2	66.3	-62.8
		3	108.7	-100.4
		-1	185.8	177.1
		-2	18.5	14.8
		-3	108.1	95.3
		-4	51.5	48.7
1	10	0	10.5	3.0
		1	138.0	141.1
		2	60.6	-51.5
		3	48.6	48.7
		4	60.7	-58.6
1	10	-1	67.4	52.0
		-2	110.4	83.9
		-3	99.5	-80.7
1	11	0	111.9	90.8
		1	26.2	-20.8
		2	24.6	23.4
		3	103.6	95.4
		4	30.3	-22.4
		-1	126.7	-99.6
		-2	26.2	18.0
		-3	113.8	-89.0
		-4	36.8	-32.4
1	12	0	40.5	-28.5
		1	125.0	-112.4
		2	91.6	78.1
		3	32.2	-29.6
		4	27.2	17.7
		-1	69.6	-56.1
		-2	36.1	-26.5
		-3	70.2	59.5
		-4	29.5	-20.3
1	13	0	56.2	-48.6
		1	11.3	6.1
		2	34.2	-26.9
		3	123.1	-117.5
		4	15.5	16.1
		-1	97.2	73.8
		-3	110.4	93.3
		-4	33.2	30.3
1	14	0	46.1	32.7
		1	145.7	126.3
		2	56.6	-40.8
		3	34.7	34.7
		4	39.8	-38.8
		-1	79.0	57.2
		-2	61.5	43.6
		-3	69.8	-59.0
1	15	0	69.1	59.2
		1	17.6	11.4
		2	54.6	47.4
		3	82.7	80.0
		-1	159.5	-123.5
		-2	22.3	-16.9
		-3	78.9	-71.4
		-4	15.3	-23.1
1	16	1	115.0	-97.9
		2	16.6	13.4
		3	26.4	-33.7
		4	47.0	48.7
		-1	57.0	-45.1
		-2	80.5	-59.2
		-3	61.4	55.8
1	17	0	43.8	-34.3
		1	27.2	-17.3
		2	25.6	-21.2
		3	45.9	-56.1
		-1	108.0	83.9
		-3	40.3	38.4
		-4	15.3	23.0
1	18	0	9.4	-9.9
		1	72.7	62.9
		2	35.7	-31.8
		3	19.8	21.3
		-1	40.6	32.6
		-2	35.3	34.5
		-3	48.2	-42.6
1	19	0	48.2	43.2
		1	9.0	-8.5
		3	39.3	48.4
		-1	66.9	-52.7
		-2	22.4	17.1
		-3	38.2	-40.2
1	20	0	11.8	-7.8
		1	67.2	-65.1
		2	42.9	40.6
		-1	27.2	-21.0
		-2	14.0	-16.3
		-3	19.8	32.9
1	21	0	33.2	-29.9
		-1	49.8	42.1
2	0	0	146.8	187.3
		1	110.2	144.8
		2	114.5	132.0
		3	86.2	-81.3
		4	19.1	-14.4
		-1	167.2	182.3
		-2	151.3	-153.1
		-3	58.0	39.5
		-4	135.2	-119.9
2	1	0	129.4	-178.3
		1	82.9	111.7
		2	101.8	96.5
		3	133.0	127.2
		4	127.6	116.5
		-2	166.8	-175.3
		-3	121.6	-116.2
		-4	16.2	17.9
2	2	0	160.7	-182.9
		1	35.8	-37.3
		2	155.5	-165.8
		3	119.9	111.6
		4	22.7	22.8
		-1	68.5	-83.6
		-2	90.2	86.4
		-3	72.8	-59.0
		-4	123.8	108.7
2	3	0	137.9	156.3
		1	134.6	-166.9
		2	69.0	-64.4
		3	45.5	-40.3
		4	87.9	-73.8
		1	44.2	-41.8
		-2	157.3	142.6
		-3	24.6	29.0
2	4	0	37.3	48.3
		1	8.4	6.7
		2	142.4	150.8
		3	90.0	-93.7
		4	42.0	-36.5
		-1	127.1	132.5
		-2	57.3	-58.2
		-3	72.7	67.3
		-4	108.4	-112.4
2	5	0	126.1	-163.2
		1	126.2	166.2
		2	76.1	75.9
		3	44.8	51.5
		4	60.8	62.0
		-1	16.6	-10.2
		-2	86.5	-100.2
		-3	39.7	-45.2
		-4	14.3	10.8
2	6	0	102.3	-130.5
		1	45.0	-54.8
		2	125.6	-145.2
		3	74.6	77.9
		4	38.8	34.9
		-1	87.3	-120.0
		-2	74.9	79.7
		-3	35.3	-38.9
		-4	103.6	115.5
2	7	0	109.6	133.4
		1	63.4	-88.9
		2	77.4	-80.3
		3	85.9	-96.1
		4	98.2	-116.9
		-1	14.8	-4.1
		-2	135.1	161.5
		-3	88.0	96.6
2	8	0	118.8	162.3
		1	62.1	81.4
		2	85.9	118.1
		3	79.4	-90.6
		-1	68.3	95.1
		-2	83.9	-109.7
		-3	29.3	37.0
		-4	76.1	-84.4
2	9	0	93.9	-126.1
		1	72.7	91.4
		2	39.3	42.9
		3	76.8	98.2
		4	75.0	77.5
		-1	23.9	31.3
		-2	94.3	-122.6
		-3	63.8	-80.5
		-4	17.7	17.7
2	10	0	88.1	-79.4
		1	14.9	-11.9
		2	95.2	-92.6
		3	70.1	68.8
		-1	78.9	-67.8
		-2	70.6	70.7
		-3	51.2	-56.2
		-4	83.8	80.6
2	11	0	99.7	99.2
		1	109.6	-104.5
		2	52.5	-43.1
		3	16.0	-20.5
		4	49.6	-45.1
		-1	31.0	-27.2
		-2	89.3	82.0
		-3	17.8	19.5
2	12	0	53.9	44.9
		1	7.4	1.3
		2	112.7	103.1
		3	74.0	-66.0
		4	22.1	-22.1
		-1	75.2	70.2
		-2	45.2	-33.7
		-3	52.3	43.4
		-4	81.3	-74.0
2	13	0	92.4	-87.8
		1	106.5	98.6
		2	50.5	38.7
		3	23.2	28.2
		4	52.8	52.9
		-1	11.9	-5.6
		-2	83.9	-72.4
		-3	41.6	-37.8
		-4	18.3	12.6
2	14	0	86.2	-63.4
		1	39.2	-31.4
		2	97.6	-91.6
		3	50.5	47.9
		4	20.5	19.1
		-1	101.7	-81.4
		-2	68.3	52.2
		-3	30.9	-23.1
		-4	76.9	78.3
2	15	0	100.2	89.4
		1	74.1	-61.9
		2	50.3	-37.6
		3	50.5	-50.8
		4	52.5	-63.0
		-1	12.3	-10.2
		-2	96.1	81.6
		-3	47.9	48.0
2	16	0	92.9	74.8
		1	20.3	20.9
		2	65.9	63.2
		3	51.7	-51.8
		-1	66.1	53.4
		-2	68.3	-59.6
		-3	24.5	26.0
		-4	39.8	-48.8
2	17	0	62.3	-57.6
		1	47.5	44.6
		2	15.2	8.5
		3	33.1	38.8
		4	31.1	42.7
		-1	42.2	34.8
		-2	75.0	-62.3
		-3	45.7	-40.6
2	18	0	39.7	-36.7
		2	45.6	-41.9
		3	40.8	43.9
		-1	49.7	-35.6
		-2	52.1	42.0
		-3	40.1	-39.0
		-4	22.5	32.2
2	19	0	49.2	46.7
		1	61.9	-50.4
		2	16.3	-15.7
		-1	19.0	-17.8
		-2	41.5	37.3
		-3	13.0	15.0
2	20	0	16.6	14.7
		2	50.5	50.7
		-1	48.2	43.0
		-2	18.9	-19.5
2	21	0	54.9	-49.1
		1	43.5	51.2
3	0	0	206.9	-245.1
		1	60.1	-68.2
		2	52.2	-52.5
		3	107.5	-95.3
		4	144.7	126.6
		-1	49.9	42.1
		-2	146.0	-128.6
		-3	136.7	140.3
		-4	121.7	114.4
3	1	0	38.2	-44.0
		1	58.1	57.1
		2	207.0	-207.8
		3	19.5	-12.2
		4	76.7	-54.7
		-1	149.9	135.1
		-2	188.1	187.6
		-3	22.0	-20.5
		-4	103.0	99.6
3	2	0	183.3	208.0
		1	43.1	42.8
		2	12.4	5.4
		3	80.1	66.7
		4	133.5	-115.1
		-1	40.8	-35.3
		-2	121.7	112.2
		-3	59.7	-86.5
		-4	90.6	-90.6
3	3	0	112.9	111.5
		1	82.5	-78.6
		2	162.9	170.3
		3	9.1	6.5
		4	50.7	38.2
		-1	56.2	-60.6
		-2	142.2	-144.2
		-4	101.4	-78.8
3	4	0	159.3	-215.3
		1	40.6	-46.5
		2	77.1	68.7
		3	55.6	-44.3
		4	103.1	90.8
		-1	67.7	55.5
		-2	96.2	-87.9
		-3	61.4	59.1
		-4	44.2	42.9
3	5	0	80.3	-91.3
		1	87.4	87.3
		2	136.6	-151.7
		3	36.2	-31.1
		4	49.6	-42.1
		-1	26.6	34.1
		-2	153.5	161.0
		-3	8.8	3.7
		-4	94.8	84.8
3	6	0	168.0	217.9
		1	45.0	46.3
		2	8.6	13.1
		3	59.8	55.5
		4	107.2	-91.2
		-1	46.3	-48.2
		-2	85.4	90.5
		-3	89.3	-86.4
		-4	67.4	-78.2
3	7	0	30.7	35.8
		1	46.4	-51.6
		2	159.7	183.3
		3	10.4	6.3
		4	56.5	54.7
		-1	104.3	-126.0
		-2	133.3	-157.6
		-3	18.7	20.0
		-4	74.1	-77.8
3	8	0	112.3	-158.3
		1	43.5	-49.8
		2	38.8	-41.5
		3	85.7	-86.6
		4	117.5	112.9
		-1	27.5	31.4
		-2	96.8	-112.2
		-3	90.6	103.4
		-4	82.4	98.0
3	9	0	40.6	-42.7
		1	38.4	44.0
		2	120.5	-150.1
		4	27.9	-28.9
		-1	75.1	93.1
		-2	86.5	117.3
		-3	22.0	-22.8
		-4	61.0	78.3
3	10	0	107.4	138.5
		1	14.3	22.5
		2	16.1	-11.3
		3	52.7	45.0
		4	89.1	-84.0
		-1	23.6	-28.6
		-2	57.5	71.9
		-3	50.2	-55.6
		-4	34.8	-55.0
3	11	0	64.7	64.2
		1	55.7	-55.9
		2	104.6	109.4
		4	29.0	26.9
		-1	16.4	-19.9
		-2	77.8	-94.6
		-4	64.5	-66.1
3	12	0	150.0	-139.5
		1	26.6	-22.0
		2	43.3	29.8
		3	35.7	-30.4
		4	62.6	64.1
		-1	51.4	48.4
		-2	73.1	-67.7
		-3	55.1	45.9
		-4	28.9	27.1
3	13	0	65.4	-59.3
		1	69.2	65.1
		2	109.1	-107.8
		3	18.1	-14.6
		4	32.4	-33.6
		-1	41.9	42.3
		-2	104.1	98.0
		-4	61.3	53.1
3	14	0	144.2	126.6
		1	44.1	35.2
		3	41.5	42.1
		4	55.9	-57.5
		-1	42.1	-38.7
		-2	66.4	52.1
		-3	60.2	-50.9
		-4	41.0	-46.3
3	15	0	20.8	17.4
		1	36.2	-37.2
		2	104.3	94.8
		3	13.5	11.4
		4	21.9	29.1
		-1	62.8	-51.9
		-2	109.8	-91.7
		-4	43.5	-46.0
3	16	0	98.5	-79.9
		1	26.7	-19.8
		2	10.7	-15.2
		3	47.6	-48.4
		4	48.4	62.7
		-1	28.5	23.0
		-2	65.2	-57.2
		-3	49.8	47.7
		-4	33.2	51.8
3	17	0	37.7	-30.9
		1	33.1	28.8
		2	93.7	-84.3
		3	10.9	15.8
		-1	66.0	50.2
		-2	57.6	48.1
		-3	21.3	-24.7
		-4	39.9	46.0
3	18	0	64.5	61.2
		2	11.1	-4.5
		3	27.0	29.2
		-1	49.2	-25.6
		-2	61.8	54.2
		-3	32.4	-32.9
3	19	0	36.4	31.0
		1	35.1	-31.7
		2	52.0	56.1
		-1	12.0	-7.1
		-2	45.9	-45.0
3	20	0	80.7	-73.1
		1	10.7	-9.9
		2	6.7	12.9
		-1	31.8	28.9
		-2	16.7	-26.1
4	0	0	34.0	25.8
		1	199.5	203.3
		2	31.9	25.0
		3	24.9	10.6
		4	14.9	8.7
		-1	62.4	63.0
		-2	47.2	-38.5
		-3	54.8	-53.1
		-4	26.6	-20.7
4	1	0	18.5	-17.8
		1	16.9	18.9
		2	23.8	-20.0
		3	190.7	169.1
		4	31.6	26.3
		-1	131.5	-120.4
		-2	22.1	-21.4
		-3	130.9	-126.0
		-4	18.4	11.8
4	2	0	32.4	28.9
		1	169.4	-172.6
		2	10.8	9.0
		3	21.8	-17.1
		4	13.4	8.4
		-1	113.5	-117.0
		-2	22.3	16.9
		-3	58.2	49.6
		-4	15.2	8.0
4	3	0	7.2	-3.1
		1	65.0	-58.4
		2	15.5	-9.4
		3	127.3	-121.3
		4	30.2	21.4
		-1	115.7	116.5
		-2	58.2	-43.3
		-3	106.5	105.0
		-4	15.4	8.3
4	4	0	44.3	-39.2
		1	111.3	109.6
		2	11.2	10.5
		3	15.0	11.7
		4	40.4	-35.3
		-1	94.8	100.6
		-2	47.8	42.0
		-3	41.7	-46.3
4	5	1	68.2	66.0
		2	15.7	9.5
		3	105.3	106.2
		4	13.8	-9.6
		-1	117.0	-118.5
		-2	18.4	14.5
		-3	74.8	-83.5
4	6	0	24.9	24.7
		1	148.9	-159.5
		2	17.6	-14.9
		3	12.1	-16.4
		4	13.9	9.2
		-1	91.1	-100.2
		-2	16.8	17.0
		-3	47.9	49.8
4	7	0	27.1	28.9
		1	20.3	-18.4
		2	24.4	22.0
		3	139.0	-156.4
		4	14.0	-16.5
		-1	101.6	106.0
		-3	98.3	117.9
4	8	0	18.0	11.9
		1	145.9	163.2
		3	11.4	15.5
		4	14.0	9.4
		-1	52.0	71.5
		-2	37.5	-43.3
		-3	37.9	-48.3
4	9	0	15.2	5.5
		1	9.1	9.2
4	9	2	12.9	-13.2
		3	104.9	118.7
		4	15.5	17.6
		-1	69.2	-85.1
		-3	68.1	-95.2
4	10	0	12.8	18.1
		1	96.1	-110.1
		-1	43.5	-58.7
		-3	26.5	28.5
4	11	0	12.5	-9.1
		1	53.1	-51.2
		3	82.8	-85.0
		4	16.1	22.3
		-1	58.8	79.3
		-2	20.8	-26.1
		-3	55.0	71.1
4	12	0	47.1	-39.0
		1	93.0	85.1
		3	15.9	14.3
		4	18.7	-20.0
		-1	71.1	86.7
		-3	27.0	-28.2
4	13	0	24.9	-18.3
		1	51.2	41.1
		2	10.5	4.2
		3	68.0	77.7
		-1	74.3	-77.6
		-2	17.4	17.3
		-3	55.9	-57.0
4	14	0	18.4	13.9
		1	93.2	-93.0
		2	16.0	-14.3
		-1	57.0	-59.6
		-2	10.6	8.8
		-3	41.9	42.7
4	15	0	15.4	8.5
		1	20.0	-14.4
		2	19.1	15.9
		3	76.7	-81.9
		-1	76.9	69.5
		-3	80.0	66.5
4	16	1	101.4	88.9
		2	12.3	-7.7
		-1	41.7	37.0
		-2	24.6	-21.1
		-3	26.1	-25.9
4	17	0	15.5	8.6
		1	12.2	6.2
		2	15.3	-15.0
		3	57.3	66.4
		-1	43.6	-38.0
		-2	10.8	7.6
		-3	57.9	-57.1
4	18	0	16.1	14.7
		1	65.4	-56.7
		2	15.3	17.8
		-1	45.0	-38.8
4	19	1	35.9	-29.0
		-1	38.5	39.3
		-2	11.5	-13.5
5	0	0	129.9	-117.1
		1	11.0	7.6
		2	136.4	-135.9
		3	13.8	-3.6
		4	35.1	31.9
		-1	40.2	-33.6
		-2	41.4	45.7
		-3	26.6	22.1
		-4	127.7	116.6
5	1	0	120.5	114.8
		1	41.9	-36.1
		2	105.1	-97.2
		4	78.0	-72.5
		-1	32.3	23.0
		-2	127.0	124.2
		-3	16.6	-11.4
		-4	18.4	2.1
5	2	0	144.4	132.7
		2	94.0	94.6
		3	37.7	-36.6
		4	30.1	-28.2
		-1	18.8	13.0
		-2	41.2	-40.9
		-4	98.7	-88.1
5	3	0	78.5	-63.3
		1	50.9	46.8
		2	64.9	62.7
		3	18.0	19.3
		4	77.9	70.6
		-1	49.8	47.5
		-2	147.1	-147.7
		-3	15.7	-0.2
		-4	18.5	-9.9
5	4	0	152.9	-145.4
		2	63.0	-67.1
		3	74.9	69.6
		4	15.6	17.8
		-1	17.7	-12.6
		-2	47.1	43.3
		-3	37.7	-39.4
		-4	72.1	71.0
5	5	0	73.3	61.8
		1	28.5	-27.1
		2	75.5	-74.3
		3	11.6	-15.2
		4	81.2	-76.9
		-1	25.4	-23.9
		-2	120.5	124.3
5	6	0	118.7	118.4
		1	15.0	10.3
		2	81.9	87.1
		3	20.7	-21.0
		4	24.4	-23.1
		-1	16.4	11.5
		-2	35.7	-40.1
		-4	90.4	-84.3
5	7	0	94.4	-95.5
		1	28.6	28.1
		2	71.4	72.1
		4	69.4	65.9
		-1	17.6	-17.0
		-2	111.4	-118.3
		-3	12.1	10.5
5	8	0	102.6	-94.2
		1	7.7	7.9
		2	110.4	-113.5
		4	21.0	22.5
		-1	17.9	-17.3
		-2	33.2	39.7
		-4	94.3	96.0
5	9	0	94.9	92.6
		1	43.0	-39.3
		2	68.9	-71.8
		4	51.7	-52.9
		-2	80.6	96.3
		-3	16.3	-17.0
		-4	17.8	11.7
5	10	0	94.4	91.8
		1	23.6	-21.0
		2	62.6	67.8
		3	22.2	-26.9
		4	21.6	-24.2
		-1	19.5	21.0
		-2	17.6	-20.0
		-4	64.5	-65.3
5	11	0	45.1	-41.0
		1	29.2	27.8
		2	55.0	52.4
		4	46.1	48.7
		-1	23.9	31.4
		-2	69.5	-87.6
5	12	0	117.2	-94.3
		2	46.1	-46.7
		3	42.7	46.0
		4	11.7	12.5
		-2	28.3	33.9
		-3	30.2	-31.9
		-4	50.7	47.7
5	13	0	51.6	42.5
		1	19.5	-16.0
		2	37.9	-35.0
		4	48.9	-59.0
		-1	23.3	-24.8
		-2	98.4	94.1
5	14	0	89.2	74.2
5	14	2	64.3	60.7
		3	18.7	-14.6
		-2	34.0	-30.3
		-4	50.8	-57.2
5	15	0	76.5	-58.2
		1	14.4	11.7
		2	58.1	45.5
		-1	14.5	-6.0
		-2	73.4	-65.5
		-3	13.2	11.9
5	16	0	62.3	-48.8
		1	21.4	17.5
		2	69.9	-62.6
		-1	16.0	-10.3
		-2	17.3	15.4
5	17	0	68.8	49.9
		1	35.3	-28.7
		2	36.2	-32.9
		-1	23.6	-16.2
		-2	56.9	54.3
5	18	0	56.4	45.4
		1	21.8	-17.3
		-1	13.1	10.2
6	0	0	73.9	68.6
		1	110.7	103.3
		2	34.1	-29.4
		3	67.3	67.2
		4	29.7	-27.7
		-1	65.8	-49.3
		-2	40.6	36.2
		-3	80.6	-82.3
6	1	0	80.0	62.7
		1	91.6	-82.4
		2	50.8	44.8
		3	52.9	49.3
		4	21.0	21.7
		-1	99.9	-88.3
		-2	54.1	-53.3
		-3	16.4	-16.0
		-4	45.1	-36.5
6	2	0	84.9	-63.5
		1	68.6	-62.6
		2	26.4	22.8
		3	66.0	-67.8
		4	48.9	50.9
		-1	39.2	30.5
		-2	80.0	-78.4
		-3	91.9	80.4
6	3	0	83.8	-62.7
		1	45.0	39.0
		2	80.5	-82.6
		3	30.4	-28.7
		-1	103.2	91.7
		-2	27.2	27.2
		-4	72.9	58.2
6	4	0	44.6	38.5
		1	59.6	55.1
		2	42.3	-34.2
		3	69.7	71.6
		4	57.6	-64.5
		-1	12.2	10.7
		-2	89.0	81.0
		-3	71.3	-72.6
6	5	0	60.7	53.5
		1	49.7	-44.0
		2	73.6	71.4
		3	37.5	35.9
		4	14.1	-10.3
		-1	79.9	-85.4
		-2	24.9	-23.5
		-3	15.5	-14.5
		-4	56.4	-53.9
6	6	0	70.4	-61.5
		1	64.4	-60.4
		2	20.1	16.7
		3	69.6	-69.0
		4	45.4	44.6
		-1	35.8	30.4
		-2	68.3	-61.2
		-3	74.4	83.8
6	7	0	54.1	-46.8
		1	56.0	74.6
		2	48.2	-46.3
		3	34.5	-34.7
		-1	77.8	81.3
		-2	43.4	46.5
		-4	41.7	38.4
6	8	0	62.3	54.6
		1	84.3	75.2
		2	25.7	-19.5
		3	44.6	54.4
		4	17.7	-24.8
		-1	44.8	-45.0
		-2	40.5	42.3
		-3	62.4	-69.7
6	9	0	61.6	59.6
		1	57.4	-60.4
		2	30.6	31.4
		3	42.3	41.6
		-1	68.1	-67.7
		-2	26.8	-34.9
		-3	17.0	-17.6
		-4	24.6	-22.8
6	10	0	45.5	-40.5
		1	63.7	-60.4
		2	36.5	38.5
		3	38.5	-48.8
		4	38.4	42.1
		-1	16.7	17.2
		-2	43.2	-48.4
		-3	37.6	45.5
		-4	25.2	11.2
6	11	0	61.6	-52.1
		1	36.0	33.0
		2	64.6	-59.4
		3	25.3	-21.2
		-1	57.1	60.6
		-2	17.9	21.1
		-4	35.0	44.4
6	12	0	42.7	34.0
		1	32.7	27.7
		2	18.5	-13.7
		3	45.7	48.8
		4	26.1	-43.1
		-1	10.9	-7.5
		-2	71.8	69.1
		-3	63.6	-60.9
		-4	11.5	-12.8
6	13	0	35.9	28.1
		1	36.6	-30.6
		2	62.2	51.4
		3	22.0	19.3
		-1	63.3	-65.6
		-4	35.0	-39.0
6	14	0	47.4	-31.7
		1	55.7	-42.1
		2	18.4	12.3
		3	36.8	-50.8
		-1	24.4	18.4
		-2	40.6	-33.6
		-3	55.3	56.3
6	15	0	41.2	-31.5
		1	66.0	51.0
		2	30.2	-26.4
		-1	57.1	46.4
		-2	23.6	20.4
6	16	1	54.0	44.2
		-1	36.4	-26.7
		-2	24.3	21.8
6	17	0	18.4	38.9
7	0	0	43.9	33.5
		1	68.4	-59.8
		2	50.6	-49.4
		3	10.4	-11.7
		-1	65.7	-57.4
		-2	53.3	49.9
		-3	23.2	17.6
		-4	26.5	28.1
7	1	0	70.0	71.5
		1	44.7	-40.8
		2	15.3	16.1
		3	55.1	-63.5
		4	12.0	-4.5
		-1	59.0	47.1
		-3	35.9	36.0
7	1	-4	17.7	-21.6
7	2	0	11.4	-8.0
		1	73.2	62.2
		2	34.9	33.6
		3	19.2	-26.8
		-1	62.0	55.5
		-2	49.8	-45.4
7	3	0	26.3	-26.1
		1	37.1	36.2
		2	19.1	-14.7
		3	60.8	68.9
		4	12.7	22.0
		-1	38.3	-31.1
		-3	66.7	-61.3
		-4	28.5	21.2
7	4	1	84.1	-79.4
		2	17.5	-17.0
		3	32.9	36.6
		4	14.6	-9.4
		-1	45.3	-44.4
		-2	37.1	34.0
7	5	0	32.9	31.9
		1	45.1	-44.3
		2	31.3	28.8
		3	60.4	-71.4
		4	9.6	-18.3
		-1	37.6	30.2
		-2	14.9	16.6
		-3	40.6	46.8
		-4	16.3	-25.7
7	6	0	22.6	-16.1
		1	64.4	64.6
		2	39.9	36.3
		3	16.2	-14.6
		4	14.9	18.7
		-1	43.8	46.5
		-2	50.5	-48.7
		-4	14.3	-20.7
7	7	0	68.3	-62.8
		1	27.9	29.3
		2	15.4	-16.0
		3	50.3	54.6
		4	14.3	8.6
		-1	44.4	-40.7
		-3	34.6	-34.1
		-4	19.0	22.8
7	8	0	28.3	22.8
		1	45.2	-44.3
		2	32.3	-34.9
		3	11.8	-9.6
		-1	40.7	-50.1
		-2	35.0	39.6
		-3	19.6	23.0
7	9	0	54.5	48.8
		1	33.3	-34.2
		3	42.2	-47.1
		-1	34.5	37.7
		-3	28.5	32.8
		-4	11.0	-14.1
7	10	1	53.4	42.7
		2	27.1	25.2
		3	17.9	-17.5
		-1	37.8	46.5
		-2	27.4	-27.5
7	11	0	26.2	-22.8
		1	43.3	37.5
		2	17.4	-16.1
		3	37.4	49.8
		-1	18.8	-16.3
		-2	7.1	-5.2
		-3	40.6	-43.9
7	12	1	60.3	-54.5
		2	11.4	-10.0
		-1	42.6	-36.4
		-2	33.1	33.0
7	13	0	26.4	21.3
		1	28.1	-21.7
		2	21.5	22.4
		-1	27.0	20.7
		-2	5.8	5.4
7	14	0	17.1	-14.7
		1	45.9	42.6
		-1	33.2	27.8
		-2	17.6	-31.9
8	0	0	83.6	73.6
		2	32.5	34.1
		3	7.5	4.0
		-1	44.8	-43.4
		-3	15.5	-16.0
		-4	27.2	-37.6
8	1	0	13.7	-10.8
		1	27.0	-25.1
		2	33.4	34.7
		3	10.6	-12.2
		-2	67.7	-69.0
		-3	16.5	20.3
		-4	11.0	-4.4
8	2	0	91.0	-63.4
		2	27.4	-28.3
		-1	14.9	16.6
		-4	34.0	42.8
8	3	0	25.3	19.6
		2	51.6	-56.0
		-2	63.5	66.4
		-3	17.8	-20.0
		-4	14.4	23.0
8	4	0	73.9	74.2
		1	26.7	-25.4
		2	32.5	33.8
		3	9.6	12.0
		-2	15.2	16.8
		-3	13.3	-19.2
		-4	32.6	-45.0
8	5	0	30.5	-24.5
		1	12.2	-9.2
		2	59.5	62.2
		3	6.9	-12.6
		-2	49.5	-55.1
		-3	18.0	21.5
		-4	13.9	-26.4
8	6	0	63.9	-59.5
		1	7.2	8.7
		2	25.8	-29.9
		3	9.5	-12.2
		-1	17.8	16.8
		-3	11.9	16.9
8	7	0	14.0	9.9
		1	27.6	23.5
		2	22.9	-25.9
		-2	48.9	57.2
		-3	9.5	-13.2
8	8	0	69.8	56.1
		1	8.9	3.2
		2	22.9	24.9
		-1	30.5	-35.9
8	9	0	12.0	-7.3
		1	20.7	-16.3
		2	29.5	28.7
		-2	54.2	-62.9
8	10	0	63.6	-56.8
		1	18.2	12.5
		-1	13.4	16.7
		-2	10.0	-10.2
8	11	0	18.5	13.5
9	0	0	11.6	9.1
		1	32.1	-32.4
		2	6.3	11.4
		-2	11.2	-17.4
9	1	0	17.4	-13.2
		1	26.7	26.4
		2	4.1	-6.4
		-1	43.0	48.9
		-2	12.1	-18.9
9	2	0	9.3	-9.7
		1	26.4	48.7
		-2	16.0	20.3
9	3	0	34.3	28.4
		1	14.2	-15.0
		-1	38.4	-47.4
9	4	1	51.0	-59.9
		-1	6.2	2.4
		-2	10.0	-16.7
9	5	0	33.1	-32.2
		1	19.1	22.5
		-1	34.8	45.6

REFERENCES.

Abrahams, S.C. (1956). Chem.Soc. Quart. Rev. p.407.

Abrahams, S.C. and Grenville-Wells, H.J. (1956). M.I.T. Laboratory for Insulation Research Tech. Rep. No.105.

Allen, P.W., Bowen, H.J.M., Sutton, L.E. and Bastiansen, O. (1952). Trans. Faraday Soc. 48, 991.

Allen, P.W. and Sutton, L.E. (1950). Acta Cryst. 3, 46.

Andersen, K.K. (1964). J.Org.Chem. 29, 1953.

Arnott, S., Davie, A.W., Robertson, J.M., Sim, G.A. and Watson, D.G. (1961). J.Chem.Soc. p.4183.

Asao, T., Buchi, G., Abdel-Kader, M.M., Chang, S.B., Wick, E.L. and Wogan, G.N. (1963). J.Amer.Chem.Soc. 85, 1706.
(1965). J.Amer.Chem.Soc. 87, 882.

Aurivillius, K.L. (1950). Acta Chem.Scand. 4, 1413.

Bak, B., Hansen, L. and Rastrup-Andersen, J. (1955). Discuss. Faraday Soc. 19, 30.

Barton, D.H.R. and Elad, D. (1956). J.Chem.Soc. p.2085; p.2090.

Bastiansen, O. and Viervoll, H. (1948). Acta Chem.Scand. 2, 702.

Bauer, S.H. (1952). Private comm. in Trans. Faraday Soc. 48, 991.

Becka, L.N. and Cruickshank, D.W.J. (1963). Proc.Roy.Soc. A 273, 435.

Beevers, C.A. and Cochran, W. (1947). Proc.Roy.Soc. A 190 257.

Blount, W.P. (1961). Turkeys 9, 52.

Bokhoven, C., Schoone, J.C. and Bijvoet, J.M. (1951). Acta Cryst. 4, 275.

Booth, A.D. (1946). Proc.Roy.Soc. A 188, 77.

Braekken, H. and Scholten, W. (1934). Z.Krist. 89, 448.

Branden, C.-I. (1964). Arkiv. Kemi. 22, 501.

Brown, C.J. and Corbridge, D.E.C. (1954). Acta Cryst. 7, 711.

Brown, I.D. (1964). Acta Cryst. 17, 654.

Brown, W.A.C. (1964). Ph.D. Thesis, Univ. of Glasgow.

Brown, W.A.C. and Sim, G.A. (1963). J.Chem.Soc. p.1050.

Cava, M.P. and Soboczenski, E.J. (1956).J.Amer.Chem.Soc. 78,5317.

Cava, M.P., Weinstein, B. and Malhotra, S.S. (1959). Tetrahedron Letters, No.15, 1.

Cochran, W. (1951). Acta Cryst. 4, 408.
(1953). Acta Cryst. 6, 260.

Craven, B.M. (1964). Acta Cryst. 17, 396.

Cruickshank, D.W.J. (1950). Acta Cryst. 3, 10.
(1956). Acta Cryst. 9, 747.

Cruickshank, D.W.J., Pilling, D.E., Bujosa, A., Lovell, F.M. and Truter, M.R. (1961). "Computing Methods and the Phase Problem," p.32. Pergamon Press, Oxford.

Davies, D.R. and Blum, J.J. (1955). Acta Cryst. 8, 129.

Darlow, S.F. (1960). Acta Cryst. 13, 683.

Dias, H.W. and Truter, M.R. (1964). Acta Cryst. 17, 937.

Eglinton, G., Lardy, I.A., Raphael, R.A. and Sim, G.A. (1964). J.Chem.Soc. p.1154.

Feist, von K., Rintelen, P. and Kuntz, E.(1935). Annalen 517, 119.

Folting, K. and Lipscomb, W.N. (1964). Acta Cryst. 17, 1263.

Giguere, P.A. and Schomaker, V. (1943). J.Amer.Chem.Soc. 65, 2025.

Gordy, W. and Sheridan, J. (1954). J.Chem.Phys. 22, 92.

Grant, I.J. (1962). Ph.D. Thesis, Univ. of Glasgow.

Grant, I.J., Hamilton, J.A., Hamor, T.A., Robertson, J.M. and Sim, G.A. (1963). J.Chem.Soc. p.2506.

Grdenic, D. (1950). Arhiv. Kemi. 2, 14.

Groth, P. and Hassel, O. (1964). Acta Chem.Scand. 18, 1327.

Hanson, A.W. (1963). Acta Cryst. 16, 939.

Harker, D. (1956). Acta Cryst. 9, 1.

Hartley, R.D., Nesbitt, B.F. and O'Kelly, J. (1963) Nature, Lond. 198, 1056.

Hassel, O. and Viervoll, H. (1947). Acta Chem.Scand. 1, 149.

Hine, R. (1962). Acta Cryst. 15, 635.

Hine, R. and Rogers, D. (1956). Chem. and Ind. p.1428.

Hope, H. (1965). Acta Cryst. 18, 259.

Hughes, E.W. (1941). J.Amer.Chem.Soc. 63, 1737.

"International Tables For X-ray Crystallography", Vol.III. (1962). The Kynoch Press, Birmingham, England.

Jeffrey, G.A. and Shiono, R. (1959). Acta Cryst. 12, 447.

Jeffery, J.W. (1963). Acta Cryst. 16, Supplement, p.A66.

Jeffreys, J.A.D., Sim, G.A., Burnell, R.H., Taylor, W.I., Corbett, R.E., Murray, J. and Sweetman, B.J. (1963). Proc.Chem.Soc. p.171.

Karle, I.L. and Karle, J. (1965). Acta Cryst. 19, 92.

Kartha, G. and Haas, D.J. (1964). J.Amer.Chem.Soc. 86, 3630.

Kartha, G. and Ramachandran, G.N. (1955). Acta Cryst. 8, 195.

Kendrew, J.C., Bodo, G., Dintzis, H.M., Parrish, R.G. and Wyckoff, H. (1958). Nature, Lond. 181, 662.

Kendrew, J.C., Dickerson, R.E., Strandberg, B.E., Hart, R.G., Davies, D.R., Phillips, D.C. and Shore, V.C. (1960). Nature, Lond. 185, 422.

Kjaer, A. and Gmelin, R. (1956). Acta Chem.Scand. 10, p.1100; P.1358; P.1614.

Kjaer, A. and Christensen, B. (1958). Acta Chem.Scand. 12, 833.
(1963). Acta Chem.Scand. 17, 846.

Klyne, W., Day, J. and Kjaer, A. (1960). Acta Chem.Scand. 14, 215.

Kunchur, N.R. and Truter, M.R. (1958). J.Chem.Soc. p.2551.

Lancaster, M.C., Jenkin, F.P. and Philip, J.McL. (1961). Nature, Lond. 192, 1095.

Lipson, H. and Cochran, W. (1953). "The Determination of Crystal Structures", G. Bell and Sons Ltd., London.

Manojlovic, Lj.M. and Edmunds, I.G. (1965). Acta Cryst. 18, 543.

Mathieson, A.McL. and Taylor, J.C. (1963). Acta Cryst. 16, 527.

Mayer, R.M. and Pratt, M.R.A. (1963). Acta Cryst. 16, 1086.

McConnell, J.F., Mathieson, A.McL. and Schoenborn, B.P. (1964). Acta Cryst. 17, 472.

McConnell, J.F. and Schoenborn, B.P.(1962). Acta Cryst. 15, 779.

Miller, R.A.L., Robertson, J.M., Sim, G.A., Clapp, R.C., Long, L. jun. and Hasselstrom, T. (1964). Nature, Lond. 202, 287.

Mislow, K., Green, M.M., Laur, P. and Chisholm, D.R. (1965). J.Amer.Chem.Soc. 87, 665.

Muirhead, H. and Perutz, M.F. (1963). Nature, Lond. 199, 633.

Nardelli, M. and Fava, G. (1962). Acta Cryst. 15, 214.

Nesbitt, B.F., O'Kelly, J., Sargeant, K. and Sheridan, A. (1962). Nature, Lond. 195, 1062.

Norton, D.A., Kartha, G. and Chia, T.L.(1964). Acta Cryst. 17,77.

Overton, K.H., Weir, N.G. and Wylie, A.(1961).Proc.Chem.Soc. p.211.

Owston, P.G. and Rowe, J.M. (1962). Acta Cryst. 15, 231.

Patterson, A.L. (1934). Phys. Rev. 46, 372.
(1935). Z. Krist. 90, 517.

Paul, I.C., Sim, G.A., Hamor, T.A. and Robertson, J.M.
(1962). J.Chem.Soc. p.4133.
(1963). J.Chem.Soc. p.5502.

Pauling, L. (1960). "The Nature of the Chemical Bond", 3rd edition, Cornell University Press.

Perutz, M.F. (1956). Acta Cryst. 9, 867.

Perutz, M.F., Rossmann, M.G., Cullis, A.F., Muirhead, H., Will, G. and North, A.C.T. (1960). Nature, Lond. 185, 416.

Prochazka, Z. (1959). Collection Czechoslov.Chem.Comm. 24, 2429.

Ramachandran, G.N. and Raman, S. (1959). Acta Cryst. 12, 957.

Robertson, J.M. (1935). J.Chem.Soc. p.615.

Robertson, J.M. and Woodward, I. (1937). J. Chem. Soc. p.219.
(1940). J. Chem. Soc. p.36.

Robertson, J.M. (1936). J.Chem.Soc. p.1195.
(1943). J.Sci.Instr. 20, 175.

Rollett, J.S. (1961). "Computing Methods and the Phase Problem in X-ray Crystal Analysis," p.87. Pergamon Press, Oxford.

Rossmann, M.G. (1960). Acta Cryst. 13, 221.

Sargeant, K., Sheridan, A., O'Kelly, J. and Carnaghan, R.B.A. (1961). Nature, Lond. 192, 1096.

Schmid, H. and Karrer, P. (1948). Helv.Chim.Acta 31, 1017.

Schomaker, V., Waser, J., Marsh, R.E. and Bergman, G. (1959). Acta Cryst. 12, 600.

Schultz, O.E. and Gmelin, R. (1954). Arch.Pharm. 287/59, 404.

Scouloudi, H. (1953). Acta Cryst. 6, 651.

Shoemaker, D.P., Donohue, J., Schomaker, V. and Corey, R.B. (1950). J.Amer.Chem.Soc. 72, 2328.

Sim, G.A. (1957). Acta Cryst. 10, 177; (1959). Acta Cryst. 12, 813.
(1961). "Computing Methods and the Phase Problem in X-ray Crystal Analysis," p.227, Pergamon Press, Oxford.

Stoicheff, B.P. (1954). Canad.J.Phys. 32, 339.

Sullivan, R.A.L. and Hargreaves, A. (1962). Acta Cryst. 15, 675.

Synge, R.L.M. and Wood, J.C. (1956). Biochem.J., Lond. 64, 252.

"Tables of Interatomic Distances and Configurations in Molecules and Ions", (1958). The Chemical Society Special Publication No.11.

Trotter, J. (1960). Acta Cryst. 13, 86.

Tunell, G. (1939). Amer.Min. 24, 448.

van den Hende, J.H. and Boutin, H. (1964). Acta Cryst. 17, 660.

van der Merwe, K.J., Fourie, L. and Scott, de B. (1963). Chem. and Ind. p.1660.

van Soest, T.C. and Peerdeman. A.F. (1964). Proc.k.ned.Akad.Westenschap 67B, 469.

Weir, N.G. (1962). Ph.D. Thesis, Univ. of Glasgow.

Wessely, F., Dinjaski, K., Isemann, W. and Singer, G. (1935). Monatsh. 66, 87.

Wheatley, P.J. (1953). Acta Cryst. <u>6</u>, 369.

Wiebenga, E.H. and Krom, C.J. (1946). Rec.trav.chim. <u>65</u>, 663.

Williams, D.E. and Rundle, R.E. (1964). J.Amer.Chem.Soc. <u>86</u>, 1660.

Wilson, A.J.C. (1942). Nature, Lond. <u>150</u>, 152.

Woolfson, M.M. (1956). Acta Cryst. <u>9</u>, 804.

Yuzawa, T. and Yamaha, M. (1953). Bull.Chem.Soc.,Jap. <u>26</u>, 414.

Zvonkova, Z.V. (1952). Zhur.Fiz.Khim. SSSR <u>26</u>, 1798.