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Molecular studies of natural products and related compounds.

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MOLECULAR STUDIES OF NATURAL PRODUCTS

AND RELATED COMPOUNDS

A thesis submitted to the Council for
National Academic Awards as part fulfilment
of the requirements for the Degree of

DOCTOR OF PHILOSOPHY

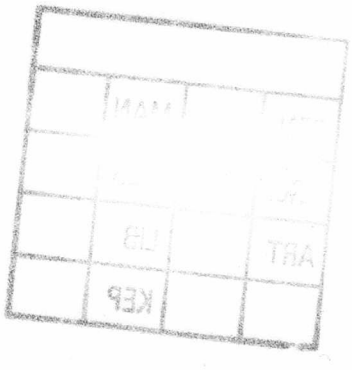
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January, 1986

TO

MY MOTHER AND FATHER

ACKNOWLEDGEMENTS

I am very grateful to Dr. P. J. Cox for his constant support, interest, advice and encouragement throughout this study. I should like to express my gratitude to Professor R. H. Thomson for his advice given during our many interesting discussions, and for his permission to spend some time in the Department of Chemistry, University of Aberdeen. I am happy to record my thanks for the patient guidance of Dr. R. A. Howie, and the hospitality extended to me during my visits to the Department of Chemistry.

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LIST OF ABBREVIATIONS

e.s.d.	-	estimated standard deviation
n.m.r.	-	nuclear magnetic resonance
i.r.	-	infrared
u.v.	-	ultraviolet

Additional abbreviations have been explained in the text.

DECLARATION

All the experimental work described in this thesis was carried out by H.J. Cowe in the laboratories of Robert Gordon's Institute of Technology, Aberdeen and the Department of Chemistry, University of Aberdeen, Old Aberdeen, and as acknowledged.

It has not been accepted in substance or concurrently submitted in candidature for any other degree.

Candidate

Director of Studies

ABSTRACT

Abstract

Molecular Studies of Natural Products and Related Compounds

Heather Johnston de Louvois Cowe

The work described in this thesis involves the use of direct methods of X-ray structure analysis for the structure elucidation of novel natural products and related compounds. Empirical force field calculations are also used for conformational and biosynthetic studies.

An outline of the development of direct methods of X-ray structure analysis is given. The implications of recent innovations in this field are discussed. The applications of molecular mechanics calculations to molecular studies are presented along with the theoretical aspects of the method.

Detailed structural information is given for several novel compounds, these having been solved using direct methods. The structures are varied in type and include alkaloids, sesquiterpenes and quinones, as well as natural product derivatives. Perspective drawings of each of the compounds allows their structure to be seen more clearly. Where possible molecular mechanics calculations have been carried out on these structures, and where appropriate the effect of crystal packing forces noted. In some instances the biosynthetic pathways of the compounds are studied in more detail.

The work involves the use of several complex computer programme packages, and the manipulation of these is described when required.

A critical survey of the various methods used for the structure elucidation of natural products is given. An attempt is made to show the interdependency of these techniques.

GENERAL SUMMARY

GENERAL SUMMARY

An outline of the theory of direct methods of X-ray structure analysis and an assessment of the implications of continuing developments in this field is given. Using direct methods, the structures of several natural products and a further three natural product derivatives are determined.

The theory of molecular mechanics calculations is outlined and this technique presented as an invaluable aid in conformational studies by application to the structures discussed.

As each of the structures solved is different, the approach to the elucidation of their structures is also different, and therefore discussed independently.

The first structure studied, a sesquiterpene lactone is unambiguously characterised by the X-ray analysis. Previous attempts to determine the structure by spectral methods had shown two possible structures. Few problems are encountered in this analysis and molecular mechanics calculations are carried out, showing that the molecule adopts a very stable conformation in the crystalline state.

Microcladallene B is a bromoallene and as such proves to be unstable in the X-ray beam. Decomposition of the cyrstal is to the extent of 68% during data collection. Despite this the structure is determined successfully. The stereochemistry at C(4) of Microcladallene is obtained. Molecular mechanics demonstrates that both rings of this molecule adopt the minimum energy conformation.

One of the two alkaloids studied is a novel alkaloid, nirurine. From the spectral analysis, an alternative structure to that found was proposed. Nirurine is found to be similar to, but quite

distinct from, the securinega alkaloids. Several ring systems are present in nirurine, and it has a highly strained valency angle between two of the rings, which is also observed in two similar structures. Molecular mechanics calculations on nirurine, securinine and phyllochrysine demonstrates that such a large valency angle is acceptable in this type of structure. A biosynthetic pathway for nirurine was obtained and this is studied using molecular mechanics. Perspective drawings of the structures involved are shown. Parameters are redefined for use in MM2 and are shown to be acceptable.

The problem of pseudosymmetry is encountered with the isofuranonaphthoquinone, Ventilone C. The procedures followed in obtaining this structure are described in detail. Despite initial problems, the structure of Ventilone C is characterised and the stereochemistry determined. Molecular mechanics calculations are not carried out due to the presence of hydrogen bonding.

Aristolindiquinone highlights one of the problems encountered in data collection and shows the value of taking preliminary photographs prior to using the four circle diffractometer. The structure of Aristolindiquinone is solved successfully, however due to the presence of hydrogen bonding, no molecular mechanics are carried out.

The second alkaloid studied, Ancistrotectorine is found to differ from the proposed structure. It is only the second member of the 7-3' linked naphthalene-isoquinoline alkaloids to be isolated. The steric hindrance around the diaryl linkage is studied further using molecular mechanics calculations, showing that the crystal structure adopts a high energy arrangement due to crystal packing forces.

Perchlororubian was first produced in the 19th century and various attempts had been made to determine its structure. Perchlororubian is shown to be a spiro-lactone. Molecular mechanics calculations show evidence for close approach between the chlorine and oxygen atoms of the structure, giving a high steric energy for the whole molecule.

Zoapatanolide B is shown to have two molecules per asymmetric unit, these two molecules differing in the arrangement of the side chain. Molecular mechanics calculations on the individual molecules are carried out, giving side chains with very similar torsion angles. This suggests that the difference in the molecules is due to crystal packing forces.

A most unsatisfactory conclusion is reached for the Leucoacetate of Ventilatone A. Disorder is found to be present, but the pattern of this disorder is not determined. The structure is concluded as conforming to proton nmr results. However with consideration of the bond angles and bond lengths found from the X-ray analysis, this conclusion is considered to be unsatisfactory.

Two structures remain unsolved in this work. Theories as to why this should be are put forward and possible remedies suggested.

The various methods used for the structure elucidation of natural products are discussed. Particular attention is paid to recent developments in nmr spectroscopy and the considerable amount of information that may be obtained from this method. A comparison is made between the techniques and the interdependency of them stressed. X-ray structure analysis is the only method that is shown to have the capability of being independent of the rest.

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CHAPTER ONE

INTRODUCTION

Introduction

Natural Products 1.1

Since the late 18th century, organic materials have been extracted from living organisms. In the intervening years, an abundance of these natural products have been isolated, studied and cited in the literature. From these studies a wide and varied branch of science has developed.

Natural products are diverse in their structure and origins. They can be purely organic, organometallic or halogen substituted organics, vegetable or animal, marine or land based. Natural products also vary substantially in size from compounds with few carbon atoms, to structures such as proteins. They show a wide range of reactivity and some have physiological properties.

Many compounds which derive from natural products are also studied within this group. These compounds may be formed naturally such as on exposure to light [1], or they may be synthesised from the natural product by a specific chemical reaction [2]. As these compounds play a vital role in the study of natural products they cannot be ignored.

The study of natural products has aided the expansion of various types of research from organic chemistry to pharmacology. Organic chemists have studied natural products to deepen and widen their knowledge of chemical reactions and mechanisms. Total synthesis of natural products is also made simpler by such research. Biosynthetic studies have advanced understanding of the pathways used by plants and animals to produce the substances they need [3].

Many natural products have been isolated which have biological activity and many have been developed for use as medicines, the first example being morphine, which was isolated from opium in 1806.

A great deal of natural product research is now carried out in the fields of medicinal chemistry and pharmacology. Bringing chemistry and pharmacology together, studies of structure activity relationships and receptor site interactions are aided by the number of processes available for study.

As well as medicinal products, many pigments which are used as dyes have been isolated from plants and animals. These are widely used in industry.

Molecular Studies 1,2

In the study of natural products, it is necessary to obtain accurate molecular data for them. The techniques involved in such studies are wide and varied.

Molecular studies may be split into two parts, structural investigations and conformational investigations. Structural studies involve the determination of the molecular formula, structure and stereochemistry of substances. Various techniques are used for this including degradative studies, mass spectroscopy, ultraviolet spectroscopy, infrared spectroscopy, nuclear magnetic resonance and X-ray diffraction analysis. Conformational studies involve finding the conformation adopted by a molecule in the solid state or in solution. The conformation can then be compared to that of similar structures. Conformations may be studied by X-ray analysis in the solid state and nmr in solution. In order to determine minimum energy conformations, molecular mechanics calculations are the most useful [4]. Quantum chemical calculations may also be used in conformational studies, however, the size of the molecules involved can make the computing time for the calculations prohibitive [3].

The methods most widely used in this thesis are X-ray diffraction analysis and molecular mechanics calculations.

X-ray Diffraction Analysis 1.3

The history of X-ray analysis can be traced back to 1912, when the first crystal structure was solved by W.L. Bragg. Since then there have been many developments which have made crystal analysis quick and efficient.

When X-ray data is collected, it is the intensities of the diffracted rays which are measured. After correction for Lorentz and polarisation effects, and after scaling, these may be related to the structure amplitude by the following equation

$$|F_{hkl}| \propto \sqrt{I_{hkl}} \qquad - (1.1)$$

The structure factor may be derived from the structure amplitude by the following equation

$$F_{hkl} = |F_{hkl}| \exp(i\phi_{hkl}) \qquad - (1.2)$$

where ϕ_{hkl} is the phase of the reflexion hkl

The atomic arrangement in the unit cell can be found if the electron density within it is known. It is possible to calculate the electron density from the following equation

$$\rho(x,y,z) = {}^{1}/_{V} \sum_{hkl} |F_{hkl}| \cos\left[2\pi(hx + ky + lz) - \phi_{hkl}\right] \qquad - (1.3)$$

where (x,y,z) are the fractional coordinates, V is the volume of the cell, and the summation is over all reflexions. The only unknowns are the phases, however, it is necessary to obtain the phases in order to obtain the electron density distribution and then the atomic arrangement. This is the phase problem, and various solutions have been formulated over the years for this problem.

The most popular method for the solution of the phase problem, used to be the heavy atom method. This method employs the Patterson function [6] which has properties such as the ability to

resolve vectors between heavy atoms, which facilitate its use in these methods. As the name suggests, the method requires heavy atoms to be present in the molecule under study. A division of the same method is that of isomorphous replacement where two or more crystalline derivatives which have the same crystal structure, but which contain different heavy atoms are used. By a method of comparison of the phases obtained from the different isomorphs, a unique solution will be found. However, natural products seldom have a heavy atom as part of the structure, and to use this method, a derivative has to be formed. Derivatisation is time consuming and not always successful - the product may not even be crystalline. For these reasons heavy atom methods are not usually employed in the study of small natural products.

Trial and error methods usually involve postulating a structure, calculating structure factors for this model and comparing their magnitude with those of the scaled observed values. The number of trial structures that require to be constructed increases with the number of parameters. This method is impracticable for all but very small and simple structures. It is not of general applicability.

The above are normally unsuitable for small natural product molecules and for this reason direct methods are usually employed.

Direct Methods 1.4

Introduction 1.4.1

Direct methods are those methods which try to evaluate the phases directly from the intensities by using relationships among these phases based solely on the observed intensities [7]. These methods do not require structural chemical information. Two

assumptions are however made in employing direct methods.

1. Given a set of observed amplitudes $|F_H|$, the corresponding phases must be consistent with positive electron density values everywhere.

2. The electron density about an atomic centre is approximately spherically symmetrical.

Direct methods had their beginnings in 1948 with the Harker-Kasper inequalities [8], later expanded by Karle and Hauptman [9]. However, the most important early papers were by Sayre [10], Cochran [11], and Zachariasen [12], which set out a mathematical relationship linking the phases of a centrosymmetric crystal. This is the sign relationship.

$$s(h_1k_1l_1)s(h_2k_2l_2)s(h_1 + h_2, k_1 + k_2, l_1 + l_2) \sim +1 \qquad -(1.4)$$

where s(hkl) is the sign of a structure factor and $\sim$ means 'probably equal to'. The probability of the relationship depends on the product of the three structure amplitudes, and where all three are large, the probability can be close to or even equal to unity. During the 1950's and early 1960's several centrosymmetric structures were solved using sign relationships.

In 1953 Hauptman and Karle put forward their theory of structure invariants and seminvariants for centrosymmetric structures [13]. A structure invariant is a quantity, the value of which remains unchanged when the origin of the unit cell is shifted. An example of a structure invariant are the structure factor amplitudes, the phases are however origin dependent. A structure seminvariant originates from space group symmetry where the choice of origin is limited to a set of special points in the unit cell. If the origin is shifted within this set of

points, some of the non-invariants become invariants. Many important structure factor products are complex numbers with origin independent phase angles, and are therefore invariants, as will be shown later. However although a quantity may be a structure invariant, its value need not necessarily be known.

One of the major advances came in 1955 when Cochran formulated the triple phase relationship [14]

$$\phi_H + \phi_{H'} + \phi_{-H-H'} \backsim 0 \text{ (modulo } 2\pi) \qquad -(1.5)$$

where $\backsim$ means is concentrated about, H represents hkl and $H + H' + -H-H' = 0$. This enables an estimate of one phase to be made when the other two are known. It is perhaps the most important invariant. These along with the sign relationship belong to the general class of Σ_2 relationships.

Up until 1956 all the theory formulated was for centrosymmetric structures, where the phase problem was one of designating the sign of the structure factor. The noncentrosymmetric case was obviously more complicated as it meant assigning a numerical value to the phase. Very little hope was held out for the solving of noncentrosymmetric crystal structures by direct methods.

In 1956 Hauptman and Karle extended their theory of structure invariants and seminvariants to encompass noncentrosymmetric structures [15]. In the same year they formulated the tangent formula [16]. It was found that a more reliable estimate of ϕ_H is obtained where there are several pairs of known phases contributing to the estimate. The formula is

$$\tan \phi_H = \frac{\sum_{H'} |E_{H'} E_{H-H'}| \sin (\phi_{H'} + \phi_{H-H'})}{\sum_{H'} |E_{H'} E_{H-H'}| \cos (\phi_{H'} + \phi_{H-H'})} \qquad -(1.6)$$

where E_H are the normalised structure factors (see later).

As well as giving a reliable estimate of ϕ_H the tangent formula can be used to refine a set of phases to self consistency. An estimate of the reliability of the tangent formula can be measured by variance, $V(\phi_H)$. A graph of variance as a function of α_H may be drawn [14,17] where

$$\alpha_H^2 = \left[\sum K_{HH'} \cos(\phi_{H'} + \phi_{H-H'})\right]^2 + \left[\sum K_{HH'} \sin(\phi_H + \phi_{H-H'})\right]^2 \quad - (1.7)$$

$$\text{and } K_{HH'} = 2\sigma_3\sigma_2^{-3/2} |E_H||E_{H'}||E_{H-H'}| \quad - (1.7a)$$

$$\text{and } \sigma_n = \sum_{j=1}^{N} Z_j^n \quad - (1.7b)$$

Z being the atomic number of the jth atom in a unit cell containing a total of N atoms. The parameter α_H gives a measure of the reliability with which ϕ_H is determined by the tangent formula. It can be shown that the $|E_{min}|$ required to obtain an acceptable α_H increases as a function of N, and therefore it is more difficult to obtain a good start, the larger the number of atoms in the unit cell.

Normalised Structure Factors 1.4.2

Implicit to any direct methods procedure, is the calculation of normalised structure amplitudes, from the structure amplitudes. The normalised structure factors are defined as

$$E_H = \frac{F_H}{(\epsilon \sum f_j^2)^{\frac{1}{2}}} \quad - (1.8)$$

where $f_j = f_j^\circ \exp(-B(\sin^2\theta)/\lambda^2)$ i.e. temperature factor expression.

ϵ is introduced to adjust for space group symmetry

E values are F values corrected for fall off in scattering at high angles, and for thermal motion. They are absolute measures of

the strength of a reflexion, and are therefore better used in direct methods.

Normalised structure amplitudes can be calculated in two ways.

(i) A Wilson plot [18] this is one of

$$\ln\left[\frac{\langle F^2_H\rangle_{rel}}{\Sigma(f^o_j)^2}\right] \text{ versus } \sin^2\theta/\lambda^2 \qquad -(1.9)$$

This should give a straight line, but in practice it is not, so the least squares straight line through the points is calculated, and from the slope, the overall temperature factor coefficient B can be obtained and from its intercept at $\sin\theta/\lambda = 0$ the scale factor k necessary for the calculation of the normalised structure amplitudes from

$$\ln\left[\frac{\langle F^2_H\rangle_{rel}}{(f^o_j)^2}\right]_\theta = 2\ln k - (2\bar{B}/\lambda^2)\sin^2\theta \qquad -(1.10)$$

(ii) A K curve [19] is a plot of

$$\left[\frac{\langle F^2_H\rangle_{rel}}{\Sigma(f^o_j)^2\epsilon_H}\right] \text{ versus } \sin^2\theta/\lambda^2 \qquad -(1.11)$$

This method divides reciprocal space into a series of fairly narrow spherical shells. Within each shell a factor is found which will convert the average observed intensity in the shell to unity. A smooth curve is fitted to these points, from which values may be obtained which will be used to produce appropriate $|E|$ values.

Direct Method Procedures 1.4.3

Structure invariants, seminvariants and the tangent formula form the basis of direct methods. However two methods may be used to develop phases in direct methods, those of symbolic addition and multisolution. With symbolic addition, new phases are developed in terms of both symbols and figures. This method

was first used to solve the structure of a noncentrosymmetric structure in 1964 [20]. The method used in this work is the multisolution method.

Multisolution Direct Methods 1.4.4

The first stage in any direct methods procedure is the calculation of $|E|$ values from the observed intensities. This is done by using one of the methods mentioned previously. However, multisolution methods [21,22] differ from symbolic addition methods, in that numerical phases rather than symbols are introduced as soon as possible. After the calculation of $|E|$ values, a Σ_2 list is compiled. This consists of all the triple phase relationships which link some of the strongest reflexions. It should be noted at this point that the triplet phase sums have probability character, and for the triplet $\phi_H + \phi_{H'} + \phi_{-H-H'}$ a probability distribution can be derived [23] which has its maximum at 0. The probability distribution is also sharper for larger values of $E_3 = N^{-\frac{1}{2}} |E_H E_{H'} E_{-H-H'}|$ in which N is the number of atoms in the unit cell. Thus the larger the $|E|$ values used in the estimate of phases, the more reliable the estimate is. The next step in the process involves the calculation of a convergence map [22]. $\alpha_H 2(\text{estimated})$ is calculated for each reflexion and is a measure of how reliably each phase can be determined in terms of all the others remaining in the data set at any one time. It can be calculated without knowledge of individual phase angles. By using this the least reliably determined phase is eliminated at each stage, leaving those reflexions which are strongly linked together at the end. These are the ones which will lead to reliable phase determination, by giving multiple indications using strong reflexions.

By recourse to symmetry considerations, all those reflexions which are structure seminvariants are determined. Using the Σ_1 formula i.e.

$$\phi_H \backsim \phi_{2H}/2 \qquad -(1.12)$$

these are expanded using restricted phases, and the probability of the phases being correct calculated. If the probability is above a preset value and the number of reflexions contributing to the calculation of the probability is greater than three, then the reflexions are accepted for use in the starting set.

The origin fixing reflexions (and the enantiomorph fixing reflexion in the case of noncentrosymmetric structures) are chosen. These origin fixing reflexions are chosen for example in 'MULTAN'[36] from as far down the convergence map as possible, and they cannot be structure seminvariants or combine to give an invariant. The origin fixing reflexions are given arbitrary phases, and the enantiomorph fixing reflexion is given a value of between 0 and π or between π and 2π. A few other phases are chosen for the starting set, and permuted trial sets of phases used. For phases with special values $0, \pi, \pm\frac{1}{2}\pi$ are used and for general phases $\pm\frac{\pi}{4}$ or $\pm\frac{3\pi}{4}$. The maximum error that is produced by this is 45°, which can easily be coped with by tangent refinement.

Further phases are then developed from the starting set of phases, producing several phase sets depending on the original phases. Each is expanded and refined by the tangent formula. Application of the Σ_2 relationships to non-centrosymmetric structures, usually yields several estimates for the value of ϕ_H Some relationships involving large E magnitudes will contribute more strongly to the estimate than others therefore it is more

usual to employ the weighted tangent formula

$$\tan \phi_H \approx \frac{\sum_{H'} \omega_H \left|E_{H'}\right| \left|E_{H-H'}\right| \sin(\phi_{H'} + \phi_{H-H'})}{\sum_{H'} \omega_H \left|E_{H'}\right| \left|E_{H-H'}\right| \cos(\phi_{H'} + \phi_{H-H'})} \quad - (1.13)$$

which takes into account the strength of the relationship being used at the time.

Due to the various phase permutations, there will be many solutions, and there must be a way of testing these trial sets of phases. This is done by means of figures of merit.

Figures of Merit 1.4.5

There are four figures of merit in general use

ABS FOM - this is given by Z where

$$Z = \sum_H (\alpha_H - \alpha_{RH}) \Big/ \sum_H (\alpha_{EH} - \alpha_{RH}) \quad - (1.14)$$

where $_{RH}$ is the value expected by random phases given by

$$\alpha_R = (\sum_r k_r 2)^{\frac{1}{2}} \quad - (1.14a)$$

and k_r is as given for (1.7)

It is a measure of the internal consistency amongst the $\sum_2$ relationships. It will be zero for random phases and unity if α_H is equal to its expectation value.

PSI ZERO [24] - this is defined as

$$\Psi_o = \sum_H \sum_{H'} \left|E_H\right| \left|E_{H-H'}\right| \quad - (1.15)$$

where $|E|$ values are either very small or zero. For small $|E_H|$, Ψ_o should have a small value for the correct phase set. It is independent of the tangent formula.

RESID [17] is given by

$$\text{RESID} = R_K = \frac{\sum_H \left|\left|E_H\right| - \left|E_H\right|_{calc}\right|}{\sum_H \left|E_H\right|} \quad - (1.16)$$

where

$$\left|E_H\right|_{calc} = K\langle \left|E_{H'}\right| \left|E_{H-H'}\right| \rangle_H \quad - (1.16a)$$

$$K = \sum_H \left|E_H\right|^2 \Big/ \sum_H \langle \left|E_{H'}\right| \left|E_{H-H'}\right| \rangle^2 \quad - (1.16b)$$

For the correct set of phases, RESID should be lowest.

A useful figure of merit for determining the correct phase set is the combined figure of merit, C, given by

$$C = \frac{(Z - Zmin)}{w_1 (Zmax - Zmin)} + \frac{\left[(\Psi_o)max - \Psi_o\right]}{w_2\left[(\Psi_o)max - (\Psi_o)min\right]} + \frac{\left[(R_K)max - R_K\right]}{w_3\left[(R_K)max - (R_K)min\right]} \quad -(1.17)$$

The final stage of any direct methods procedure is the calculation of an E map [25], for the phase set with the most favourable figures of merit. The E map is a Fourier map calculated with E values instead of F values.

Advances in Direct Methods 1.4.6

In recent years direct methods have been modified and improved.

As seen earlier, the triplet relationship is an invariant, however higher invariants can be generated, which increase the power of resolution. The most useful of these invariants are the quartets. The general relationship is

$$\phi_H + \phi_{H'} + \phi_{H''} + \phi_{-H-H'-H''} = p \quad -(1.18)$$

where the most probable value of p is 0 for $E_4 = N^{-1} \left| E_H E_{H'} E_{H''} E_{-H-H'-H''} \right|$

These quartets have been known since 1953 [26], but their full power was realised in 1973 [27], when it was discovered that they become more reliable when considered simultaneously with three identical quartets, constructed from triplets, such as

$$\left.\begin{matrix} \phi_H + \phi_{H'} + \phi_{H-H'} \\ \phi_{H''} + \phi_{-H-H'-H''} + \phi_{H+H'} \end{matrix}\right] \quad \phi_H + \phi_{H'} + \phi_{H''} + \phi_{-H-H'-H''} \quad -(1.19)$$

This indicates that p depends not only on E4, but on the cross term $\left|E_{H+H'}\right|$. As two other identical quartets including

the phases $\phi_{H+H''}$ and $\phi_{H'+H''}$ may be constructed, the value of p is also dependent on $|E_{H+H''}|$ and $|E_{H'+H''}|$ This forms part of the very important neighbourhood principle. The first neighbourhood of the quartet (1.19) consists of the four magnitudes

$$|E_H|, |E_{H'}|, |E_{H''}|, |E_{-H-H'-H''}| \quad - (1.20)$$

In the favourable case where these four magnitudes are large then $p \wr 0$. However the magnitudes are rarely large enough to satisfy this. A more useful relationship involves the cross terms already mentioned where

$$|E_{H+H'}|, |E_{H+H''}|, |E_{H'+H''}| \quad - (1.21)$$

and the four magnitudes above are the second neighbourhood of the quartet (1.19) Where all seven magnitudes are large then $p \wr 0$ and where (1.20) are large and (1.21) are small, then $p \wr \pi$ Those where $p \wr \pi$ are negative quartets, and Hauptman has shown [28] that although the estimates are subject to systematic errors, this is a very powerful tool. There also exists a third neighbourhood [29,30] which shows that the value of p can depend on 13 E magnitudes. This can be useful as allowance is made for missing E magnitudes. This formula is not yet widely used. A special quartet with

$$2\phi_H + \phi_{-H+H'} + \phi_{-H-H'} = p \quad - (1.22)$$

where for large $|E_H|$, $|E_{-H+H'}|$, $|E_{-H-H'}|$ and small $|E_{H'}|$, [31,32], $p \wr \pi$ is also useful.

Quintet invariants also exist, and these take the form

$$\phi_H + \phi_{H'} + \phi_{H''} + \phi_{H'''} + \phi_{-H-H'-H''-H'''} = p \quad - (1.23)$$

However these have a $N^{-3/2}$ reliability dependence (as compared to $N^{-\frac{1}{2}}$ for triplets and N^{-1} for quartets) and there is difficulty in generating accurate relationships so they are not

widely used.

Quartets and Quintets can be used for the figures of merit NQEST [33] and NQINT. Again NQEST is the most useful and is given by

$$NQEST = \sum_{H,H',H''}^{n} B \cos(\phi_H + \phi_{H'} + \phi_{H''}) \Big/ \sum_{H,H',H''}^{n} B \qquad - (1.24)$$

NQEST ranges from -1 to +1 and is expected to be negative for a correct set of phases, provided that the E magnitudes of the cross terms are sufficiently small, and B min (derived from the probability relationships) [34] is sufficiently large.

Magic integers [35] are now widely used in direct methods programmes for the permutation of phases. They allow estimated phases to be assigned to a large set of reflexions. This is useful for complex structures where an increase in the size of the starting set is desirable. Approximate values of several phase angles can be expressed in terms of a single parameter x by

$$\phi_r = n_r x \text{ (modulo 1)} \qquad - (1.25)$$

If the integers n are chosen wisely e.g.

$$\begin{aligned} \phi_1 &= 3x \bmod(1) \\ \phi_2 &= 4x \bmod(1) \\ \phi_3 &= 5x \bmod(1) \end{aligned} \qquad - (1.26)$$

then no matter the value of ϕ for some value of the coefficient x in the range $0 \leq x < 1$ the three equations can be approximately satisfied. Thus three phases may be expressed in terms of one undefined variable (another three may be defined in terms of the variable y or z). In order to determine the value of x, the highest peaks in a type of Fourier series [36] are located. In

general any relationship involving the phases represented by magic integers and fixed phases can be expressed as an equation of the type

$$Hx + H'y + H''z + b = 0 \mod(1) \qquad - (1.27)$$

(b arises because of translational symmetry)

It is not possible usually to find x,y,z values which simultaneously satisfy all of several of these relationships, but the best solutions can be identified with the x,y,z coordinates of the highest positive peaks of the function.

$$\Psi(x,y,z) = \sum |E_{1r}\ E_{2r}\ E_{3r}|\ \cos 2\ (H_r x + H'_r y + H''_r z + b_r) \qquad - (1.28)$$

This method has been extended by the primary - secondary method [37]. Here a number of phases called primaries are represented directly by magic integers. Strong triple phase relationships are found which link two primaries with one other reflexion, and that third reflexion (a secondary) may be represented in magic integer form. The Ψ map may then be calculated with all relationships linking the P and S reflexions. These principles have been incorporated into the programmes 'MAGIC' and 'MAGEX' [38].

An alternative exists to that of phase permutation, the method of assigning random phases to the starting set. The method may proceed in two ways from here.

(i) Apply least squares techniques to a set of linear equations representing the triple phase relationships. Trial sets of random phases are obtained, and then extended as normal by the tangent formula and refined. This is the basis of the programme 'YZARC' [39].

(ii) Assign unknown phases random values, and only refine these [40]. This is the basis of the programme 'RANTAN'.

Quartets, Quintets, YZARC and RANTAN, were not available for

most of this work, but latterly with the use of 'MITHRIL' [41] these became available.

Refinement of Crystal Structures 1.5

Introduction 1.5.1

Once a trial structure has been obtained using the methods described earlier, it is necessary to refine the coordinates in order to obtain more accurate structural parameters. Refinement involves adjusting the parameters that define the proposed structure to attain the maximum agreement with the observed X-ray data. From the model, a set of structure factors is obtained, allowing their magnitudes $|F_H|_c$ to be compared with the observed structure amplitudes $|F_H|_o$. This is usually done using the reliability index

$$R = \frac{\Sigma \big| |Fo| - |Fc| \big|}{\Sigma |Fo|} \qquad - (1.29)$$

where Fo and Fc are the observed and calculated structure factors respectively. Although this index is useful in measuring whether the adjustment made to a model is an improvement, it is not a good indicator of the correctness of a model.

Two main methods are used for the refinement of structures. those of (i) difference synthesis (Fo - Fc) (ii) least squares. The difference synthesis involves a step by step adjustment of the electron density distribution expected for the model structure, to that of the observed electron density distribution. Least squares involves a step by step adjustment of the calculated $|F|$ values to the observed values, (it is carried out in reciprocal space). The method used in this work is least squares refinement coupled with a difference map for the location of missing atoms.

Least Squares Refinement 1.5.2

Given a linear function with n variables $x_1, x_2, \ldots x_n$ the variables can be thought of as defining a space whose value at any point is determined both by the location $x_1, x_2, \ldots, x_n$ and by independent parameters $p_1, p_2, \ldots, p_n$ which define the function. This gives

$$f = p_1x_1 + p_2x_2 + \ldots + p_nx_n \qquad - (1.30)$$

if the values of the function are measured at m different points with $m > n$ then least squares theory states that the best values for the parameters $p_1, p_2, \ldots, p_n$ are those which minimise the sums of the squares of the properly weighted differences between the observed and calculated values of the function for all the observational points. The quantity to be minimised is

$$D = \sum_{r=1}^{m} \omega_r (fo_r - fc_r)^2 \qquad - (1.31)$$

where ω_r = weight to be assigned to the observation

fo_r = one of the m observed values of the function

fc_r = corresponding calculated value to fo_r

The parameters p need to be adjusted to minimise D. This is achieved by differentiating the right side of (1.31) with respect to each of the parameters in turn, and setting the derivative equal to zero, giving

$$\sum_{r=1}^{m} \omega_r (fo_r - fc_r) \delta fc_r / \delta p_j = 0 \quad (j = 1,2,\ldots,n) \qquad - (1.32)$$

This gives a set of n equations in n unknowns, known as the normal equations. Normally partial derivatives are taken for each of the equations such as (1.30), and substituting in (1.32) to give the normal equations. Solution of these equations gives directly the best values of the parameters p_j in the least squares sense.

If the functional form of the equations (1.30) is not linear

in the p's then the normal equations are not linear. They can however be made linear by approximating the function as a Taylor series. This happens in X-ray diffraction, where the quantity most commonly minimised is

$$D = \sum_H \omega_H \left(|Fo| - |kFc| \right)^2 \qquad - (1.33)$$

where ω_H is the weight of the observation and $_H$ indicates summation over all the observed reflexions. The n normal equations are of the form

$$\sum_H \omega_H \left(|Fo| - |k\ Fc\ (p_1, p_2, \ldots, p_n)| \right) \delta |k\ Fc\ (p_1, \ldots, p_n)| / \delta p_j = 0$$
$$(j = 1, 2, \ldots, n) \qquad - (1.34)$$

The function $|Fc|$ may be expressed as a Taylor series to give a system of n equations in n unknowns $(\Delta p_j's)$. These equations are linear in the $\Delta p_j's$ and solvable for them. By using a Taylor series, the terms a_j are generated, which are approximate values of p_j If the a_j are sufficiently good approximations, application of a least squares process to the linear equations, gives values for the quantities Δp_j such that the $a_j's$ given by

$$a_j' = a_j + \Delta p_j \qquad - (1.35)$$

are better approximations to the best values for the parameters p_j than the initial $a_j's$. Combination of the linear equations with the approximate $a_j's$ gives better values for the various parameters. These may then be used to repeat the process until convergence is obtained, and no further changes are produced. Sometimes the intensities may be minimised instead of the amplitudes. However the approximations made in constructing the least squares equations constrain its application to the refinement of reasonably accurate initial parameters.

The normal equations are very complex, and they are usually described in terms of matrix notation. The matrix of the normal equations is a square array. The principle diagonal has the elements a_{ii} lying on it and are sums of squares and the terms must always be positive. The off diagonal elements are sums of products and can therefore be positive or negative. Although there are several types of least squares analysis depending on the type of matrix used, full matrix least squares is used in these studies. The normal equations may be written

$$\begin{aligned} a_{11}x_1 + a_{12}x_2 + \ldots + a_{1n}x_n &= v_1 \\ a_{21}x_1 + a_{22}x_2 + \ldots + a_{2n}x_n &= v_2 \\ &\vdots \\ a_{n1}x_1 + a_{n2}x_2 + \ldots + a_{nn}x_n &= v_n \end{aligned} \qquad - (1.36)$$

where

$$a_{1j} = \sum_{r=1}^{m} \omega_r \frac{\delta |Fc_r|}{\delta p_i} \frac{\delta |Fc_r|}{\delta p_j} \qquad x_j = \Delta p_j \qquad - (1.36a)$$

$$v_i = \sum_{r=1}^{m} \omega_r (\Delta F_r) \frac{\delta |Fc_r|}{\delta p_i} \qquad - (1.36b)$$

This may be written in matrix form.

$$\begin{pmatrix} a_{11} & a_{12} & \ldots & a_{1n} \\ a_{21} & a_{22} & \ldots & a_{2n} \\ \vdots & \vdots & & \vdots \\ a_{n1} & a_{n2} & \ldots & a_{nn} \end{pmatrix} \begin{pmatrix} x_1 \\ x_2 \\ \vdots \\ x_n \end{pmatrix} = \begin{pmatrix} v_1 \\ v_2 \\ \vdots \\ v_n \end{pmatrix} \qquad - (1.36c)$$

or as $AX = V$ - (1.36d)

If the equations (1.36) have a solution an inverse matrix A^{-1} exists such that $A^{-1}A$ equals the matrix equivalent of 1 then

$$\begin{aligned} A^{-1}AX &= A^{-1}V \\ X &= A^{-1}V \end{aligned} \qquad - (1.37)$$

A^{-1} is a matrix with elements b_{ij}.

The parameters p_j in X-ray analysis include fractional

positional coordinates of all the atoms, isotropic or anisotropic temperature factor parameters and an overall scale factor. The full matrix least squares minimisation process thus involves calculations of the elements of a normal equations matrix of very large dimensionality and also the inversion of such a matrix. Conversion to the optimal parameters can be slow and occur over many cycles. This process can however be carried out readily by computer.

Estimated Standard Deviations 1.5.3

As it is normal practice to solve the normal equations by matrix methods, both the new values of the parameters varied and their estimated standard deviations may be found. The equation used is

$$\sigma p_i = \sqrt{b_{ii} \left(\sum_{r=1}^{m} \omega_r \Delta F_r^2 \right) / (m - n)} \qquad - (1.38)$$

where b_{ii} is the ith diagonal element of the inverse matrix, ω_r the weight of the rth ΔF, m the number of observations and n the number of parameters.

Correlation Coefficients 1.5.4

The correlation coefficient may be defined by the equation

$$\delta ij = b_{ij} / \sqrt{b_{ii}} \sqrt{b_{jj}} \qquad - (1.39)$$

It is a measure of the interdependence or correlation of parameters i and j. If correlation coefficients are large interdependence among the parameters may seriously impair the refinement.

Weighting Schemes 1.5.5

The functions as in (1.31) and (1.33) are minimised in least squares analysis, and they carry a weighting factor for each observation. The weighting should be a measure of the reliability of the

observation, and can if properly chosen adjust the contribution of each observation to the normal equations in such a way as to produce the most reliable results. This however is often insufficient and other methods exist for defining weights [43].

Scale Factor 1.5.6

$|Fc|$ values are on an absolute scale, and therefore a scale factor should be applied to the $|Fo|$ values. However during least squares analysis, the structural model is adjusted to fit the observations, therefore it is the scale of the $|Fc|$ values that has to be adjusted. Once the scale factor has been determined the $|Fo|$ values have to be rescaled to almost an absolute scale by multiplying by $1/$ scale factor.

Difference Map 1.5.7

The difference synthesis involves the use of the Fourier transform of (Fo-Fc) , thus

$$\Delta\rho = {}^{1}/_{V_{h\,k\,l}} (Fo - Fc) e^{i\phi c} e^{-2\pi i(hx + ky + lz)} \quad - (1.40)$$

where ϕc is the phase of Fc.

Even when the phases are partly in error, the ΔF synthesis can provide useful information. A partial set of data, for which the probabilities are especially high that the assigned phases are correct, may be used. A difference synthesis is normally carried out at the end of a routine least squares analysis and as is obvious from its nature, it is useful for locating missing atoms, even hydrogen atoms may be located by difference synthesis and the production of a difference map.

Empirical Force Field Calculations 1.6

Introduction 1.6.1

The Force Field or molecular mechanics method, provides a useful method for determining the structure and energy of a molecule. The basis of these calculations is that bonds have natural lengths and angles and that molecules will adjust their geometries to take these up as nearly as possible. Obviously as a molecule becomes more complicated, other forces come into play, but the above is still a good approximation, if steric interactions are included.

Although the basic ideas of Force Field calculations go back to the 1930's, the most important papers appeared in 1946. T.L. Hill proposed that van der Waals interactions together with stretching and bending deformations, should be used to minimise steric energies, leading to information about structure and energy [44]. Westheimer and Mayer also formulated the method for studying racemization rates [45]. However, these methods were not widely applicable as there were no computers. However, after the 1950's this method grew considerably in use.

In molecular mechanics, the motions of the nuclei are studied, and the electrons are assumed to find an optimum distribution about the nuclei. The Born-Oppenheimer surface describes the energy of the molecule in terms of the nuclear positions. Molecular mechanics calculations employ an empirically derived set of equations for the Born-Oppenheimer surface. This set of equations is called the Force Field.

Force Fields 1.6.2

The Force Field contains parameters that may be adjusted to

obtain the best fit of calculated and experimental properties of the molecules. The assumption is often made in molecular mechanics that corresponding parameters and force constants may be transferred from one molecule to another. The simplest equation formulated is the Westheimer equation

$$E = \sum Es + \sum Eb + \sum E_{\omega} + \sum Enb \qquad - (1.41)$$

where E - overall molecular potential energy, the steric energy

Es - energy of bond deformation

Eb - energy of bending

E_{ω} - torsional energy

Enb - energy of non bonded interactions

These are summed over all bonds, bond angles, torsion angles and non-bonded interactions between all atoms not bound to each other or to a common atom. Other terms may also be introduced which take into account coulombic and solvent interactions and also out of plane bending.

In molecular mechanics, the atoms of a molecule may be thought of as being joined by mutually independent springs, restoring the natural values of bond lengths and angles. Every molecular mechanics force field contains the following functions

$$Es = \tfrac{1}{2} Kl\ (l - lo)^2 \qquad - (1.42)$$

where Kl = force constant, l = bond length. lo = strain free bond length.

$$Eb = \tfrac{1}{2} F\theta\ (\theta - \theta o)^2 - F\theta'\ (\theta - \theta o)^3 \qquad - (1.43)$$

where $F\theta$ and $F\theta'$ are force constants, θ = valency angle, θo = strain free valency angle.

The Force constants and natural lengths/angles must be the same for all bonds of the same type.

Another essential term in any force field is the van der Waals interaction term. This may be represented by the equation

$$Enb = Fr \{-2/\alpha^6 + \exp [12 (1 - \alpha)]\} \qquad - (1.44)$$

where Fr is a force constant, $\alpha = r/(r_1^* + r_2^*)$, r being interatomic non-bonded distances and r_1^* and r_2^* are van der Waals interaction constants.

The van der Waals interaction constants are calculated in different ways by different workers [46 50].

The final standard term in any molecular mechanics force field, is the torsional term. This may be represented by

$$E\omega = \tfrac{1}{2} F\omega (1 + s \cos n\omega) \qquad - (1.45)$$

where $F\omega$ = force constant, n = periodicity of $F\omega$, ω = torsion angle, s = +1 (minimum energy when staggered), s = -1 (minimum energy in eclipsed conformation).

The force constants required for the above equation are either picked from fitting to known structures [51,52] or spectroscopic values [53,54] are used.

The above equations have been developed considerably over the last few years, to make them more powerful and applicable to more structures. This has been done by modifying the parameters used.

Parameters 1.6.3

A molecular mechanics force field is only as good as the potential functions, and the parameters used. Different force fields usually differ in their parameters and also in the mathematical form of the

potential functions, therefore the parameters are not transferable. It is obviously advantageous to optimise the parameters used, and this is done by using a combination of trial and error and least squares techniques [55].

There has been considerable contention as to the values for strainless bond lengths and angles, but the application of additional experimental data to determining and improving these values is leading to accepted best values. The parameters used for non-bonded interactions have been the most difficult to determine, as there are four to find, these being (i) the depth, (ii) the van der Waals radius, (iii) steepness of the repulsive potential, (iv) correlation factor between the attractive and repulsive potentials. Numerical values for the parameters for different atoms have to be found, and this has been done in two different ways (i) by quantum mechanical methods [56,57], and (ii) empirically [58]. Both of these worked quite well, and whichever van der Waals functions are used in modern force fields, they are all relatively similar. Torsional parameters must also be fixed, and this is done relatively simply by fitting the rotational barriers about single bonds in simple molecules.

It must be stressed again that parameters and constants vary from force field to force field, and one that may describe a particular type of molecule well, may not be as good for another.

Conjugated systems produce special problems in parameterisation, but two methods have been used to overcome this. The first involves a self consistent field calculation on the pi system, and then a separate molecular mechanics treatment for the rest of the molecule [59,60]. This works reasonably well. The second is that of Allinger and Sprague [55], and this overcomes problems of non-planarity in the pi system.

Minimisation of Molecular Geometry 1.6.4

Using the potential energy functions described earlier and a set of coordinates, an initial energy for a molecule may be calculated. Energies calculated with standard geometries are however acknowledged as being only qualitatively correct. There therefore has to be some way of optimising geometry with respect to energy, and this is part of the standard molecular mechanics procedure. Minimisation techniques are of two types (i) first derivative minimisation and (ii) second derivative minimisation.

First derivative minimisation as its name suggests, makes use only of the slope of the potential surface. The object of this is to find x, the coordinate where the energy (f(x)) is a minimum, starting from x_o, the initial value of $x.f(x_o)$ is calculated and then a technique such as steepest descent used to compare it with a value $f(x_o + \Delta x)$ the sign of the change in the value of the function giving the direction of the minimum, and its value how much nearer the minimum it is [61]. This is the numerical approach. The energy is calculated each time an atom is moved in turn by small increments in the directions of the three coordinate axes, the process being repeated until a minimum is determined. Steepest descents methods converge rapidly when derivatives are large, but when they are small, convergence is slow. A modified steepest descent method formulated by Schleyer, et al. [62,63] called pattern search does not have this problem.

The method most often used is second derivative techniques. These utilise both the slope and the curvature of the potential surface. It is usually called the Newton - Raphson (NR) minimisation technique. The method is applied in molecular mechanics for the minimisation of the sum of potential energy functions. In this

method, the function of the energy with respect to the coordinates is analytically differentiated with respect to each coordinate. The second derivatives are then taken, again with respect to each coordinate. The atoms are then moved to points where all of the first partial derivatives are simultaneously equal to zero, which is accomplished by solution of $3N-6$ simultaneous equations. Because of approximations used in deriving the equations, the energy minimum is not reached on the first try, so the process is repeated with the improved coordinates, as many times as is necessary. This method is much faster than steepest descents. Variations of this method have been used by different authors [53,64-70].

Uses of Molecular Mechanics Calculations 1.6.5

Molecular mechanics calculations have been employed in a wide range of structural studies. Conformational minimums can be determined accurately by this method. Heats of formation, reaction rates, interconversion pathways and energy profiles may also be determined by this method. In this work molecular mechanics calculations have been used in conformational studies, to compare X-ray structures with proposed energy minimum structures and also to produce accurate structural parameters for proposed structures in biosynthetic pathways. It should be stressed that molecular mechanics is an empirical method, and the molecular model obtained relates to a hypothetical motionless state at absolute zero, however for the atoms and systems for which the parameters have been fully developed, it is an extremely good way of obtaining molecular models.

CHAPTER TWO

DATA COLLECTION USING THE FOUR CIRCLE DIFFRACTOMETER

Data Collection Using the Four Circle Diffractometer

Instrumentation 2.1

The four circle diffractometer has four arcs, which may be adjusted independently or together, to bring any crystal plane into the desired reflecting position. The four circles are ω, 2θ, ϕ and χ. ω and 2θ are mounted about a common axis and their movements may be correlated. The orienter i.e. the ϕ and χ circles is mounted within the ω circle and rotates with ω This may be shown more clearly in Fig. 2.1 The particular four circle diffractometer used in these studies was a NICOLET P3 diffractometer.

The goniometer head to which the crystal is mounted is attached about the ϕ axis. It is very important for the alignment of the crystal that the axes of the $\omega(2\theta)$ and ϕ circles and the centre of the χ arc intersect precisely.

The remaining parts of the diffractometer are an X-ray tube and detector being so positioned that the centre of the orienter system, centre of the detector aperture and the focal point of the source all lie in a plane accurately perpendicular to the $\omega(2\theta)$ axis.

Method 2.2

The first stage in the data collection is to select a suitable crystal. This is done by initially examining the crystals under the microscope and selecting a crystal of suitable dimensions and with no obvious defects. The crystal is then studied using the polarizing microscope to determine if it extinguishes evenly. If these conditions are met, then the crystal is accepted.

The crystal is mounted on a pip which is inserted into the

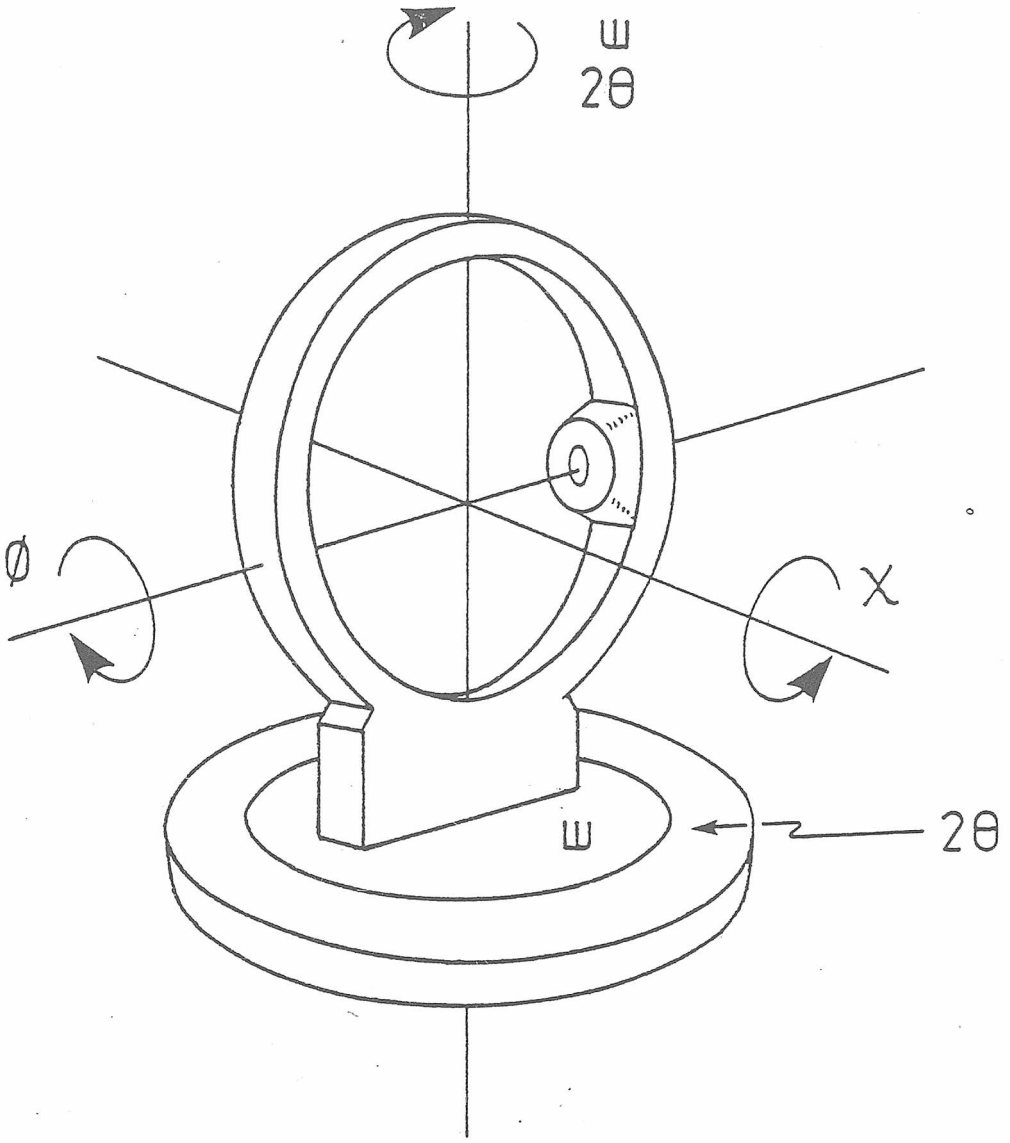


FIG. 2.1

goniometer head. The goniometer is mounted in the centre of the diffractometer and the crystal aligned in the X-ray beam. The crystal is aligned optically using a microscope with a crosswire gauge. The ϕ, ω and χ circles are set at zero degrees and 2θ is rotated, the crystal being realigned as necessary until at no time in 360° rotation of 2θ does the crystal move from the X-ray beam.

In order to find a few reflexions from the crystal, a rotation photograph is taken, the time taken for this and the number of rotations depending on the size of the crystal. The 2θ, ω and χ axes are set at zero degrees and ϕ is rotated continuously. From the polaroid photograph, the approximate distances between equivalent reflexions are measured. From these measurements and with the distance from the crystal to the centre of the film, the computer calculates values of χ and 2θ

The centering programme is then used. This performs all the necessary functions for defining the reflexion array. After calculating the values of χ and 2θ the reflexions are searched for by driving to these angles and rotating ϕ at slow speed in 120° steps up to 360°. Reflexions are accepted as being observed if they are 1000 counts per second or over. This minimum may of course be reduced if the reflexions are particularly weak. The centres of the reflexions are determined by fixing ϕ and moving the 2θ, ω and χ axes one at a time to find the individual centres. These centres are found in an iterative manner, the process being terminated when on the last iteration the changes in 2θ, ω and χ are less than 0.02°, 0.01° and 0.04° respectively.

The next step in the data collection process is the determination

of the axes. For this purpose, it is necessary to assign indices to the reflexions for which the refined angular settings have been obtained. The programme produces a set of up to 30 possible axial vectors and the angles between these vectors. Three solutions that are orthogonal and/or short are chosen and identified by number as a,b,c. Axial photographs may be taken at this point in order to confirm the presence of symmetry axes and also to determine from the layer line spacing whether any of the axial lengths need to be doubled. The axes identified, the programme indexes each reflexion in the reflexion array and outputs a calculated cell volume. The data is now in a suitable form for the least squares calculations. From the indices and refined angles, the orientation matrix, unit cell dimensions and volume are calculated as are the standard deviations of these, if required. Calculated hkl values are also printed.

A fast data set is then collected. This is done using a $2\theta:\theta$ scan. Here the detector and the crystal move simultaneously, the ω axis driving at half the speed of the 2θ axis. The data are collected to $2\theta \leq 10^{\circ}$. The scan rate is very high, so that only those reflexions of high intensity are accepted as being observed. Several reflexions with the highest intensity and which represent all quadrants of reciprocal space are chosen and expanded in accordance with symmetry considerations. These are then reinput into the centering programme for centering, and thereafter a more accurate orientation matrix determined.

The next step is the data collection. This is again carried out using the $2\theta:\theta$ scan. Firstly standard reflexions are chosen, the intensities of these being measured at regular

intervals during the data collection. The maximum 2θ value and the starting and finishing hkl values are set and the data collection proceeds automatically.

Once the data has been collected, it is introduced into a data reduction programme. Firstly the intensities are converted to structure factor amplitudes, and a corresponding correction done on the esd's of the intensities. The F observed ($|Fo|$) values are then converted to F relative ($|Frel|$) values by correcting for Lorentz and polarisation effects. The Lorentz correction is as for any set of data , but the polarisation factor is dependent on the angle of incidence of the radiation and hence is modified for use with the diffractometer data. The equation used is

$$p = \left[f \frac{1 + \cos^2 2\theta m \cdot \cos^2 2\theta c}{1 + \cos^2 2\theta m} + 1-f \frac{1 + \cos 2\theta m \cdot \cos^2 2\theta c}{1 + \cos 2\theta m} \right] \quad - (2.1)$$

where m and c refer to monochromator crystal and sample crystal respectively and f takes into account that the graphite crystal may be considered partly ideally mosaic and partly perfect.
The values used are f = 0.5

$$2\theta m(MoK\alpha) = 12.160 \quad - (2.2)$$

The reflexions are then examined, and a reflexion is considered as being observed if $I > 2\sigma(I)$. Unobserved reflexions are removed, and the space group determined on the basis of systematic absences where possible.

CHAPTER THREE

A BRIEF OUTLINE OF PROGRAMMES AND PROCEDURES USED IN THIS WORK

A Brief Outline of Programmes and Procedures used in this Work

Introduction 3.1

Many programmes were used in the course of this work, these being

'MULTAN 78' [71]

'MULTAN 80' [72]

'MITHRIL' [41]

'SHELX' [73]

'MM2' [74]

'MM2P' [75]

'MODEL' [76]

'BONDLA' [77]

'GEOMX' [78]

'ORTEP' [79]

Direct Methods Packages 3.2

'MULTAN 78', 'MULTAN 80' and MITHRIL are all direct methods programmes. Most of the work was carried out using 'MULTAN 80'. All three are suites of programmes designed to carry out the following functions.

1. Normalisation of structure factors. Opportunities exist at this point to input any structural information about the substance being studied. Scaling of the structure factors to obtain the normalised structure factors may be carried out in two ways. a. By drawing least squares straight line through the Debye points. b. By using a k curve.
2. Generation of triplet phase relationships (and higher invariants, i.e. quartets and quintets in 'MITHRIL') and application of space group symmetry to determine phase restrictions.
3. Σ_1 formula is applied to structure seminvariants and the

origin and enantiomorph defining reflexions chosen.

4. Phase relationships set up which will be used later to test the solutions produced by the tangent formula.

5. Generation of starting sets of phases by assigning phase values given by a magic integer sequence to those reflexions chosen earlier. The tangent formula is then used to develop these into a complete set of phases using the previously determined phases.

6. Figures of merit are computed and used to decide whether a set will yield a correct solution, and in which order to compute the E maps.

7. E map with the largest combined figure of merit is computed.

8. Highest peaks in the map are searched for, and bonded sequences among these peaks located. The interpretation of this is printed.

Structure Refinement 3.3

Whilst 'SHELX' is a completely integrated direct methods package, it is not used in this work for that purpose. It is used mainly for full matrix least squares refinement and difference fourier synthesis.

1. Input to 'SHELX' consists of raw intensity data and an instruction file containing the coordinates of the structure/partial structure to be refined.

2. Systematic absences are rejected.

3. Full matrix (or blocked full matrix) refinement is carried out. The form of the anisotropic temperature factors are shown in Fig. 3.1.

4. A difference fourier map is computed, allowing location of missing non-hydrogen atoms if necessary.

5. Bond lengths and bond angles are calculated.

6. Any missing atoms are input and the refined parameters are used to repeat the above process. Anisotropic temperature factors may be input for non-hydrogen atoms, and hydrogen atom positions may be calculated and constrained. Refinement continues until convergence is achieved.

7. Analysis of variance is calculated and printed out.

8. Full F(obs)/F(calc) lists are printed out.

Molecular Mechanics Calculations 3.4

Molecular mechanics calculations are carried out using one of two programmes 'MM2' or 'MM2P'. 'MM2' is not suitable for calculations involving conjugated systems, but 'MM2P' is a modified version of 'MM2' which is suitable for such calculations. The 'MM2' force field is very complex, but it has been fully developed for alkanes, non-conjugated alkenes and alkynes, non-conjugated carbonyl compounds, alcohols, ethers, sulfides, mercaptans, monohalides and amines. Both programmes have been

Thermal Parameters

The anisotropic temperature factors are expressed as

$$\underline{T} = \exp\left[-2\pi^2(\underline{U}_{11}\underline{h}^2\underline{a}^{*2} + \underline{U}_{22}\underline{k}^2\underline{b}^{*2} + \underline{U}_{33}\underline{l}^2\underline{c}^{*2} + 2\underline{U}_{12}\underline{hk}\underline{a}^*\underline{b}^* + 2\underline{U}_{13}\underline{hl}\underline{a}^*\underline{c}^* + 2\underline{U}_{23}\underline{kl}\underline{b}^*\underline{c}^*)\right]$$

with final parameters $\underline{U}_{ij} \times 10^3$

FIG. 3.1

used successfully but only for minimum energy calculations. It is however also possible to conduct heat of formation calculations using these programmes. The following steps are taken in the calculations.

1. Coordinates of structure input, and an initial energy is calculated using the standard geometries of the 'MM2' force field.

2. Energy minimisation takes place by full matrix Newton - Raphson minimisation, and a final energy calculated.

Molecular Geometry Calculations 3.5

Geometry calculations are carried out using the program 'GEOMX'. This programme was developed at Glasgow University. The programme uses previously calculated coordinates for a set of atoms (usually the atoms are the crystallographic asymmetric unit) to obtain their various geometric functions. The functions which may be calculated are bond lengths, bond angles, torsion angles, (the sign convention used for an angle (1)-(2)-(3)-(4) is such that the sign is negative if an anticlockwise rotation of (1) required to eclipse atom (4) whilst looking down the (2)-(3) bond) mean planes, and intermolecular contacts. The above functions may be printed out in publication format with standard deviations.

Calculation of Hydrogen Positions 3.6

Within 'SHELX' there is no function available for the calculation of methylene hydrogen positions. This is therefore carried out using the programme 'BONDLA'. By consideration of the geometries of surrounding atoms, this programme calculates hydrogen atom positions.

Structural Models 3.7

Sometimes it is necessary to obtain coordinates for a proposed structure e.g. for use in 'MULTAN'. The calculations necessary to obtain these coordinates are done using the programme 'MODEL'. The input for 'MODEL' consists of atom connections, and proposed bond lengths, valency angles and torsion angles for the structure.

Perspective Drawings and Stereopacking Diagrams 3.8

Perspective drawings and stereopacking diagrams are produced using the programme 'ORTEP'. This programme uses the atom coordinates and thermal motion data for the data to draw the thermal ellipsoid diagrams. For crystal structures, it also requires a knowledge of the unit cell dimensions and symmetry operations. The types of spheres drawn by the programme may be specified.

Other Computational Procedures Utilized 3.9

During the course of this work, the crystal structure search and retrieval programme [80] prepared by the Cambridge Crystallographic Data Centre, has been used for information search and retrieval. This programme is an on-line interactive programme for retrieving information from the databank of organic and organometallic crystal structures. The databank contains three types of information. 1. Bibliographic information, 2. Connectivity information and 3. Numeric information. The bibliographic information has proved to be the most useful, saving considerable time in obtaining references for structures. Various commands are available for accessing the system, depending on the information that is required.

CHAPTER FOUR

A SESQUITERPENE LACTONE FROM PARAMURICEA CHAMAELEON

A Sesquiterpene Lactone from Paramuricea Chamaeleon

Introduction 4.1

This sesquiterpene lactone (Fig. 4.1) is thought to be a photo-oxidation product of Linderazulene (Fig. 4.2). It was isolated from the coral Paramuricea Chamaeleon as an orange crystalline solid.

From spectral analysis the lactone was thought to have the structure shown in Fig. 4.1. However, the evidence was equivocal, so an X-ray analysis was carried out to confirm the structure.

Experimental 4.2

A crystal of dimensions 0.8 x 0.225 x 0.0625mm was chosen for analysis. The data were collected according to the procedure described in Chapter 2, for 998 reflexions to $2\theta \leq 50^{\circ}$. Oscillation photographs were taken. The structure was solved by using direct methods[71], the E map being computed for 172 reflexions with $|E|_{min} > 1.5$. The non-hydrogen atoms were clearly visible from the E map, and the coordinates of these were refined by full matrix least squares[73]. Anisotropic temperature factors were introduced for all non-hydrogen atoms. The hydrogen atom positions were calculated, but on refinement, the methyl hydrogen atoms had anomalous temperature factors associated with them. The methyl hydrogens were removed and reinserted in the positions visible from the difference map. The methyl hydrogens were given common temperature factors of 0.01 and allowed to refine, and the other hydrogens were given common temperature factors of 0.05 and allowed to refine. The hydrogen atom bond lengths were fixed at 1.00 $\mathring{A} \pm 0.02 \mathring{A}$ and the methyl hydrogen atom separation at 1.633 $\mathring{A} \pm 0.02 \mathring{A}$.

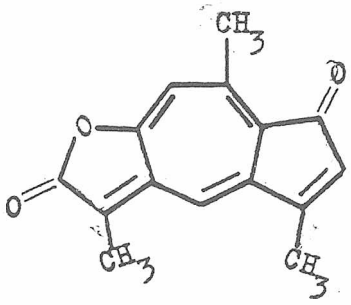


FIG. 4.1

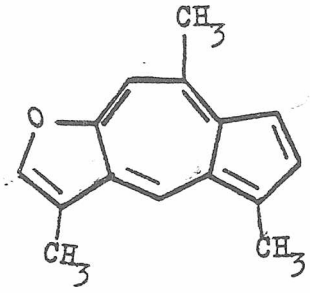


FIG. 4.2

The final weighting scheme used was

$$\omega = 1/\sigma^2(F)$$

Molecular mechanics calculations were carried out using the programme MM2P [75].

Results 4.3

Crystallographic Data 4.3.1

$C_{15}H_{12}O_3$, Mr = 240.258, Dc = 1.38gcm^{-3}, monoclinic, C2/c, Z = 8, a = 11.676(8)Å, b = 15.911(15)Å, c = 13.898(6)Å, β = 116.31(4)°, U = 2314.46Å^3, μ(MoKα) = 0.56cm^{-1}, F(000) = 1008.0, Final R value = 5.5% for 904 unique reflexions ($I>3\sigma(I)$), ωR = 4.17%, maximum height on difference map = 0.170 eÅ^{-3}, minimum height on difference map = -0.264 eÅ^{-3}, maximum shift/error ratio = 0.033.

Structural Data 4.3.2

The sesquiterpene lactone was found to have the structure shown in Fig. 4.1. The atomic arrangement is shown in Fig. 4.3 and a stereoscopic view of crystal packing is shown in Fig. 4.4. The various structural parameters are listed in Table 4.1 - 4.5. Vibrational spectra and their major assignments are shown in Figs. 4.5, 4.6 and Table 4.6 respectively.

Molecular mechanics calculations for this lactone gave a final steric energy of 14.1408 KCal mol^{-1}.

Discussion 4.4

X-ray Analysis 4.4.1

Few problems were encountered in the X-ray analysis of this lactone. The final R value of 5.5% indicates that the data collected were of a reasonably high standard and that the results are reliable.

Although the methyl hydrogens were unstable in their calculated

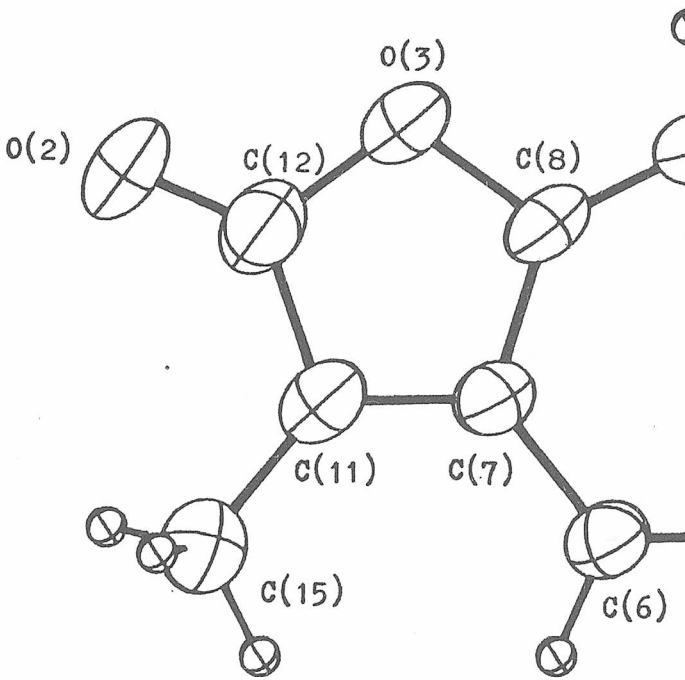
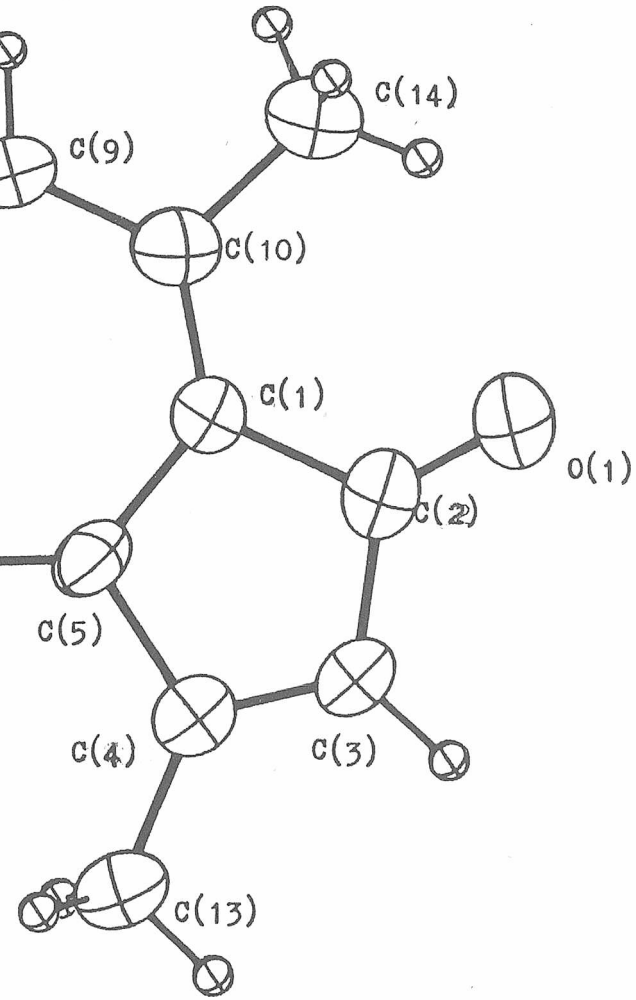


FIG. 4.3


C(14)
C(9)
C(10)
C(1)
O(1)
C(2)
C(5)
C(4)
C(3)
C(13)

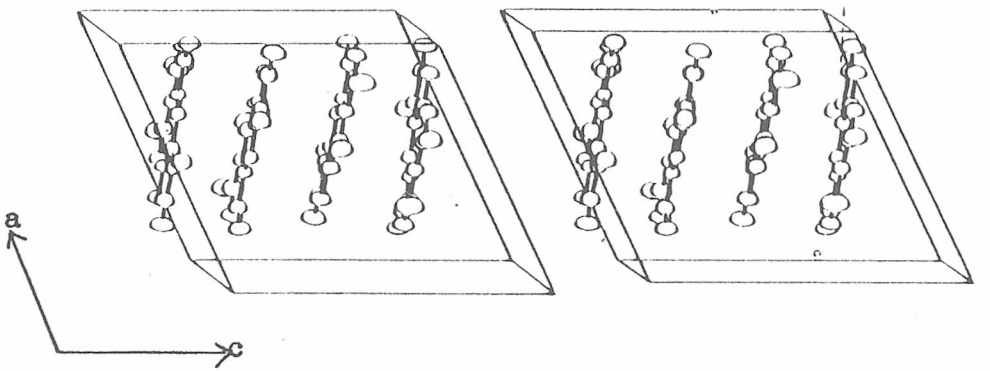


FIG. 4.4

Atom	Fractional
O(1)	
O(2)	
O(3)	
C(1)	
C(2)	
C(3)	
C(4)	
C(5)	
C(6)	
C(7)	
C(8)	
C(9)	
C(10)	
C(11)	
C(12)	
C(13)	
C(14)	
C(15)	
H(3)	
H(6)	
H(9)	
H(13A)	
H(13B)	
H(13C)	
H(14A)	
H(14B)	
H(14C)	
H(15A)	
H(15B)	
H(15C)	

TABLE 4.1

x	y	z
2539(3)	2709(2)	-2639(4)
8444(3)	-1089(2)	0480(3)
7525(3)	0193(2)	0102(3)
3831(4)	1461(2)	-1834(4)
2634(5)	1952(3)	-2500(5)
1613(4)	1335(3)	-2979(4)
2054(4)	0561(3)	-2728(4)
3447(4)	0578(3)	-2017(3)
4133(4)	-0107(3)	-1686(4)
5476(4)	-0209(3)	-1012(3)
6304(4)	0496(3)	-0531(4)
6121(4)	1328(3)	-0569(4)
4981(4)	1795(3)	-1189(4)
6187(4)	-0923(3)	-0696(3)
7502(5)	-0685(3)	0010(4)
1287(4)	-0228(3)	-3051(4)
5186(5)	2738(3)	-1041(5)
5830(5)	-1820(3)	-0971(5)
0718(22)	1490(25)	-3459(29)
3634(31)	-0673(16)	-1930(30)
6891(26)	1651(22)	-0079(28)
1346(40)	-0526(27)	-2390(23)
1624(40)	-0617(26)	-3432(29)
0377(24)	-0089(30)	-3527(28)
4405(28)	3060(29)	-1140(33)
5857(31)	2829(31)	-0283(17)
5511(37)	2940(31)	-1548(27)
6146(37)	-2009(29)	-1503(26)
6254(37)	-2175(28)	-0312(23)
4886(17)	-1897(31)	-1299(32)

Atomic Coordinates ($x10^4$) with e.s.d.'s

Atom	U_{11}
O(1)	56(2)
O(2)	39(2)
O(3)	33(2)
C(1)	37(3)
C(2)	45(3)
C(3)	31(2)
C(4)	38(3)
C(5)	27(3)
C(6)	35(3)
C(7)	33(3)
C(8)	26(2)
C(9)	35(3)
C(10)	44(3)
C(11)	35(3)
C(12)	42(3)
C(13)	36(3)
C(14)	54(3)
C(15)	52(3)
H(3)	55(8)
H(6)	55(8)
H(9)	55(8)
H(13A)	104(7)
H(13B)	104(7)
H(13C)	104(7)
H(14A)	104(7)
H(14B)	104(7)
H(14C)	104(7)
H(15A)	104(7)
H(15B)	104(7)
H(15C)	104(7)

Anisotropic

TABLE 4.2

U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
45(2)	138(4)	4(2)	19(2)	12(2)
79(2)	98(3)	18(2)	9(2)	12(2)
63(2)	73(2)	2(2)	9(2)	6(2)
38(3)	56(3)	1(2)	16(2)	0(2)
49(3)	73(3)	8(2)	18(3)	6(3)
50(3)	73(3)	3(2)	8(2)	2(3)
51(3)	59(3)	1(2)	16(3)	-1(2)
51(3)	47(3)	3(2)	12(2)	2(2)
49(3)	49(3)	-2(2)	17(2)	0(2)
47(3)	46(3)	-2(2)	14(2)	-2(2)
61(4)	49(3)	2(2)	11(2)	3(2)
50(3)	61(3)	-6(2)	11(2)	-3(2)
49(3)	62(3)	-7(3)	21(2)	-4(3)
61(3)	42(3)	4(2)	13(2)	-2(2)
65(4)	63(3)	7(3)	18(2)	12(3)
60(3)	86(4)	-6(2)	16(3)	1(3)
54(3)	101(4)	-10(3)	24(3)	-2(3)
58(3)	74(4)	6(2)	21(3)	-6(3)

Temperature Factor Coefficients ($\times 10^3$) with e.s.d.'s

TABLE 4.3

O(1)	C(2)	1.217(6)	C(4)	C(13)	1.491(7)
O(2)	C(12)	1.189(6)	C(5)	C(6)	1.349(6)
O(3)	C(8)	1.388(5)	C(6)	C(7)	1.431(5)
O(3)	C(12)	1.402(6)	C(7)	C(8)	1.438(6)
C(1)	C(2)	1.508(6)	C(7)	C(11)	1.360(6)
C(1)	C(5)	1.462(5)	C(8)	C(9)	1.338(7)
C(1)	C(10)	1.350(5)	C(9)	C(10)	1.432(6)
C(2)	C(3)	1.458(6)	C(10)	C(14)	1.519(6)
C(3)	C(4)	1.321(6)	C(11)	C(12)	1.459(6)
C(4)	C(5)	1.483(5)	C(11)	C(15)	1.488(7)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 4.4

C(1)	C(2)	O(1)	127.2(5)	C(3)	C(2)	O(1)	126.5(5)
O(3)	C(12)	O(2)	120.9(5)	C(11)	C(12)	O(2)	132.1(5)
C(12)	O(3)	C(8)	108.4(4)	C(7)	C(8)	O(3)	108.3(4)
C(9)	C(8)	O(3)	118.0(4)	C(11)	C(12)	O(3)	107.0(4)
C(5)	C(1)	C(2)	105.2(4)	C(10)	C(1)	C(2)	125.6(4)
C(3)	C(2)	C(1)	106.2(4)	C(10)	C(1)	C(5)	129.2(4)
C(4)	C(5)	C(1)	107.1(4)	C(6)	C(5)	C(1)	131.4(4)
C(9)	C(10)	C(1)	125.5(4)	C(14)	C(10)	C(1)	122.0(4)
C(4)	C(3)	C(2)	111.3(5)	C(5)	C(4)	C(3)	110.1(4)
C(13)	C(4)	C(3)	126.4(5)	C(13)	C(4)	C(5)	123.4(4)
C(6)	C(5)	C(4)	121.5(4)	C(7)	C(6)	C(5)	127.1(4)
C(8)	C(7)	C(6)	123.9(4)	C(11)	C(7)	C(6)	127.8(4)
C(11)	C(7)	C(8)	108.3(4)	C(9)	C(8)	C(7)	133.7(5)
C(12)	C(11)	C(7)	108.1(4)	C(15)	C(11)	C(7)	131.0(4)
C(10)	C(9)	C(8)	128.8(5)	C(14)	C(10)	C(9)	112.5(4)
C(15)	C(11)	C(12)	120.9(4)				

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 4.5

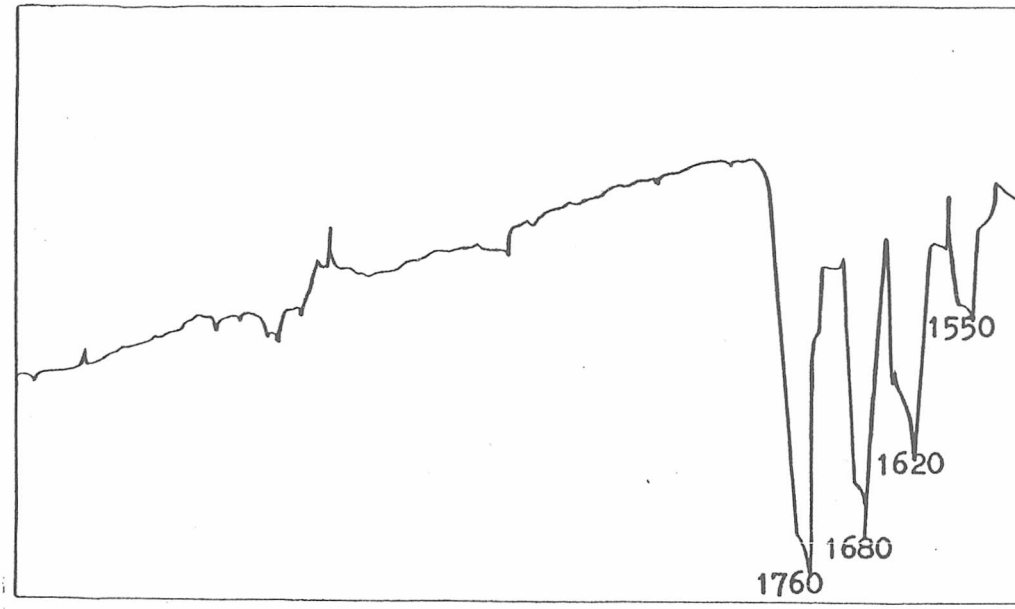
C(12) - O(3) - C(8) - C(7)	1.7(5)	C(12)
C(8) - O(3) - C(12) - O(2)	179.8(5)	C(8)
C(5) - C(1) - C(2) - O(1)	-174.8(5)	C(5)
C(10) - C(1) - C(2) - O(1)	7.3(9)	C(10)
C(2) - C(1) - C(5) - C(4)	-2.6(5)	C(2)
C(10) - C(1) - C(5) - C(4)	175.3(5)	C(10)
C(2) - C(1) - C(10) - C(9)	-179.1(5)	C(2)
C(5) - C(1) - C(10) - C(9)	3.5(8)	C(5)
O(1) - C(2) - C(3) - C(4)	175.2(5)	C(1)
C(2) - C(3) - C(4) - C(5)	1.3(6)	C(2)
C(3) - C(4) - C(5) - C(1)	0.9(5)	C(3)
C(13) - C(4) - C(5) - C(1)	-176.4(4)	C(13)
C(1) - C(5) - C(6) - C(7)	2.0(8)	C(4)
C(5) - C(6) - C(7) - C(8)	3.3(8)	C(5)
C(6) - C(7) - C(8) - O(3)	177.6(4)	C(6)
C(11) - C(7) - C(8) - O(3)	-1.3(5)	C(11)
C(6) - C(7) - C(11) - C(12)	-178.5(5)	C(6)
C(8) - C(7) - C(11) - C(12)	0.4(5)	C(8)
O(3) - C(8) - C(9) - C(10)	177.1(5)	C(7)
C(8) - C(9) - C(10) - C(1)	3.2(9)	C(8)
C(7) - C(11) - C(12) - O(2)	179.2(6)	C(7)
C(15) - C(11) - C(12) - O(2)	-1.9(9)	C(15)

Torsion Angles (°) (Non Hydrogen Atoms)

-	O(3)	- C(8)	- C(9)	-179.1(5)
-	O(3)	- C(12)	- C(11)	-1.5(5)
-	C(1)	- C(2)	- C(3)	3.2(5)
-	C(1)	- C(2)	- C(3)	-174.7(5)
-	C(1)	- C(5)	- C(6)	175.1(5)
-	C(1)	- C(5)	- C(6)	-7.0(9)
-	C(1)	- C(10)	- C(14)	1.6(8)
-	C(1)	- C(10)	- C(14)	-175.8(5)
-	C(2)	- C(3)	- C(4)	-2.9(6)
-	C(3)	- C(4)	- C(13)	178.5(5)
-	C(4)	- C(5)	- C(6)	-177.1(5)
-	C(4)	- C(5)	- C(6)	5.6(7)
-	C(5)	- C(6)	- C(7)	179.4(4)
-	C(6)	- C(7)	- C(11)	-178.0(5)
-	C(7)	- C(8)	- C(9)	-1.5(9)
-	C(7)	- C(8)	- C(9)	179.7(5)
-	C(7)	- C(11)	- C(15)	2.8(8)
-	C(7)	- C(11)	- C(15)	-178.3(5)
-	C(8)	- C(9)	- C(10)	-4.0(10)
-	C(9)	- C(10)	- C(14)	-177.4(5)
-	C(11)	- C(12)	- O(3)	0.7(5)
-	C(11)	- C(12)	- O(3)	179.5(4)

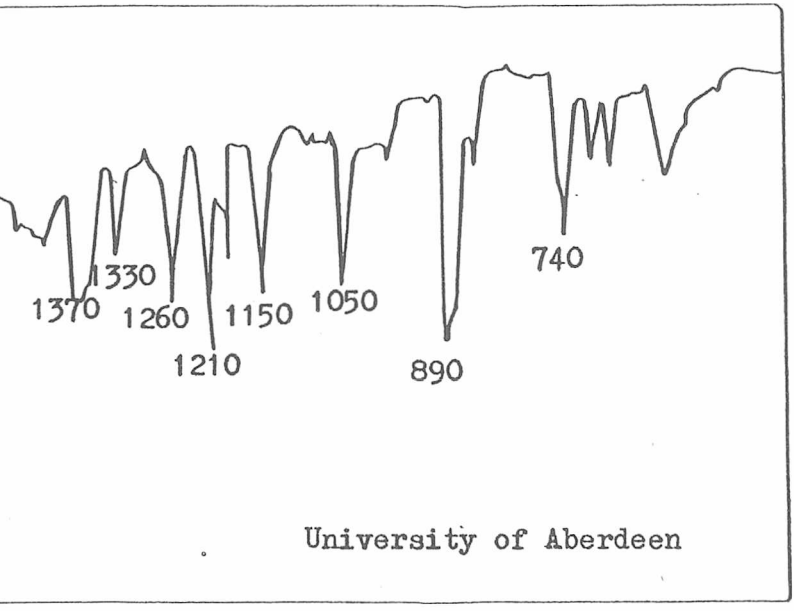
with e.s.d.'s

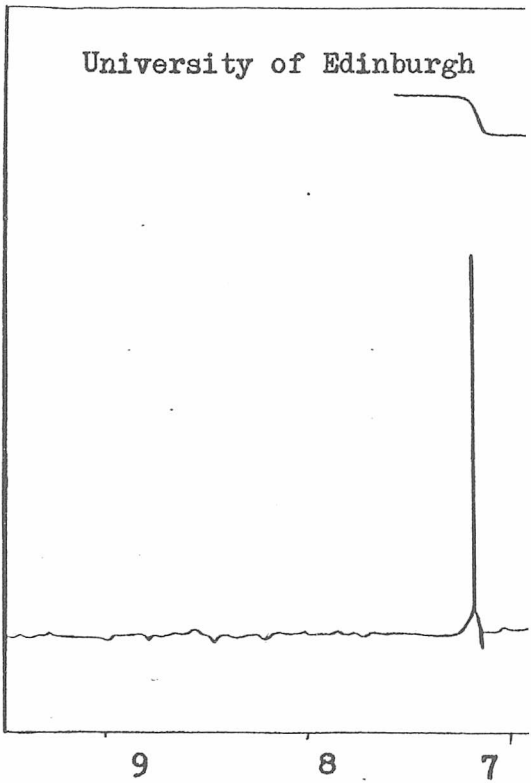
Infra Red Spectrum



Wave number cm^{-1}

FIG. 4.5


1370
1330
1260
1210
1150
1050
890
740
University of Aberdeen


University of Edinburgh
9
8
7

^{1}Hnmr Spectrum

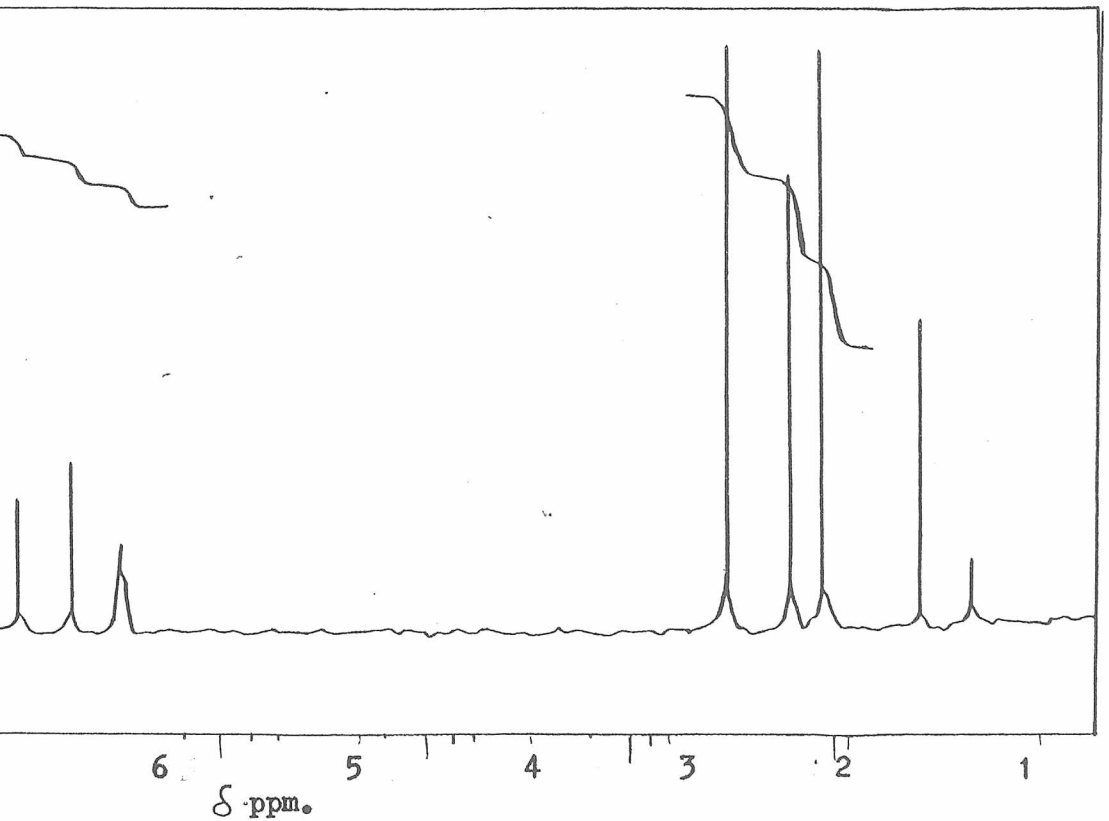


FIG. 4.6

TABLE 4.6

Major Assignments for the photo-oxidation product of Linderazulene

IR ν_{max} KBr	1760cm^{-1}	lactone
	1685cm^{-1}	- ketone
	1210cm^{-1}	- ether function
	1620cm^{-1} 1550cm^{-1}	c - c double bond
UV λ_{max}	208nm	conjugated c=c
Solvent CH_3OH	216nm	
^{1}Hnmr	δ 2.15	
Solvent $CDCl_3$	δ 2.35	methyl group protons
360MHz	δ 2.70	
	δ 6.25	
	δ 6.55	single protons
	δ 6.75	

positions, they were stable in the positions obtained from the difference map, and caused no further problems.

Structural 4.4.2

It was necessary to undertake an X-ray structure analysis of this substance, as the results from the spectral analysis were ambiguous. Although the spectra were consistent with the structure shown in Fig. 4.1, an alternative structure also fitted the spectral data, although it would have required skeletal rearrangement of the structure to produce this. Most of the evidence however suggested the structure shown in Fig. 4.1,as a very similar structure mallophyllidin [81] isolated from Fenila malacophylla produces spectral data which are similar to the structure under study. The X-ray analysis confirmed the structure.

The sesquiterpene lactone was found to be a very planar structure, ring A being planar to within 0.016Å, ring B to within 0.038Å, and ring C to within 0.077Å. The greatest deviation from planarity is in ring B, probably due to the greater flexibility within the seven membered ring. The γ lactone ring is more planar than the cyclopentone ring, probably due to the extra constraint of the cyclic oxygen. All the torsion angles are very close to 0° or 180°.

Many similar structures to this lactone have been studied, however the majority of these structures have a saturated or at least an unconjugated central ring system. This makes it difficult to compare them to the structure under study, as the central ring affects the conformation of the other two rings. Examples of such structures are Deacetylneotanulin [82], Hymenalone [83], 4-o-acetyl-2-o-p-iodobenzoylflorilenalin [84].

The sesquiterpene lactone is thought to be photoartefact of Linderazulene formed by exposing an ethanolic solution of Linderazulene to direct sunlight for six days. A proposed mechanism for this reaction is shown in Fig. 4.7. This is not yet proven, though from earlier evidence about the method of formation of the endoperoxide by attack on the furan ring [85], and the photosensitiser effects of azulene and linderazulene [86], it seems likely that this is the true mechanism. However a recent paper by Scheuer and Li suggests that the lactone is a natural product [87].

Molecular Mechanics 4.4.3

The steric energy of this sesquiterpene lactone is very low, suggesting that it is a very stable molecule. The crystal coordinates moved very little on minimisation, suggesting that the conformation of the molecule is very similar in the crystalline and free state.

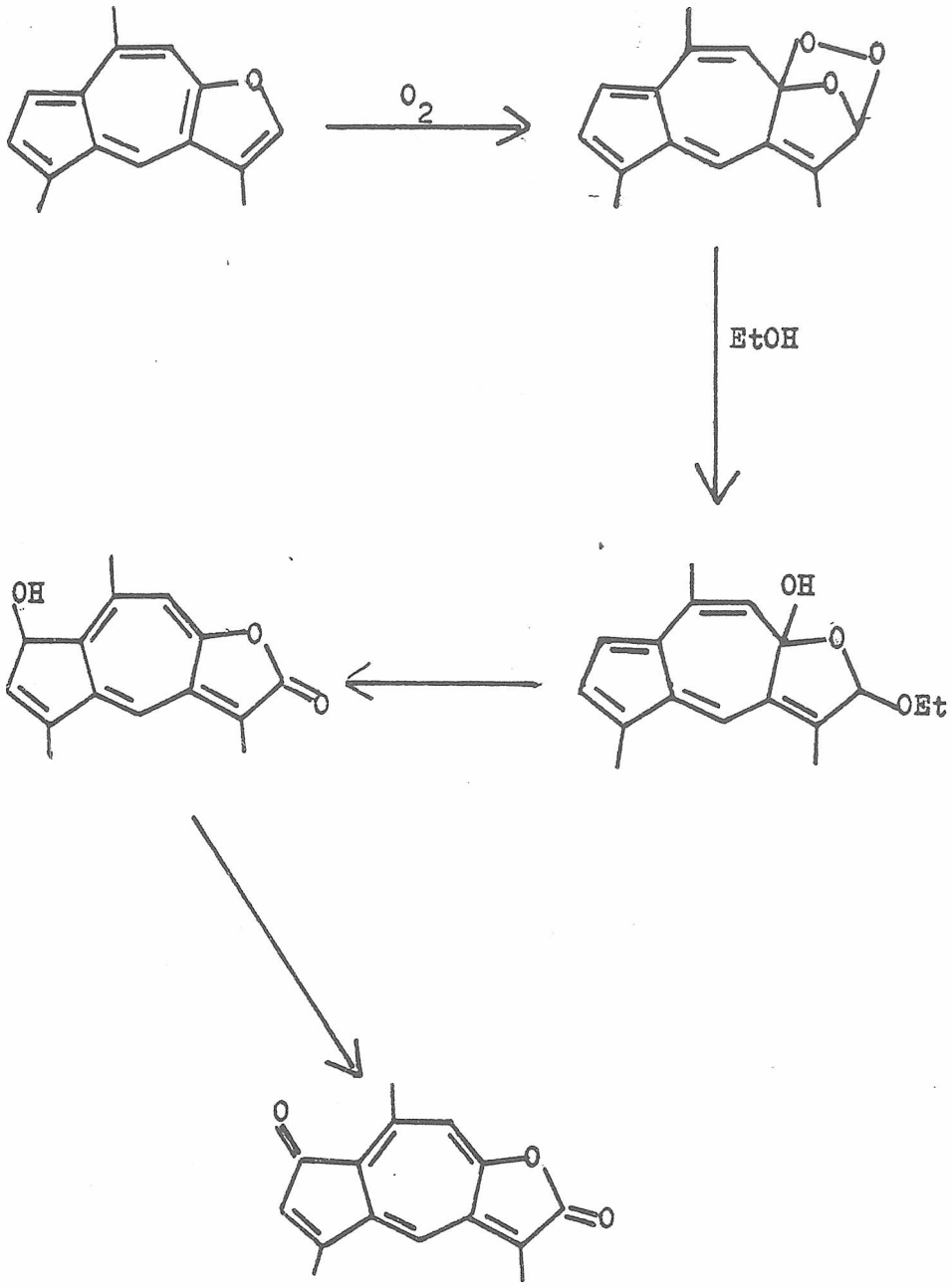


FIG. 4.7

CHAPTER FIVE

MICROCLADALLENE B. A BROMOALLENE FROM THE ALGA LAURENCIA MICROCLADIA

Microcladallene B, a Bromoallene from the alga Laurencia Microcladia

Introduction 5.1

Microcladallene B (Fig. 5.1) is a bromoallene obtained from the alga Laurencia microcladia. It is a crystalline substance the crystals being red and platey. It is one of three similar structures. Spectral analysis gave the structure as shown in Fig. 5.1.

It was necessary to undertake an X-ray analysis in order to confirm the structure and obtain the absolute configuration. The molecular formula had been determined by mass spectroscopy to be $C_{15}H_{18}Br_2O_2$.

Experimental 5.2

A crystal was chosen from a sample of poor quality. The data were collected as detailed in Chapter 2. The data were measured to $2\theta \leq 50^{\circ}$ for 1366 unique reflexions. The structure was solved using direct methods [71], the E map being computed for 176 reflexions, with $|E|_{min} > 1.4$. Difficulty was experienced at first with 'MULTAN' [72] where the reflexions in the starting set were not sufficient to fix the enantiomorph. The number of largest E's was increased from 150 to 300 to try and overcome this, but was unsuccessful. It was apparent that there were suitable reflexions for the starting set, but the programme was not choosing them. One starting set reflexion was selected and input. This produced an E map with the bromine atoms clearly visible. The coordinates of the bromine atoms were used in 'SHELX' [73] to produce a difference map with all non-hydrogen atoms clearly visible. The structure was refined by full matrix least squares [73]. Anisotropic temperature factors were introduced for the bromine atoms, and then for the remaining

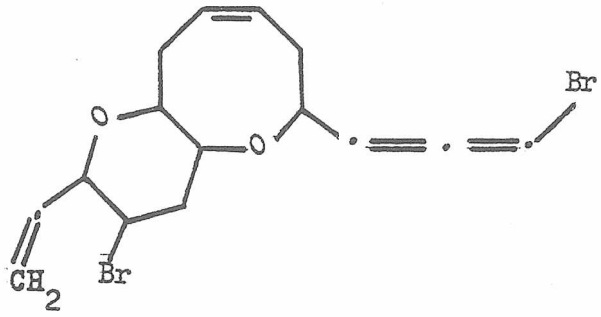


FIG. 5.1

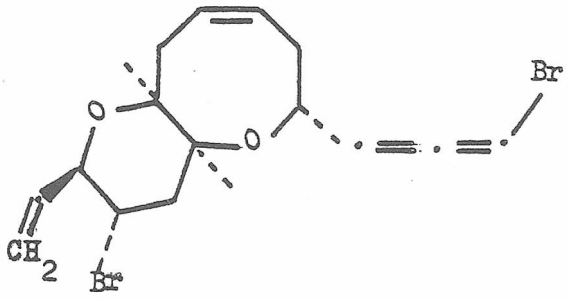


FIG. 5.2

non-hydrogen atoms. The hydrogen atom positions were calculated. The absolute configuration was determined.

The final weighting scheme used was

$$\omega = 1.4766/(\ ^{2}(F))$$

Molecular mechanics calculations were carried out using the programme MM2 [74].

Results 5.3

Crystallographic data 5.3.1

$C_{15}H_{18}O_2Br_2$, Mr = 390.114 Dc = 1.63gcm^{-3}, orthorhombic $P2_12_12_1$, Z = 4, a = 5.174(5)Å, b = 14.234(8)Å, c = 21.479(14)Å, U = 1581.9Å^3, μ(MoKα) = 50cm^{-1}, F(000) = 776.0, Final R. value = 7.24% for 832 unique reflexions ($I>3\sigma(I)$), ωR = 5.36%, maximum height on difference map = 0.734 eÅ^{-3} minimum height on difference map = -0.681 eÅ^{-3} maximum shift/error ratio = 0.121.

Structural Data 5.3.2

Microcladallene B was found to have the structure shown in Fig. 5.2. Its atomic arrangement is shown in Fig. 5.3 and a stereoscopic view of crystal packing is shown in Fig. 5.4.

The various structural parameters for Microcladallene B are listed in Tables 5.1 - 5.5.

Molecular mechanics calculations for Microcladallene B gave a final steric energy of 29.2645 KCal mol^{-1}.

Discussion 5.4

X-ray Analysis 5.4.1

Few problems were encountered in the analysis of Microcladallene B. Why it was not possible for the enantiomorph to be defined is not at all clear, as suitable reflexions were obviously present. Even increasing the number of reflexions available did not result in a suitable one being chosen. The reflexions that were suitable also

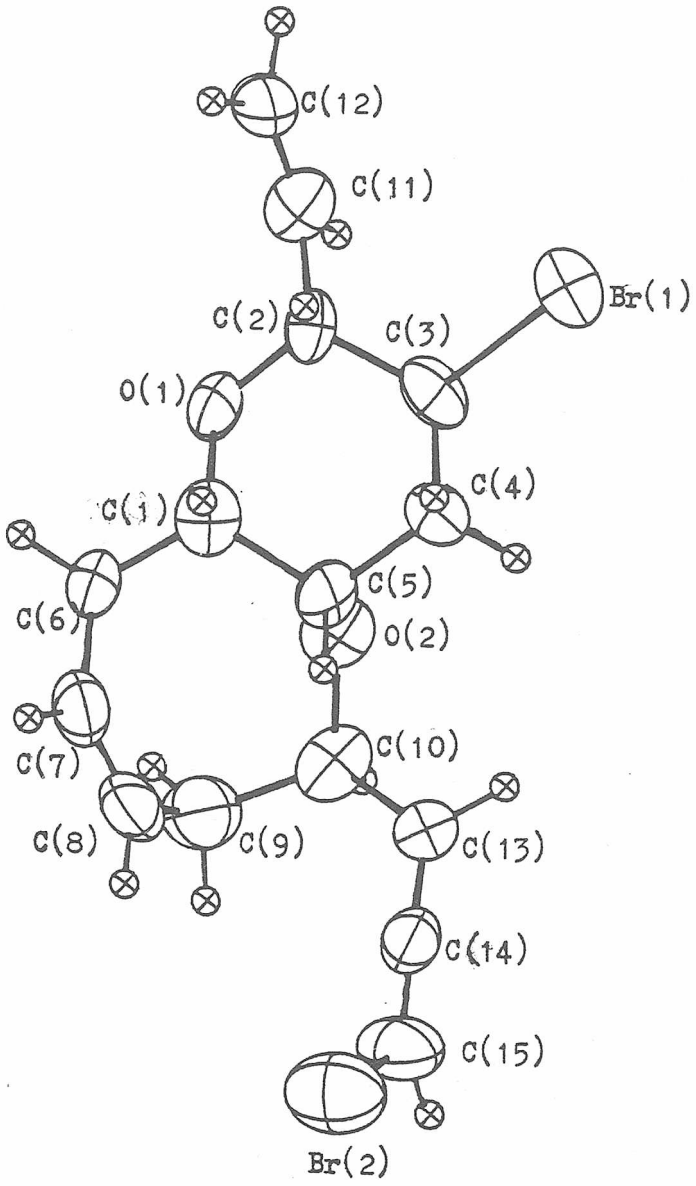
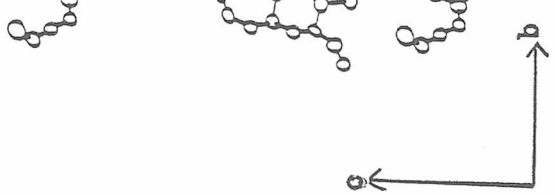


FIG. 5.3


b
a

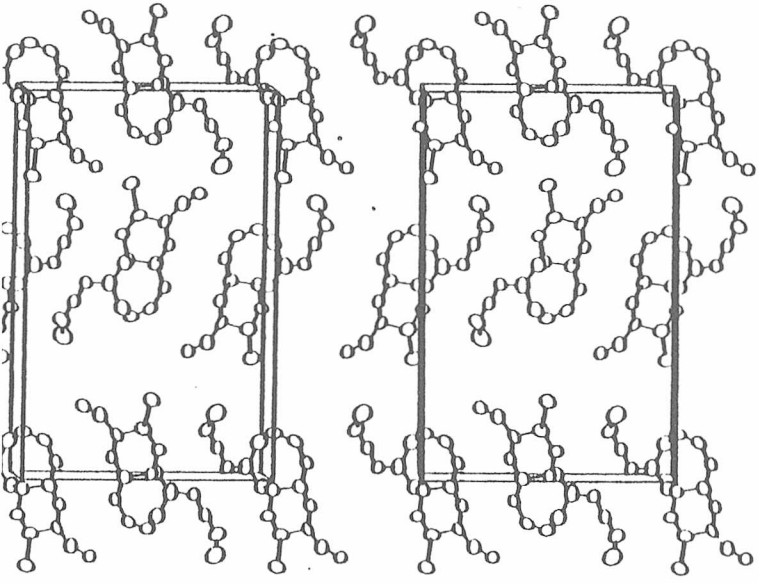


FIG. 5.4

Atom
BR(1)
BR(2)
O(1)
O(2)
C(1)
C(2)
C(3)
C(4)
C(5)
C(6)
C(7)
C(8)
C(9)
C(10)
C(11)
C(12)
C(13)
C(14)
C(15)
H(1)
H(2)
H(3)
H(4A)
H(4B)
H(5)
H(6A)
H(6B)
H(7)
H(8)
H(9A)
H(9B)
H(10)
H(11)
H(12A)
H(12B)
H(13)
H(15)

Fractional

TABLE 5.1

x	y	z
-0723(7)	5168(1)	7185(1)
-1856(5)	2597(2)	3227(1)
2075(31)	6598(7)	5660(5)
3614(31)	4750(9)	5226(5)
0876(40)	6116(12)	5162(7)
0898(51)	6455(10)	6246(7)
1155(46)	5409(11)	6397(6)
-0116(52)	4816(11)	5890(6)
1041(49)	5046(12)	5254(7)
2103(50)	6435(10)	4569(6)
1269(51)	5916(13)	4009(7)
2193(56)	5117(16)	3820(7)
4491(58)	4637(15)	4100(9)
4188(46)	4113(12)	4716(8)
2176(49)	7090(13)	6728(8)
1266(68)	7801(13)	6974(7)
2387(52)	3283(12)	4695(7)
1684(45)	2844(10)	4191(8)
1095(44)	2382(13)	3713(8)
-1010(40)	6267(12)	5145(7)
-0977(51)	6625(10)	6242(7)
3028(46)	5243(11)	6431(6)
-2013(52)	4950(11)	5884(6)
0171(52)	4135(11)	5983(6)
0061(49)	4715(12)	4918(7)
4017(50)	6361(10)	4611(6)
1674(50)	7114(10)	4508(6)
-0136(51)	6204(13)	3752(7)
1310(56)	4800(16)	3463(7)
5113(58)	4168(15)	3787(9)
5846(58)	5128(15)	4164(9)
5923(46)	3832(12)	4800(8)
3764(49)	6699(13)	6784(8)
-0314	8160	6766
1868	8112	7372
1675(52)	3051(12)	5100(7)
2286(44)	1865(13)	3583(8)

Atomic Coordinates ($x10^4$) with e.s.d.'s

TABLE 5.2

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
BR(1)	193(4)	82(1)	58(1)	-15(2)	22(2)	11(1)
BR(2)	92(2)	111(2)	94(1)	10(2)	-15(2)	-19(1)
O(1)	112(13)	38(6)	57(6)	-25(9)	-5(9)	7(5)
O(2)	37(11)	62(7)	69(7)	-8(10)	-16(8)	-3(6)
C(1)	22(15)	61(10)	63(9)	-14(12)	-15(11)	9(9)
C(2)	106(24)	39(9)	57(9)	10(14)	-4(14)	18(8)
C(3)	64(18)	79(13)	51(8)	-4(15)	38(11)	5(8)
C(4)	127(24)	59(9)	42(7)	-16(16)	8(12)	-1(8)
C(5)	52(19)	47(12)	57(9)	-29(14)	-9(12)	1(8)
C(6)	113(25)	39(9)	55(9)	-16(14)	-2(14)	8(7)
C(7)	103(24)	62(11)	48(8)	-24(17)	-15(14)	16(9)
C(8)	119(25)	75(14)	41(9)	-43(19)	10(13)	1(10)
C(9)	93(24)	75(15)	79(12)	-19(18)	25(16)	-10(11)
C(10)	29(16)	54(11)	92(13)	4(13)	6(13)	-2(10)
C(11)	100(22)	53(11)	77(12)	6(14)	11(16)	2(10)
C(12)	207(32)	53(10)	51(9)	35(18)	29(14)	3(8)
C(13)	111(26)	45(10)	51(9)	-4(15)	7(13)	-4(8)
C(14)	66(17)	40(9)	63(10)	1(13)	18(13)	4(8)
C(15)	105(21)	76(12)	61(9)	36(19)	-10(14)	-15(10)
H(1)	65(12)					
H(2)	65(12)					
H(3)	65(12)					
H(4A)	65(12)					
H(4B)	65(12)					
H(5)	65(12)					
H(6A)	65(12)					
H(6B)	65(12)					
H(7)	65(12)					
H(8)	65(12)					
H(9A)	65(12)					
H(9B)	65(12)					
H(10)	65(12)					
H(11)	65(12)					
H(12A.	65(12)					
H(12B)	65(12)					
H(13)	65(12)					
H(15)	65(12)					

Anisotropic Temperature Factor Coefficients: ($x10^3$) with e.s.d.'s

TABLE 5.3

BR(1) - C(3)	1.982(17)	C(3) - C(4)	1.527(24)
BR(2) - C(15)	1.875(21)	C(4) - C(5)	1.527(23)
O(1) - C(1)	1.414(20)	C(6) - C(7)	1.476(23)
O(1) - C(2)	1.413(21)	C(7) - C(8)	1.299(31)
O(2) - C(5)	1.398(29)	C(8) - C(9)	1.497(37)
O(2) - C(10)	1.453(21)	C(9) - C(10)	1.527(27)
C(1) - C(5)	1.538(24)	C(10) - C(13)	1.505(29)
C(1) - C(6)	1.494(23)	C(11) - C(12)	1.235(28)
C(2) - C(3)	1.530(21)	C(13) - C(14)	1.302(24)
C(2) - C(11)	1.525(26)	C(14) - C(15)	1.257(24)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 5.4

C(2) - C(3) - BR(1)	107.9(12)	C(4) - C(3) - BR(1)	107.6(14)
C(14) - C(15) - BR(2)	124.5(17)	C(2) - O(1) - C(1)	114.5(14)
C(5) - C(1) - O(1)	111.0(13)	C(6) - C(1) - O(1)	108.1(15)
C(3) - C(2) - O(1)	107.0(13)	C(11) - C(2) - O(1)	109.4(17)
C(10) - O(2) - C(5)	114.5(15)	C(1) - C(5) - O(2)	110.2(17)
C(4) - C(5) - O(2)	110.3(15)	C(9) - C(10) - O(2)	111.7(14)
C(13) - C(10) - O(2)	112.7(17)	C(6) - C(1) - C(5)	112.8(14)
C(4) - C(5) - C(1)	107.8(14)	C(7) - C(6) - C(1)	114.7(17)
C(11) - C(2) - C(3)	113.3(14)	C(4) - C(3) - C(2)	110.5(13)
C(12) - C(11) - C(2)	127.6(25)	C(5) - C(4) - C(3)	110.5(17)
C(8) - C(7) - C(6)	125.8(21)	C(9) - C(8) - C(7)	124.5(19)
C(10) - C(9) - C(8)	119.3(22)	C(13) - C(10) - C(9)	114.9(16)
C(14) - C(13) - C(10)	125.1(17)	C(15) - C(14) - C(13)	176.6(21)

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 5.5

C(2)	O(1)	C(1)	C(5)	-63.3(18)	C(2)	O(1)	C(1)	C(6)	172.5(14)
C(1)	O(1)	C(2)	C(3)	63.0(18)	C(1)	O(1)	C(2)	C(11)	-173.9(14)
C(10)	O(2)	C(5)	C(1)	-117.1(16)	C(10)	O(2)	C(5)	C(4)	124.0(16)
C(5)	O(2)	C(10)	C(9)	78.7(20)	C(5)	O(2)	C(10)	C(13)	-52.4(21)
O(1)	C(1)	C(5)	O(2)	-65.5(19)	O(1)	C(1)	C(5)	C(4)	55.0(19)
C(6)	C(1)	C(5)	O(2)	56.0(20)	C(6)	C(1)	C(5)	C(4)	176.5(15)
O(1)	C(1)	C(6)	C(7)	173.3(15)	C(5)	C(1)	C(6)	C(7)	50.2(22)
O(1)	C(2)	C(3)	BR(1)	-175.6(11)	O(1)	C(2)	C(3)	C(4)	-58.2(19)
C(11)	C(2)	C(3)	BR(1)	63.8(17)	C(11)	C(2)	C(3)	C(4)	-178.9(16)
O(1)	C(2)	C(11)	C(12)	107.5(25)	C(3)	C(2)	C(11)	C(12)	-133.3(24)
BR(1)	C(3)	C(4)	C(5)	173.8(12)	C(2)	C(3)	C(4)	C(5)	56.3(20)
C(3)	C(4)	C(5)	O(2)	67.6(20)	C(3)	C(4)	C(5)	C(1)	-52.8(20)
C(1)	C(6)	C(7)	C(8)	-83.1(27)	C(6)	C(7)	C(8)	C(9)	-7.5(37)
C(7)	C(8)	C(9)	C(10)	77.3(30)	C(8)	C(9)	C(10)	O(2)	-66.3(25)
C(8)	C(9)	C(10)	C(13)	63.6(26)	O(2)	C(10)	C(13)	C(14)	149.1(20)
C(9)	C(10)	C(13)	C(14)	19.6(30)	C(10)	C(13)	C(14)	C(15)	104.0(355)
C(13)	C(14)	C(15)	BR(2)	163.1(348)					

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

entered into phase relationships with other reflexions, therefore it can only be concluded that the algorithm used in 'MULTAN'[72] was inappropriate for this structure. Another problem encountered was that of defining the thermal motion of the non-hydrogen atoms. It was found initially when anisotropic temperature factors were introduced for these, that the thermal motion of some of the atoms could not be described correctly by anisotropic temperature factors, for different atoms at different times. When this occurred, the anisotropic temperature factors were removed and reintroduced successfully later in the refinement.

Eventually refinement proceeded with anisotropic temperature factors for all non-hydrogen atoms. This problem could have been due to the fact that no absorption corrections were carried out on the data. The absorption coefficient was relatively high and absorption could have been a significant factor. Atoms associated with non-positive definite statements are those designated to have negative thermal motion. This arises because absorption effects are more severe at low 2θ angles, so the general decrease in intensity with increased 2θ is to some extent cancelled out, and as this decrease is caused by thermal motion, the apparent thermal motion is artificially reduced [88]. A great deal of spurious electron density was present on the final difference map, and again this could have been as a result of a lack of absorption corrections.

Decomposition of the crystal occurred during data collection to the extent of 68%. This could explain the high esd.'s associated with the structural parameters. More satisfactory results could possibly be obtained by recollecting the data.

Structural 5.4.2

Both of the ring systems in Microcladallene B adopt minimum energy conformations [89,90]. This is shown more clearly in Table 5.6. As the stereochemistry at C(4) in Microcladallene B was determined, it was possible to determine the stereochemistry of the other members of the series, as they had very similar spectral data to Microcladallene B. These structures are shown in Fig. 5.7.

Molecular Mechanics 5.4.3

Molecular mechanics calculations also demonstrated that Microcladallene B adopts the minimum energy conformation. This is again shown in Table 5.6. The final steric energy is low and reflects this stability. Rotation of the allene side chain produced no significant change in the overall energy of the system, as at no point in the rotation did any atom come into close contact with another. Rotation of the allene side chain in no way affects the conformation of the two ring systems.

Biosynthesis 5.4.4

Characterisation of the full structure of Microcladallene B made it possible to apply Bu'Lock's theory [91] of the cyclisation of diepoxides to these substances and a theoretical biogenesis to be proposed. This is shown in Fig. 5.8. Thus the determination of the X-ray structure of Microcladallene B allowed knowledge of a whole group of substance to be expanded. It had been hoped to study the biogenetic pathway by molecular mechanics, but as can be seen, the precursors are aliphatic compounds and do not lend themselves readily to such a study.

TABLE 5.6

Six membered ring

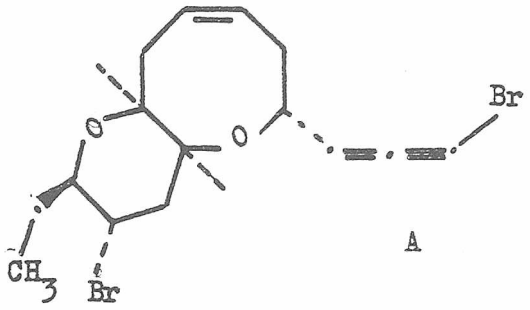
Torsion angle	X-ray	Molecular mechanics	Minimum energy
1234	63.0°	61.87°	55.2°
2345	-58.2°	-57.26°	-55.2°
3456	56.3°	53.82°	55.2°
4561	-52.8°	-52.05°	-55.2°
5612	55.0°	54.30°	55.2°
6123	63.3°	-60.76°	-55.2°

Chair conformation

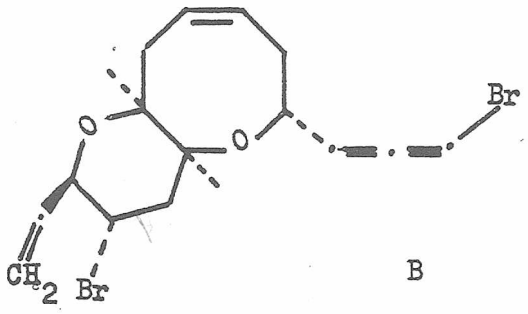
Eight membered ring

Torsion angle	X-ray	Molecular mechanics	Minimum energy
1234	57.0°	55.16°	-56.0°
2345	-117.1°	-119.75°	103.0°
3456	78.7°	82.06°	-71.0°
4567	-66.3°	-66.83°	73.0°
5678	77.3°	74.92°	-83.0°
6781	- 7.5°	- 1.48°	2.0°
7812	-83.1°	-89.02°	91.0°
8123	50.2°	49.49°	-49.0°

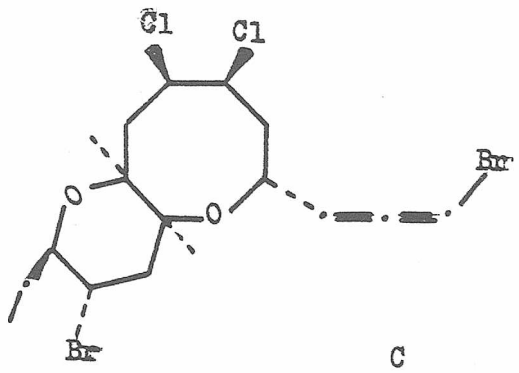
Twist boat chair conformation



A



B



C

FIG. 5.7

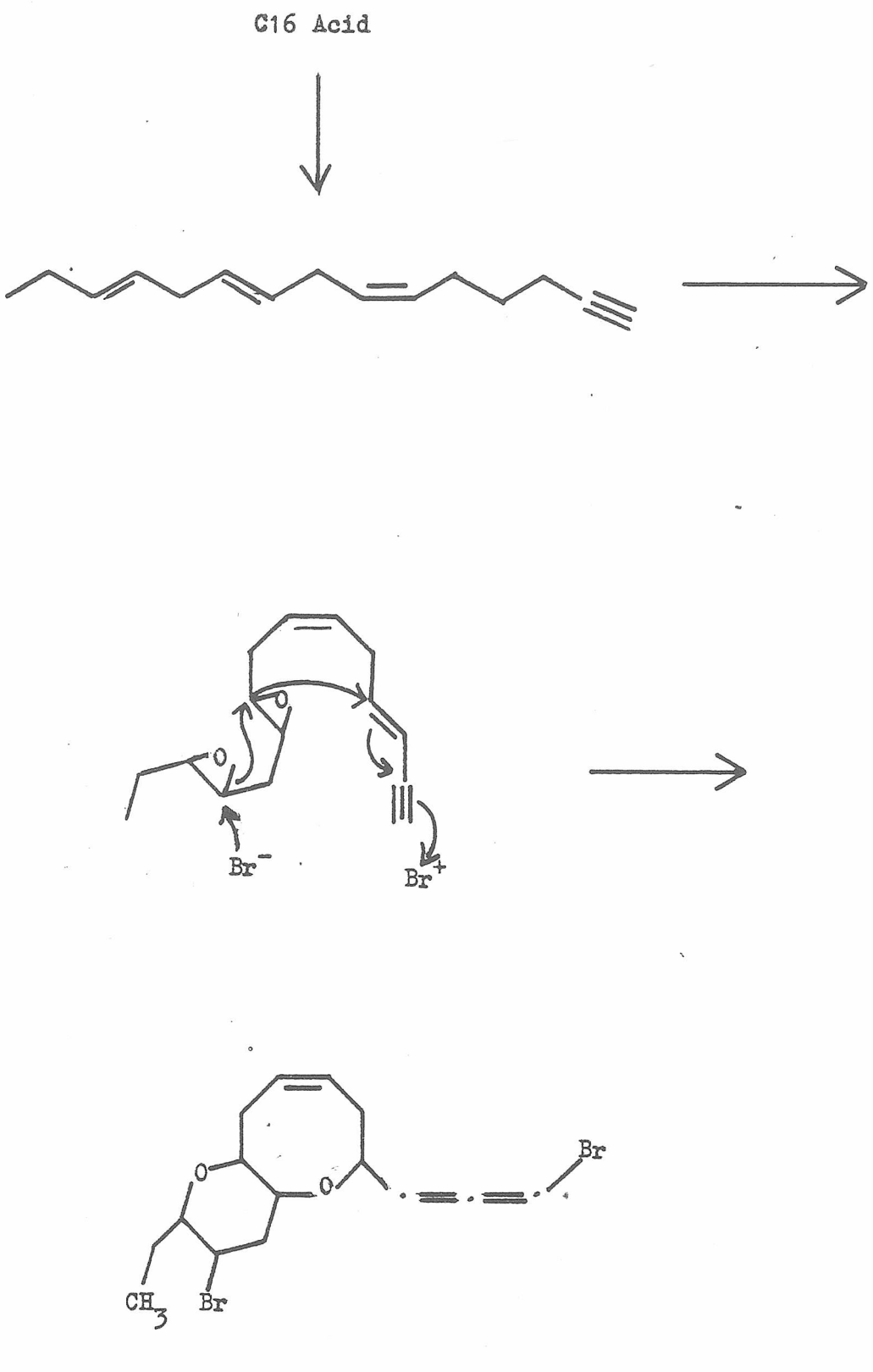


FIG. 5.8

CHAPTER SIX

NIRURINE A NOVEL ALKALOID FROM PHYLLANTHUS NIRURI

Nirurine a Novel Alkaloid from Phyllanthus niruri

Introduction 6.1

Nirurine (Fig 6.2) is a new type of alkaloid isolated from the aerial parts of Phyllanthus niruri L (Euphorbiaceae). It is closely related to, but quite distinct from, the securinega alkaloids.

Various spectral analyses were carred out on nirurine, and these suggested the structure as shown in Fig. 6.1. The molecular formula was determined by mass spectroscopy to be $C_{12}H_{13}NO_3$. There was however considerable ambiguity as to the actual structure, so an X-ray analysis was conducted to determine this unequivocally.

Experimental 6.2

A crystal of dimensions 2.75 x 0.6 x 0.8 x 1.56mm was chosen for the X-ray analysis. The data were collected according to the procedure in Chapter 2, for 1051 unique reflexions to $2\theta \leq 50^{\circ}$ Oscillation photographs were taken to confirm the presence of symmetry axes. The structure was solved by direct methods [71], the E map being computed for 164 reflexions with $|E| > 1.2$.

The structure was refined using full matrix least squares [74]. Anisotropic temperature factors were introduced for all non-hydrogen atoms, allowing the hydrogen atoms to be seen clearly from the difference map. The hydrogen atom positions were calculated and found to correspond to the observed positions. The hydrogen atom positions were allowed to refine, but their bond lengths were fixed at $1.00\text{Å} \pm 0.02\text{Å}$.

The final weighting scheme used was

$$\omega = 1.8144/(\sigma^2(F) + 0.0151F^2)$$

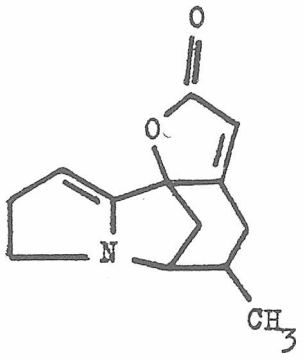


FIG. 6.1

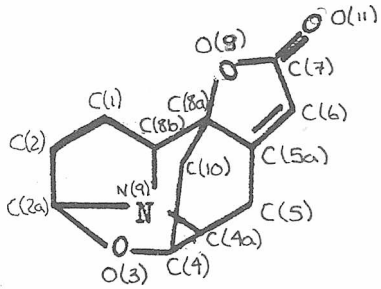


FIG. 6.2

Molecular Mechanics calculations were carried out using the programme MM2 [74]. To utilize this programme for nirurine certain parameters had to be developed, the changes made being shown in Table 6.1.

Results 6.3

Crystallographic Data 6.3.1

$C_{12}H_{13}NO_3$, Mr = 219.239, Dc = 1.41gcm^{-3}, orthorhobmic $P2_12_12_1$, Z = 4, a = 6.710(5)Å, b = 9.431(8)Å, c = 16.192(22)Å, U = 1024.7Å^3, μ(MoKα) = 0.62cm^{-1}, F(000) = 464.0, Final R value = 4.32% for 1051 unique reflexions, ωR = 4.29%, maximum height on difference map = 0.263 eÅ^{-3}, minimum height on difference map = -0.185 eÅ^{-3}. maximum shift/error ratio = 0.068.

Structural Data 6.3.2

Nirurine was found to have the structure as shown in Fig. 6.2. The atomic arrangement is shown in Fig. 6.3 and a stereoscopic view of crystal packing is shown in Fig. 6.4. The structural parameters are shown in Tables 6.2 - 6.6. The major spectral assignments are listed in Table 6.7.

Molecular mechanics calculations for nirurine gave a final steric energy of 29.2645 KCal mol^{-1}.

Discussion 6.4

X-ray Analysis 6.4.1

Few problems were encountered during the structure determination of nirurine. As the crystals were large and well shaped, a good data set was obtained, allowing the structure to be solved with ease. The esd's associated with the various structural parameters are very low, again indicative of the quality of the data. It was not found possible to determine the absolute configuration of

TABLE 6.1

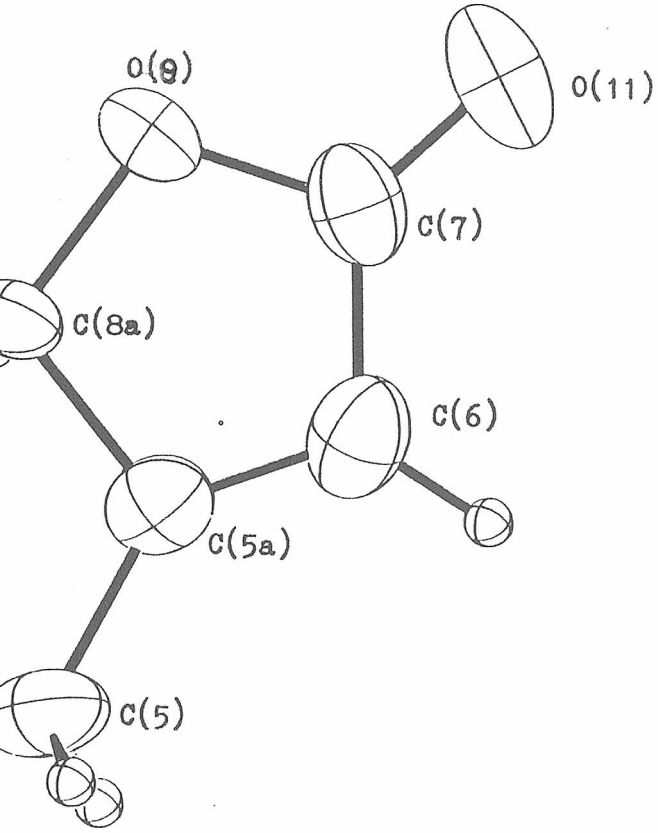
TORSION ANGLES

ATOM TYPES INVOLVED IN TORSION ANGLE	REDEFINED ATOM TYPES
Csp^2-Csp^2-Csp^2(carbonyl)-ether type O	Csp^2-Csp^3-Csp^2(carbonyl)-ether type O
Csp^2-Csp^3-Csp^2(carbonyl)-ether type O	Csp^3-Csp^3-Csp^2(carbonyl)-ether type O
Csp^2-Csp^2(carbonyl)-ether type O-Csp^3	Csp^3-Csp^2(carbonyl)-ether type O-Csp^3
ether type O-Csp^2(carbonly)-Csp^2-H	ether type O-Csp^2(carbonyl)-Csp^3 H
ether type O-Csp^3-Nsp^3-Csp^3	Nsp^3-Csp^3-Nsp^3-Csp^3
Csp^3-ether type O-Csp^3-Nsp^3	Csp^3-ether type O-Csp^3-ether type O

VALENCY ANGLES

ATOMS INVOLVED IN VALENCY ANGLE	REDEFINED ATOM TYPES	ANGLE (°)
Csp^2-Csp^2(carbonyl)-ether type O	Csp^3-Csp^2(carbonyl)-ether type O	111.0
ether type O-Csp^3-Nsp^3	ether type O-Csp3-ether type O	108.0

FIG. 6.3


O(9)
O(11)
C(7)
C(8a)
C(6)
C(5a)
C(5)

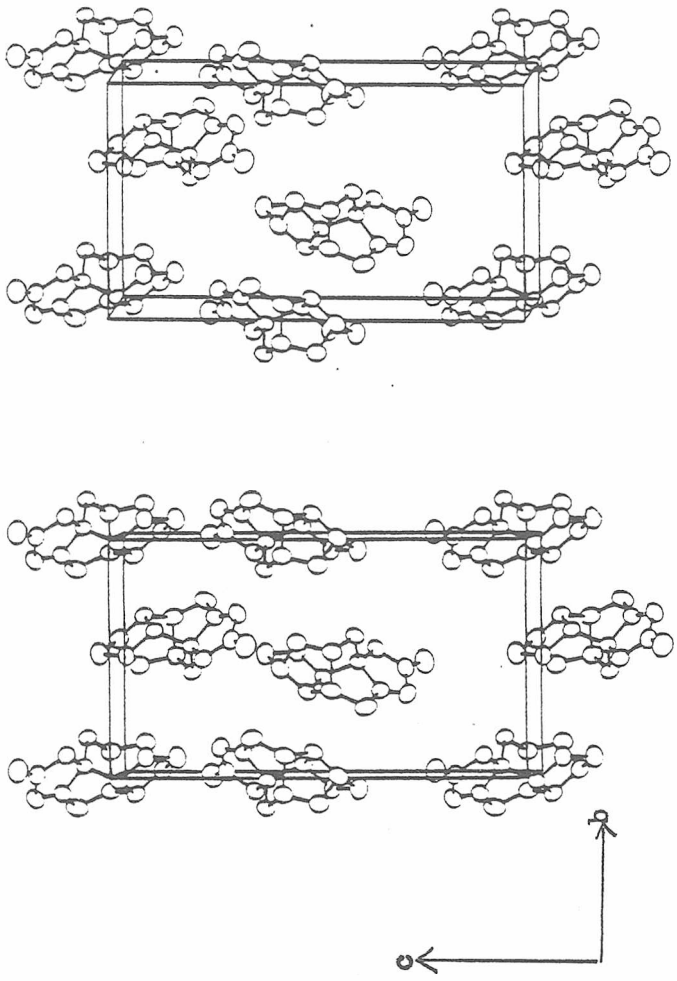


FIG. 6.4

Atom	Fractional
C(1)	
C(2)	
C(2A)	
O(3)	
C(4)	
C(4A)	
C(5)	
C(5A)	
C(6)	
C(7)	
O(8)	
C(8A)	
C(8B)	
N(9)	
C(10)	
O(11)	
H(11)	
H(12)	
H(21)	
H(22)	
H(2A)	
H(4)	
H(4A)	
H(51)	
H(52)	
H(6)	
H(8B)	
H(101)	
H(102)	

TABLE 6.2

x	y	z
9906(4)	0450(3)	5319(2)
11445(5)	0405(4)	6022(2)
13306(4)	-0167(3)	5604(2)
14357(3)	0954(2)	5165(1)
14463(4)	0558(3)	4313(2)
14230(4)	-1065(3)	4345(2)
13635(4)	-1776(3)	3536(2)
11710(4)	-1126(3)	3261(2)
10386(5)	-1312(3)	2660(2)
8795(4)	-0270(3)	2761(2)
9210(2)	0552(2)	3446(1)
11046(3)	0049(2)	3809(1)
10665(3)	-0595(3)	4667(2)
12644(3)	-1174(2)	4985(1)
12684(4)	1191(3)	3827(2)
7304(4)	-0042(3)	2359(1)
9785(55)	1408(23)	5054(18)
8607(34)	0162(33)	5565(19)
11074(52)	-0238(30)	6485(16)
11711(59)	1367(24)	6256(20)
14205(39)	-0596(28)	6029(14)
15741(31)	0882(28)	4068(16)
15435(38)	-1513(33)	4588(18)
13455(68)	-2818(21)	3596(23)
14650(48)	-1548(45)	3113(18)
10398(49)	-2032(26)	2219(15)
9773(40)	-1398(25)	4594(17)
12243(49)	2087(24)	4101(16)
13038(41)	1435(28)	3247(11)

Atomic Coordinates ($x10^4$) with e.s.d.'s

TABLE 6.3

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	35(1)	60(2)	43(1)	5(1)	3(1)	0(1)
C(2)	46(2)	76(2)	48(1)	-2(2)	-2(1)	-4(1)
C(2A)	35(1)	55(1)	53(1)	-2(1)	-9(1)	8(1)
O(3)	38(1)	49(1)	63(1)	-9(1)	-9(1)	1(1)
C(4)	25(1)	47(1)	61(1)	-5(1)	1(1)	4(1)
C(4A)	26(1)	42(1)	70(2)	6(1)	3(1)	6(1)
C(5)	39(1)	46(1)	78(2)	7(1)	12(2)	-10(1)
C(5A)	35(1)	43(1)	49(1)	-5(1)	11(1)	-2(1)
C(6)	51(2)	57(1)	48(1)	-13(1)	8(1)	-10(1)
C(7)	42(1)	59(2)	43(1)	-13(1)	-4(1)	7(1)
O(8)	30(1)	48(1)	49(1)	2(1)	-6(1)	5(1)
C(8A)	23(1)	34(1)	46(1)	2(1)	-1(1)	2(1)
C(8B)	23(1)	37(1)	48(1)	-1(1)	-2(1)	5(1)
N(9)	27(1)	41(1)	54(1)	1(1)	-4(1)	9(1)
C(10)	34(1)	38(1)	53(1)	-4(1)	1(1)	6(1)
O(11)	60(1)	92(2)	63(1)	-9(1)	-26(1)	8(1)
H(11)	57(9)					
H(12)	55(9)					
H(21)	59(9)					
H(22)	67(10)					
H(2A)	40(7)					
H(4)	41(7)					
H(4A)	56(9)					
H(51)	78(11)					
H(52)	80(12)					
H(6)	50(8)					
H(8B)	44(8)					
H(101)	52(8)					
H(102)	34(6)					

Anisotropic Temperature Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 6.4

C(1) - C(2)	1.538(4)	C(5) - C(5A)	1.497(4)
C(1) - C(8B)	1.531(4)	C(5A) - C(6)	1.329(4)
C(2) - C(2A)	1.519(4)	C(5A) - C(8A)	1.488(3)
C(2A) - O(3)	1.456(3)	C(6) - C(7)	1.460(4)
C(2A) - N(9)	1.450(4)	C(7) - O(8)	1.382(3)
O(3) - C(4)	1.431(4)	C(7) - O(11)	1.213(4)
C(4) - C(4A)	1.539(4)	O(8) - C(8A)	1.445(3)
C(4) - C(10)	1.549(4)	C(8A) - C(8B)	1.538(3)
C(4A) - C(5)	1.525(5)	C(8A) - C(10)	1.539(3)
C(4A) - N(9)	1.489(4)	C(8B) - N(9)	1.525(3)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 6.5

C(8B) - C(1) - C(2)	105.6(2)	C(2A) - C(2) - C(1)	103.4(2)
C(8A) - C(8B) - C(1)	115.1(2)	N(9) - C(8B) - C(1)	106.7(2)
O(3) - C(2A) - C(2)	111.0(2)	N(9) - C(2A) - C(2)	106.8(2)
N(9) - C(2A) - O(3)	106.6(2)	C(4) - O(3) - C(2A)	107.8(2)
C(4A) - N(9) - C(2A)	102.5(2)	C(8B) - N(9) - C(2A)	105.4(2)
C(4A) - C(4) - O(3)	102.8(2)	C(10) - C(4) - O(3)	110.5(2)
C(10) - C(4) - C(4A)	108.8(2)	C(5) - C(4A) - C(4)	115.8(2)
N(9) - C(4A) - C(4)	99.5(2)	C(8A) - C(10) - C(4)	106.9(2)
N(9) - C(4A) - C(5)	112.4(2)	C(5A) - C(5) - C(4A)	107.5(2)
C(8B) - N(9) - C(4A)	111.3(2)	C(6) - C(5A) - C(5)	137.7(3)
C(8A) - C(5A) - C(5)	112.7(2)	C(8A) - C(5A) - C(6)	109.6(2)
C(7) - C(6) - C(5A)	108.5(2)	O(8) - C(8A) - C(5A)	104.9(2)
C(8B) - C(8A) - C(5A)	107.1(2)	C(10) - C(8A) - C(5A)	108.6(2)
O(8) - C(7) - C(6)	108.7(2)	O(11) - C(7) - C(6)	131.5(3)
O(11) - C(7) - O(8)	119.8(3)	C(8A) - O(8) - C(7)	108.3(2)
C(8B) - C(8A) - O(8)	110.8(2)	C(10) - C(8A) - O(8)	112.8(2)
C(10) - C(8A) - C(8B)	112.2(2)	N(9) - C(8B) - C(8A)	107.6(2)

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 6.6

C(8B)	C(1)	C(2)	C(2A)	21.5(3)	C(2)	C(1)	C(8B)	C(8A)	-121.4(2)
C(2)	C(1)	C(8B)	N(9)	-2.2(3)	C(1)	C(2)	C(2A)	O(3)	81.2(3)
C(1)	C(2)	C(2A)	N(9)	-34.6(3)	C(2)	C(2A)	O(3)	C(4)	-120.7(2)
N(9)	C(2A)	O(3)	C(4)	-4.8(3)	C(2)	C(2A)	N(9)	C(4A)	150.1(2)
C(2)	C(2A)	N(9)	C(8B)	33.6(3)	O(3)	C(2A)	N(9)	C(4A)	31.4(3)
O(3)	C(2A)	N(9)	C(8B)	-85.1(2)	C(2A)	O(3)	C(4)	C(4A)	-22.9(3)
C(2A)	O(3)	C(4)	C(10)	93.1(2)	O(3)	C(4)	C(4A)	C(5)	161.7(2)
O(3)	C(4)	C(4A)	N(9)	41.1(2)	C(10)	C(4)	C(4A)	C(5)	44.4(3)
C(10)	C(4)	C(4A)	N(9)	-76.1(2)	O(3)	C(4)	C(10)	C(8A)	-94.1(2)
C(4A)	C(4)	C(10)	C(8A)	18.1(3)	C(4)	C(4A)	C(5)	C(5A)	-57.6(3)
N(9)	C(4A)	C(5)	C(5A)	55.8(3)	C(4)	C(4A)	N(9)	C(2A)	-43.8(2)
C(4)	C(4A)	N(9)	C(8B)	68.4(2)	C(5)	C(4A)	N(9)	C(2A)	-166.9(2)
C(5)	C(4A)	N(9)	C(8B)	-54.7(3)	C(4A)	C(5)	C(5A)	C(6)	-175.2(3)
C(4A)	C(5)	C(5A)	C(8A)	3.9(3)	C(5)	C(5A)	C(6)	C(7)	-180.0(3)
C(8A)	C(5A)	C(6)	C(7)	0.9(3)	C(5)	C(5A)	C(8A)	O(8)	179.2(2)
C(5)	C(5A)	C(8A)	C(8B)	-63.0(3)	C(5)	C(5A)	C(8A)	C(10)	58.4(3)
C(6)	C(5A)	C(8A)	O(8)	-1.5(3)	C(6)	C(5A)	C(8A)	C(8B)	116.3(2)
C(6)	C(5A)	C(8A)	C(10)	-122.3(2)	C(5A)	C(6)	C(7)	O(8)	0.0(3)
C(5A)	C(6)	C(7)	O(11)	179.4(3)	C(6)	C(7)	O(8)	C(8A)	-1.0(3)
O(11)	C(7)	O(8)	C(8A)	179.5(2)	C(7)	O(8)	C(8A)	C(5A)	1.5(2)
C(7)	O(8)	C(8A)	C(8B)	-113.8(2)	C(7)	O(8)	C(8A)	C(10)	119.4(2)
C(5A)	C(8A)	C(8B)	C(1)	-178.4(2)	C(5A)	C(8A)	C(8B)	N(9)	62.9(2)
O(8)	C(8A)	C(8B)	C(1)	-64.5(3)	O(8)	C(8A)	C(8B)	N(9)	176.8(2)
C(10)	C(8A)	C(8B)	C(1)	62.6(3)	C(10)	C(8A)	C(8B)	N(9)	-56.2(2)
C(5A)	C(8A)	C(10)	C(4)	-70.2(2)	O(8)	C(8A)	C(10)	C(4)	174.0(2)
C(8B)	C(8A)	C(10)	C(4)	48.0(3)	C(1)	C(8B)	N(9)	C(2A)	-19.2(3)
C(1)	C(8B)	N(9)	C(4A)	-127.6(2)	C(8A)	C(8B)	N(9)	C(2A)	104.8(2)
C(8A)	C(8B)	N(9)	C(4A)	-5.6(3)					

Torsion Angles (^{0}) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 6.7

Major Assignments for Nirurine

IR ν max	1755cm^{-1}	-	α,β unsaturated γ lactone
UV λ max	243nm	-	α,β unsaturated γ lactone
^{1}Hnmr Solvent $CDCl_3$ 360MHz	δ 1.625	(ddd)	proton of C(10) β
	δ 2.023	(m)	proton of C(2) α
	δ 2.076	(m)	proton of C(2) β
	δ 2.112	(m)	proton of C(1) α
	δ 2.200	(m)	proton of C(1) β
	δ 2.287	(d)	proton of C(10) α
	δ 2.936 δ 3.338	(dd) (dd)	methylene protons of C(5)
	δ 3.265	(m)	proton of C(8b)
	δ 3.542	(dd)	proton of C(4a)
	δ 4.220	(dd)	proton of C(4)
	δ 4.964	(d)	proton of C(2a)
	δ 5.740	(t)	proton of C(6)

The assignments shown above are the modified assignments, with knowledge of the true structure.

The spectra were run at the University of Illinois at Chicago

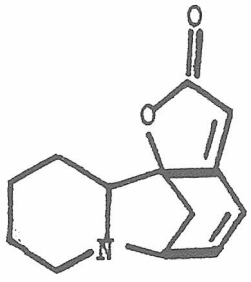
Nirurine as both configurations had the same R value.

Structural 6.4.2

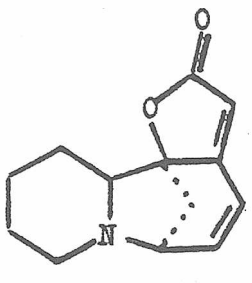
The structure first proposed for Nirurine from the spectral data was incorrect although it was consistent with the results of the spectral analysis. The X-ray analysis allowed the structure to be successfully elucidated and for the ^{1}Hnmr to be reassigned and expanded by selective radiation experiments.

Nirurine is a very rigid structure, and has several different ring systems. The two piperidine rings and the cyclohexane ring adopt the boat conformation, which although not the minimum energy conformation [89], is stable. The γ lactone ring is planar to within 0.01Å. The pyrrolidine ring adopts the envelope conformation with C(2a) at the flap, as does the oxazolidine ring with C(4a) at the flap. Again these are stable systems. The bridged perhydro-oxepine ring system adopts the chair conformation with the symmetry axis intersecting C(8a) and the mid-point of the O(3)-C(2a) bond. This is the minimum energy conformation [89]. The bridged perhydro-oxecine ring adopts a distorted twist-boat-chair conformation, the twist-boat-chair conformation being of minimum energy for this system [89]. Thus none of the ring systems present are distorted greatly from their minimum energy conformations, which serves to give a very stable molecule as reflected in its low steric energy.

An interesting feature of Nirurine is the particularly large angle at C(5)-C(5a)-C(6) of 137.7(3)°. This is obviously highly strained, but is shown to be common in this type of structure by comparison with two similar structures, Securinine [92], and Phyllochrysine [93,94] shown in Fig. 6.5. The valency angles for the structures are shown in Table 6.8. All these molecules possess



SECURININE



PHYLLOCHRYSINE

FIG. 6.5

TABLE 6.8

Angle	C(5) - C(5a) - C(6)	C(6) - C(5a) - C(8a)	C(5) - C(5a) - C(8a)
Nirurine	137.7°	109.6°	112.7°
Securinine	135.0°	110.0°	115.0°
Phyllochrysine	131.0° (137.0°)	110.0° (109.0°)	119.0° (112.0°)

a γ lactone function and a bridge system, and it is thought that this could be the cause of the strain. Although highly strained, the distortion produced by this angle is very small in nirurine, as the C(5a) atom in nirurine is only 0.0058Å out of the C(5), C(6), C(8a) plane causing little effect on the stability of the system.

Molecular mechanics 6.4.3

Molecular mechanics calculations carried out on nirurine showed a reduction in the C(5) - C(5a) - C(6) angle from 137.7° to 129.6°. It is found that a similar situation arises with phyllochrysine where the angle remains at 131.0° after minimisation. This again indicates the acceptability of such high angles.

From the molecular mechanics calculations carried out on phyllochrysine, it is possible to determine the conformations of the ring systems in the structure, and compare them with those in nirurine. The piperidine ring in phyllochrysine is a skew boat, the pyrrolidine ring an envelope with C(10) the out of plane atom. The cyclohexene ring adopts the half chair conformation and the γ lactone ring is planar. Obviously the change in ring sequence in nirurine affected the piperidine ring system as in phyllochrysine it adopts a lower energy form [95]. However, the other rings in the system behave similarly in both compounds.

Molecular mechanics calculations were carried out on phyllochrysine using the coordinates from the paper of Riche [93], and it is shown to have a steric energy of 25.87 KCal mol^{-1}, the coordinates moving little from their original positions. This energy is lower than that for nirurine. Nirurine is closely related to the norsecurinine alkaloids which are less stable [96,97]

than the securinine type alkaloids to which phyllochrysine belongs.

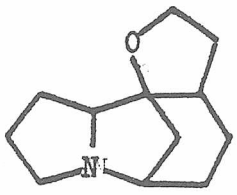
As stated previously certain parameters had to be redefined in order to carry out the molecular mechanics calculations of these structures. MM2 is not fully developed and some of the systems present in these structures are not available in the parameter lists. It could be argued that these changes make the results invalid, however it is justified in that these parameters produce bond lengths and valency angles in agreement with accepted values for lactones [98], as shown in Table 6.9.

Biosynthesis 6.4.4

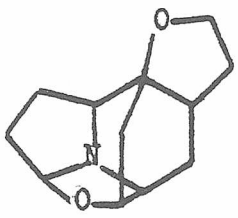
When nirurine was first isolated, it was thought to belong to a group of substances known as the securinega alkaloids, and in particular the norsecurinine alkaloids. However it is found to represent a new type of alkaloid closely resembling the securinega alkaloids. This similarity may be seen from Fig. 6.6. As this is a new type of structure, a biosynthetic pathway has not been documented, however a pathway has been formulated [99], although it is purely speculative and has still to be confirmed. In this pathway it is proposed that unlike the securinine alkaloids where the linkage of the amine is to tyrosine at the para position [100,101], it is to the 3 position of dopamine. This would explain the skeletal formation and the retention of an oxygen at C(4). The proposed biogenesis is shown in Fig. 6.7. Coordinates were obtained for each of these structures by building up a model using the programme 'MODEL' [76]. Molecular mechanics calculations were then carried out on each of the structures using the programme MM2 [74]. The results of these calculations are shown in Table 6.10. The energy calculated for step 5 is not a true energy, as N^+ had to be defined as an sp^3

TABLE 6.9

	COMPARISON OF BOND LENGTHS & VALENCY ANGLES WITH ACCEPTED VALUES		
TYPE	ACCEPTED	BEFORE MINIMIZATION	AFTER MINIMIZATION
1 - 1	1.3350Å	1.3289Å	1.3310Å
2 - 5	1.3340Å	1.3819Å	1.3584Å
2 - 4	1.210Å	1.2123Å	1.2074Å
3 - 5	1.470Å	1.4449Å	1.4125Å
1 - 2 - 5	115.60°	108.662°	109.905°
2 - 5 - 3	114.80°	108.286°	110.067°
4 - 2 - 5	120.00°	119.856°	123.010°
5 - 3 - 1	110.20°	104.930°	101.521°



NOR-SECURININE SKELETON



NIRURINE SKELETON

FIG. 6.6

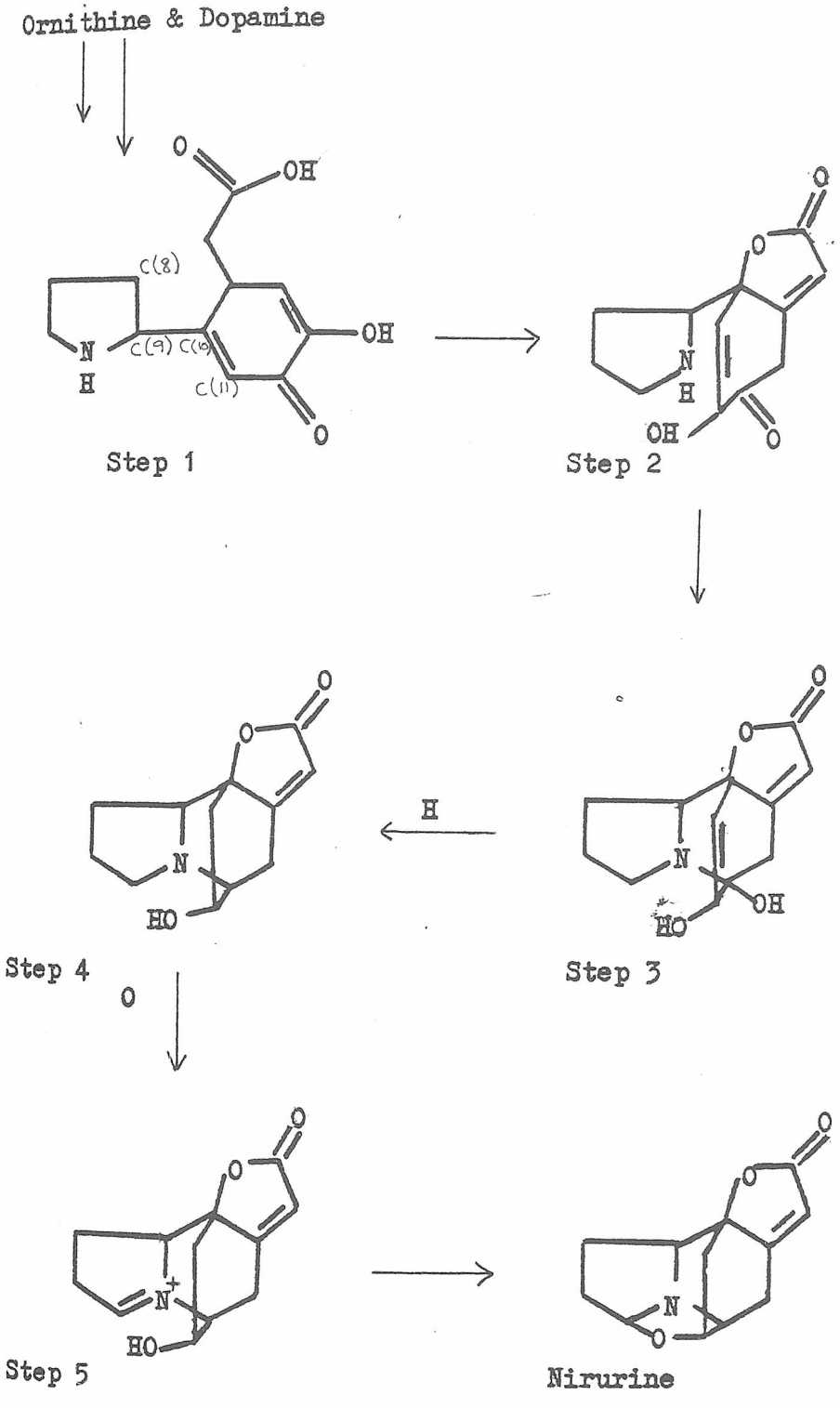

Ornithine & Dopamine
Step 1
Step 2
Step 3
Step 4
Step 5
Nirurine
H
O

FIG. 6.7

TABLE 6.10

STEP (FIG. 6.7)	STERIC ENERGY KCal mol^{-1}.
1	24.350
2	25.200
3	43.365
4	41.983
5	37.991

carbon, as MM2 has no facility for accepting 'whole' charges on an atom. The energy is lower than would be expected for such a transition molecule. The rest of the structures are stable, the first two having lower steric energies than nirurine.

Structures one and two were studied further by varying the C(8) - C(9) - C(10) - C(11) torsion angle and observing the change in energy produced. The energy profiles are shown in Fig. 6.8 and 6.9. The energy minima in structure 1 are not as well defined as those for structure 2. With structure 2 there are three definite minima, the global minimum occurring at a torsion angle of 60° and the local minima at -60° and -175°. With structure 1 the global minimum occurs at a torsion angle of 76° and the local minima at 160° and -68°. Perspective drawings for the minima are shown in Fig. 6.10 for structure 1 and Fig 6.11 for structure 2.

Perspective drawings for the structures of the biogenetic pathway of nirurine are shown in Fig. 6.12, and demonstrate more clearly the proposed formation of the alkaloid.

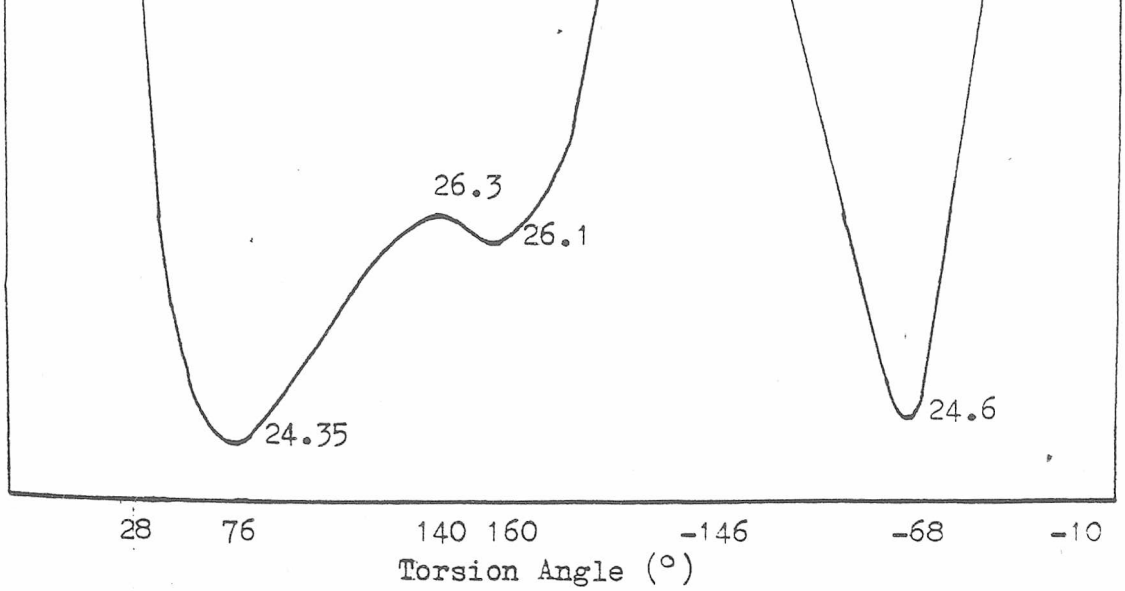
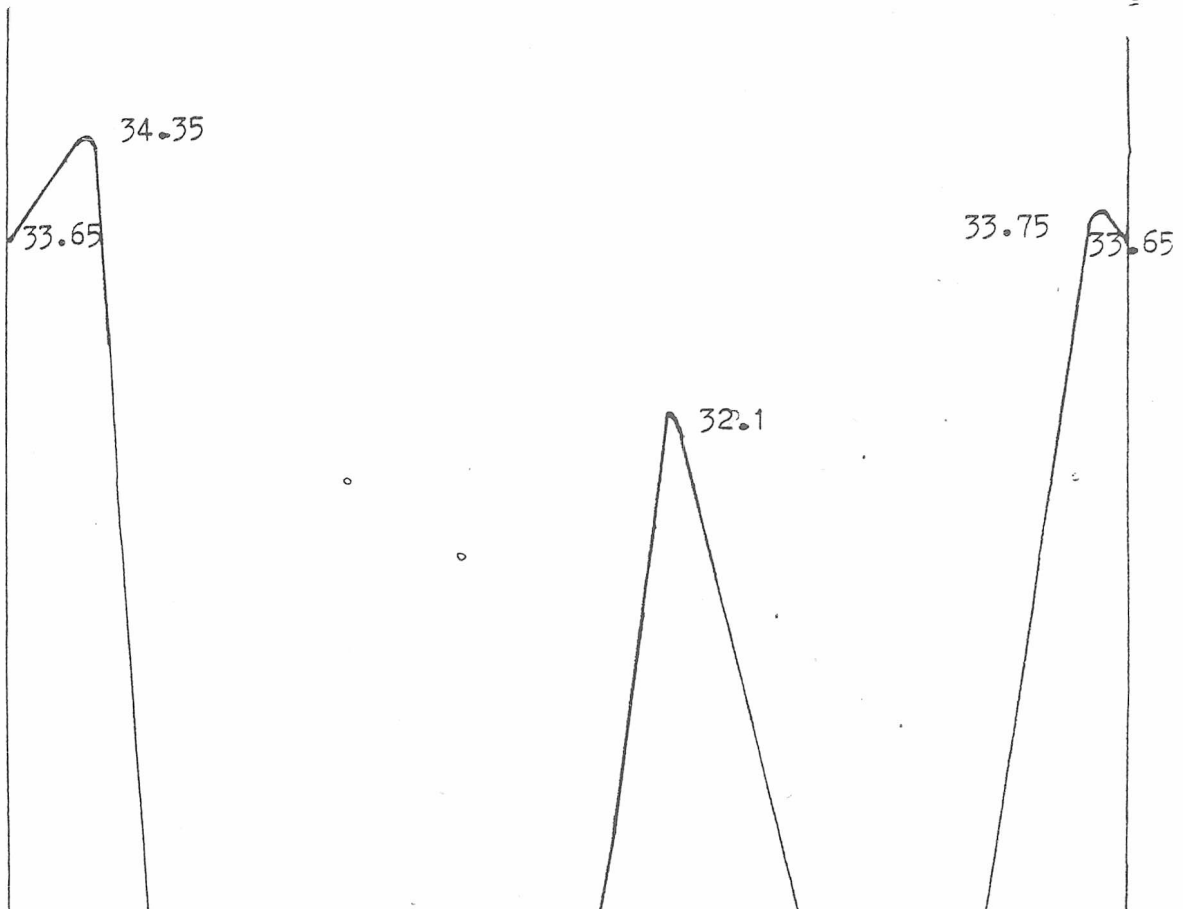


FIG. 6.8


34.35
33.65
32.1
33.75
33.65
Energy KCal mol^{-1}

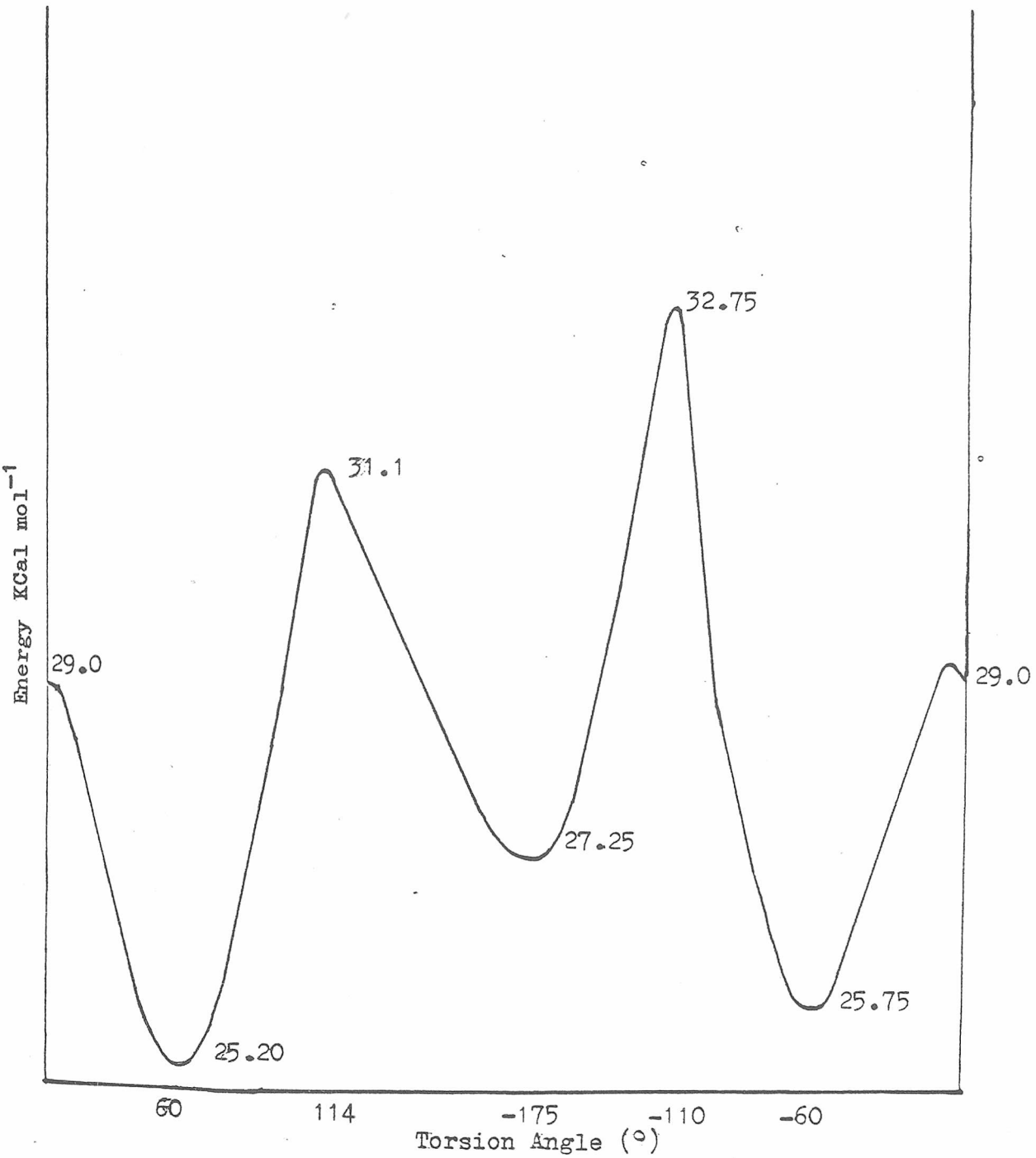
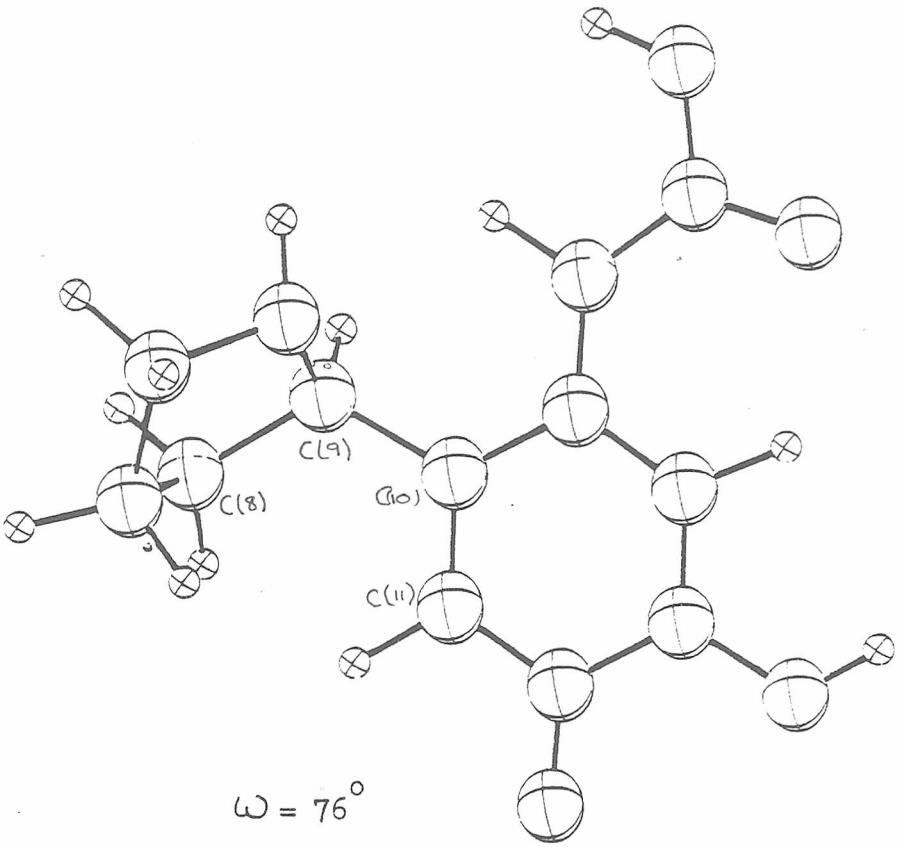
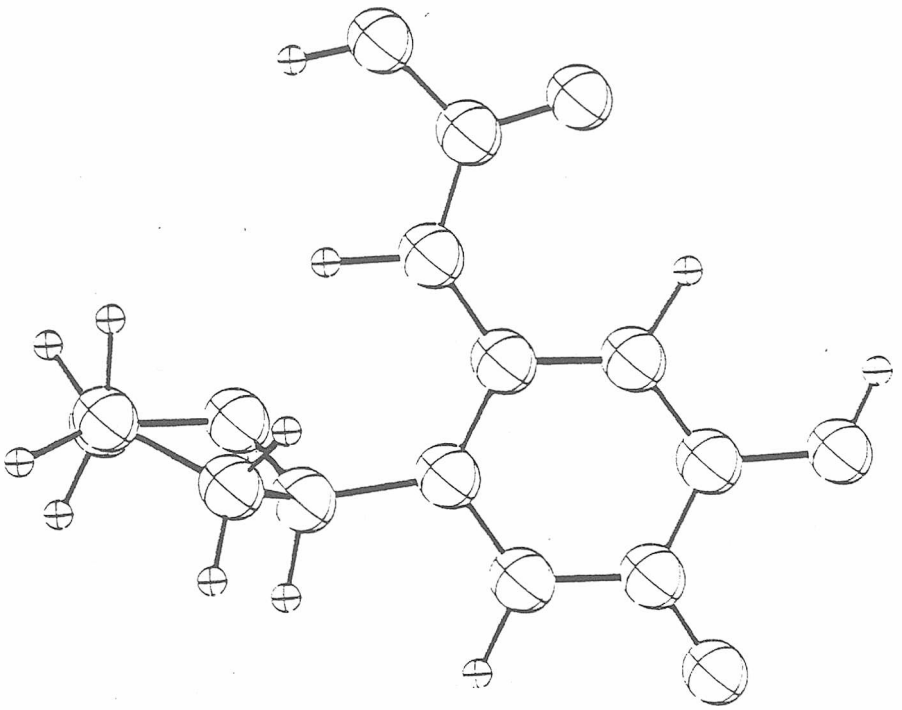

Energy KCal mol⁻¹
29.0
25.20
31.1
27.25
32.75
25.75
29.0
60
114
-175
-110
-60
Torsion Angle (°)

FIG. 6.9

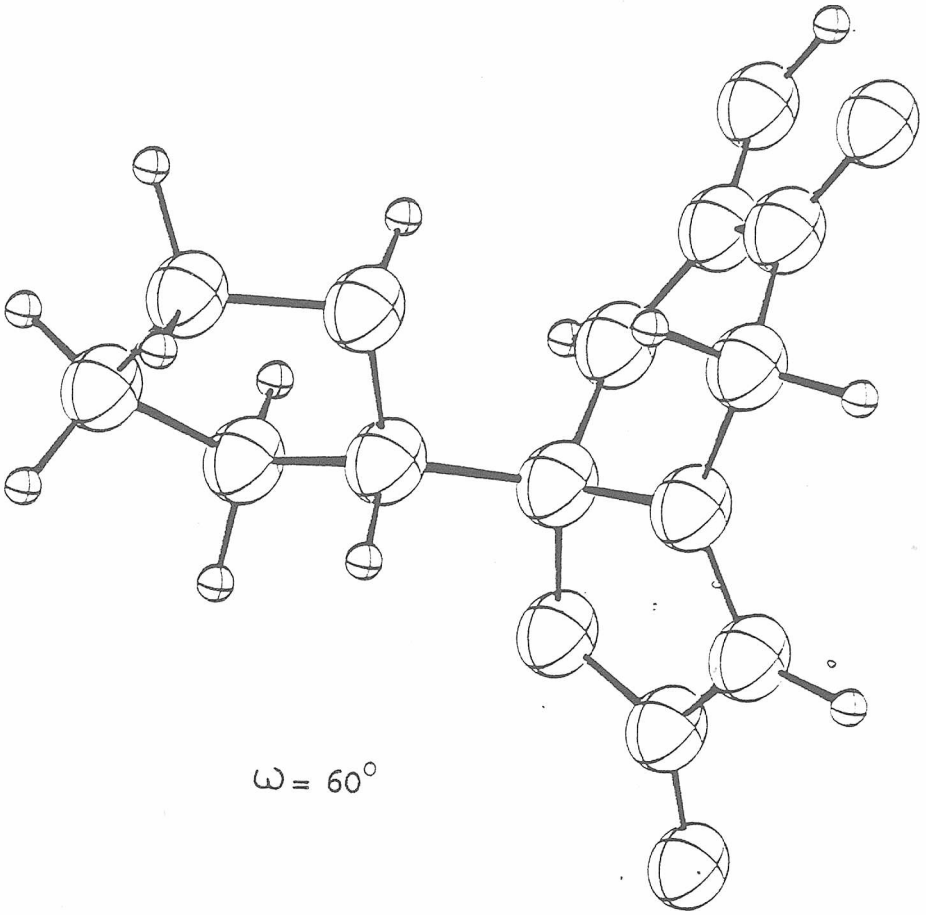


$\omega = 76^{\circ}$

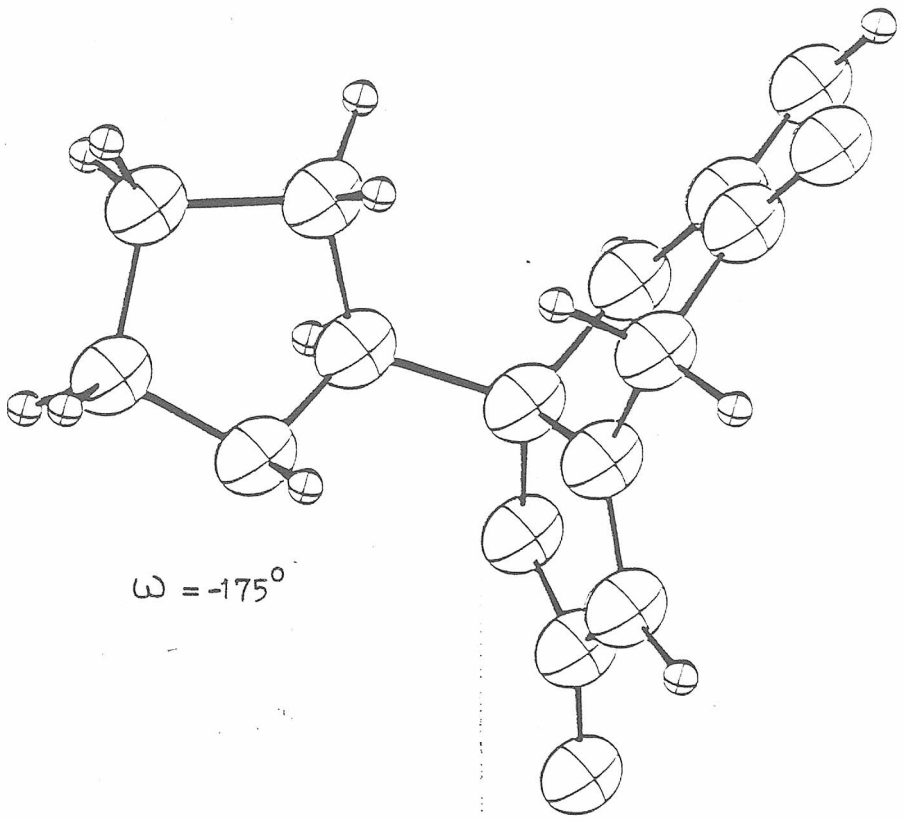


$\omega = -68^{\circ}$

FIG. 6.10

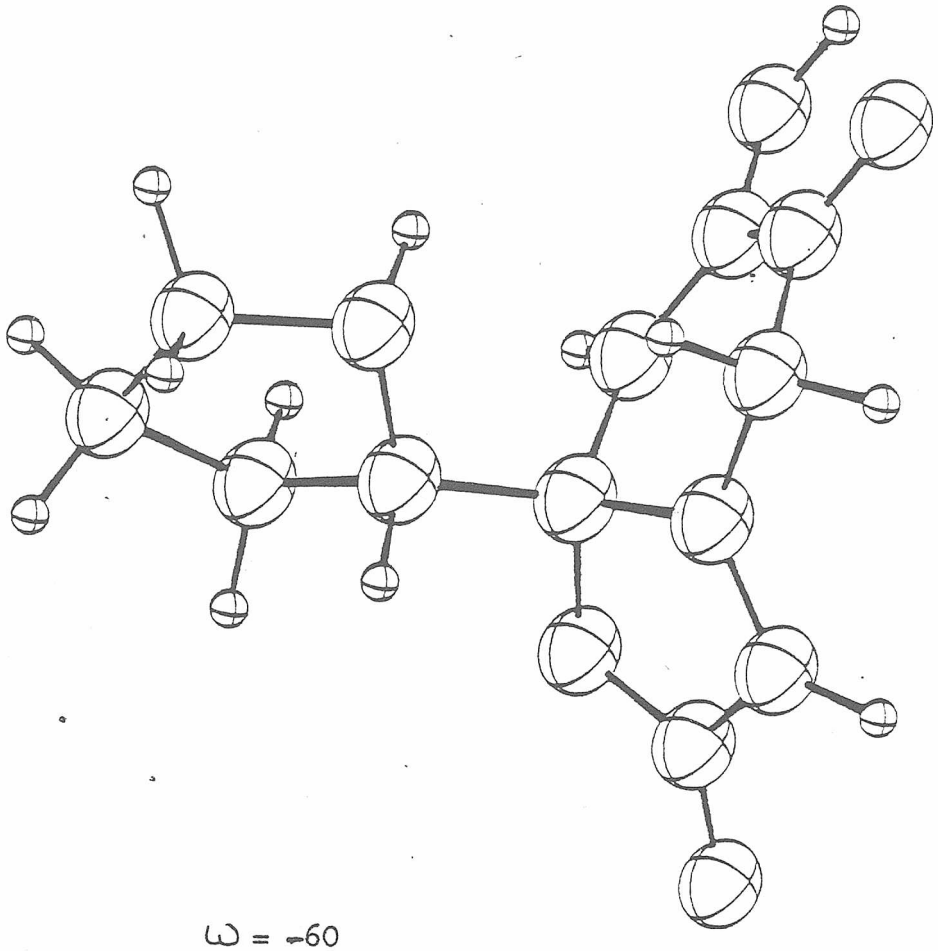


$\omega = 60^{\circ}$



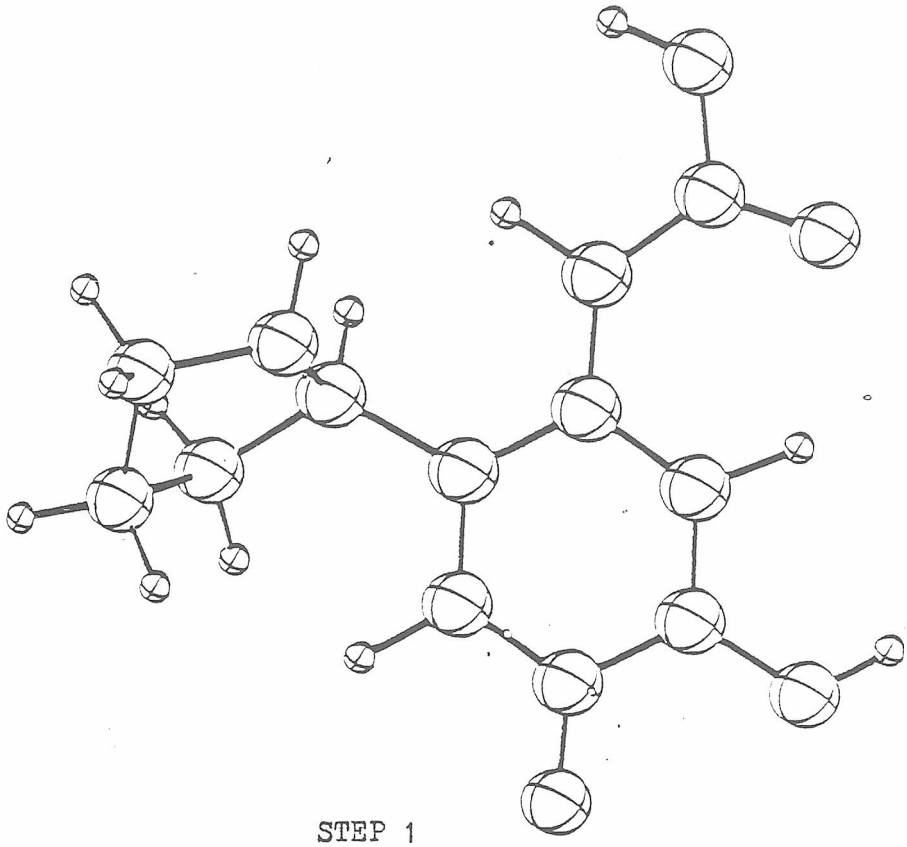
$\omega = -175^{\circ}$

FIG. 6.1.1

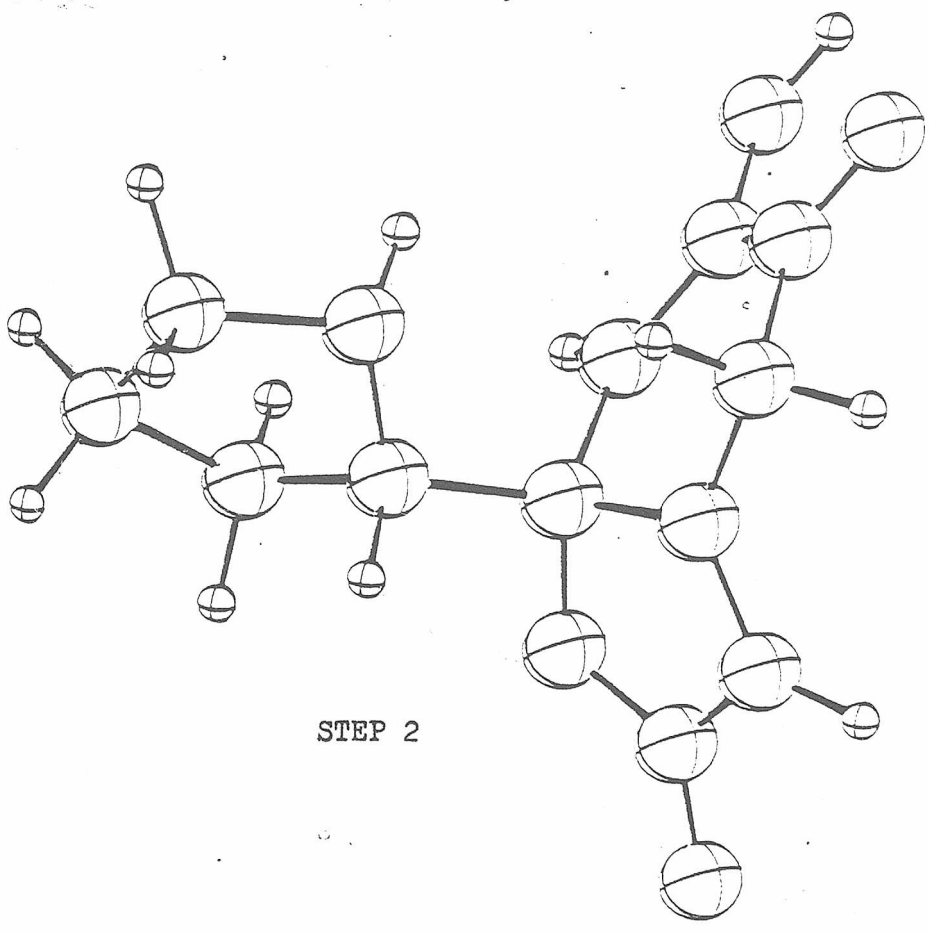


$\omega = -60$

FIG. 6.11 (cont.)

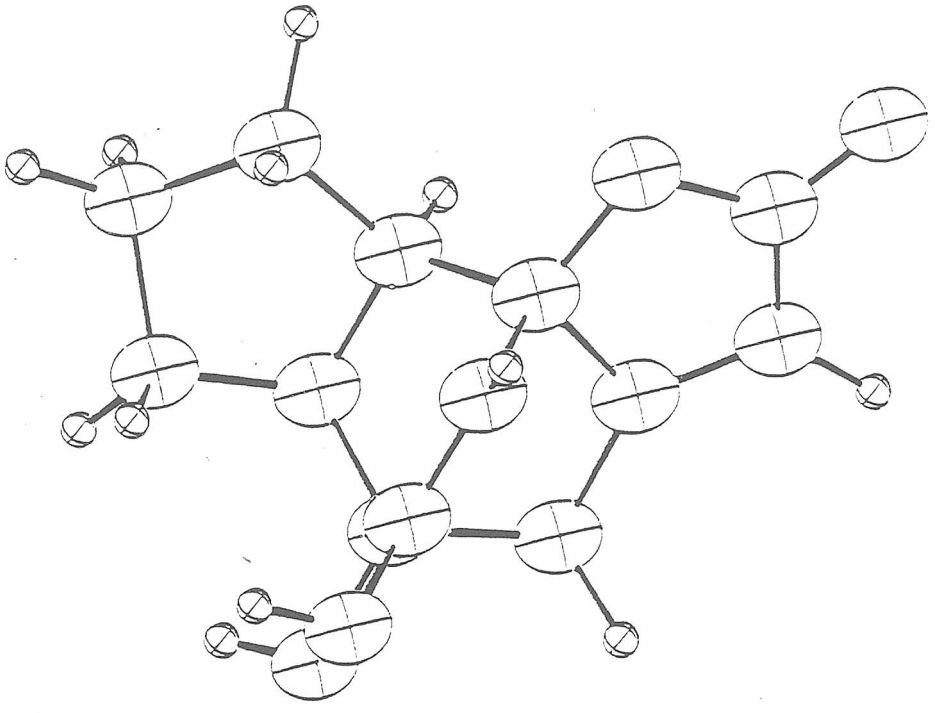


STEP 1

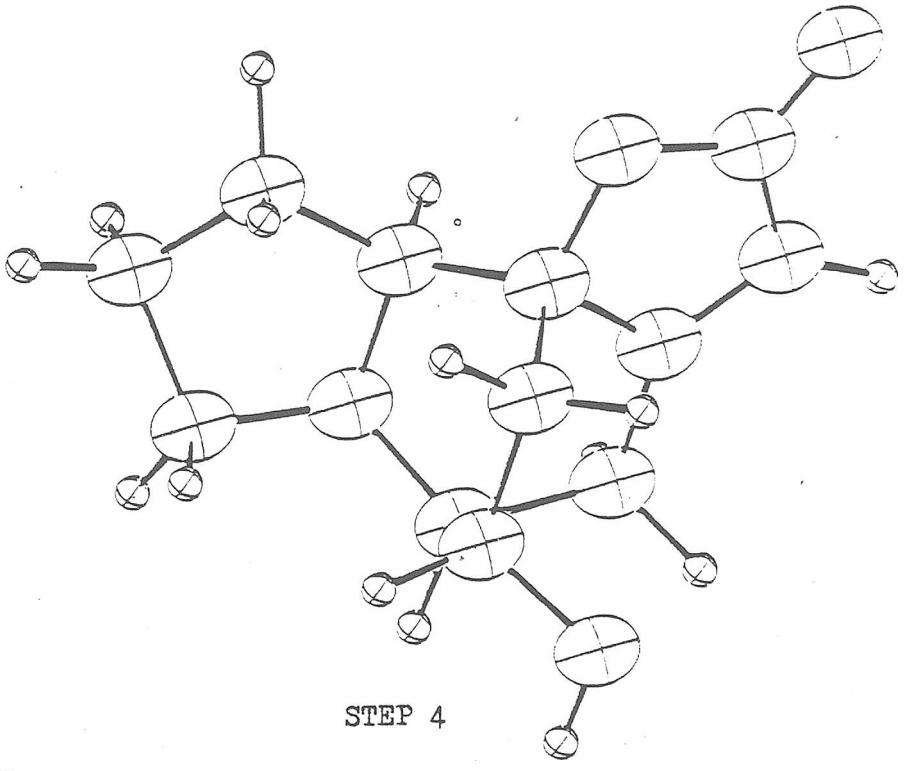


STEP 2

FIG. 6.12

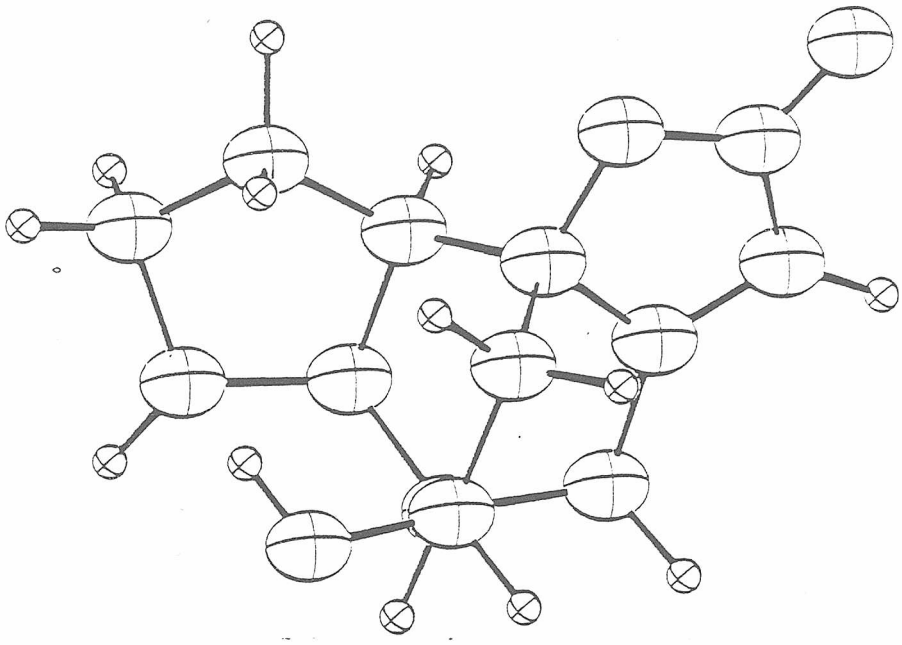


STEP 3

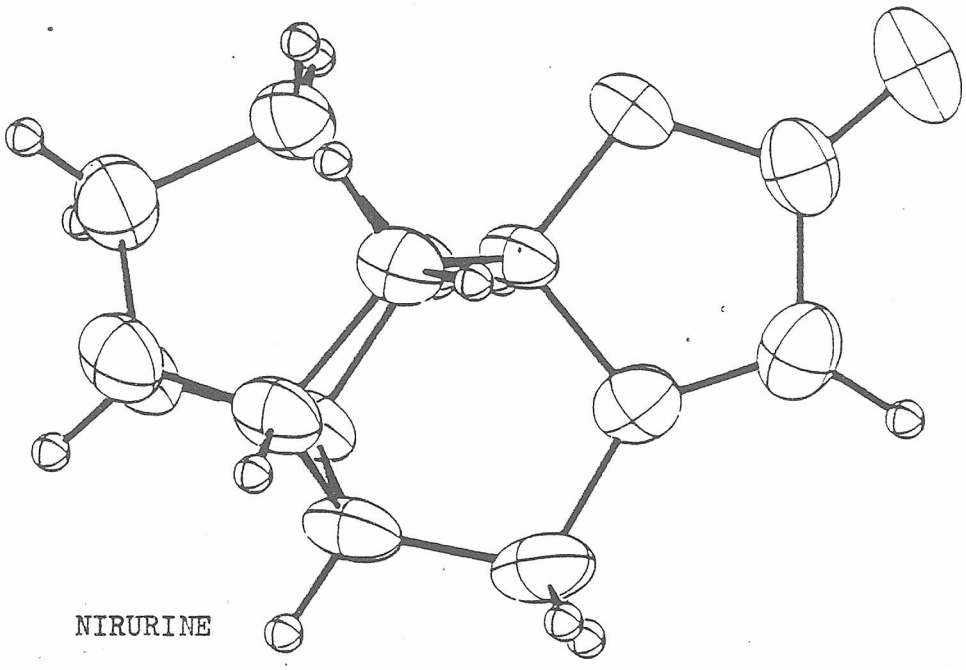


STEP 4

FIG. 6.12 (cont.)



STEP 5



NIRURINE

FIG. 6.12 (cont.)

CHAPTER SEVEN

VENTILONE C, AN ISOFURANONAPHTHOQUINONE FROM VENTILAGO MADERASPATANA

Ventilone C, an Isofuranonaphthoquinone from Ventilago maderaspatana

Introduction 7.1

Ventilone C (Fig. 7.3) is one of five isonaphthoquinones which have been isolated from the root bark of ventilago maderaspatana (Rhamnaceae). It is a red crystalline substance.

The skeleton of the structure was determined by spectral analysis to be that shown in Fig. 7.1, but there were uncertainties about the positions of the methoxy/hydroxy groups, and also about the stereochemistry of the system. The molecular formula had been previously determined by mass spectroscopy to be C15H1207.

Experimental 7.2

The crystals of ventilone C were platey; the one chosen for analysis being 0.1mm x 0.175mm x 0.65mm. The method outlined in Chapter 2 was used for data collection with $2\theta \leq 50^{\circ}$. 1164 reflexions were measured.

The structure was solved by direct methods using 'MULTAN 80' [72], however problems were encountered during the analysis. The E values exhibited a hypercentric distribution. In the first instance, a K curve was implemented instead of a Wilson plot ('MULTAN 78' was being used at this point). The probability for the acceptance of the Σ_1 relationships was increased from 0.95 to 2.00. Both of these attempts were unsuccessful, so the number of reflexions used in the computation of the E map was increased. These adjustments were also unsuccessful. A model of the structure was then built up using the programme 'MODEL' [76], and the coordinates of this input to 'MULTAN' [72] as a randomly oriented group. Although this produced a slight shift away from the hypercentric distribution of E values, no solution was obtained. Four reflexions were omitted from the

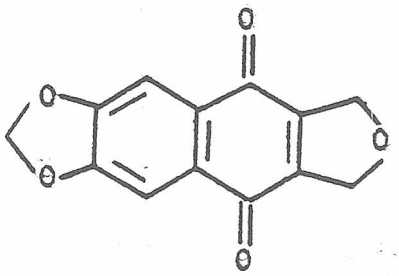


FIG. 7.1

data set, as they appeared to have very large E values. All phase relationships were used by setting Kmin to 0. This did not progress the analysis. The statistically weighted tangent formula was implemented, but nothing sensible was obtained from the E maps. (Five E maps were printed each time). The temperature factor was increased from the calculated value of 3.5 to 5.00, and a tricyclic fragment was clearly visible from the E map. The coordinates of this were entered into 'MULTAN'[72] as an oriented group. This produced an E map with the skeleton of the structure and all the attached oxygen atoms clearly visible. These coordinates were then used in 'SHELX'[73] to produce a starting R value of 42.64%, with all non-hydrogen atoms being clearly visible from the difference map.

The structure was refined by full matrix least squares[73]. At the introduction of anisotropic temperature factors for the non-hydrogen atoms, the temperature factors of O(2), O(10), O(12) became very high. The positions of the oxygens were transposed as shown in Fig. 7.2, however the temperature factors went non-positive definite, so it was assumed that the first positions were correct. The hydrogen atom positions were calculated and allowed to refine. Their bond lengths were fixed at 1.00Å ± 0.02Å. Common temperature factors were introduced for the methyl and hydroxy hydrogens of 0.1 and for the remaining hydrogens of 0.05.

The final weighting scheme used was

$$\omega = 1.00/(\sigma^2(F) + 0.00987F^2)$$

Results 7.3

Crystallographic Data 7.3.1

$C_{15}H_{12}O_7$, Mr = 304.25, Dc = 1.58gcm^{-3}, orthorhombic $P2_12_12_1$,

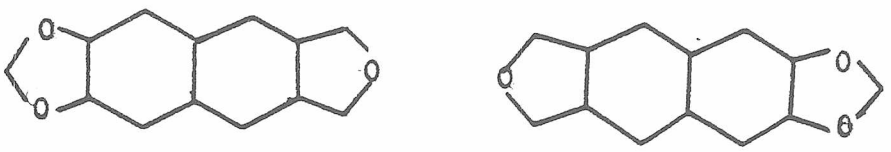


FIG. 7.2

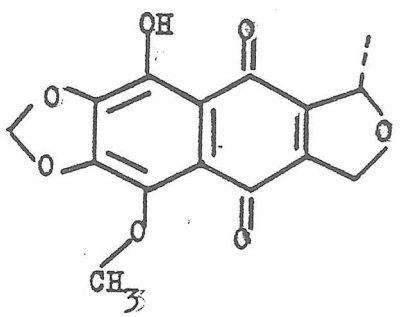


FIG. 7.3

Z= 4, a = 6.715(5)Å, b = 19.365(11)Å, c = 9.658(8)Å, U = 1255.89Å^3, μ(MoKα) = 0.82cm^{-1}, F(000) = 632.00, Final R value = 5.33% for 820 unique reflexions ($I > 3\sigma(I)$), ωR = 6.55%, maximum height on difference map =0.218 eÅ^{-3} minimum height on difference map =-0.308 eÅ^{-3}, maximum shift/error ratio = 0.025.

Structural Data 7.3.2

Ventilone C was found to have the structure as shown in Fig. 7.3. Fig. 7.4 shows the atomic arrangement of ventilone C, and a stereoscopic view of crystal packing is shown in Fig. 7.5. The various structural parameters are listed in Tables 7.1 - 7.5.

Ventilone C possesses intramolecular hydrogen bonding, as shown below

A - - - D		A - - - H
O(18) - - - O(16) 2.519(7)Å		O(18) - - - HO(16) 1.596(79)Å
O(18) - - - HO(16)	___	O(16) 152.1°(96)

The vibrational spectra and their major assignments are shown in Figs. 7.6 and 7.7 and Table 7.6 respectively.

Structure factors for Ventilone C are listed in Appendix A.

Discussion 7.4

X-ray Analysis 7.4.1

The X-ray analysis of ventilone C was problematic. It is thought that difficulties arose because it is a very planar structure, and it is possible that pseudo-symmetry caused by the symmetrical arrangement of the rings and the positions of the oxygens around the rings, caused the problems. The hypercentric distribution of E values is a result of this assumed symmetry, as this could cause the intensity distribution to tend towards that of a centric crystal [102].

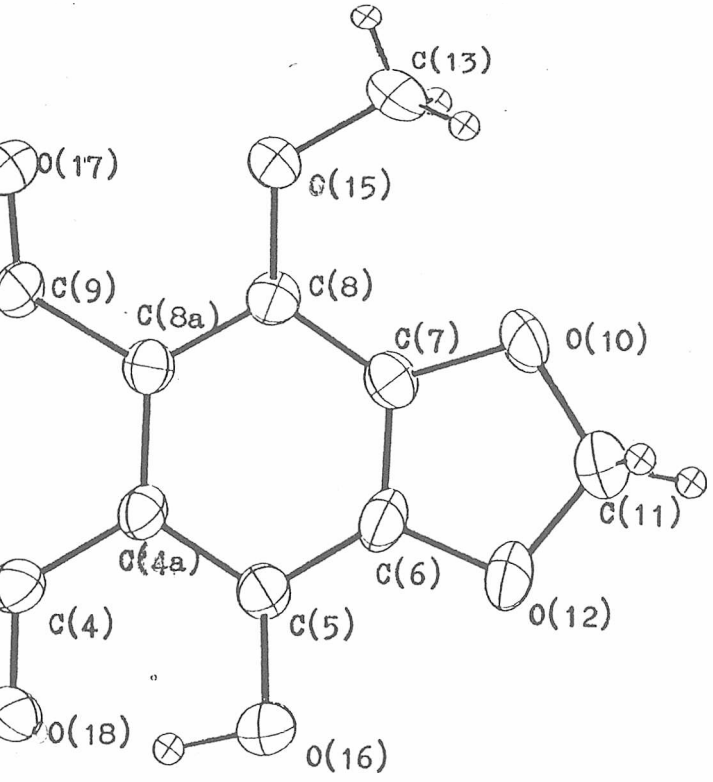
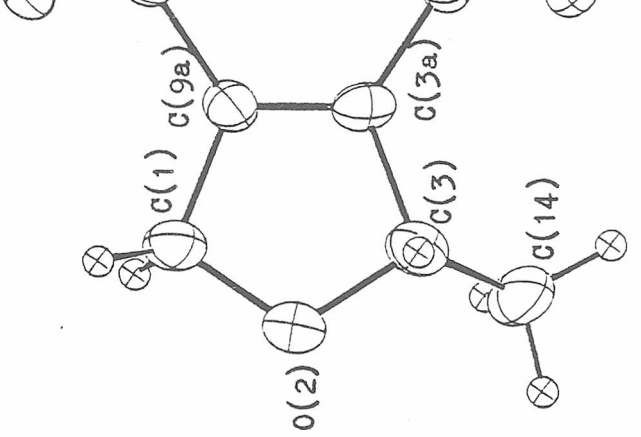


FIG. 7.4


C(9a)
C(3a)
C(1)
C(3)
C(14)
O(2)

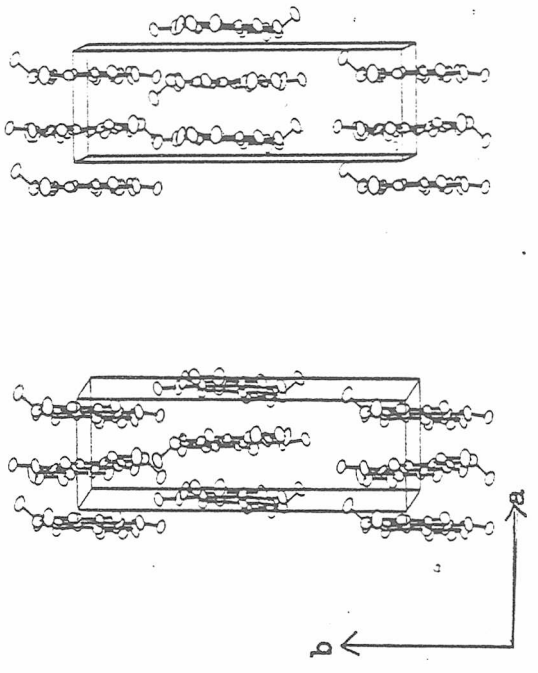


FIG 7.5

Atom	Fractional
C(1)	
O(2)	
C(3)	
C(3A)	
C(4)	
C(4A)	
C(5)	
C(6)	
C(7)	
C(8)	
C(8A)	
C(9)	
C(9A)	
O(10)	
C(11)	
O(12)	
C(13)	
C(14)	
O(15)	
O(16)	
O(17)	
O(18)	
H(1A)	
H(1B)	
H(3)	
H(11A)	
H(11B)	
H(13A)	
H(13B)	
H(13C)	
H(14A)	
H(14B)	
H(14C)	
HO(16)	

TABLE 7.1

x	y	z
2787(15)	-0611(4)	10612(8)
2772(19)	-1349(3)	10602(6)
2858(17)	-1629(4)	9212(9)
2671(15)	-0982(3)	8336(7)
2605(18)	-0950(3)	6832(7)
2465(18)	-0267(3)	6208(6)
2443(20)	-0236(3)	4737(7)
2465(20)	0409(3)	4155(7)
2534(22)	1008(3)	4905(8)
2488(18)	1005(3)	6370(7)
2471(16)	0354(3)	7013(7)
2476(16)	0292(3)	8552(7)
2568(16)	-0422(3)	9113(7)
2444(19)	1564(2)	4024(5)
2413(27)	1281(4)	2659(9)
2404(18)	0551(3)	2781(6)
2739(21)	2245(4)	6554(8)
1252(17)	-2162(5)	9027(10)
2509(16)	1581(2)	7165(6)
2538(14)	-0795(2)	3912(5)
2396(12)	0775(2)	9364(5)
2551(15)	-1487(2)	6120(5)
1517(76)	-0502(43)	11109(85)
3814(96)	-0365(38)	11182(80)
4350(37)	-1681(41)	9209(89)
3820(62)	1383(43)	2349(90)
1760(117)	1484(38)	1820(55)
3104(113)	2534(39)	7347(55)
1375(66)	2361(50)	6201(80)
3713(96)	2279(52)	5781(58)
1167(136)	-2381(40)	8078(40)
-0110(69)	-1984(40)	9269(85)
1591(129)	-2545(33)	9702(64)
2297(228)	-1163(38)	4611(82)

Atomic Coordinates ($x10^4$) with e.s.d.'s

Atom	U_{11}
C(1)	41(5)
O(2)	136(7)
C(3)	59(6)
C(3A)	26(4)
C(4)	38(5)
C(4A)	30(4)
C(5)	40(5)
C(6)	59(6)
C(7)	57(6)
C(8)	41(5)
C(8A)	30(4)
C(9)	35(4)
C(9A)	38(4)
O(10)	134(7)
C(11)	99(9)
O(12)	126(6)
C(13)	71(7)
C(14)	86(8)
O(15)	90(5)
O(16)	78(4)
O(17)	66(4)
O(18)	76(4)
H(1A)	38(10)
H(1B)	38(10)
H(3)	38(10)
H(11A)	38(10)
H(11B)	38(10)
H(13A)	76(12)
H(13B)	76(12)
H(13C)	76(12)
H(14A)	76(12)
H(14B)	76(12)
H(14C)	76(12)
HO(16)	76(12)

Anisotropic

TABLE 7.2

U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
37(3)	31(4)	1(4)	-7(5)	8(3)
38(3)	30(3)	-1(5)	-7(6)	5(2)
43(4)	31(4)	-8(5)	-14(4)	5(3)
32(3)	33(3)	11(4)	2(4)	9(2)
34(3)	30(3)	5(6)	-7(5)	-4(3)
33(3)	24(3)	-9(6)	0(4)	2(2)
30(3)	30(3)	6(6)	15(5)	0(2)
42(4)	22(3)	9(6)	4(6)	4(3)
32(3)	34(3)	-22(7)	6(5)	3(3)
34(4)	29(3)	-25(6)	-6(5)	1(2)
30(3)	28(3)	3(6)	-7(4)	2(3)
33(3)	25(3)	3(6)	14(5)	1(2)
34(3)	26(3)	-15(5)	3(5)	0(2)
24(2)	37(3)	24(6)	13(6)	9(2)
40(4)	39(4)	-16(8)	-1(9)	10(3)
43(3)	26(3)	9(6)	16(5)	9(2)
28(4)	45(4)	-12(6)	4(6)	1(3)
36(5)	50(5)	-23(5)	-28(5)	9(4)
25(2)	31(2)	8(5)	2(5)	1(2)
36(3)	31(3)	-16(5)	-2(4)	-4(2)
39(2)	29(3)	5(4)	4(5)	-3(2)
30(2)	38(3)	-16(5)	14(5)	-2(2)

Temperature Factor Coefficients ($\times 10^3$) with e.s.d.'s

TABLE 7.3

C(1)	O(2)	1.429(9)	C(5)	O(16)	1.346(8)
C(1)	C(9A)	1.501(10)	C(6)	C(7)	1.368(10)
O(2)	C(3)	1.449(10)	C(6)	O(12)	1.356(9)
C(3)	C(3A)	1.517(10)	C(7)	C(8)	1.415(10)
C(3)	C(14)	1.503(14)	C(7)	O(10)	1.374(8)
C(3A)	C(4)	1.455(10)	C(8)	C(8A)	1.405(9)
C(3A)	C(9A)	1.321(9)	C(8)	O(15)	1.354(8)
C(4)	C(4A)	1.456(9)	C(8A)	C(9)	1.491(9)
C(4)	O(18)	1.247(8)	C(9)	C(9A)	1.486(9)
C(4A)	C(5)	1.422(9)	C(9)	O(17)	1.222(8)
C(4A)	C(8A)	1.432(8)	O(10)	C(11)	1.428(10)
C(5)	C(6)	1.370(9)	C(11)	O(12)	1.419(10)
C(13)	O(15)	1.423(9)			

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d's

TABLE 7.4

C(9A) - C(1) - O(2)	103.7(6)	C(3) - O(2) - C(1)	112.3(6)
C(3A) - C(9A) - C(1)	110.0(6)	C(9) - C(9A) - C(1)	125.6(6)
C(3A) - C(3) - O(2)	101.8(6)	C(14) - C(3) - O(2)	109.8(8)
C(14) - C(3) - C(3A)	116.2(8)	C(4) - C(3A) - C(3)	126.5(6)
C(9A) - C(3A) - C(3)	111.4(6)	C(9A) - C(3A) - C(4)	122.1(6)
C(4A) - C(4) - C(3A)	117.0(6)	O(18) - C(4) - C(3A)	121.1(6)
C(9) - C(9A) - C(3A)	124.0(6)	O(18) - C(4) - C(4A)	121.8(6)
C(5) - C(4A) - C(4)	116.9(5)	C(8A) - C(4A) - C(4)	122.5(6)
C(8A) - C(4A) - C(5)	120.5(5)	C(6) - C(5) - C(4A)	116.6(6)
O(16) - C(5) - C(4A)	123.9(5)	C(8) - C(8A) - C(4A)	120.9(6)
C(9) - C(8A) - C(4A)	118.3(5)	O(16) - C(5) - C(6)	119.3(6)
C(7) - C(6) - C(5)	123.8(6)	O(12) - C(6) - C(5)	125.9(6)
O(12) - C(6) - C(7)	110.3(6)	C(8) - C(7) - C(6)	121.7(6)
O(10) - C(7) - C(6)	109.6(6)	C(11) - O(12) - C(6)	106.5(6)
O(10) - C(7) - C(8)	128.4(6)	C(8A) - C(8) - C(7)	116.5(6)
O(15) - C(8) - C(7)	124.3(6)	C(11) - O(10) - C(7)	105.8(5)
O(15) - C(8) - C(8A)	119.2(6)	C(9) - C(8A) - C(8)	120.8(5)
C(13) - O(15) - C(8)	120.7(6)	C(9A) - C(9) - C(8A)	116.0(5)
O(17) - C(9) - C(8A)	125.3(6)	O(17) - C(9) - C(9A)	118.7(6)
O(12) - C(11) - O(10)	107.8(6)		

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 7.5

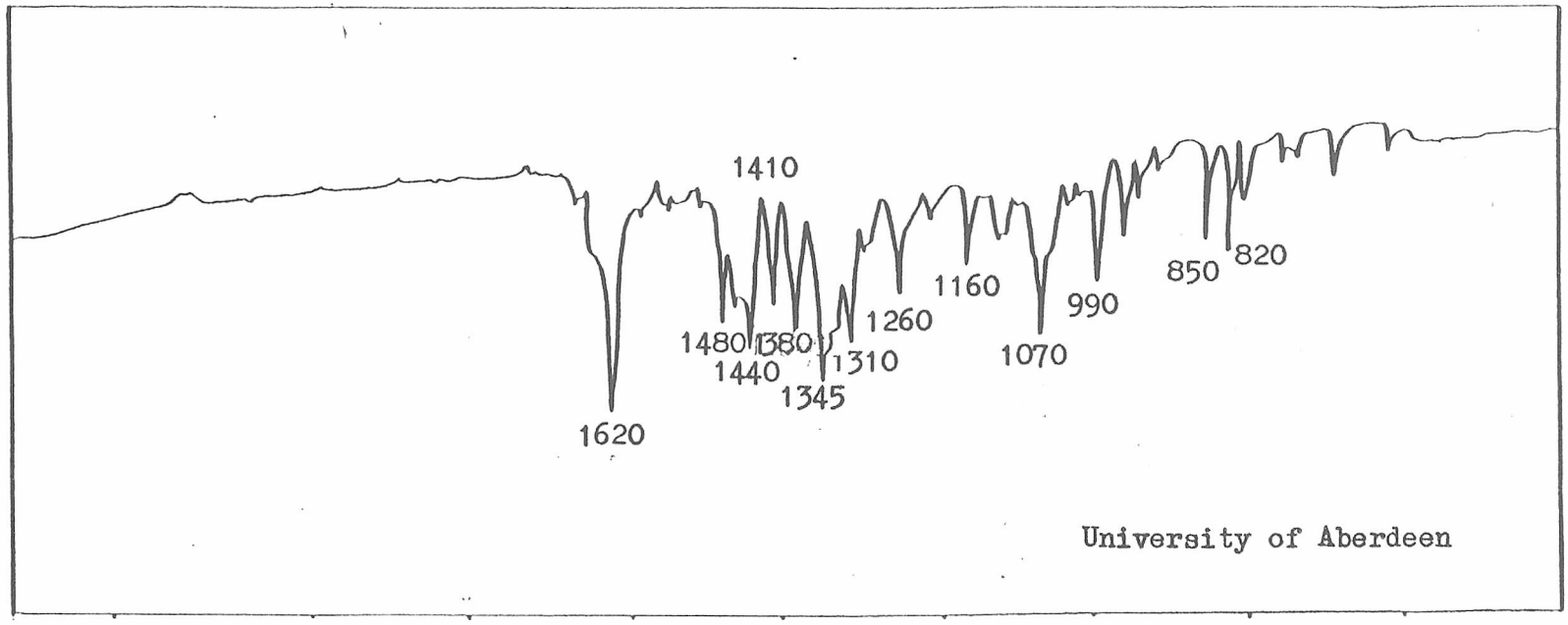
C(9A) - C(1) - O(2) - C(3)	8.5(9)
O(2) - C(1) - C(9A) - C(9)	179.3(8)
C(1) - O(2) - C(3) - C(14)	-130.4(8)
O(2) - C(3) - C(3A) - C(9A)	2.0(10)
C(14) - C(3) - C(3A) - C(9A)	121.2(9)
C(3) - C(3A) - C(4) - O(18)	5.5(14)
C(9A) - C(3A) - C(4) - O(18)	-174.5(8)
C(3) - C(3A) - C(9A) - C(9)	176.9(8)
C(4) - C(3A) - C(9A) - C(9)	-3.1(13)
C(3A) - C(4) - C(4A) - C(8A)	2.5(12)
O(18) - C(4) - C(4A) - C(8A)	178.3(8)
C(4) - C(4A) - C(5) - O(16)	0.2(13)
C(8A) - C(4A) - C(5) - O(16)	176.5(8)
C(4) - C(4A) - C(8A) - C(9)	-4.4(12)
C(5) - C(4A) - C(8A) - C(9)	179.5(8)
C(4A) - C(5) - C(6) - O(12)	-178.4(9)
O(16) - C(5) - C(6) - O(12)	6.1(15)
C(5) - C(6) - C(7) - O(10)	-177.2(9)
O(12) - C(6) - C(7) - O(10)	2.2(12)
C(7) - C(6) - O(12) - C(11)	-0.7(11)
C(6) - C(7) - C(8) - O(15)	-178.5(9)
O(10) - C(7) - C(8) - O(15)	-5.8(16)
C(8) - C(7) - O(10) - C(11)	-176.2(10)
C(7) - C(8) - C(8A) - C(9)	178.4(8)
O(15) - C(8) - C(8A) - C(9)	0.0(12)
C(8A) - C(8) - O(15) - C(13)	173.3(8)
C(4A) - C(8A) - C(9) - O(17)	-176.7(8)
C(8) - C(8A) - C(9) - O(17)	3.8(13)
C(8A) - C(9) - C(9A) - C(3A)	1.0(12)
O(17) - C(9) - C(9A) - C(3A)	-179.6(8)
O(10) - C(11) - O(12) - C(6)	-1.1(11)

Torsion Angles (°) (Non

O(2)	- C(1)	- C(9A)	- C(3A)	-7.1(9)
C(1)	- O(2)	- C(3)	- C(3A)	-6.7(9)
O(2)	- C(3)	- C(3A)	- C(4)	-178.0(8)
C(14)	- C(3)	- C(3A)	- C(4)	-58.8(12)
C(3)	- C(3A)	- C(4)	- C(4A)	-178.6(8)
C(9A)	- C(3A)	- C(4)	- C(4A)	1.4(12)
C(3)	- C(3A)	- C(9A)	- C(1)	3.2(10)
C(4)	- C(3A)	- C(9A)	- C(1)	-176.8(7)
C(3A)	- C(4)	- C(4A)	- C(5)	178.7(8)
O(18)	- C(4)	- C(4A)	- C(5)	-5.5(13)
C(4)	- C(4A)	- C(5)	- C(6)	-175.2(8)
C(8A)	- C(4A)	- C(5)	- C(6)	1.2(13)
C(4)	- C(4A)	- C(8A)	- C(8)	175.1(8)
C(5)	- C(4A)	- C(8A)	- C(8)	-1.0(12)
C(4A)	- C(5)	- C(6)	- C(7)	0.9(14)
O(16)	- C(5)	- C(6)	- C(7)	-174.6(9)
C(5)	- C(6)	- C(7)	- C(8)	-3.2(16)
O(12)	- C(6)	- C(7)	- C(8)	176.2(9)
C(5)	- C(6)	- O(12)	- C(11)	178.7(10)
C(6)	- C(7)	- C(8)	- C(8A)	3.2(14)
O(10)	- C(7)	- C(8)	- C(8A)	175.9(9)
C(6)	- C(7)	- O(10)	- C(11)	-2.8(11)
C(7)	- C(8)	- C(8A)	- C(4A)	-1.1(12)
O(15)	- C(8)	- C(8A)	- C(4A)	-179.5(8)
C(7)	- C(8)	- O(15)	- C(13)	-5.0(13)
C(4A)	- C(8A)	- C(9)	- C(9A)	2.7(11)
C(8)	- C(8A)	- C(9)	- C(9A)	-176.8(7)
C(8A)	- C(9)	- C(9A)	- C(1)	173.8(7)
O(17)	- C(9)	- C(9A)	- C(1)	-6.8(12)
C(7)	- O(10)	- C(11)	- O(12)	2.4(11)

Hydrogen Atoms) with e.s.d.'s

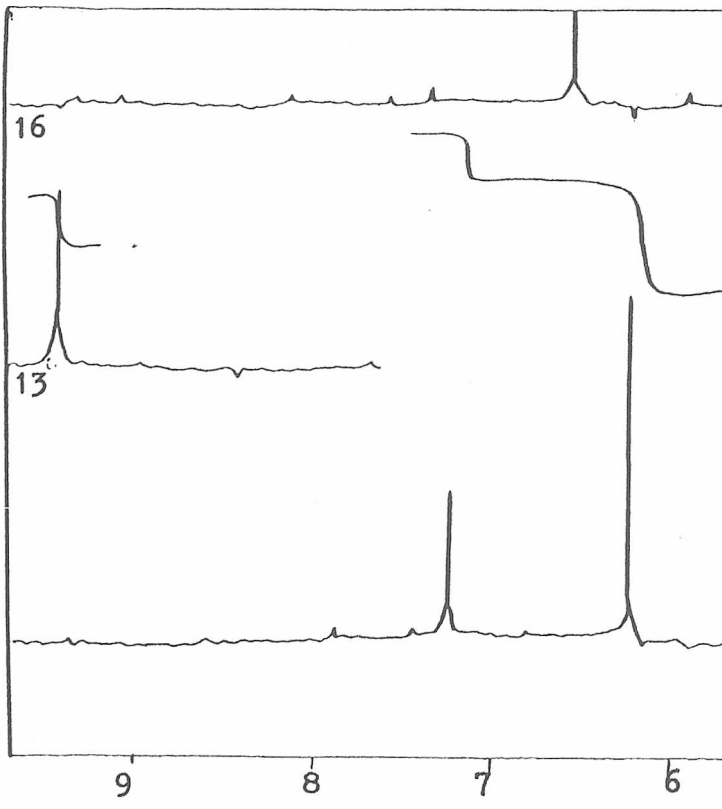
Infra Red Spectrum



Wave number cm^{-1}

FIG. 7.6

^{1}Hnmr Spectrum



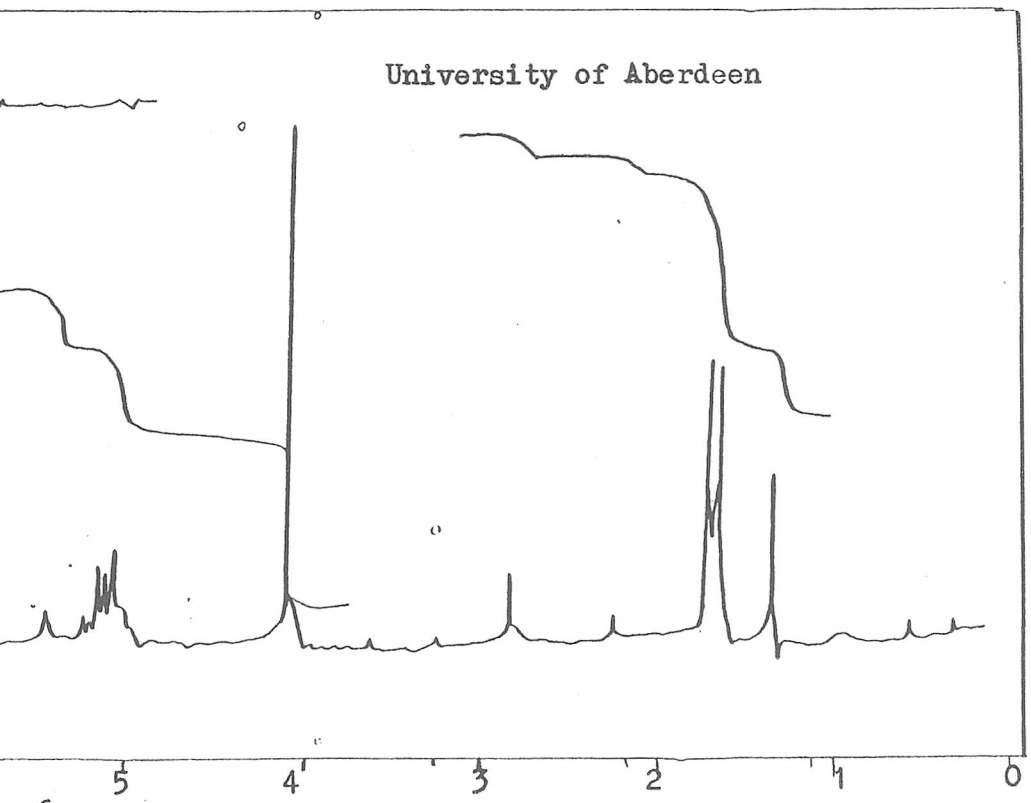


FIG. 7.7

TABLE 7.6

Major Assignments for Ventilone C

IR νmax KBr	930cm^{-1}	-	methylenedioxy group
	1625cm^{-1}	-	carbonyl bond
^{1}Hnmr Solvent $CDCl_3$ 220MHz	δ 1.54 (d)	-	methyl protons C(14)
	δ 4.0 (s)	-	methyl protons C(13)
	δ 5.04 (ddd)	-	methylene protons
	δ 5.40 (m)	-	methine proton
	δ 6.22 (s)	-	methylene protons
	δ 12.52 (s)	-	hydroxy proton

It was thought that input of a model of the structure might be successful, as it is a very planar molecule and would be expected to be difficult to solve. Input of a model allowed molecular scattering factors to be used in the calculation of E values, which is sometimes successful in clarifying the E map. This however was unsuccessful.

A Wilson plot in fitting a least squares straight line to a curve can produce considerable errors in the estimation of the temperature and scale factors used in normalisation. The K curve goes through every Debye point, and is extrapolated at low angles by the least squares straight line. This helps to produce a more reliable estimate of the temperature and scale factors. This was why a K curve was used.

As Σ_1 relationships are often unreliable and produce incorrect phases, the probability level at which Σ_1 phases were accepted was increased.

The maximum number of phase relationships were used on the assumption that it was a difficult structure, and that the weaker relationships would have to be employed in order to obtain a solution.

It was thought that perhaps the correct phases were being generated, but that the tangent formula was not working efficiently for this structure. In an attempt to overcome this, the statistically weighted tangent formula [5] was implemented. As this is stabilised by weighting throughout, it tends to work better for difficult structures.

The temperature factor was raised in an attempt to change the phasing pathway, and perhaps give a solution. Increasing or decreasing the temperature factor effectively introduces random

phasing into the system [103], which when used in a more deliberate and logical manner may aid structure solution. This proved to be successful.

During the course of the analysis, it was thought that the structure could be incorrect because of the eratic behaviour of the cyclic oxygens and their associated carbon atoms. However it is now thought that the structure has very slight disorder in its oxygen positions, and that a very small proportion of the molecules exist in the alternative structure as shown previously.

Despite the problems encountered in this analysis, the final R value obtained is reasonably low. The esd's associated with the structural parameters are quite high, but not high enough to invalidate the results of the analysis.

Structural 7.4.2

Ventilone C is a very planar structure. The dihydroisofuran ring is the least planar of the rings, with O(2) being the out of plane atom. No similar structures have been studied by X-ray diffraction analysis.

Ventilone C has intramolecular hydrogen bonding. The hydrogen bonding explains the downfield shift of the hydroxy hydrogen shown in the ^{1}Hnmr spectrum.

An interesting feature of the Hnmr is the long range coupling evident in the dihydroisofuran ring. The long range coupling is consistent with that found in benzoisofurans [104], although the couplings are larger than those found in phthalans [105]. This may be explained by the dihydroisofuran ring being fused to a quinone and not a benzene.

Molecular Mechanics 7.4.3

It is not possible to carry out molecular mechanics studies

on this molecule, as neither of the programmes available for use are able to compensate for the presence of hydrogen bonding.

CHAPTER EIGHT

ARISTOLINDIQUINONE, A NAPHTHOQUINONE FROM ARISTOLOCHIA INDICA

Aristolindiquinone a Naphthoquinone from Aristolochia indica

Introduction 8.1

Aristolindiquinone (Fig 8.1) was isolated from the roots of Aristolochia indica. It was thought to have the structure as shown in Fig. 8.1, this having been determined on the basis of spectral evidence. An X-ray analysis was undertaken to confirm this.

Experimental 8.2

Three crystals were mounted and preliminary photographs taken, before a satisfactory crystal was found. The dimensions of the crystal chosen were 1.0 x 0.1 x 0.5mm. Preliminary zero and first layer Weissenberg photographs were taken to ensure the crystal was suitable for data collection. The data was collected as outlined in Chapter 2, the intensities of 1424 reflexions being measured, scanning to $2\theta \leq 50^{\circ}$.

The structure was solved using 'MULTAN',[72] the E map being computed for 164 reflexions with $|E| > 1.48$. All the non-hydrogen atoms were visible from the E map. The oxygen atoms were assumed to be in the proposed positions and the coordinates input to 'SHELX'.[73] The structure was refined by full matrix least squares. Anisotropic temperature factors were introduced for the non-hydrogen atoms, allowing all but one hydroxy hydrogen to be located from the difference map. The F map instruction was modified so that only the low angle reflexions were looked at, and the missing hydrogen was located. The hydroxy hydrogen atoms were given common temperature factors of 0.1 and the other hydrogens of 0.05, and allowed to refine. The hydrogen atom bond lengths were constrained to $1.00\text{Å} \pm 0.02\text{Å}$, and the methyl hydrogen separation to $1.633\text{Å} \pm 0.02\text{Å}$.

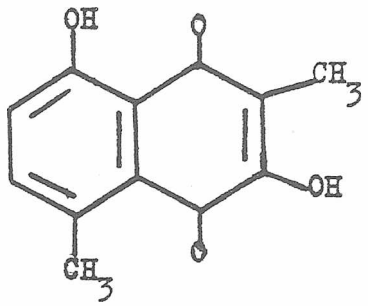


FIG. 8.1

Results 8.3

Crystallographic Data 8.3.1

$C_{12}H_{10}O_4$, Mr = 218.208, Dc = 1.47gcm^{-3}, monoclinic $P2_1/n$ Z = 4, a = 4.894(2)Å, b = 11.595(6)Å, c = 18.039(6)Å, β = 106.27(3)°, U = 982.64Å^3, μ(MoKα) = 0.68cm^{-1}, F(000) = 456.000, Final R value = 7.16% for 866 unique reflexions ($I > 3\sigma(I)$), ωR = 7.16%, maximum height on difference map = 0.127 eÅ^{-3}, minimum height on difference map = -0.123 eÅ^{-3}, maximum shift/error ratio = 0.082.

Structural Data 8.3.2

Aristolindiquinone was found to have the structure as shown in Fig. 8.1. The atomic arrangement is shown in Fig. 8.2, and Fig 8.3 shows a stereoscopic view of crystal packing. The structural parameters are listed in Tables 8.1 - 8.5. The major spectral assignments are shown in Table 8.6.

Aristolindiquinone was found to have both intra and intermolecular hydrogen bonding, as shown below.

Intermolecular

A - - - D		A - - - H	
O(1) - - - O(2)	2.734(8)Å	O(1) - - - HO(2)	1.903(79)Å
O(1) - - - HO(2)		O(2)	139.1(70)°

Coordinates transposed by -x + 2, -y, -z + 1

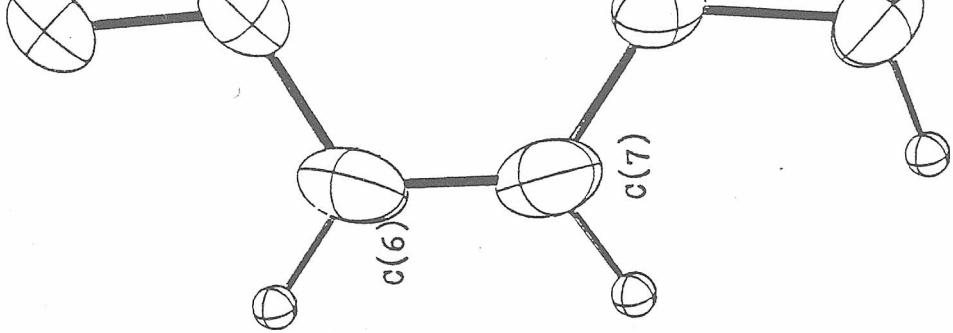
Intramolecular

A - - - D		A - - - H	
O(3) - - - O(4)	2.570(7)Å	O(3) - - - HO(4)	1.701(65)Å
O(3) - - - HO(4)		O(4)	143.1(76)°

Discussion 8.4

X-ray Analysis 8.4.1

Initially problems were experienced with the crystals chosen


C(6)
C(7)

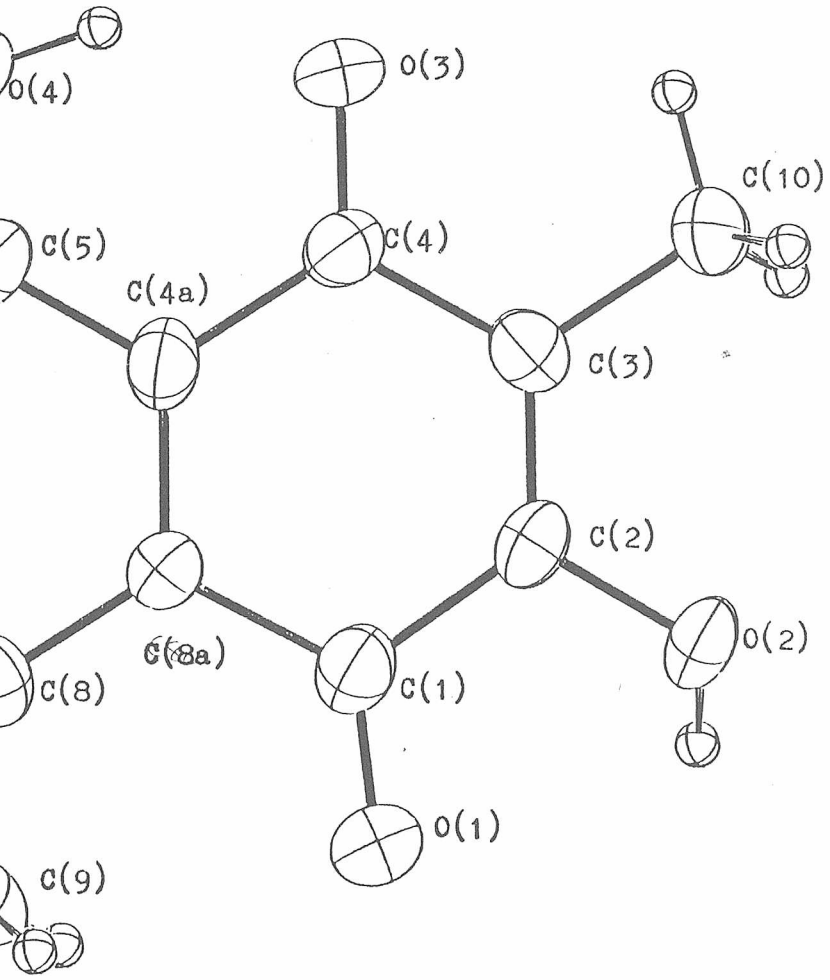


FIG. 8.2

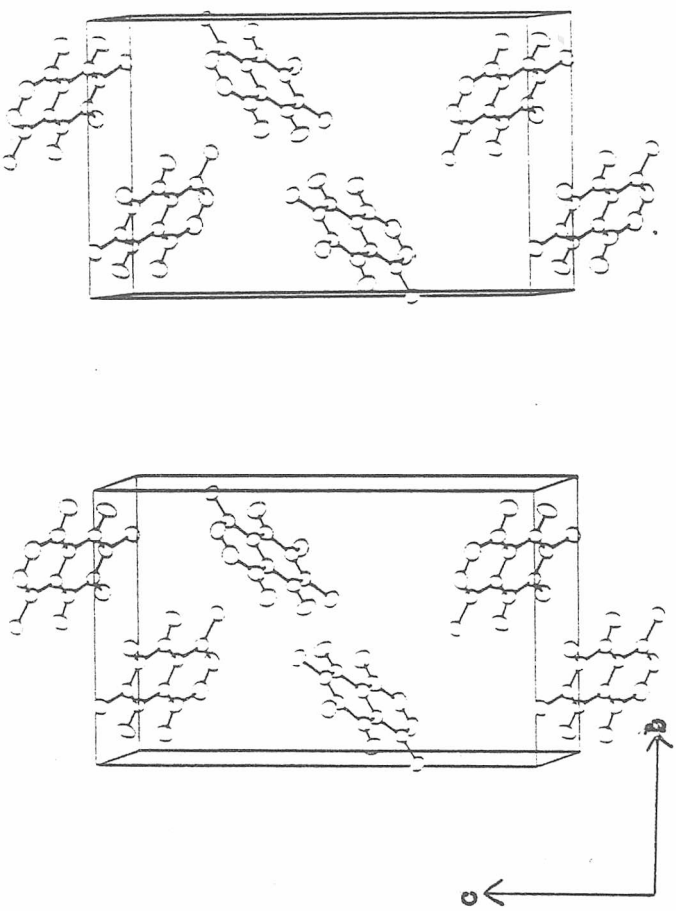


FIG. 8.3

TABLE 8.1

Atom	x	y	z
O(1)	7059(12)	0322(5)	4412(3)
O(2)	9599(11)	1544(5)	5651(3)
O(3)	2007(12)	4127(5)	5013(3)
O(4)	-1989(12)	3999(5)	3740(3)
C(1)	5835(16)	1206(6)	4488(4)
C(2)	7093(16)	1924(6)	5187(4)
C(3)	5884(16)	2876(6)	5359(4)
C(4)	3174(15)	3273(6)	4845(4)
C(4A)	1900(16)	2627(6)	4123(4)
C(5)	-0614(16)	3043(6)	3618(4)
C(6)	-1832(18)	2436(8)	2924(5)
C(7)	-0586(17)	1463(8)	2769(4)
C(8)	1916(16)	0991(6)	3250(4)
C(8A)	3146(15)	1604(6)	3938(4)
C(9)	3103(19)	-0094(7)	3006(4)
C(10)	7161(20)	3538(7)	6090(4)
HO(2)	10799(176)	1071(67)	5405(50)
HO(4)	-1117(186)	4165(80)	4299(19)
H(6)	-3706(103)	2732(78)	2601(44)
H(7)	-1297(192)	1034(73)	2276(30)
H(9A)	5141(63)	0073(63)	3020(37)
H(9B)	3080(137)	-0725(47)	3382(30)
H(9B)	2069(129)	-0356(61)	2473(17)
H(10A)	7109(143)	3112(54)	6568(29)
H(10B)	9166(66)	3706(56)	6096(44)
H(10C)	6067(121)	4274(35)	6064(41)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

TABLE 8.2

Atom	U_{11}	U_{22}
O(1)	50(4)	45(3)
O(2)	38(3)	51(3)
O(3)	52(4)	40(3)
O(4)	51(4)	52(3)
C(1)	34(4)	37(4)
C(2)	35(4)	36(4)
C(3)	38(4)	38(4)
C(4)	33(4)	32(4)
C(4A)	36(5)	39(4)
C(5)	35(4)	41(4)
C(6)	30(4)	65(6)
C(7)	41(5)	62(5)
C(8)	41(5)	34(4)
C(8A)	36(4)	33(4)
C(9)	61(5)	40(5)
C(10)	62(6)	46(5)
HO(2)	96(16)	
HO(4)	96(16)	
H(6)	96(16)	
H(7)	96(16)	
H(9A)	78(12)	
H(9B)	78(12)	
H(9B)	78(12)	
H(10A)	78(12)	
H(10B)	78(12)	
H(10C)	78(12)	

Anisotropic Temperature

U_{33}	U_{12}	U_{13}	U_{23}
54(3)	17(3)	1(3)	-7(3)
36(3)	17(3)	-3(2)	2(3)
57(4)	20(3)	-1(3)	-12(3)
52(3)	20(3)	-5(3)	-1(3)
38(4)	1(3)	5(3)	4(3)
31(4)	6(3)	3(3)	9(3)
33(4)	-1(3)	9(3)	-7(3)
38(4)	5(3)	7(3)	3(3)
31(4)	1(3)	-1(3)	2(3)
49(5)	6(4)	7(4)	9(4)
41(4)	2(4)	-2(3)	4(4)
35(4)	-6(4)	4(4)	-11(4)
36(4)	-9(3)	7(3)	-1(3)
32(4)	7(3)	4(3)	0(3)
37(4)	-5(4)	15(4)	-9(4)
30(4)	14(4)	-3(4)	-3(4)

Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 8.3

O(1)	C(1)	1.214(9)	C(3)	C(10)	1.503(10)
O(2)	C(2)	1.352(8)	C(4)	C(4A)	1.480(9)
O(3)	C(4)	1.222(9)	C(4A)	C(5)	1.395(10)
O(4)	C(5)	1.346(10)	C(4A)	C(8A)	1.418(11)
C(1)	C(2)	1.492(10)	C(5)	C(6)	1.416(11)
C(1)	C(8A)	1.483(9)	C(6)	C(7)	1.350(13)
C(2)	C(3)	1.329(11)	C(7)	C(8)	1.398(10)
C(3)	C(4)	1.465(9)	C(8)	C(8A)	1.410(9)
C(8)	C(9)	1.502(11)			

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 8.4

C(2)	C(1)	O(1)	117.9(7)	C(8A)	C(1)	O(1)	124.0(7)
C(1)	C(2)	O(2)	115.3(6)	C(3)	C(2)	O(2)	120.9(6)
C(3)	C(4)	O(3)	120.1(6)	C(4A)	C(4)	O(3)	121.1(7)
C(4A)	C(5)	O(4)	124.6(7)	C(6)	C(5)	O(4)	116.6(7)
C(8A)	C(1)	C(2)	118.1(6)	C(3)	C(2)	C(1)	123.8(7)
C(4A)	C(8A)	C(1)	117.5(6)	C(8)	C(8A)	C(1)	120.8(6)
C(4)	C(3)	C(2)	119.7(6)	C(10)	C(3)	C(2)	121.8(7)
C(10)	C(3)	C(4)	118.4(7)	C(4A)	C(4)	C(3)	118.8(6)
C(5)	C(4A)	C(4)	118.5(7)	C(8A)	C(4A)	C(4)	121.9(6)
C(8A)	C(4A)	C(5)	119.6(7)	C(6)	C(5)	C(4A)	118.8(7)
C(8)	C(8A)	C(4A)	121.7(7)	C(7)	C(6)	C(5)	119.7(8)
C(8)	C(7)	C(6)	124.6(7)	C(8A)	C(8)	C(7)	115.6(7)
C(9)	C(8)	C(7)	119.3(7)	C(9)	C(8)	C(8A)	125.1(7)

Valency Angles (^{o}) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 8.5

O(1)	C(1)	C(2)	O(2)	3.5(10)	O(1)	C(1)	C(2)	C(3)	-176.4(7)
C(8A)	C(1)	C(2)	O(2)	-177.5(6)	C(8A)	C(1)	C(2)	C(3)	2.6(11)
O(1)	C(1)	C(8A)	C(4A)	177.1(7)	O(1)	C(1)	C(8A)	C(8)	-3.8(12)
C(2)	C(1)	C(8A)	C(4A)	-1.8(10)	C(2)	C(1)	C(8A)	C(8)	177.3(7)
O(2)	C(2)	C(3)	C(4)	180.0(6)	O(2)	C(2)	C(3)	C(10)	-2.9(11)
C(1)	C(2)	C(3)	C(4)	-0.1(11)	C(1)	C(2)	C(3)	C(10)	177.0(7)
C(2)	C(3)	C(4)	O(3)	177.6(7)	C(2)	C(3)	C(4)	C(4A)	-2.9(11)
C(10)	C(3)	C(4)	O(3)	0.4(11)	C(10)	C(3)	C(4)	C(4A)	179.8(7)
O(3)	C(4)	C(4A)	C(5)	2.5(11)	O(3)	C(4)	C(4A)	C(8A)	-176.9(7)
C(3)	C(4)	C(4A)	C(5)	-176.9(7)	C(3)	C(4)	C(4A)	C(8A)	3.6(11)
C(4)	C(4A)	C(5)	O(4)	0.2(11)	C(4)	C(4A)	C(5)	C(6)	179.5(7)
C(8A)	C(4A)	C(5)	O(4)	179.6(7)	C(8A)	C(4A)	C(5)	C(6)	-1.1(11)
C(4)	C(4A)	C(8A)	C(1)	-1.2(10)	C(4)	C(4A)	C(8A)	C(8)	179.7(7)
C(5)	C(4A)	C(8A)	C(1)	179.4(7)	C(5)	C(4A)	C(8A)	C(8)	0.3(11)
O(4)	C(5)	C(6)	C(7)	-179.4(7)	C(4A)	C(5)	C(6)	C(7)	1.3(12)
C(5)	C(6)	C(7)	C(8)	-0.7(13)	C(6)	C(7)	C(8)	C(8A)	-0.1(12)
C(6)	C(7)	C(8)	C(9)	-179.4(8)	C(7)	C(8)	C(8A)	C(1)	-178.7(7)
C(7)	C(8)	C(8A)	C(4A)	0.3(11)	C(9)	C(8)	C(8A)	C(1)	0.5(11)
C(9)	C(8)	C(8A)	C(4A)	179.5(7)					

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 8.6

Major Spectral Assignments for Aristolindiquinone

IR ν max	1645cm^{-1} (s)	carbonyl absorption
	1615cm^{-1} (s)	
	3340cm^{-1} (b)	phenolic group
UV λ max	208 nm	naphthoquinone moiety
	236 nm	
	284 nm	
	433 nm	
	Bathochromic shift on addition of base	
	220 nm	phenolic group
	238 nm	
	270 nm	
	394 nm	
	495 nm	
^{1}H nmr 100MHz Solvent $CDCl_3$	δ 2.06 (s)	methyl hydrogens C(10)
	δ 2.64 (s)	methyl hydrogens C(9)
	δ 7.17 (d)	aromatic hydrogen
	δ 7.32 (d)	aromatic hydrogen
	δ 7.65 (s)	hydroxy hydrogen
	δ 13.03 (s)	hydroxy hydrogen (hydrogen bonded)
^{13}C nmr 25.05MHz	δ 114.8	C(4a)
	δ 118.5	C(3)
	δ 125.9	C(8a)
	δ 126.3	C(6)
	δ 135.2	C(8)
	δ 139.3	C(7)
	δ 154.0	C(2)
	δ 160.8	C(5)
	δ 181.5	C(1)
	δ 191.2	C(4)

The spectra were run at the University of Illinois at Chicago.

for the X-ray analysis. They were found not to obey Friedel's law. The situation with these crystals was as follows

Present	017	$0\bar{1}7$	136	$1\bar{3}6$	202	$\bar{2}0\bar{2}$
Absent	$01\bar{7}$	$0\bar{1}\bar{7}$	$\bar{1}3\bar{6}$	$\bar{1}36$		

It is thought that these crystals may have been twinned with the crystals lying back to back, as the breakdown in Friedel's law could not be caused by an anomalous scatterer in a structure such as this. This problem illustrates the value of doing preliminary photographic investigations on a crystal, before transfer to the diffractometer. A crystal may appear suitable, but show discrepancies and unusual absences, after considerable work has been carried out. If preliminary photographs had been taken, the absences would have been observed. However this situation arises so infrequently that it is not standard procedure to take preliminary photographs.

The data collected for Aristolindiquinone were of good quality, shown by the low esd.'s associated with the structural parameters. This is not reflected in the R value. but the structure is centrosymmetric and could be expected to have a high R value [106]. Various weighting schemes were tried in an endeavour to lower the R value, however this had the effect of increasing the esd's. As the esd's are a better indication of the quality of the data [88], the weighting schemes were abandoned.

Structural 8.4.2

The structure was found to have been correctly determined by the spectral analysis. However the X-ray analysis successfully determined the hydrogen bonding sequence. As seen from Table 8.1 the assignments for the spectral analysis were extensive and the ^{1}Hnmr spectrum showed clearly that hydrogen bonding was present.

However, the presence of intermolecular hydrogen bonding was also established by X-ray analysis.

Aristolindiquinone is a very planar structure, ring A being planar to within 0.124Å and ring B to within 0.0403Å. This is as would be expected for structures of this type, as may be seen by comparison with Naphthazarine [107], Phthiocol [108], and Juglone [109].

It was thought that the density was high for a structure of this type, as there are no heavy atoms present. However, after consulting tabulated densities of similar quinone and naphthoquinone structures [110], it was found that they were similar.

Molecular Mechanics 8.4.3

It was found impossible to carry out molecular mechanics calculations on Aristolindiquinone, as MM2 has no facilities for calculating the effects of hydrogen bonding. Various authors have made attempts to include hydrogen bonding potentials in a variety of different molecular mechanics programmes, but those of particular interest are those implemented in modified versions of MM1 [111] and MM2 [112]. A system used in MM1 was found to give very good agreement with standard hydrogen bond geometries [113] and could possibly be used more extensively in the future. MM1 was not however available for these studies. Recent modifications to MM2 as in the force field MM2HB [112] have made it possible to use MM2 for hydrogen bonded structures. In order to implement this, it would have been necessary to modify MM2 extensively [114], which it was not possible to do. This force field has been used successfully on α-D-Glucose.

CHAPTER NINE

ANCISTROTECTORINE, A NAPHTHALENE-ISOQUINOLINE ALKALOID FROM ANCISTROCLADUS TECTORIUS (ANCISTROCLADACEAE)

Ancistrotectorine, a Naphthalene-Isoquinoline Alkaloid from Ancistrocladus tectorius (Ancistrocladaceae)

Introduction 9.1

Ancistrotectorine (Fig. 9.1) was isolated from the leaves of Ancistracladus tectorius. From the UV spectrum, ancistrotectorine was found to be a naphthalene-isoquinoline alkaloid containing a phenolic hydroxy group [116]. The proton nmr firmly established the naphthalene-isoquinoline nature of the compound and it was thought to be similar to the structure shown in Fig. 9.2. However it was not possible to completely determine the structure from these spectra. It was not certain if the molecular mass of ancistrotectorine was 407 or 421amu.

Experimental 9.2

The crystals of ancistrotectorine were well shaped and clear. The first crystal mounted showed no evidence of diffraction from the rotation photograph. A second crystal was chosen, and although the spots on the rotation photograph were very faint, it was decided to proceed, the intensity limit for the acceptance of a reflexion being lowered. The procedure as in Chapter 2 was followed, the data being collected to $2\theta \leq 40^{\circ}$ for 1088 reflexions.

The data were input to 'MULTAN 80' [72] to produce an E map computed for 230 reflexions with $|E|_{min} > 1.2$. An elevated temperature factor of 4.0 (raised from 3.2) was necessary to effect a solution. A satisfactory refinement was not achieved with the first cycle of least squares, and an alternative arrangement of the atoms was visible from the difference map. The coordinates of this arrangement were used in 'SHELX' [73] refinement proceeding using full matrix least squares analysis [73], to produce a starting R value of 34% .

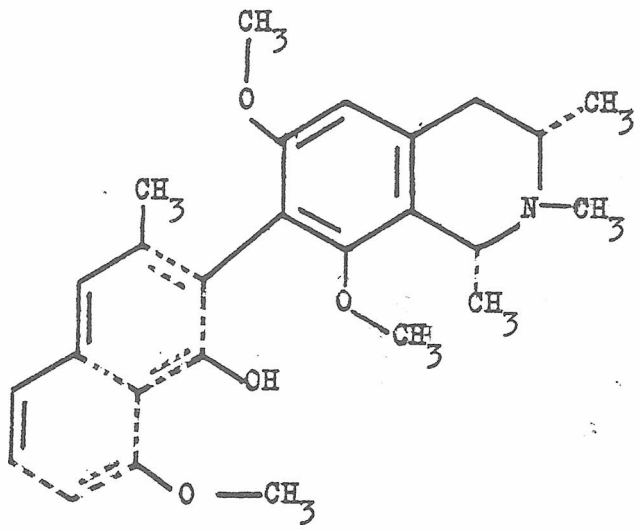


FIG. 9.1

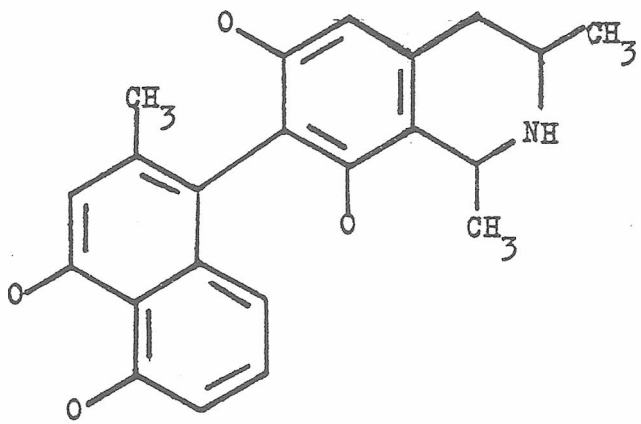


FIG. 9.2

All the non-hydrogen atoms were clearly visible from the difference map. Anisotropic temperature factors were introduced for all non-hydrogen atoms, allowing all the hydrogens except the hydroxy hydrogen to be identified. When the hydrogen atom positions were allowed to refine, the hydroxy hydrogen was visible from the difference map. Refinement proceeded with unit weights, the methyl and hydroxy hydrogens being given common temperature factors of 0.1 and the remaining hydrogens, common temperature factors of 0.05.

Molecular mechanics calculations were carried out on the central ring systems only, using the programme MM2 [74]. All aromatic C = C bond lengths were defined as 1.39Å.

Results 9.3

Crystallographic Data 9.3.1

$C_{26}H_{31}O_4N$, Mr = 421.535, Dc = 1.18gcm^{-3}, monoclinic, $P2_1$, Z = 2, a = 11858(14)Å, b = 7.043(7)Å, c = 14.693(28)Å, β = 111.43(12)°. U = 1142.26Å^3, μ(MoKα) = 0.46cm^{-1}, F(000) = 434.00, Final R value = 8.02% for 1087 unique reflexions, ωR = 8.02%, maximum height on difference map = 0.115 eÅ^{-3}, minimum height on difference map = -0.113 eÅ^{-3}, maximum shift/error ratio = 0.115.

Structural Data 9.3.2

Ancistrotectorine has the structure as shown in Fig. 9.1. The atomic arrangement is shown in Fig. 9.3 and a stereoscopic view of crystal packing is shown in Fig. 9.4. The structural parameters are listed in Tables 9.1 - 9.5. The ^{1}Hnmr assignments are shown in Fig. 9.5.

Discussion 9.4

X-ray Analysis 9.4.1

The quality of the data collected for ancistrotectorine

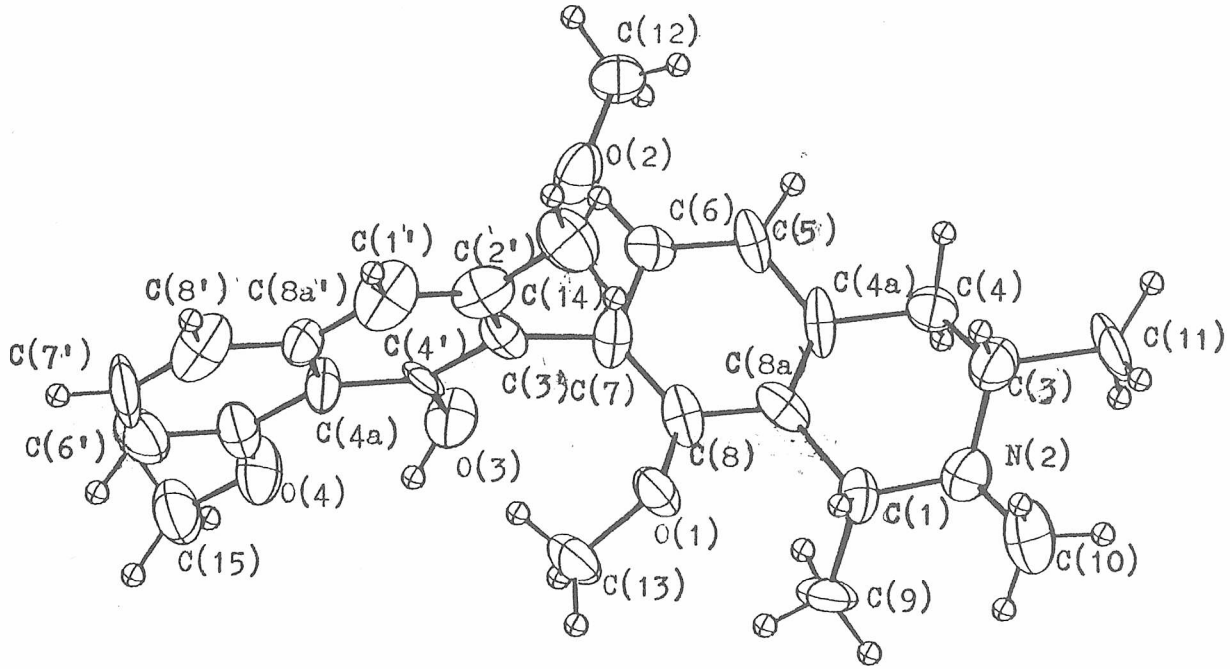

C(12)
O(2)
C(6)
C(5)
C(1')
C(2')
C(14)
C(4a)
C(4)
C(11)
C(8')
C(8a')
C(4')
C(3')
C(7)
C(8a)
C(3)
C(7')
C(6')
C(4a)
O(3)
C(8)
N(2)
O(4)
C(1)
O(1)
C(10)
C(15)
C(13)
C(9)

FIG. 9.3

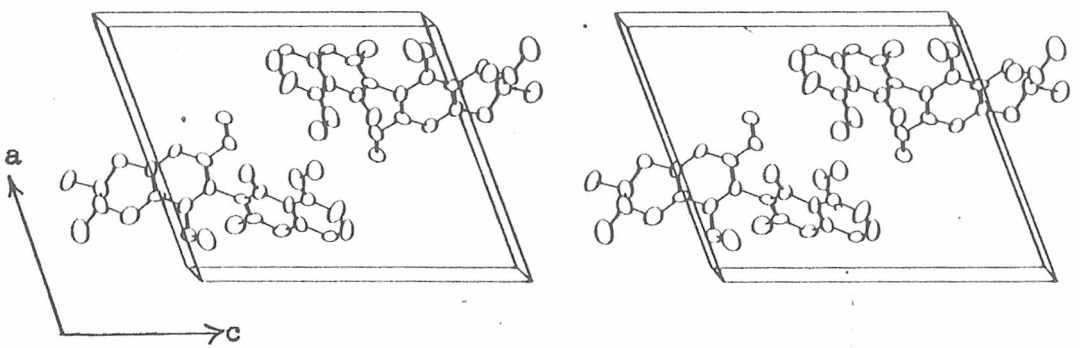
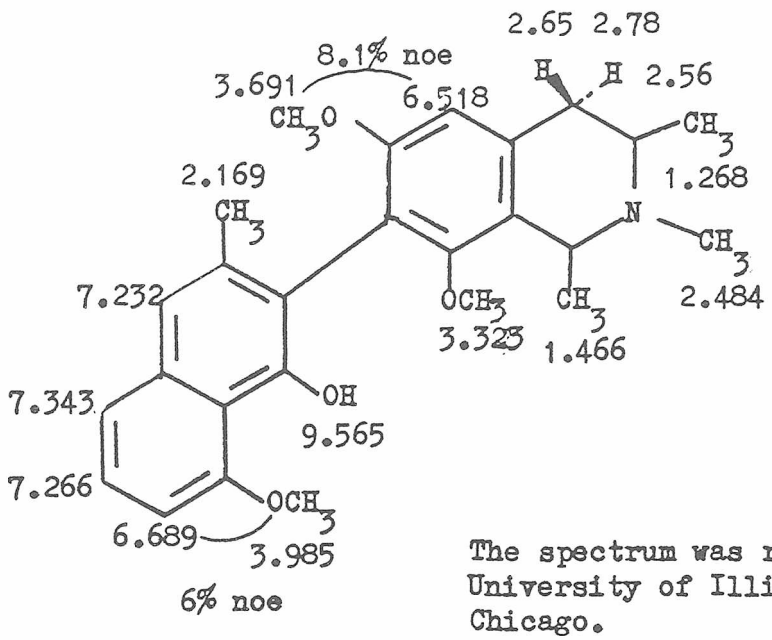


FIG. 9.4



The spectrum was run at the University of Illinois at Chicago.

FIG. 9.5

TABLE 9.1

Atom	x	y
O(1)	1240(8)	9594(20)
O(2)	4670(7)	6549(22)
O(3)	3793(7)	11387(23)
O(4)	3645(10)	13613(23)
C(1)	1943(12)	8794(27)
N(2)	2331(11)	7931(24)
C(3)	2868(13)	6042(27)
C(4)	3963(11)	6199(29)
C(4A)	3682(12)	7021(28)
C(5)	4374(11)	6427(28)
C(6)	4056(11)	7043(25)
C(7)	3025(12)	8244(29)
C(8)	2347(12)	8766(27)
C(8A)	2689(11)	8158(25)
C(1')	1312(11)	7786(30)
C(2')	1769(11)	7380(27)
C(3')	2589(10)	8651(25)
C(4')	2962(11)	10278(23)
C(4A')	2490(13)	10752(27)
C(5')	2789(12)	12383(27)
C(6')	2339(18)	12833(30)
C(7')	1546(16)	11543(32)
C(8')	1207(11)	9955(35)
C(8A')	1656(13)	9490(26)
C(9)	2074(14)	10965(24)
C(10)	1288(16)	7917(32)
C(11)	3135(14)	5103(30)
C(12)	5591(12)	5143(29)
C(13)	1163(16)	11377(31)
C(14)	1417(14)	5579(28)
C(15)	4087(14)	15272(29)

Fractional Atomic Coordinates ($\times 10^4$) with

z
0433(6)
2800(6)
2826(7)
4207(6)
-1116(9)
-1864(8)
-1609(10)
-0718(10)
0111(9)
1072(10)
1837(9)
1655(9)
0679(9)
-0056(9)
3363(11)
2658(10)
2481(9)
3052(9)
3776(9)
4364(10)
5069(13)
5221(11)
4686(10)
3945(9)
-1185(11)
-2780(12)
-2472(10)
2970(9)
0861(11)
2077(12)
4814(11)

e.s.d.'s

TABLE 9.1(cont.)

HO(3)	3799(102)	12787(37)	2937(80)
H(1)	1102(41)	8521(175)	-1144(71)
H(3)	2306(81)	5109(127)	-1480(79)
H(4A)	4436(81)	5016(86)	-0463(68)
H(4B)	4559(77)	7121(122)	-0795(77)
H(5)	5014(69)	5531(121)	1053(74)
H(1')	0662(71)	6984(134)	3440(73)
H(6')	2342(100)	14212(56)	5234(79)
H(7')	1116(75)	11783(176)	5685(55)
H(8')	0583(72)	9069(124)	4760(74)
H(9A)	1546(77)	11586(160)	-0873(64)
H(9B)	2922(39)	10879(185)	-0680(52)
H(9C)	2105(94)	11794(152)	-1731(60)
H(10A)	0896(106)	9195(78)	-2902(88)
H(10B)	0712(97)	6960(116)	-2689(90)
H(10C)	1516(101)	7534(144)	-3343(62)
H(11A)	3642(84)	3971(104)	-2491(95)
H(11B)	2300(38)	4926(158)	-2963(65)
H(11C)	3510(89)	6260(106)	-2646(91)
H(12A)	5926(88)	4500(150)	3619(40)
H(12B)	5199(96)	4183(126)	2448(56)
H(12C)	6272(70)	5770(154)	2829(70)
H(13A)	0396(52)	12051(147)	0480(69)
H(13B)	1102(88)	10847(161)	1476(52)
H(13C)	1854(67)	12306(135)	1050(79)
H(14A)	2230(49)	5140(165)	2110(79)
H(14B)	0997(82)	4533(126)	2288(81)
H(14C)	0919(74)	5983(177)	1399(43)
H(15A)	4807(59)	15680(158)	4660(75)
H(15B)	3619(94)	16424(107)	4869(83)
H(15C)	4383(87)	14600(147)	5464(49)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Atom	U_{11}	U_{22}
O(1)	42(6)	43(6)
O(2)	42(9)	86(9)
O(3)	68(6)	42(6)
O(4)	88(8)	74(9)
C(1)	48(9)	48(10)
N(2)	66(8)	54(9)
C(3)	61(10)	50(12)
C(4)	45(9)	55(11)
C(4A)	48(9)	66(11)
C(5)	41(8)	54(10)
C(6)	35(8)	27(8)
C(7)	49(9)	66(11)
C(8)	50(9)	52(10)
C(8A)	23(7)	25(8)
C(1')	27(8)	89(15)
C(2')	23(7)	67(12)
C(3')	23(7)	37(9)
C(4')	45(8)	10(8)
C(4A')	55(9)	81(11)
C(5')	35(8)	56(12)
C(6')	100(13)	42(11)
C(7')	91(13)	80(14)
C(8')	22(7)	118(17)
C(8A')	51(9)	55(11)
C(9)	68(10)	27(9)
C(10)	121(16)	81(14)
C(11)	88(11)	59(12)
C(12)	38(8)	63(11)
C(13)	102(13)	63(13)
C(14)	64(10)	44(11)
C(15)	79(11)	62(12)

Anisotropic Temperature

BLE 9.2

U_{33}	U_{12}	U_{13}	U_{23}
47(5)	16(5)	5(4)	-16(5)
31(5)	5(6)	5(4)	-2(6)
91(6)	-26(6)	32(5)	-2(5)
41(6)	-4(8)	13(6)	-25(7)
37(8)	-6(8)	25(7)	-6(8)
38(7)	4(8)	9(7)	-2(7)
38(8)	-5(9)	15(8)	0(8)
45(9)	22(8)	13(8)	6(8)
37(8)	-17(10)	25(7)	-31(9)
47(9)	-4(8)	25(7)	-26(8)
40(9)	-8(8)	-2(6)	-9(8)
33(8)	-3(10)	20(7)	-11(8)
50(10)	-6(9)	27(8)	-20(9)
64(10)	-7(7)	14(7)	-15(8)
60(10)	-25(9)	17(8)	3(10)
58(9)	4(9)	13(7)	14(9)
44(8)	7(7)	1(6)	-13(8)
46(8)	7(7)	24(7)	-4(7)
28(8)	-2(8)	20(7)	-9(8)
41(9)	4(9)	10(7)	-16(9)
87(13)	9(11)	69(11)	5(11)
48(10)	7(12)	37(9)	-34(11)
46(9)	2(11)	11(7)	14(12)
27(8)	17(9)	9(7)	0(8)
71(10)	18(8)	33(9)	19(8)
63(11)	-2(13)	32(11)	-30(12)
73(11)	12(11)	53(9)	-28(10)
47(8)	20(9)	6(7)	6(9)
59(10)	51(12)	12(9)	-11(10)
81(11)	-17(9)	26(9)	-17(10)
58(10)	4(11)	24(9)	-22(10)

Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 9.2 (cont.)

HO(3)	38(11)
H(1)	38(11)
H(3)	38(11)
H(4A)	38(11)
H(4B)	38(11)
H(5)	38(11)
H(1')	38(11)
H(6')	38(11)
H(7')	38(11)
H(8')	38(11)
H(9A)	88(11)
H(9B)	88(11)
H(9C)	88(11)
H(10A)	88(11)
H(10B)	88(11)
H(10C)	88(11)
H(11A)	88(11)
H(11B)	88(11)
H(11C)	88(11)
H(12A)	88(11)
H(12B)	88(11)
H(12C)	88(11)
H(13A)	88(11)
H(13B)	88(11)
H(13C)	88(11)
H(14A)	88(11)
H(14B)	88(11)
H(14C)	88(11)
H(15A)	88(11)
H(15B)	88(11)
H(15C)	88(11)

Anisotropic Temperature Coefficients ($x10^3$) with e.s.d.'s

TABLE 9.3

O(1)	C(8)	1.359(18)	C(5)	C(6)	1.380(22)
O(1)	C(13)	1.422(25)	C(6)	C(7)	1.429(22)
O(2)	C(6)	1.379(14)	C(7)	C(8)	1.413(17)
O(2)	C(12)	1.427(21)	C(7)	C(3′)	1.510(21)
O(3)	C(4′)	1.352(18)	C(8)	C(8A)	1.354(22)
O(4)	C(5′)	1.416(22)	C(1′)	C(2′)	1.363(24)
O(4)	C(15)	1.447(23)	C(1′)	C(8A′)	1.443(25)
C(1)	N(2)	1.469(22)	C(2′)	C(3′)	1.414(23)
C(1)	C(8A)	1.550(17)	C(2′)	C(14)	1.501(25)
C(1)	C(9)	1.544(25)	C(3′)	C(4′)	1.393(21)
N(2)	C(3)	1.463(24)	C(4′)	C(4A′)	1.411(22)
N(2)	C(10)	1.458(17)	C(4A′)	C(5′)	1.403(25)
C(3)	C(4)	1.473(16)	C(4A′)	C(8A′)	1.417(25)
C(3)	C(11)	1.562(25)	C(5′)	C(6′)	1.365(28)
C(4)	C(4A)	1.492(23)	C(6′)	C(7′)	1.383(31)
C(4A)	C(5)	1.413(18)	C(7′)	C(8′)	1.341(30)
C(4A)	C(8A)	1.370(21)	C(8′)	C(8A′)	1.414(23)

Bond Lengths: (Å) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 9.4

C(13) – O(1) – C(8)	118.2(13)	C(7) – C(8) – O(1)	121.2(12)
C(8A) – C(8) – O(1)	117.7(12)	C(12) – O(2) – C(6)	116.0(11)
C(5) – C(6) – O(2)	123.9(14)	C(7) – C(6) – O(2)	115.9(11)
C(3′) – C(4′) – O(3)	115.0(11)	C(4A′)– C(4′) – O(3)	123.4(14)
C(15) – O(4) – C(5′)	120.9(12)	C(4A′)– C(5′) – O(4)	115.8(13)
C(6′) – C(5′) – O(4)	118.9(16)	C(8A) – C(1) – N(2)	114.4(13)
C(9) – C(1) – N(2)	107.1(13)	C(3) – N(2) – C(1)	114.2(12)
C(10) – N(2) – C(1)	107.5(13)	C(9) – C(1) – C(8A)	108.5(12)
C(4A) – C(8A) – C(1)	119.1(12)	C(8) – C(8A) – C(1)	118.9(13)
C(10) – N(2) – C(3)	111.9(14)	C(4) – C(3) – N(2)	108.6(15)
C(11) – C(3) – N(2)	112.1(12)	C(11) – C(3) – C(4)	112.3(14)
C(4A) – C(4) – C(3)	111.6(13)	C(5) – C(4A) – C(4)	118.7(15)
C(8A) – C(4A) – C(4)	120.9(12)	C(8A) – C(4A) – C(5)	120.0(13)
C(6) – C(5) – C(4A)	119.3(14)	C(8) – C(8A) – C(4A)	122.0(13)
C(7) – C(6) – C(5)	120.2(12)	C(8) – C(7) – C(6)	118.3(13)
C(3′) – C(7) – C(6)	118.9(12)	C(3′) – C(7) – C(8)	122.1(14)
C(8A) – C(8) – C(7)	120.1(15)	C(2′) – C(3′) – C(7)	119.2(14)
C(4′) – C(3′) – C(7)	120.9(14)	C(8A′)– C(1′) – C(2′)	121.3(16)
C(3′) – C(2′) – C(1′)	119.9(16)	C(14) – C(2′) – C(1′)	120.3(16)
C(4A′)– C(8A′)– C(1′)	118.8(13)	C(8′) – C(8A′)– C(1′)	123.0(16)
C(14) – C(2′) – C(3′)	119.8(13)	C(4′) – C(3′) – C(2′)	119.9(12)
C(4A′)– C(4′) – C(3′)	121.5(14)	C(5′) – C(4A′)– C(4′)	125.3(15)
C(8A′)– C(4A′)– C(4′)	118.6(15)	C(8A′)– C(4A′)– C(5′)	116.2(13)
C(6′) – C(5′) – C(4A′)	125.2(16)	C(8′) – C(8A′)– C(4A′)	118.2(16)
C(7′) – C(6′) – C(5′)	116.6(18)	C(8′) – C(7′) – C(6′)	121.9(16)
C(8A′)– C(8′) – C(7′)	121.9(17)		

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 9.5

C(13)	O(1)	C(8)	C(7)	61.5(20)	C(13)	O(1)	C(8)	C(8A)	-129.7(16)
C(12)	O(2)	C(6)	C(5)	-7.6(21)	C(12)	O(2)	C(6)	C(7)	171.5(13)
C(15)	O(4)	C(5')	C(4A')	-175.7(14)	C(15)	O(4)	C(5')	C(6')	3.1(23)
C(8A)	C(1)	N(2)	C(3)	29.3(18)	C(8A)	C(1)	N(2)	C(10)	154.2(14)
C(9)	C(1)	N(2)	C(3)	149.6(13)	C(9)	C(1)	N(2)	C(10)	-85.5(15)
N(2)	C(1)	C(8A)	C(4A)	5.6(20)	N(2)	C(1)	C(8A)	C(8)	-175.3(14)
C(9)	C(1)	C(8A)	C(4A)	-113.9(16)	C(9)	C(1)	C(8A)	C(8)	65.2(18)
C(1)	N(2)	C(3)	C(4)	-61.6(17)	C(1)	N(2)	C(3)	C(11)	173.8(13)
C(10)	N(2)	C(3)	C(4)	175.9(14)	C(10)	N(2)	C(3)	C(11)	51.3(18)
N(2)	C(3)	C(4)	C(4A)	58.0(17)	C(11)	C(3)	C(4)	C(4A)	-177.5(13)
C(3)	C(4)	C(4A)	C(5)	147.9(15)	C(3)	C(4)	C(4A)	C(8A)	-24.6(21)
C(4)	C(4A)	C(5)	C(6)	-174.2(14)	C(8A)	C(4A)	C(5)	C(6)	-1.6(23)
C(4)	C(4A)	C(8A)	C(1)	-7.6(22)	C(4)	C(4A)	C(8A)	C(8)	173.3(15)
C(5)	C(4A)	C(8A)	C(1)	180.0(14)	C(5)	C(4A)	C(8A)	C(8)	0.9(24)
C(4A)	C(5)	C(6)	O(2)	-180.0(14)	C(4A)	C(5)	C(6)	C(7)	1.0(23)
O(2)	C(6)	C(7)	C(8)	-178.8(14)	O(2)	C(6)	C(7)	C(3')	-8.3(20)
C(5)	C(6)	C(7)	C(8)	0.3(23)	C(5)	C(6)	C(7)	C(3')	170.8(14)
C(6)	C(7)	C(8)	O(1)	167.4(14)	C(6)	C(7)	C(8)	C(8A)	-1.1(23)
C(3')	C(7)	C(8)	O(1)	-2.8(24)	C(3')	C(7)	C(8)	C(8A)	-171.3(14)
C(6)	C(7)	C(3')	C(2')	-86.6(18)	C(6)	C(7)	C(3')	C(4')	94.9(18)
C(8)	C(7)	C(3')	C(2')	83.6(20)	C(8)	C(7)	C(3')	C(4')	-94.9(19)
O(1)	C(8)	C(8A)	C(1)	12.5(21)	O(1)	C(8)	C(8A)	C(4A)	-168.4(14)
C(7)	C(8)	C(8A)	C(1)	-178.6(14)	C(7)	C(8)	C(8A)	C(4A)	0.5(24)
C(8A')	C(1')	C(2')	C(3')	-0.6(24)	C(8A')	C(1')	C(2')	C(14)	178.7(15)
C(2')	C(1')	C(8A')	C(4A')	-0.2(24)	C(2')	C(1')	C(8A')	C(8')	-179.6(16)
C(1')	C(2')	C(3')	C(7)	-176.1(15)	C(1')	C(2')	C(3')	C(4')	2.4(23)
C(14)	C(2')	C(3')	C(7)	4.6(22)	C(14)	C(2')	C(3')	C(4')	-176.9(14)
C(7)	C(3')	C(4')	O(3)	-2.0(20)	C(7)	C(3')	C(4')	C(4A')	175.0(14)
C(2')	C(3')	C(4')	O(3)	179.5(13)	C(2')	C(3')	C(4')	C(4A')	-3.5(22)
O(3)	C(4')	C(4A')	C(5')	-1.4(24)	O(3)	C(4')	C(4A')	C(8A')	179.4(13)
C(3')	C(4')	C(4A')	C(5')	-178.2(15)	C(3')	C(4')	C(4A')	C(8A')	2.6(22)
C(4')	C(4A')	C(5')	O(4)	-0.7(23)	C(4')	C(4A')	C(5')	C(6')	-179.5(17)

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 9.5 (cont.)

C(8A')-	C(4A')-	C(5') -	O(4)	178.7(14)	C(8A')-	C(4A')-	C(5') -	C(6')	-0.3(25)
C(4') -	C(4A')-	C(8A')-	C(1')	-0.8(23)	C(4') -	C(4A')-	C(8A')-	C(8')	178.7(15)
C(5') -	C(4A')-	C(8A')-	C(1')	180.0(15)	C(5') -	C(4A')-	C(8A')-	C(8')	-0.6(22)
O(4) -	C(5') -	C(6') -	C(7')	-177.3(16)	C(4A')-	C(5') -	C(6') -	C(7')	1.5(28)
C(5') -	C(6') -	C(7') -	C(8')	-1.8(29)	C(6') -	C(7') -	C(8') -	C(8A')	1.1(30)
C(7') -	C(8') -	C(8A')-	C(1')	179.6(17)	C(7') -	C(8') -	C(8A')-	C(4A')	0.2(26)

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

was very poor, the intensities of the reflexions being low. A raised temperature factor, which has been discussed in Chapter 7, was required to effect a solution. The analysis does however stress the importance of the difference map in X-ray structure determination. The final R value was high, but this could be attributed directly to the quality of the data. It should be possible to improve upon the results with better crystals.

Structural 9.4.2

Prior to the X-ray structure determination, all the structural features of ancistrotectorine had been identified from the proton nmr spectrum, however the estimation of the structure was incorrect. After the X-ray analysis, the proton nmr spectrum was reassigned and the structure found to correspomd very well to this.

Ancistrotectorine is a 7 - 3' linked naphthalene-isoquinoline alkaloid, being only the second of this type to be isolated. The other 7 - 3'linked alkaloid is Ancistrocladidine [117].

It was found that rings A, B and C are planar and ring D adopts a sofa conformation. Considerable steric hindrance exists about the diaryl linkage, which results in a dihedral angle between the two aromatic rings of 56.4°. This was studied further using molecular mechanics calculations.

Molecular Mechanics 9.4.3

As the programme MM2P [75] could not be used to drive a torsion angle through a series of values, MM2 [74] was used, carrying out molecular mechanics calculations on only the central aromatic rings, as is shown more clearly in Fig. 9.8. It was thought that this could be justified,as very little steric interaction with the other two rings was evident from the Dreiding model.

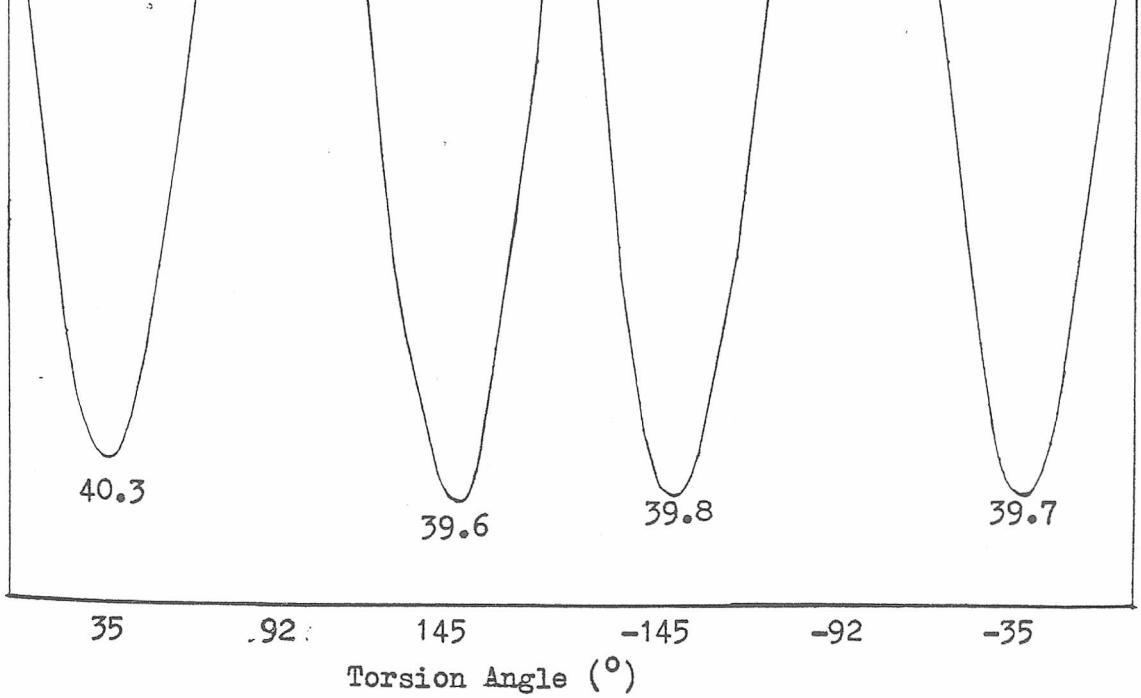
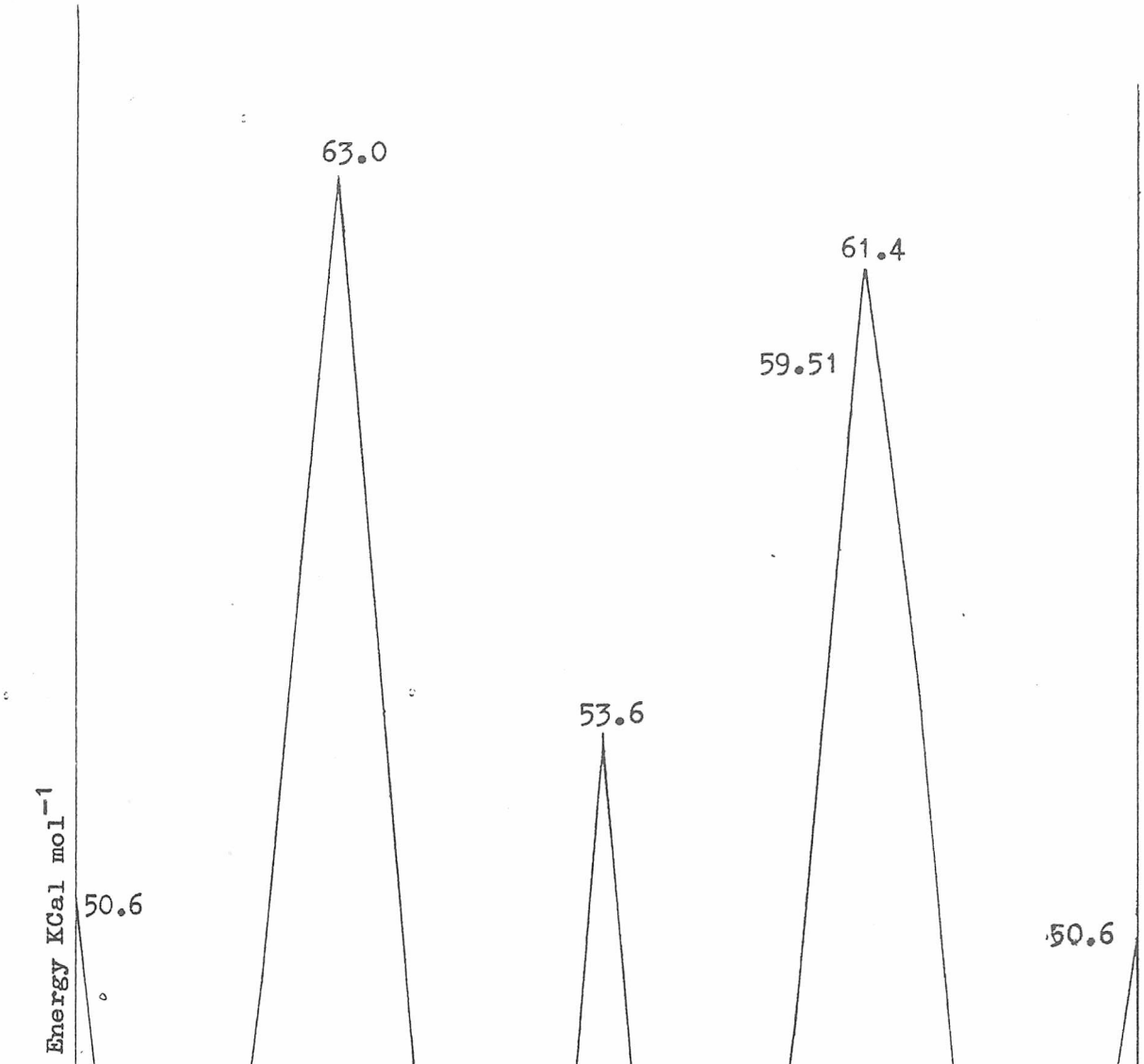
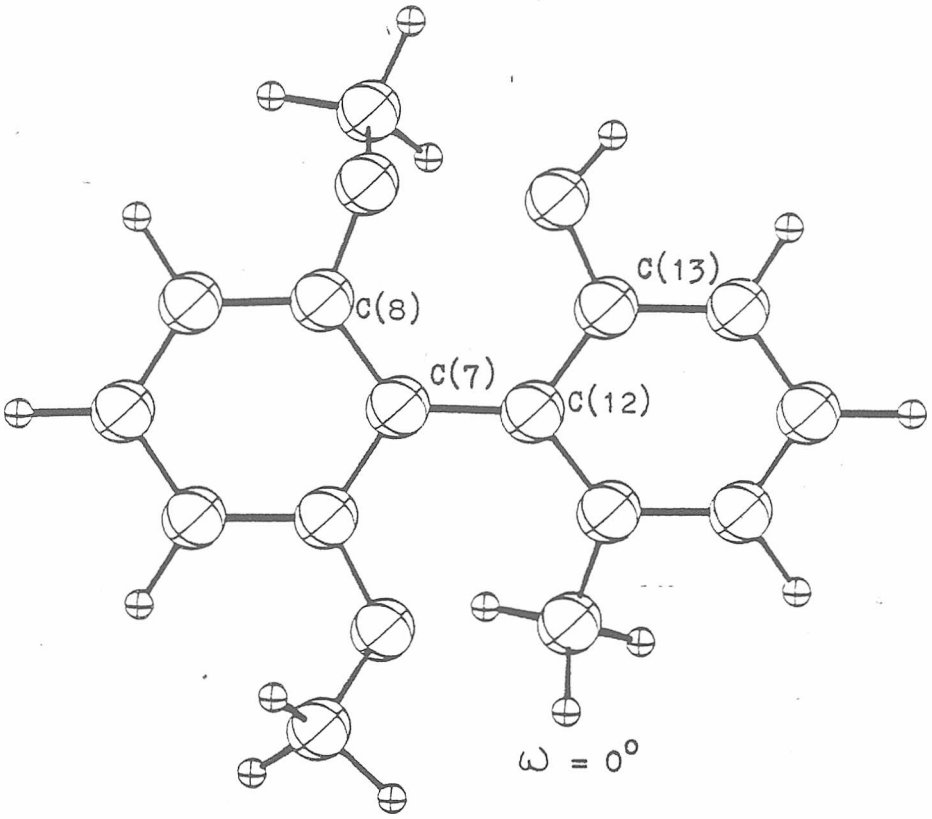


FIG. 9.7


Energy KCal mol^{-1}
50.6
63.0
53.6
59.51
61.4
50.6



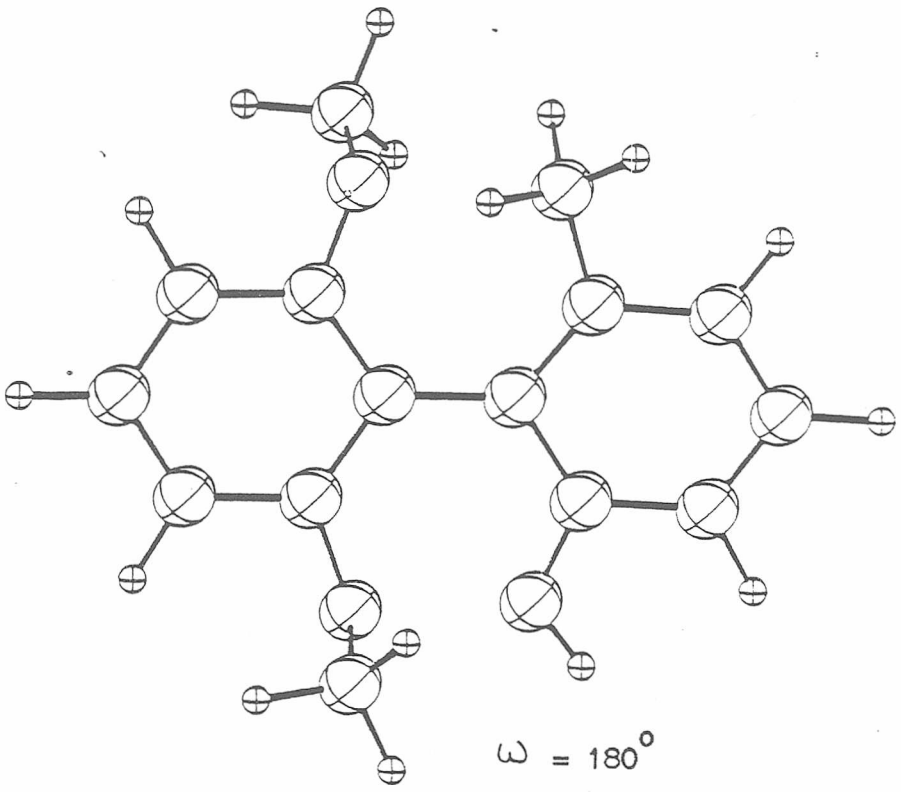
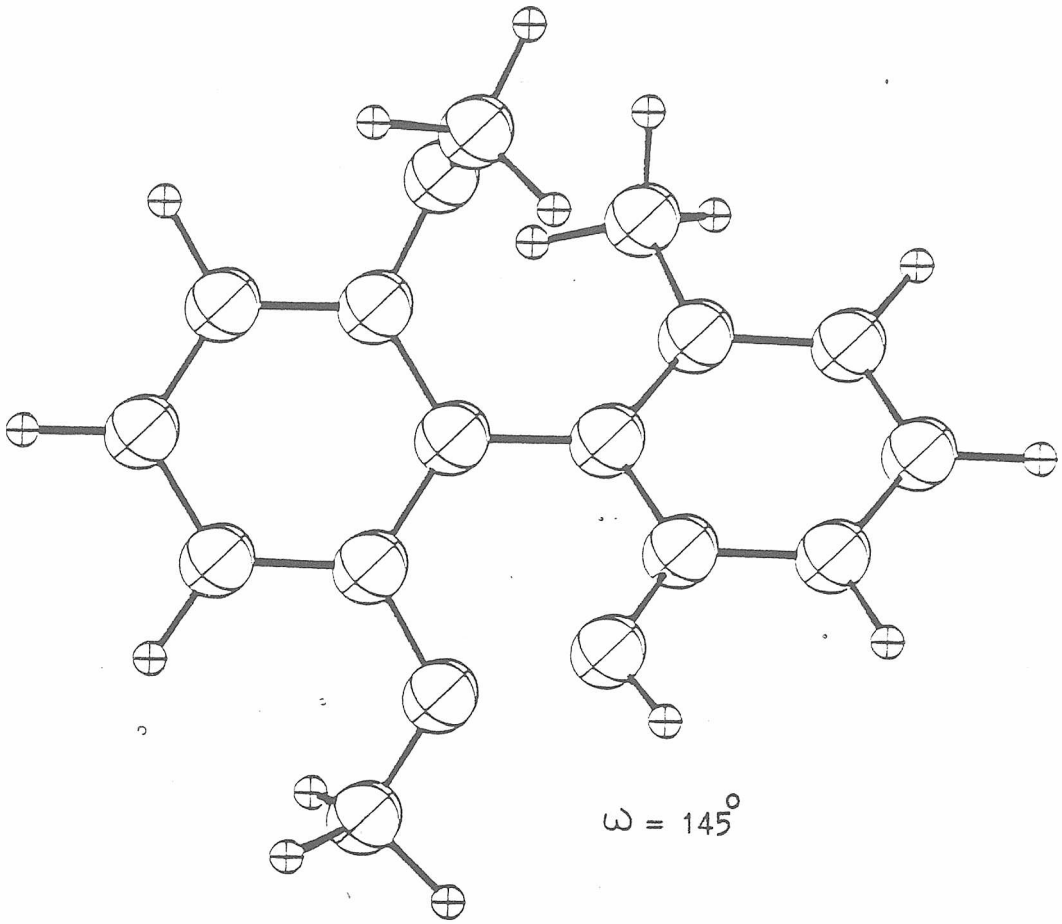


FIG. 9.8



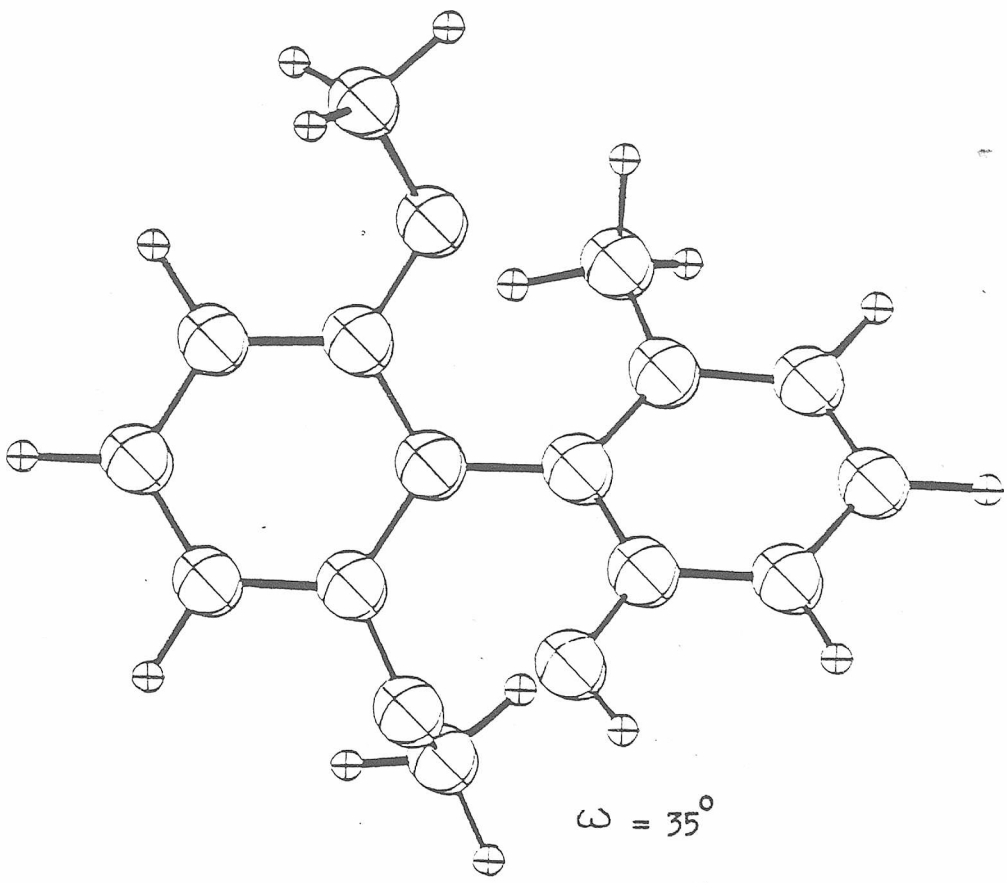


FIG. 9.8 (cont.)

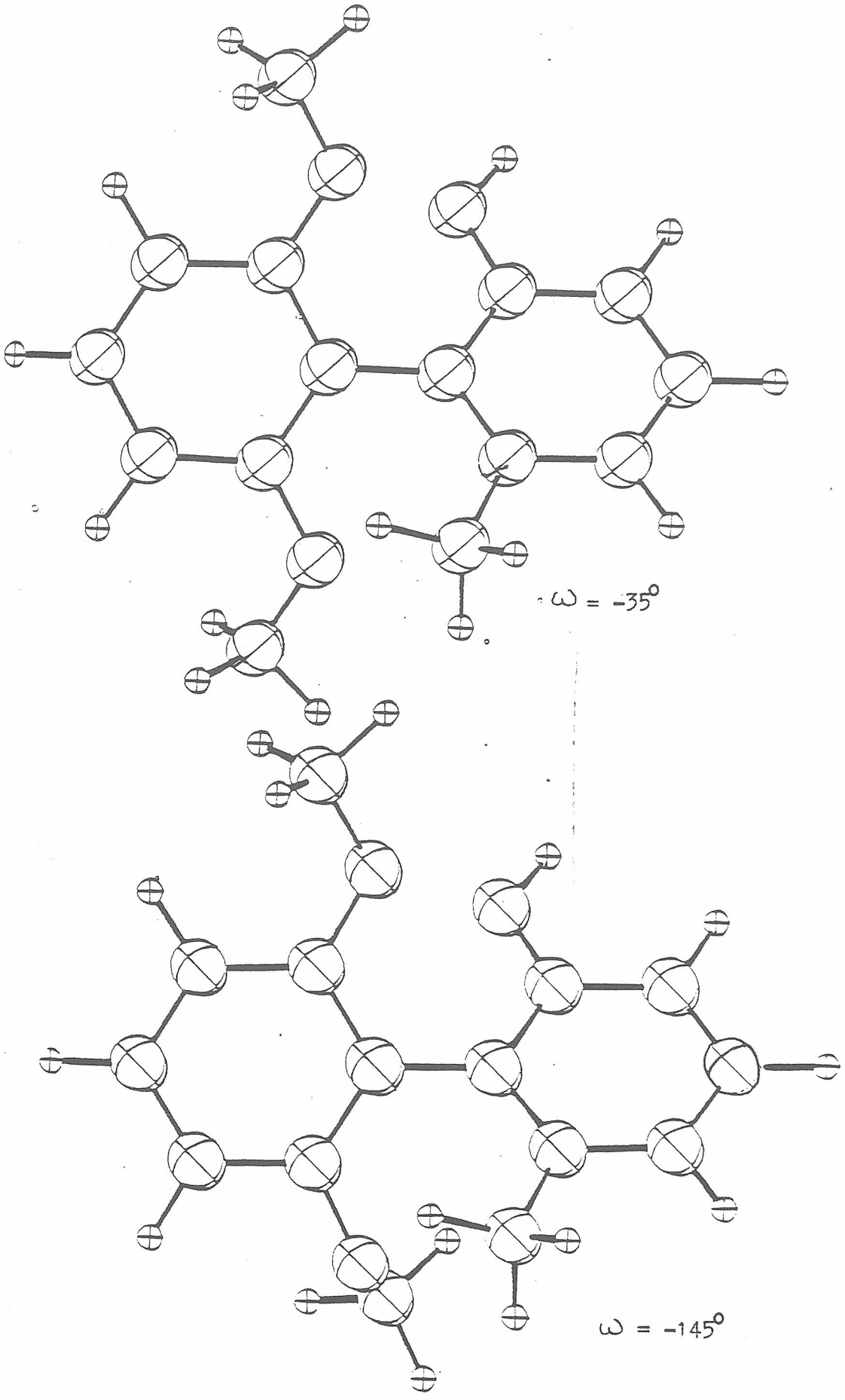


FIG. 9.8 (cont.)

By rotating the torsion angle C(8)-C(7)-C(12)-C(13) the energy profile shown in Fig. 9.7 was obtained. The crystal structure lies part way up a maximum, with a steric energy of 59.51 Kcal mol^{-1}. Perspective drawings for the energy maxima and energy minima are shown in Fig. 9.8. It can be concluded that the crystal structure adopts a high energy due to crystal packing forces. The same situation arises with Biphenyl [118,119,120], where in the crystal the molecule is planar, but in the gas phase it is not. Molecular mechanics calculations on an isolated molecule gave the minimum energy as the gas phase value .

Biosynthesis 9.4.4

A biosynthetic pathway for the naphthalene-isoquinoline [121] alkaloids has been postulated, but this is very general and does not apply exclusively to the 7 - 3'linked substances. This pathway is shown in Fig. 9.9.

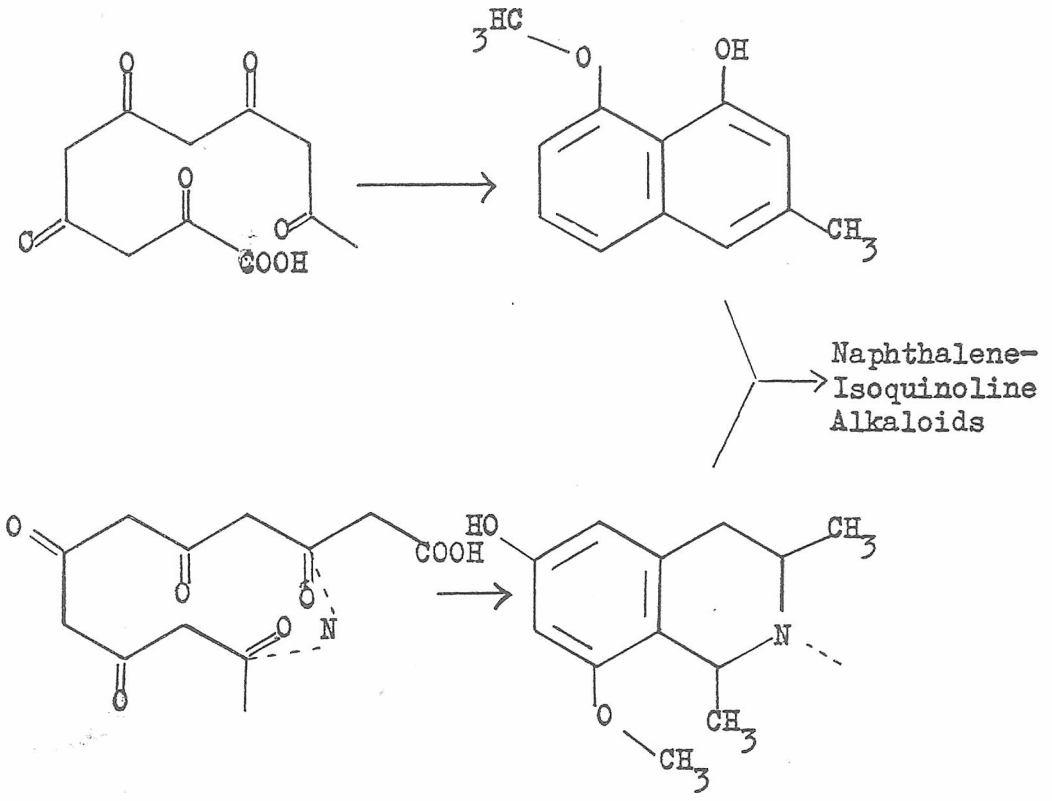

3HC
O
OH
CH3
COOH
Naphthalene-
Isoquinoline
Alkaloids
HO
COOH
CH3
N
CH3
O
CH3

FIG. 9.9

CHAPTER TEN

PERCHLORORUBIAN, A SPIRO-LACTONE FROM RUBIA TINCTORUM

Perchlororubian a Spiro-Lactone from Rubia tinctorum

Introduction 10.1

Perchlororubian (Fig. 10.1) was first produced after chlorination of Chlororubian [122] between 1852 and 1856 by H.E. Schunck [123,124,125]. An attempt was made much later to identify the structure of perchlororubian by spectral methods and degradative studies [122], but with very little success. Later spectral analysis suggested the structures shown in Fig. 10.2, as possibilities for that of perchlororubian. From the mass spectrum, the molecular formula was determined as $C_{14}H_{4}O_{5}Cl_{6}$. An X-ray analysis was undertaken to identify the structure.

Experimental 10.2

A large rectangular crystal was chosen for the X-ray structure determination. Data was collected for 3696 unique reflexions, scanning to $2\theta \leq 60^{\circ}$. From the E map computed for 300 reflexions $|E|_{min} > 1.7$ using 'MULTAN 80' [72], the positions of the six chlorine atoms and one six membered ring were obtained. The coordinates of these were input to 'SHELX' [73] for the calculation of a difference map, which allowed all the non-hydrogen atoms to be identified. The coordinates of all the non-hydrogen atoms were used in 'SHELX' [73] for least squares refinement. The non-hydrogen atoms were given anisotropic temperature factors. The hydrogen atom positions were calculated and their bond lengths then fixed at $1.00\text{Å} \pm 0.02\text{Å}$ and allowed to refine.

The final weighting scheme used was

$$\omega = 11.4358/(\sigma^2(F))$$

Molecular mechanics calculations were carried out using the programme MM2P [75]. Certain parameters had to be redefined,

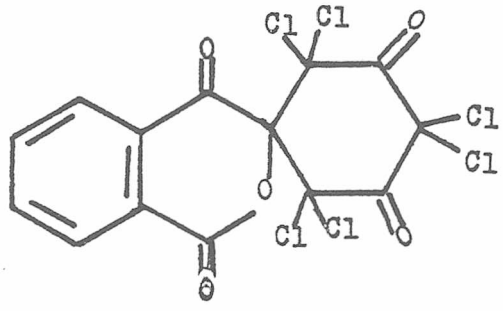
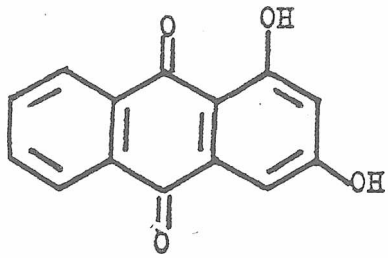
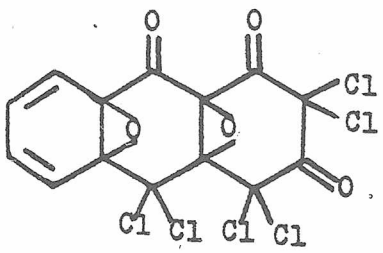
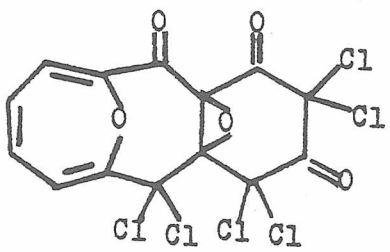


FIG. 10.1


O
OH
OH
O


O
O
Cl
Cl
O
O
Cl
Cl
Cl
Cl


O
O
Cl
Cl
O
O
Cl
Cl
Cl
Cl

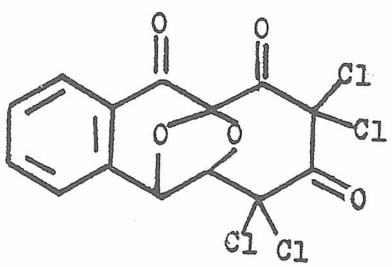

O
O
Cl
Cl
O
O
O
Cl
Cl

FIG. 10.2

these being as shown in Table 10.1

Results 10.3

Crystallographic Data 10.3.1

$C_{14}H_4O_5Cl_6$ Mr =464.900 , Dc = 1.876gcm^{-3}, orthorhombic Pbca Z = 8, a = 11.051(8)Å, b = 11.380(9)Å, c = 26.213(20)Å, U = 3290.6Å^3, μ(MoKα) = 9.75cm^{-1}, F(000) = 1840.00, Final R value = 7.14% for 2970 unique reflexions ($I > 3\sigma(I)$), ω R = 6.22%, maximum height on difference map = 0.633 eÅ^{-3}, minimum height on difference map = -0.620 eÅ^{-3}, maximum shift/error ratio = 0.006.

Structural Data 10.3.2

Perchlororubian has the structure as shown in Fig. 10.1. The atomic arrangement is shown in Fig. 10.3 and Fig. 10.4 shows a stereoscopic view of crystal packing. Tables 10.2 - 10.6 list the structural parameters for perchlororubian. The vibrational spectra are shown in Figs. 10.5 - 10.6, and their major assignments are in Table 10.7.

Molecular mechanics calculations gave a final steric energy of 94.57 K Cal mol^{-1}.

Discussion 10.4

X-ray Analysis 10.4.1

The crystals of perchlororubian were large and an extensive data set was obtained. The chlorine atoms aided the initial analysis as their positions were easily identified from the E map. The esd's associated with the structural parameters are very low, indicating that the results are very reliable.

Structural 10.4.2

It was possible to fully identify the structure using X-ray

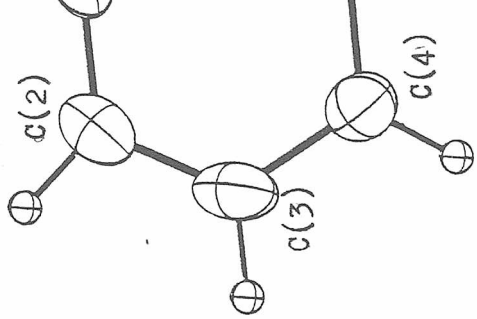
TABLE 10.1

TORSION ANGLES

ATOM TYPES INVOLVED IN TORSION ANGLE	REDEFINED ATOM TYPES
Csp^2-Csp^2-Csp^2(carbonyl)-ether type O	Csp^2-Csp^3-Csp^2(carbonyl)-ether type O
Csp^2-Csp^2-(carbonyl) ether type O-Csp^3	Csp^3-Csp^2(carbonyl)ether type O-Csp^3
Csp^2(carbonyl)-ether type O-Csp^3-Csp^2(carbonyl)	Csp^3-ether type O-Csp^3-Csp^2(carbonyl)
ether type O-Csp^3-Csp^2(carbonyl)-Csp^2	ether type O-Csp^3-Csp^2(carbonyl)-Csp^3
Csp^2(carbonyl)-Csp^3-Csp^3-C1	Csp^3-Csp^3-Csp^3-C1

VALENCY ANGLES

ATOMS INVOLVED IN VALENCY ANGLE	REDEFINED ATOM TYPES	ANGLE (°)
Csp^2-Csp^2(carbonyl)-ether type O	Csp^3-Csp^2(carbonyl)-ether type O	111.00


C(2)
C(3)
C(4)

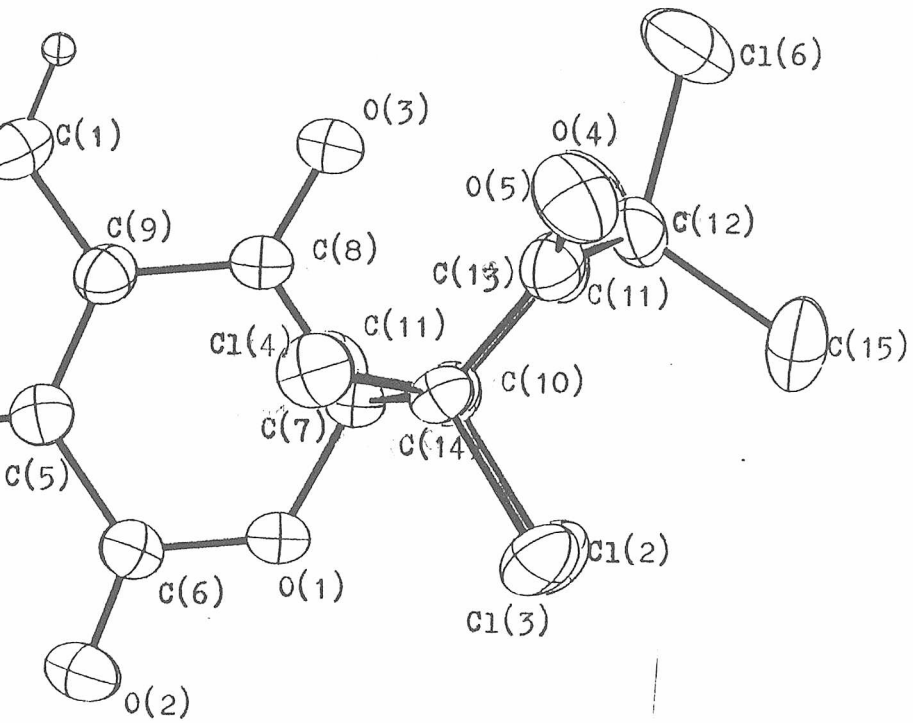

Cl(6)
O(3)
C(1)
O(4)
O(5)
C(9)
C(8)
C(12)
C(13)
C(11)
Cl(4)
C(11)
C(15)
C(10)
C(7)
C(14)
C(5)
Cl(2)
C(6)
O(1)
Cl(3)
O(2)

FIG. 10.3

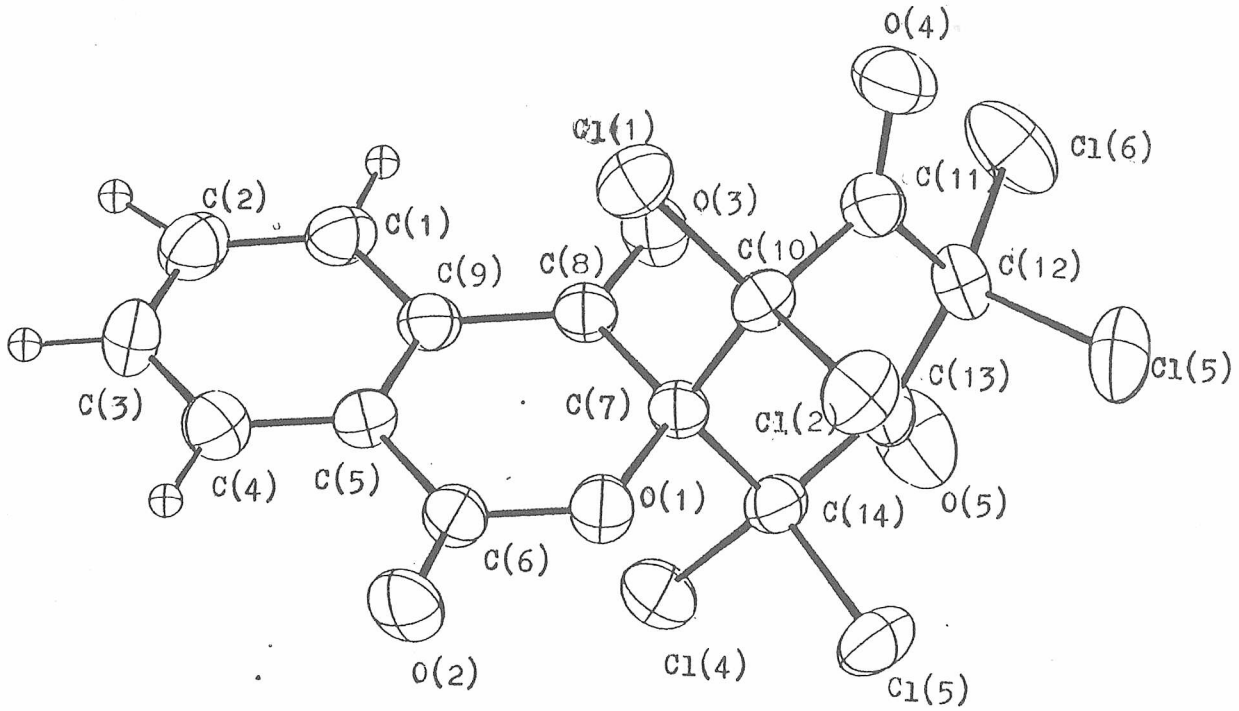
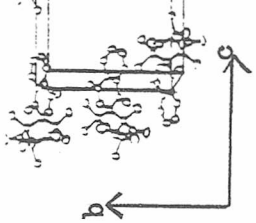

O(4)
Cl(1)
Cl(6)
C(2)
C(1)
O(3)
C(11)
C(9)
C(8)
C(10)
C(12)
C(13)
Cl(5)
C(3)
C(7)
Cl(2)
C(4)
C(5)
O(1)
C(14)
O(5)
C(6)
O(2)
Cl(4)
Cl(5)

FIG. 10.3 (cont.)


c
a

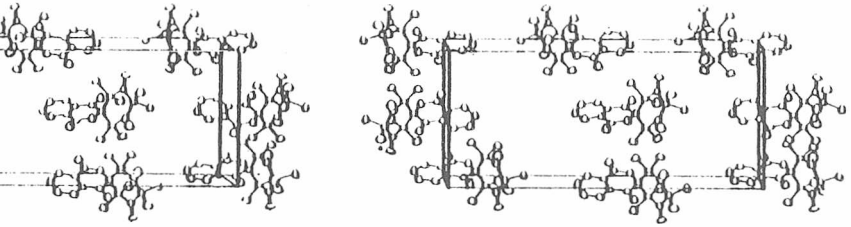


FIG. 10.4

TABLE 10.2

Atom	x	y	z
CL(1)	6956(1)	1100(1)	5936(1)
CL(2)	5421(1)	0402(1)	6760(1)
CL(3)	2880(1)	3569(1)	5950(1)
CL(4)	2938(1)	1887(1)	6763(1)
CL(5)	5176(1)	2718(2)	7596(1)
CL(6)	6449(2)	4683(2)	7147(1)
O(1)	4183(3)	1175(3)	5822(1)
O(2)	3496(4)	0211(3)	5168(2)
O(3)	5913(3)	3843(3)	5919(1)
O(4)	7522(3)	2531(5)	6801(2)
O(5)	3987(4)	4674(4)	6814(2)
C(1)	5751(5)	3722(4)	4819(2)
C(2)	5588(5)	3582(5)	4307(2)
C(3)	4880(6)	2672(5)	4123(2)
C(4)	4370(5)	1849(5)	4450(2)
C(5)	4544(4)	1966(4)	4968(2)
C(6)	4031(4)	1067(4)	5312(2)
C(7)	4791(4)	2132(4)	6062(2)
C(8)	5367(4)	3041(4)	5703(2)
C(9)	5236(4)	2912(4)	5158(2)
C(10)	5866(4)	1610(4)	6377(2)
C(11)	6465(5)	2527(5)	6734(2)
C(12)	5630(5)	3396(5)	7014(2)
C(13)	4434(5)	3749(4)	6739(2)
C(14)	3847(4)	2825(4)	6380(2)
H(1)	6187(56)	4428(40)	4942(26)
H(2)	5868(54)	4045(49)	4015(17)
H(3)	4754(43)	2571(45)	3754(8)
H(4)	3999(51)	1147(36)	4294(23)

Fractional Atomic Coordinates ($\times 10^4$) with e.s.d.'s

TABLE 10.2

Atom	x	y	z
CL(1)	6956(1)	1100(1)	5936(1)
CL(2)	5421(1)	0402(1)	6760(1)
CL(3)	2880(1)	3569(1)	5950(1)
CL(4)	2938(1)	1887(1)	6763(1)
CL(5)	5176(1)	2718(2)	7596(1)
CL(6)	6449(2)	4683(2)	7147(1)
O(1)	4183(3)	1175(3)	5822(1)
O(2)	3496(4)	0211(3)	5168(2)
O(3)	5913(3)	3843(3)	5919(1)
O(4)	7522(3)	2531(5)	6801(2)
O(5)	3987(4)	4674(4)	6814(2)
C(1)	5751(5)	3722(4)	4819(2)
C(2)	5588(5)	3582(5)	4307(2)
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C(4)	4370(5)	1849(5)	4450(2)
C(5)	4544(4)	1966(4)	4968(2)
C(6)	4031(4)	1067(4)	5312(2)
C(7)	4791(4)	2132(4)	6062(2)
C(8)	5367(4)	3041(4)	5703(2)
C(9)	5236(4)	2912(4)	5158(2)
C(10)	5866(4)	1610(4)	6377(2)
C(11)	6465(5)	2527(5)	6734(2)
C(12)	5630(5)	3396(5)	7014(2)
C(13)	4434(5)	3749(4)	6739(2)
C(14)	3847(4)	2825(4)	6380(2)
H(1)	6187(56)	4428(40)	4942(26)
H(2)	5868(54)	4045(49)	4015(17)
H(3)	4754(43)	2571(45)	3754(8)
H(4)	3999(51)	1147(36)	4294(23)

Fractional Atomic Coordinates ($\times 10^4$) with e.s.d.'s

TABLE 10.2

Atom	x	y	z
CL(1)	6956(1)	1100(1)	5936(1)
CL(2)	5421(1)	0402(1)	6760(1)
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CL(4)	2938(1)	1887(1)	6763(1)
CL(5)	5176(1)	2718(2)	7596(1)
CL(6)	6449(2)	4683(2)	7147(1)
O(1)	4183(3)	1175(3)	5822(1)
O(2)	3496(4)	0211(3)	5168(2)
O(3)	5913(3)	3843(3)	5919(1)
O(4)	7522(3)	2531(5)	6801(2)
O(5)	3987(4)	4674(4)	6814(2)
C(1)	5751(5)	3722(4)	4819(2)
C(2)	5588(5)	3582(5)	4307(2)
C(3)	4880(6)	2672(5)	4123(2)
C(4)	4370(5)	1849(5)	4450(2)
C(5)	4544(4)	1966(4)	4968(2)
C(6)	4031(4)	1067(4)	5312(2)
C(7)	4791(4)	2132(4)	6062(2)
C(8)	5367(4)	3041(4)	5703(2)
C(9)	5236(4)	2912(4)	5158(2)
C(10)	5866(4)	1610(4)	6377(2)
C(11)	6465(5)	2527(5)	6734(2)
C(12)	5630(5)	3396(5)	7014(2)
C(13)	4434(5)	3749(4)	6739(2)
C(14)	3847(4)	2825(4)	6380(2)
H(1)	6187(56)	4428(40)	4942(26)
H(2)	5868(54)	4045(49)	4015(17)
H(3)	4754(43)	2571(45)	3754(8)
H(4)	3999(51)	1147(36)	4294(23)

Fractional Atomic Coordinates ($\times 10^4$) with e.s.d.'s

TABLE 10.3

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
CL(1)	46(1)	46(1)	51(1)	7(1)	8(1)	-9(1)
CL(2)	62(1)	44(1)	53(1)	4(1)	1(1)	18(1)
CL(3)	38(1)	50(1)	55(1)	3(1)	-8(1)	8(1)
CL(4)	39(1)	59(1)	54(1)	-8(1)	10(1)	12(1)
CL(5)	63(1)	84(1)	33(1)	3(1)	4(1)	4(1)
CL(6)	58(1)	67(1)	78(1)	-20(1)	6(1)	-34(1)
O(1)	51(2)	33(2)	37(2)	-17(1)	-2(1)	1(1)
O(2)	56(2)	38(2)	53(2)	-15(2)	0(2)	-8(2)
O(3)	51(2)	38(2)	41(2)	-17(2)	1(2)	-3(1)
O(4)	29(2)	91(3)	70(3)	2(2)	-11(2)	-27(2)
O(5)	70(3)	47(2)	68(3)	18(2)	-9(2)	-14(2)
C(1)	43(3)	31(2)	45(3)	0(2)	9(2)	6(2)
C(2)	53(3)	44(3)	43(3)	7(3)	11(2)	10(2)
C(3)	66(3)	56(3)	30(2)	7(3)	-3(2)	4(2)
C(4)	49(3)	42(3)	43(3)	1(2)	-4(2)	-3(2)
C(5)	33(2)	32(2)	37(2)	2(2)	-1(2)	2(2)
C(6)	35(2)	33(2)	38(2)	-4(2)	-1(2)	-2(2)
C(7)	38(2)	26(2)	33(2)	-7(2)	0(2)	0(2)
C(8)	38(3)	28(2)	36(2)	-6(2)	0(2)	0(2)
C(9)	39(3)	29(2)	36(2)	1(2)	4(2)	1(2)
C(10)	36(3)	33(2)	36(2)	4(2)	0(2)	3(2)
C(11)	40(3)	50(3)	30(2)	3(2)	1(2)	-4(2)
C(12)	40(3)	48(3)	31(2)	-4(2)	3(2)	-8(2)
C(13)	48(3)	37(2)	37(2)	2(2)	6(2)	0(2)
C(14)	29(2)	34(2)	37(2)	-3(2)	3(2)	5(2)
H(1)	69(20)					
H(2)	60(18)					
H(3)	32(12)					
H(4)	57(17)					

Anisotropic Temperature Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 10.4

CL(1) - C(10)	1.768(5)	C(1) - C(9)	1.401(7)
CL(2) - C(10)	1.772(5)	C(2) - C(3)	1.385(8)
CL(3) - C(14)	1.769(5)	C(3) - C(4)	1.389(8)
CL(4) - C(14)	1.777(5)	C(4) - C(5)	1.378(7)
CL(5) - C(12)	1.782(5)	C(5) - C(6)	1.477(6)
CL(6) - C(12)	1.757(5)	C(5) - C(9)	1.411(6)
O(1) - C(6)	1.353(6)	C(7) - C(8)	1.536(6)
O(1) - C(7)	1.426(5)	C(7) - C(10)	1.564(6)
O(2) - C(6)	1.200(6)	C(7) - C(14)	1.551(6)
O(3) - C(8)	1.232(6)	C(8) - C(9)	1.443(6)
O(4) - C(11)	1.181(6)	C(10) - C(11)	1.550(7)
O(5) - C(13)	1.179(6)	C(11) - C(12)	1.539(7)
C(1) - C(2)	1.363(8)	C(12) - C(13)	1.558(7)
C(13) - C(14)	1.553(7)		

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

CL(2) - C(10) - CL(1)	107.8(2)
C(11) - C(10) - CL(1)	109.0(3)
C(11) - C(10) - CL(2)	107.4(3)
C(7) - C(14) - CL(3)	107.9(3)
C(7) - C(14) - CL(4)	112.2(3)
CL(6) - C(12) - CL(5)	109.6(3)
C(13) - C(12) - CL(5)	105.6(3)
C(13) - C(12) - CL(6)	108.3(3)
O(2) - C(6) - O(1)	116.5(4)
C(8) - C(7) - O(1)	116.1(4)
C(14) - C(7) - O(1)	108.0(4)
C(7) - C(8) - O(3)	114.8(4)
C(10) - C(11) - O(4)	121.0(5)
C(12) - C(13) - O(5)	120.6(5)
C(9) - C(1) - C(2)	119.6(5)
C(5) - C(9) - C(1)	119.9(4)
C(4) - C(3) - C(2)	121.2(5)
C(6) - C(5) - C(4)	118.8(4)
C(9) - C(5) - C(6)	121.4(4)
C(10) - C(7) - C(8)	105.3(4)
C(9) - C(8) - C(7)	119.7(4)
C(11) - C(10) - C(7)	112.8(4)
C(12) - C(11) - C(10)	117.7(4)
C(14) - C(13) - C(12)	117.4(4)

Valency Angles (°)

TABLE 10.5

C(7) - C(10) - CL(1)	107.3(3)
C(7) - C(10) - CL(2)	112.5(3)
CL(4) - C(14) - CL(3)	107.8(2)
C(13) - C(14) - CL(3)	108.3(3)
C(13) - C(14) - CL(4)	107.5(3)
C(11) - C(12) - CL(5)	107.4(3)
C(11) - C(12) - CL(6)	108.8(4)
C(7) - O(1) - C(6)	124.3(4)
C(5) - C(6) - O(1)	119.5(4)
C(10) - C(7) - O(1)	107.5(3)
C(5) - C(6) - O(2)	124.0(4)
C(9) - C(8) - O(3)	125.4(4)
C(12) - C(11) - O(4)	121.3(5)
C(14) - C(13) - O(5)	122.0(5)
C(3) - C(2) - C(1)	120.3(5)
C(8) - C(9) - C(1)	121.3(4)
C(5) - C(4) - C(3)	119.1(5)
C(9) - C(5) - C(4)	119.8(4)
C(8) - C(9) - C(5)	118.8(4)
C(14) - C(7) - C(8)	105.4(3)
C(14) - C(7) - C(10)	114.9(4)
C(13) - C(14) - C(7)	112.9(4)
C(13) - C(12) - C(11)	117.0(4)

(Non Hydrogen Atoms) with e.s.d.'s

TABLE 10.6

C(7)	O(1)	C(6)	O(2)	-179.8(4)	C(7)	O(1)	C(6)	C(5)	1.7(6)
C(6)	O(1)	C(7)	C(8)	-4.0(6)	C(6)	O(1)	C(7)	C(10)	-121.5(4)
C(6)	O(1)	C(7)	C(14)	114.0(4)	C(9)	C(1)	C(2)	C(3)	2.5(8)
C(2)	C(1)	C(9)	C(5)	-0.6(7)	C(2)	C(1)	C(9)	C(8)	-178.8(5)
C(1)	C(2)	C(3)	C(4)	-3.2(9)	C(2)	C(3)	C(4)	C(5)	2.0(8)
C(3)	C(4)	C(5)	C(6)	-178.1(5)	C(3)	C(4)	C(5)	C(9)	-0.2(7)
C(4)	C(5)	C(6)	O(1)	-179.3(4)	C(4)	C(5)	C(6)	O(2)	2.3(7)
C(9)	C(5)	C(6)	O(1)	2.8(7)	C(9)	C(5)	C(6)	O(2)	-175.6(5)
C(4)	C(5)	C(9)	C(1)	-0.5(7)	C(4)	C(5)	C(9)	C(8)	177.7(4)
C(6)	C(5)	C(9)	C(1)	177.4(4)	C(6)	C(5)	C(9)	C(8)	-4.4(7)
O(1)	C(7)	C(8)	O(3)	-178.2(4)	O(1)	C(7)	C(8)	C(9)	2.1(6)
C(10)	C(7)	C(8)	O(3)	-59.5(5)	C(10)	C(7)	C(8)	C(9)	120.8(4)
C(14)	C(7)	C(8)	O(3)	62.4(5)	C(14)	C(7)	C(8)	C(9)	-117.3(4)
O(1)	C(7)	C(10)	CL(1)	70.3(4)	O(1)	C(7)	C(10)	CL(2)	-48.1(4)
O(1)	C(7)	C(10)	C(11)	-169.8(4)	C(8)	C(7)	C(10)	CL(1)	-54.1(4)
C(8)	C(7)	C(10)	CL(2)	-172.4(3)	C(8)	C(7)	C(10)	C(11)	65.9(5)
C(14)	C(7)	C(10)	CL(1)	-169.6(3)	C(14)	C(7)	C(10)	CL(2)	72.1(4)
C(14)	C(7)	C(10)	C(11)	-49.6(5)	O(1)	C(7)	C(14)	CL(3)	-71.0(4)
O(1)	C(7)	C(14)	CL(4)	47.7(4)	O(1)	C(7)	C(14)	C(13)	169.4(4)
C(8)	C(7)	C(14)	CL(3)	53.6(4)	C(8)	C(7)	C(14)	CL(4)	172.3(3)
C(8)	C(7)	C(14)	C(13)	-66.0(5)	C(10)	C(7)	C(14)	CL(3)	169.1(3)
C(10)	C(7)	C(14)	CL(4)	-72.2(4)	C(10)	C(7)	C(14)	C(13)	49.4(5)
O(3)	C(8)	C(9)	C(1)	0.5(7)	O(3)	C(8)	C(9)	C(5)	-177.7(4)
C(7)	C(8)	C(9)	C(1)	-179.9(4)	C(7)	C(8)	C(9)	C(5)	2.0(6)
CL(1)	C(10)	C(11)	O(4)	-23.7(6)	CL(1)	C(10)	C(11)	C(12)	158.8(4)
CL(2)	C(10)	C(11)	O(4)	92.8(5)	CL(2)	C(10)	C(11)	C(12)	-84.8(5)
C(7)	C(10)	C(11)	O(4)	-142.7(5)	C(7)	C(10)	C(11)	C(12)	39.8(6)
O(4)	C(11)	C(12)	CL(5)	-89.8(5)	O(4)	C(11)	C(12)	CL(6)	28.8(6)
O(4)	C(11)	C(12)	C(13)	151.8(5)	C(10)	C(11)	C(12)	CL(5)	87.7(4)
C(10)	C(11)	C(12)	CL(6)	-153.7(4)	C(10)	C(11)	C(12)	C(13)	-30.7(6)
CL(5)	C(12)	C(13)	O(5)	89.3(5)	CL(5)	C(12)	C(13)	C(14)	-88.9(4)
CL(6)	C(12)	C(13)	O(5)	-28.1(6)	CL(6)	C(12)	C(13)	C(14)	153.7(4)

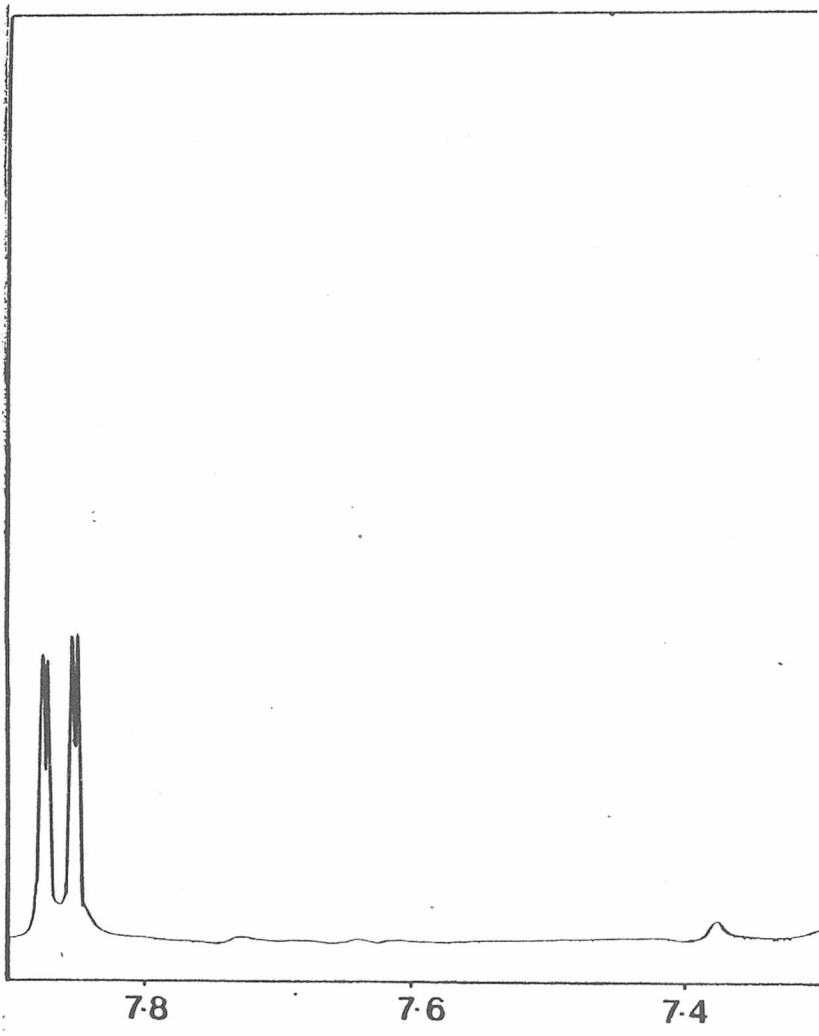
Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 10.6 (cont.)

C(11) - C(12) - C(13) - O(5)	-151.4(5)	C(11) - C(12) - C(13) - C(14)	30.4(6)
O(5) - C(13) - C(14) - CL(3)	23.0(6)	O(5) - C(13) - C(14) - CL(4)	-93.3(5)
O(5) - C(13) - C(14) - C(7)	142.4(5)	C(12) - C(13) - C(14) - CL(3)	-158.8(4)
C(12) - C(13) - C(14) - CL(4)	84.9(4)	C(12) - C(13) - C(14) - C(7)	-39.4(6)

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

^{1}H nmr Spectrum



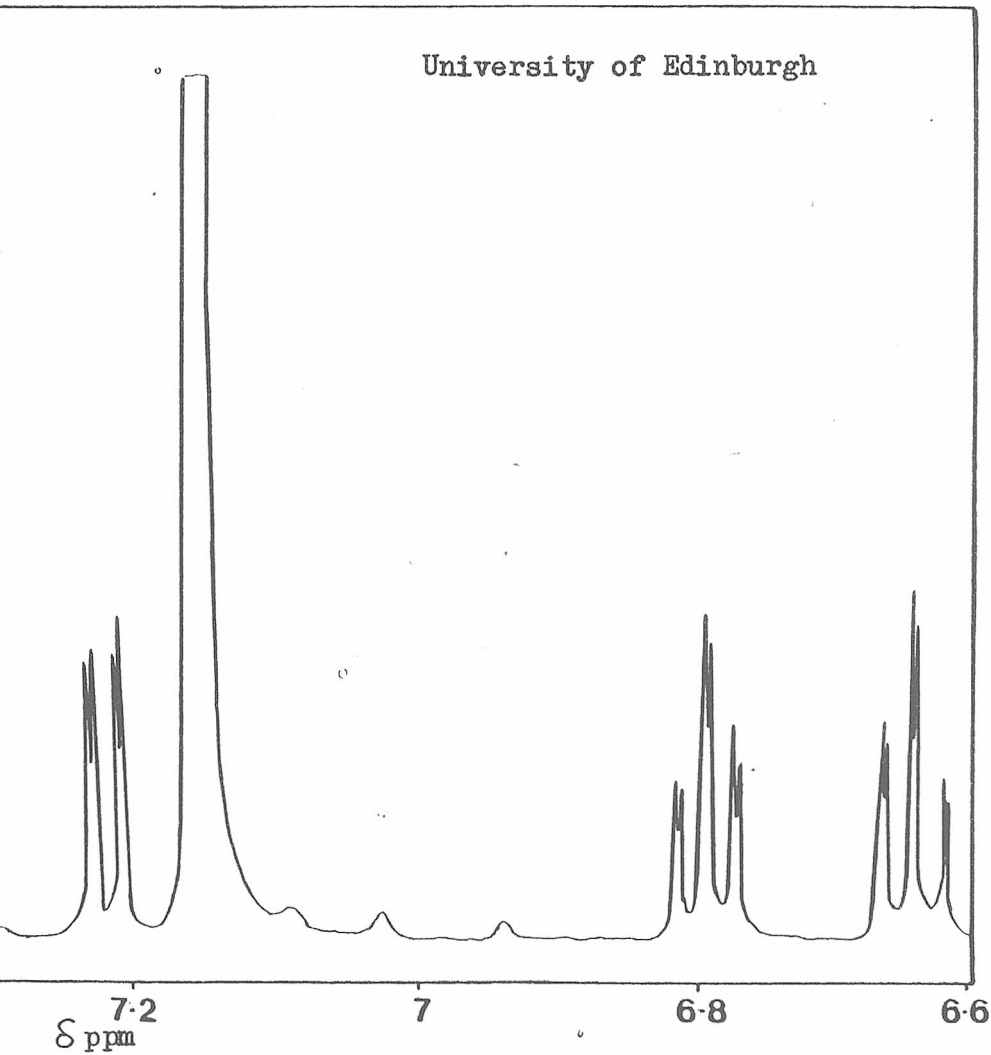


FIG. 10.5

Infra Red Spectrum

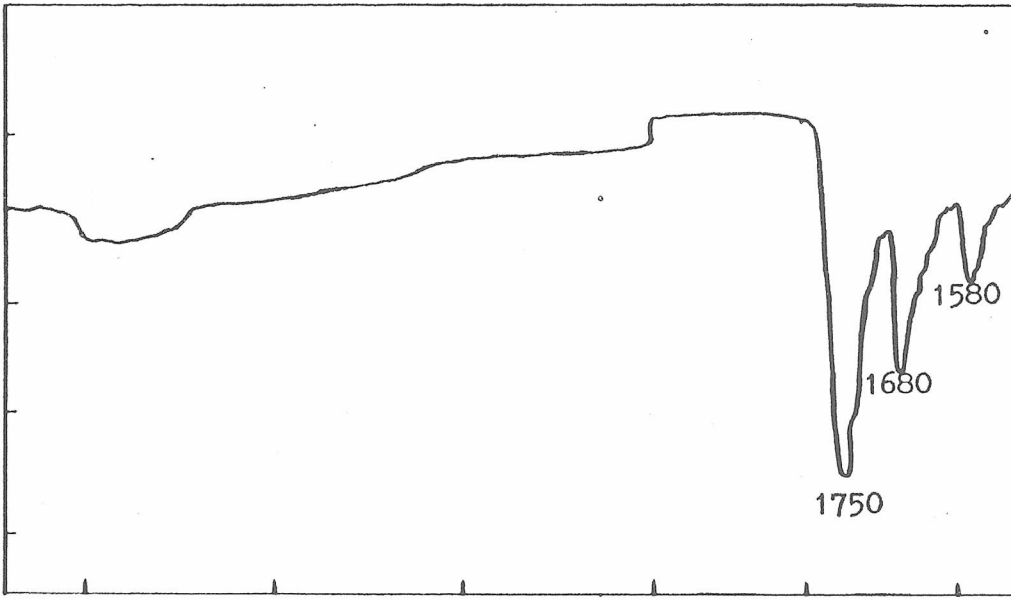


FIG. 10.6

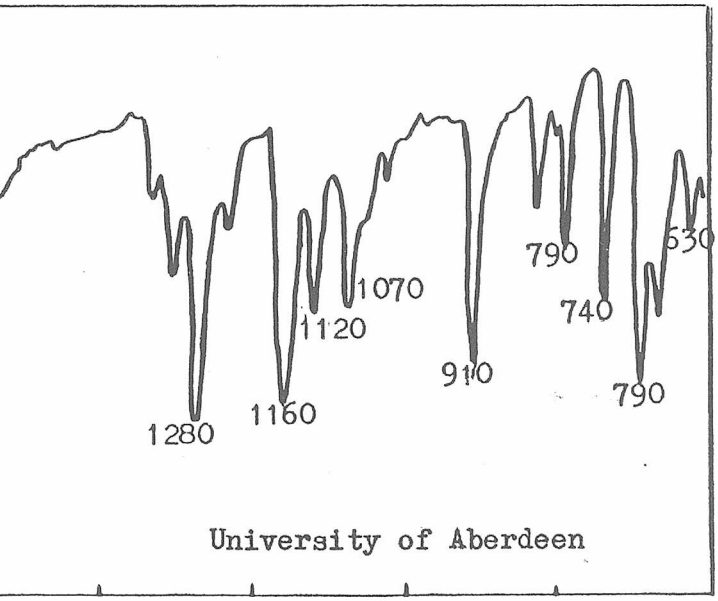

1280
1160
1120
1070
910
790
740
790
530
University of Aberdeen

TABLE 10.7

Major Spectral Assignments for Perchlororubian

IR ν max	630cm^{-1}		
	690cm^{-1}		Chlorine atoms
	740cm^{-1}		
	790cm^{-1}		
	1070 - 1160cm^{-1}	-	ether function
	1240 - 1330cm^{-1}	-	carbonyl function
	1580cm^{-1}	-	aromatic function
	1680cm^{-1}	-	carbonyl function
UV λ max	226 nm	-	conjugated system
	277 nm	-	carbonyl functions
^{1}H nmr 360MHz	δ 6.6 td		
	δ 6.79 td	-	C(2), C(3)
Solvent C_6D_6	δ 7.15 s	-	Solvent
	δ 7.22 ddd		
	δ 7.85 ddd	-	C(1), C(4)

diffraction data. The failure to characterise the structure from the spectra was probably due to the limited amount of information available from them.

Both ring A and ring B are planar and ring C adopts a chair conformation. Due to the conformation of ring C, there is little interaction between the chlorine and the oxygen atoms. Two similar structures to perchlororubian, a spirocyclic chroman [126], and a nitroxyl biradical [127], both have rings A and B planar, but they differ in ring C. In the spirocyclic chroman, ring C adopts an envelope conformation, and in the nitroxyl biradical it adopts a half chair. However, the differences in substituents on ring C are sufficient to explain the conformations.

Molecular Mechanics 10.4.3

Molecular mechanics calculations were carried out using the programme MM2P which is suitable for use with conjugated structures. The steric energy for perchlororubian is high. It was noted that the energies of the non-bonded interactions between the chlorines and oxygens are high, suggesting these are closer than would be expected in the free state. Comparison of the coordinates of the molecule before and after minimisation showed that very little movement of the coordinates took place during minimisation.

CHAPTER ELEVEN

ZOAPATANOLIDE B, A SESQUITERPENE LACTONE FROM MONTANOA TOMENTOSA

Zoapatanolide B, a Sesquiterpene Lactone from Montanoa tomentosa

Introduction 11.1

Zoapatanolide B (Fig. 11.1) is a germacranolide sesquiterpene lactone from the leaves of the zoapatle plant Montanoa tomentosa. The structure was already known [128], but the spectral data did not fully support the stereochemistry as it had been proposed. It was hoped that X-ray analysis would clarify this situation.

Experimental 11.2

Data were collected according to the procedure in Chapter 2. The intensities of 2619 reflexions were measured ($2\theta \leq 50^{\circ}$). The structure was solved using the direct methods programme 'MITHRIL' [41]. Two molecules were clearly visible from the E map, (these being shown in Fig. 11.2), which was computed for 800 reflexions $|E|_{min} > 1.00$. Refinement proceeded by full matrix least squares analysis [73]. Anisotropic temperature factors were input for all non-hydrogen atoms. The hydrogen atom positions were calculated, the methylene hydrogen positions being calculated using the programme 'BONDLA' [77]. The hydroxy hydrogen positions were obtained by examining the low angle reflexions. The methyl and hydroxy hydrogen atoms were given common temperature factors of 0.1 and the remaining hydrogen atoms common temperature factors of 0.05. The hydrogen atom bond lengths were fixed at $1.00\text{Å} \pm 0.02\text{Å}$, and the methyl hydrogen separation at $1.63\text{Å} \pm 0.02\text{Å}$. The hydrogen atom positions were allowed to refine.

The final weighting scheme used was

$$\omega = 1.00/\ (\sigma^2(F))$$

Molecular mechanics calculations were carried out using the programme MM2 [74]. Some of the parameters had to be redefined, and these are shown in Table 11.1.

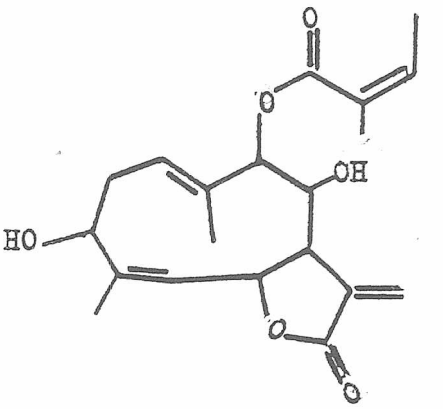


FIG. 11.1

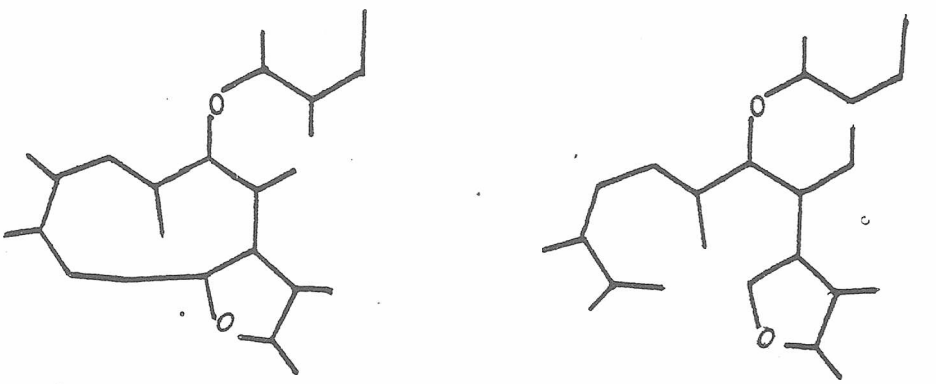


FIG. 11.2

TABLE 11.1

TORSION ANGLES

ATOM TYPES INVOLVED IN TORSION ANGLE	REDEFINED ATOM TYPES
Csp^2-Csp^2-Csp^2(carbonyl)-ether type O	Csp^2-Csp^3-Csp^2(carbonyl)-ether type O
Csp^2-Csp^2(carbonyl)-ether type O-Csp^3	Csp^3-Csp^2(carbonyl)ether type O-Csp^3
Csp^2-Csp^3-ether type O-Csp^2(carbonyl)	
ether type O-Csp^2(carbonyl-Csp^2-Csp^3	

VALENCY ANGLES

ATOMS INVOLVED IN VALENCY ANGLE	REDEFINED ATOM TYPES	ANGLE (°)
Csp^2-Csp^2(carbonyl)-ether type O	Csp^3-Csp^2(carbonyl)-ether type O	111.00

Results 11.3

Crystallographic Data 11.3.1

$C_{20}H_{26}O_6$, Mr = 362.421, Dc = 1.19gcm^{-3}, monoclinic, $P2_1$, Z = 4, a = 10.117(5)Å, b = 16.426(22)Å, c = 14.945(6)Å, β = 125.63(5)°, U = 2018.65Å^3, μ(MoKα) = 0.52cm^{-1}, F(000) = 776.00, Final R value = 5.13% for 2619 unique reflexions ($I > 3\sigma(I)$), ωR = 4.16%, maximum height on difference map = 0.191 eÅ^{-3}, minimum height on difference map = -0.177 eÅ^{-3}, maximum shift/error ratio = 0.187.

Structural Data 11.3.2

The structure of zoapatanolide B is shown in Fig. 11.1. Perspective drawings of molecules 1 and 2 are shown in Fig. 11.3. A stereoscopic view of crystal packing is shown in Fig. 11.4. Structural parameters are listed in Tables 11.2 - 11.6.

Molecular mechanics calculations gave the following steric energies for molecules 1 and 2.

Molecule 1 = 21.1385 KCal mol^{-1} Molecule 2 = 21.2964 KCal mol^{-1}

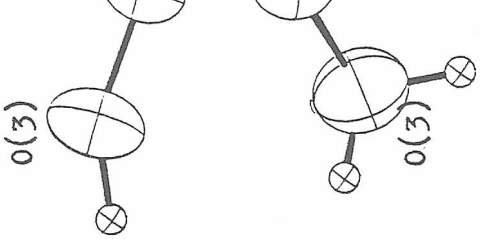
Zoapatanolide B was found to possess hydrogen bonding.

A - - - D		A - - - H	
O(6) - - - O(3A)	2.832(5)Å	O(6) - - - HO(3A)	1.847(64)Å
O(6) - - - HO(3A)		O(3A)	156.5(64)Å

Discussion 11.4

X-ray Analysis 11.4.1

A few problems were encountered during the refinement stage of the analysis. From the geometries obtained after the first four cycles of least squares, it appeared that one of the molecules had the structure of zoapatanolide A and one of the structure zoapatanolide B, however this was resolved after a few more cycles of least squares. The molecules exhibited a difference in the


O(3)
O(3)

MOLECULE 1

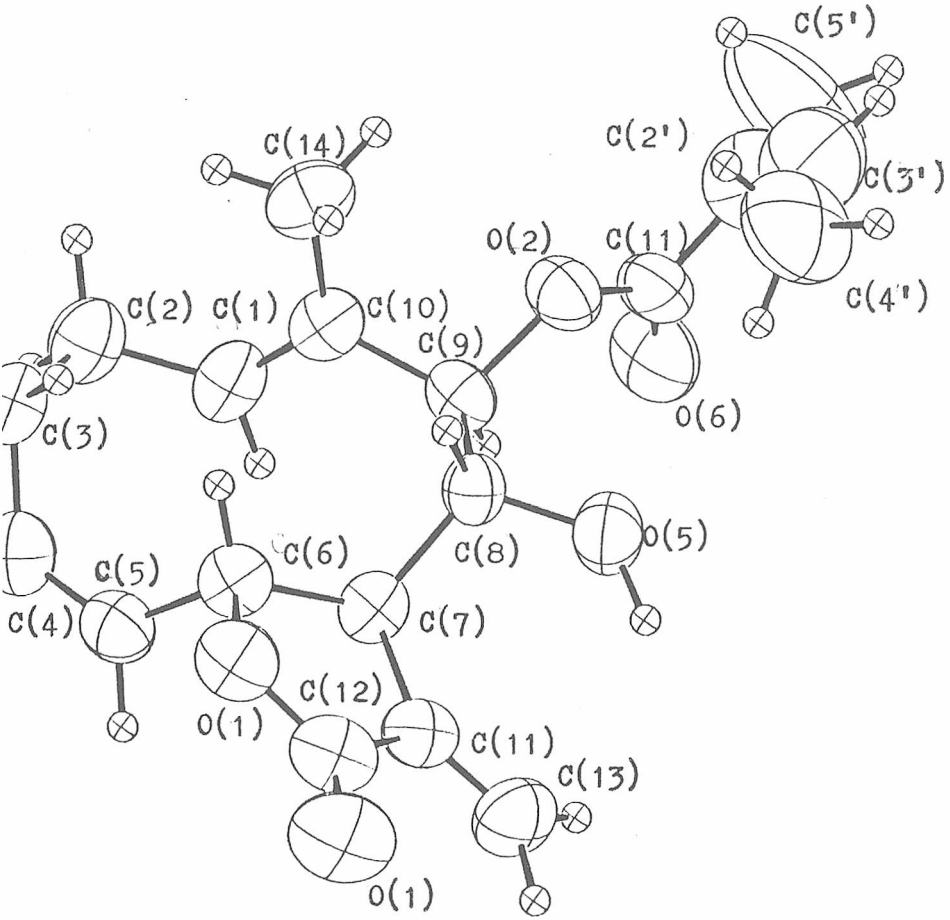


FIG. 11.3

MOLECULE 2

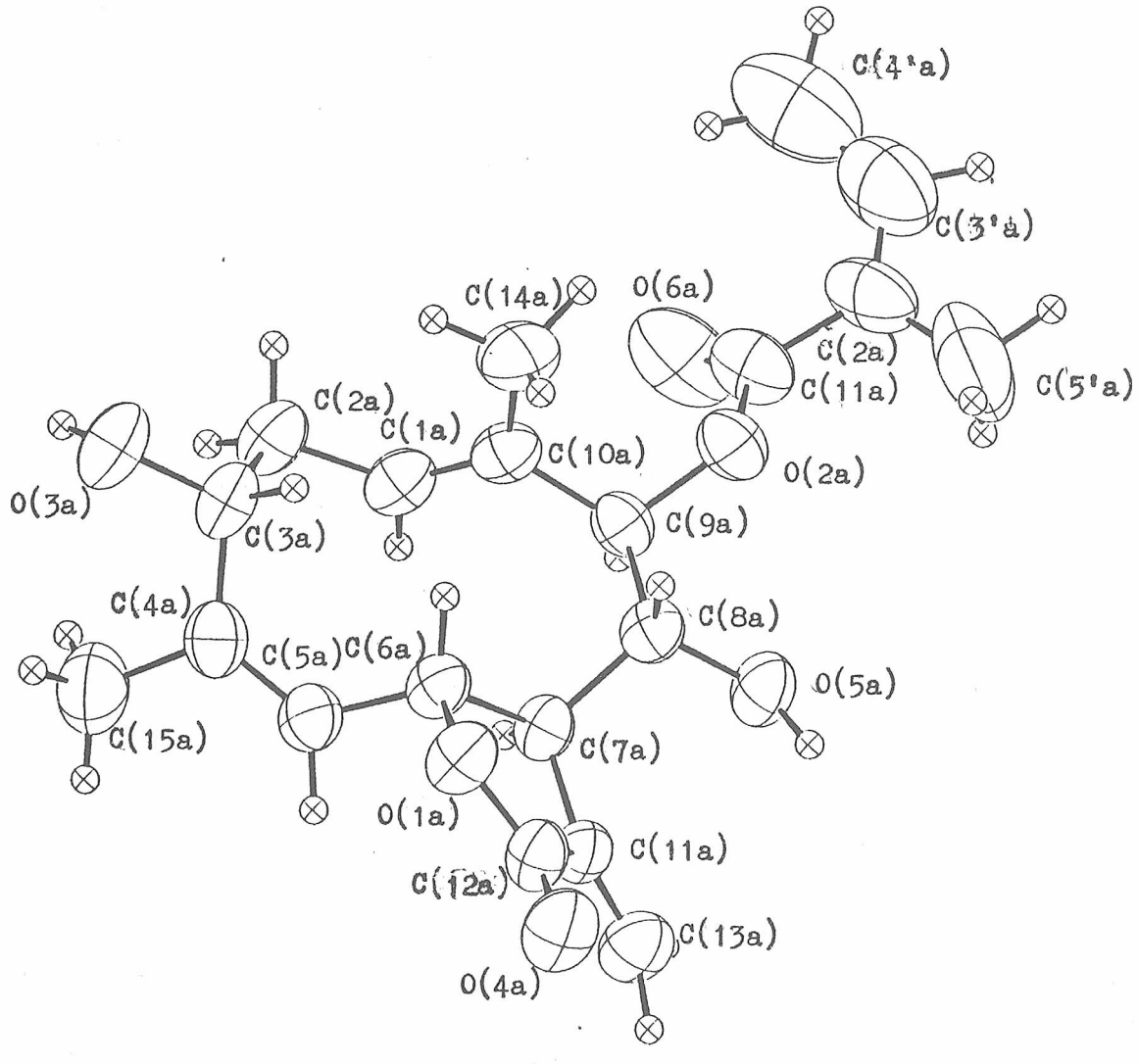
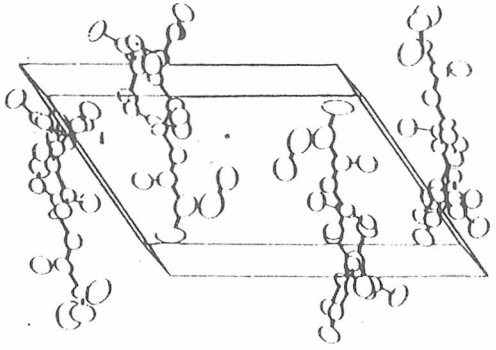


FIG. 11.3

FIG.



11.4

TABLE 11.2

Atom	x	y	z
O(1)	12191(3)	2268(2)	5648(2)
O(2)	5349(3)	2456(2)	2977(2)
O(3)	10486(4)	-0709(2)	3923(3)
O(4)	13480(4)	3453(2)	6250(3)
O(5)	7685(4)	3572(2)	3843(3)
O(6)	4066(4)	3019(2)	1307(3)
C(1)	7647(5)	1058(3)	2556(3)
C(2)	8253(6)	0208(3)	2703(4)
C(3)	9979(6)	0125(3)	3759(3)
C(4)	11132(5)	0681(3)	3735(3)
C(5)	11425(5)	1452(3)	4083(3)
C(6)	10778(5)	1917(3)	4615(3)
C(7)	9707(5)	2660(2)	3954(3)
C(8)	8189(4)	2754(3)	3965(3)
C(9)	6768(4)	2295(3)	2987(3)
C(10)	6998(5)	1378(3)	3036(3)
C(11)	10859(5)	3367(3)	4487(3)
C(12)	12323(6)	3074(3)	5549(3)
C(13)	10732(7)	4111(3)	4131(5)
C(14)	6596(7)	0905(3)	3708(4)
C(15)	11862(9)	0340(4)	3174(6)
C(1′)	4121(5)	2841(3)	2101(5)
C(2′)	2696(9)	2968(5)	2123(7)
C(3′)	2835(10)	3249(5)	2927(8)
C(4′)	4264(11)	3463(6)	4002(7)
C(5′)	1011(9)	2842(10)	0986(9)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 1

TABLE 11.2 (cont.)

HO(3)	11071(75)	-1139(31)	3811(55)
HO(5)	8342(68)	4076(28)	4070(54)
H(1)	7833(44)	1469(19)	2148(28)
H(2A)	7529(46)	-0190(24)	2742(36)
H(2B)	8342(49)	-0011(25)	2111(26)
H(3)	10018(47)	0311(24)	4411(24)
H(5)	12156(43)	1822(22)	4029(34)
H(6)	10278(40)	1516(18)	4848(28)
H(7)	9335(34)	2599(19)	3193(16)
H(8)	8362(36)	2455(18)	4616(20)
H(9)	6530(34)	2549(17)	2321(18)
H(13A)	9858(36)	4375(22)	3440(21)
H(13B)	11596(35)	4530(18)	4546(27)
H(14A)	5421(25)	0976(33)	3366(41)
H(14B)	7243(52)	1118(32)	4473(24)
H(14C)	6880(59)	0329(18)	3690(45)
H(15A)	12585(53)	-0119(22)	3646(36)
H(15B)	12507(55)	0774(25)	3122(41)
H(15C)	11068(54)	0110(29)	2405(22)
H(3')	1616(34)	3342(58)	2475(67)
H(4'A)	4053(70)	3887(23)	4383(48)
H(4'B)	5300(48)	3606(30)	4074(49)
H(4'C)	4450(68)	2922(18)	4371(48)
H(5'A)	0850(70)	2777(33)	0265(31)
H(5'B)	0125(58)	3180(28)	0891(43)
H(5'C)	1003(84)	2287(21)	1273(51)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 1

TABLE 11.2 (cont.)

O(1A)	7125(3)	2463(2)	0744(2)
O(2A)	0551(3)	2191(2)	-2159(2)
O(3A)	6718(4)	-0645(2)	0030(2)
O(4A)	8069(4)	3731(2)	1172(2)
O(5A)	2455(3)	3444(2)	-1848(2)
O(6A)	-0908(4)	1463(3)	-3680(3)
C(1A)	3340(5)	0764(3)	-2049(3)
C(2A)	4278(6)	-0016(3)	-1582(4)
C(3A)	5939(5)	0117(3)	-0463(3)
C(4A)	7012(5)	0672(3)	-0576(3)
C(5A)	6992(5)	1479(3)	-0487(3)
C(6A)	6001(5)	1971(3)	-0244(3)
C(7A)	4870(5)	2605(2)	-1150(3)
C(8A)	3191(4)	2686(3)	-1344(3)
C(9A)	2021(5)	2022(3)	-2091(3)
C(10A)	2616(5)	1168(3)	-1682(3)
C(11A)	5891(5)	3367(2)	-0705(3)
C(12A)	7148(5)	3243(3)	0498(3)
C(13A)	5844(6)	4016(3)	-1215(4)
C(14A)	2438(7)	0856(3)	-0814(5)
C(15A)	8091(8)	0281(4)	-0834(5)
C(1'A)	-0838(5)	1887(4)	-3002(4)
C(2'A)	-2252(7)	2150(4)	-2995(4)
C(3'A)	-3502(10)	1719(7)	-3382(7)
C(4'A)	-3779(11)	0904(9)	-3823(8)
C(5'A)	-2063(10)	2970(6)	-2447(8)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 2

HO(3A)
HO(5A)
H(1A)
H(2A)
H(2B)
H(3A)
H(5A)
H(6A)
H(7A)
H(8A)
H(9A)
H(13A)
H(13A)
H(14A)
H(14A)
H(14A)
H(15A)
H(15A)
H(15A)
H(3'A)
H(4'A)
H(4'A)
H(4'A)
H(5'A)
H(5'A)
H(5'A)

Fractional

TABLE 11.2 (cont.)

6475(95)	-1023(38)	-0568(46)
2889(88)	3940(29)	-1391(55)
3424(46)	1007(23)	-2615(26)
3639(48)	-0413(23)	-1472(37)
4420(52)	-0334(25)	-2083(30)
5708(46)	0415(23)	0009(28)
7627(57)	1900(26)	-0554(43)
5415(35)	1620(17)	-0041(26)
4705(40)	2402(19)	-1823(20)
3316(39)	2572(21)	-0642(19)
1736(36)	2100(19)	-2840(17)
5026(42)	4098(27)	-2018(18)
6647(44)	4466(20)	-0863(33)
1270(28)	0852(36)	-1079(49)
3091(57)	1169(33)	-0109(34)
2857(64)	0292(18)	-0701(50)
7398(63)	-0114(28)	-1442(32)
8905(56)	-0044(30)	-0149(30)
8706(63)	0633(33)	-1017(43)
-4371(70)	2072(37)	-3441(60)
-4574(53)	0625(39)	-3748(47)
-2793(52)	0577(39)	-3550(49)
-4283(57)	1096(44)	-4589(29)
-1181(49)	3188(41)	-1680(24)
-1992(63)	3293(38)	-2987(35)
-3128(37)	3104(41)	-2566(42)

Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 2

TABLE 11.3

Atom	U_{11}	U_{22}
O(1)	51(2)	78(2)
O(2)	52(2)	71(2)
O(3)	89(2)	56(2)
O(4)	71(2)	114(3)
O(5)	64(2)	57(2)
O(6)	64(2)	106(3)
C(1)	55(2)	48(3)
C(2)	72(3)	57(3)
C(3)	70(3)	50(3)
C(4)	62(3)	64(3)
C(5)	51(3)	65(3)
C(6)	47(2)	58(3)
C(7)	52(2)	49(2)
C(8)	47(2)	46(2)
C(9)	40(2)	64(3)
C(10)	42(2)	54(2)
C(11)	60(3)	61(3)
C(12)	61(3)	84(4)
C(13)	80(4)	59(3)
C(14)	85(3)	67(3)
C(15)	121(5)	86(4)
C(1')	45(3)	71(3)
C(2')	107(5)	112(5)
C(3')	108(6)	116(6)
C(4')	147(7)	156(7)
C(5')	59(4)	321(15)

Anisotropic Temperature

Molecule 1

U_{33}	U_{12}	U_{13}	U_{23}
45(1)	-3(2)	17(1)	-4(1)
61(2)	8(2)	33(2)	12(1)
77(2)	19(2)	43(2)	11(2)
57(2)	-29(2)	25(2)	-32(2)
77(2)	5(2)	38(2)	-5(2)
66(2)	19(2)	30(2)	26(2)
37(2)	-3(2)	11(2)	-6(2)
56(3)	-1(3)	29(2)	-6(2)
53(2)	6(2)	34(2)	1(2)
55(2)	6(2)	30(2)	-1(2)
58(2)	1(2)	31(2)	2(2)
36(2)	-2(2)	16(2)	0(2)
31(2)	-4(2)	20(2)	-7(2)
43(2)	4(2)	23(2)	-1(2)
38(2)	5(2)	19(2)	6(2)
35(2)	-3(2)	8(2)	4(2)
48(2)	-11(2)	37(2)	-16(2)
45(2)	-11(3)	30(2)	-13(2)
77(3)	-16(3)	46(3)	-17(3)
71(3)	-6(3)	46(3)	10(2)
134(5)	4(3)	100(4)	-16(4)
82(4)	5(2)	36(3)	15(3)
100(5)	-5(4)	56(4)	1(4)
116(6)	0(5)	47(5)	4(5)
137(7)	1(6)	110(6)	-22(6)
166(8)	-11(7)	41(5)	-79(10)

Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 11.3(cont.)

HO(3)	135(6)
HO(5)	135(6)
H(1)	54(11)
H(2A)	73(14)
H(2B)	69(13)
H(3)	59(12)
H(5)	70(13)
H(6)	48(11)
H(7)	34(8)
H(8)	34(9)
H(9)	25(8)
H(13A)	50(7)
H(13B)	50(7)
H(14A)	135(6)
H(14B)	135(6)
H(14C)	135(6)
H(15A)	135(6)
H(15B)	135(6)
H(15C)	135(6)
H(3')	180(36)
H(4'A)	135(6)
H(4'B)	135(6)
H(4'C)	135(6)
H(5'A)	135(6)
H(5'B)	135(6)
H(5'C)	135(6)

Anisotropic Temperature Factor Coefficients ($x10^3$) with e.s.d.'s

Molecule 1

TABLE 11.3 (cont.)

O(1A)	52(2)	52(2)	42(1)	-2(1)	11(1)	0(1)
O(2A)	45(2)	74(2)	55(2)	-1(1)	25(1)	-4(1)
O(3A)	95(2)	49(2)	62(2)	15(2)	38(2)	8(1)
O(4A)	63(2)	63(2)	58(2)	-6(2)	16(1)	-17(2)
O(5A)	56(2)	51(2)	55(2)	9(1)	24(1)	8(1)
O(6A)	61(2)	166(4)	69(2)	-26(2)	22(2)	-36(3)
C(1A)	60(3)	47(2)	44(2)	-5(2)	23(2)	-2(2)
C(2A)	76(3)	45(3)	63(3)	-1(2)	35(3)	-5(2)
C(3A)	69(3)	41(2)	54(2)	9(2)	30(2)	1(2)
C(4A)	55(3)	61(3)	52(2)	12(2)	26(2)	6(2)
C(5A)	44(2)	52(3)	55(2)	5(2)	21(2)	7(2)
C(6A)	47(2)	46(2)	43(2)	1(2)	20(2)	4(2)
C(7A)	47(2)	45(2)	38(2)	5(2)	21(2)	4(2)
C(8A)	50(2)	48(2)	41(2)	3(2)	23(2)	7(2)
C(9A)	42(2)	58(3)	44(2)	1(2)	24(2)	-3(2)
C(10A)	50(2)	48(2)	46(2)	-8(2)	21(2)	-2(2)
C(11A)	43(2)	42(2)	50(2)	-2(2)	21(2)	-3(2)
C(12A)	51(3)	50(3)	48(2)	4(2)	22(2)	-3(2)
C(13A)	64(3)	55(3)	64(3)	-10(2)	30(3)	-9(2)
C(14A)	86(3)	72(3)	95(4)	3(3)	59(3)	25(3)
C(15A)	102(4)	78(4)	106(4)	19(3)	71(4)	6(3)
C(1'A)	49(3)	96(4)	48(2)	-3(3)	20(2)	7(3)
C(2'A)	43(3)	117(5)	74(3)	-4(3)	21(3)	23(3)
C(3'A)	79(6)	161(8)	119(6)	16(6)	51(5)	33(6)
C(4'A)	101(6)	247(14)	135(7)	-21(8)	63(5)	5(9)
C(5'A)	115(6)	177(9)	176(8)	56(6)	104(6)	30(7)

Anisotropic Temperature Factor Coefficients ($x10^3$) with e.s.d.'s

Molecule 2

TABLE 11.3 (cont.)

HO(3A)	135(8)
HO(5A)	135(8)
H(1A)	57(11)
H(2A)	78(15)
H(2B)	71(13)
H(3A)	60(12)
H(5A)	97(17)
H(6A)	37(9)
H(7A)	39(9)
H(8A)	45(9)
H(9A)	33(8)
H(13A)	50(9)
H(13A)	50(9)
H(14A)	135(8)
H(14A)	135(8)
H(14A)	135(8)
H(15A)	135(8)
H(15A)	135(8)
H(15A)	135(8)
H(3'A)	135(25)
H(4'A)	135(8)
H(4'A)	135(8)
H(4'A)	135(8)
H(5'A)	135(8)
H(5'A)	135(8)
H(5'A)	135(8)

Anisotropic Temperature Coefficients ($x10^3$) with e.s.d.'s

Molecule 2

TABLE 11.4

O(1)	–	C(6)	1.547(9)	O(1A) – C(6A)	1.519(10)
O(1)	–	C(12)	1.351(15)	O(1A) – C(12A)	1.368(13)
O(2)	–	C(9)	1.489(15)	O(2A) – C(9A)	1.483(14)
O(2)	–	C(1′)	1.366(11)	O(2A) – C(1A′)	1.379(11)
O(3)	–	C(3)	1.450(12)	O(3A) – C(3A)	1.431(11)
O(4)	–	C(12)	1.243(12)	O(4A) – C(12A)	1.233(11)
O(5)	–	C(8)	1.415(12)	O(5A) – C(8A)	1.448(11)
O(6)	–	C(1′)	1.278(20)	O(6A) – C(1A′)	1.289(19)
C(1)	–	C(2)	1.485(14)	C(1A) – C(2A)	1.526(14)
C(1)	–	C(10)	1.374(18)	C(1A) – C(10A)	1.350(18)
C(2)	–	C(3)	1.607(12)	C(2A) – C(3A)	1.656(11)
C(3)	–	C(4)	1.516(19)	C(3A) – C(4A)	1.532(18)
C(4)	–	C(5)	1.342(16)	C(4A) – C(5A)	1.345(15)
C(4)	–	C(15)	1.590(24)	C(4A) – C(15A)	1.543(22)
C(5)	–	C(6)	1.556(19)	C(5A) – C(6A)	1.533(18)
C(6)	–	C(7)	1.560(12)	C(6A) – C(7A)	1.618(11)
C(7)	–	C(8)	1.566(17)	C(7A) – C(8A)	1.547(16)
C(7)	–	C(11)	1.532(13)	C(7A) – C(11A)	1.529(12)
C(8)	–	C(9)	1.590(11)	C(8A) – C(9A)	1.534(12)
C(9)	–	C(10)	1.528(14)	C(9A) – C(10A)	1.527(13)
C(10)	–	C(14)	1.599(20)	C(10A) – C(14A)	1.632(21)
C(11)	–	C(12)	1.543(12)	C(11A) – C(12A)	1.568(12)
C(11)	–	C(13)	1.333(16)	C(11A) – C(13A)	1.346(16)
C(1′)	–	C(2′)	1.489(28)	C(1A′) – C(2A′)	1.508(23)
C(2′)	–	C(3′)	1.344(37)	C(2A′) – C(3A′)	1.379(25)
C(2′)	–	C(5′)	1.582(19)	C(2A′) – C(5A′)	1.522(27)
C(3′)	–	C(4′)	1.576(22)	C(3A′) – C(4A′)	1.443(34)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

Molecule 1 Molecule 2

C(12) - O(1) - C(6)	109.4(7)
C(7) - C(6) - O(1)	106.1(7)
C(11) - C(12) - O(1)	111.2(9)
C(8) - C(9) - O(2)	102.0(8)
O(6) - C(1') - O(2)	126.6(13)
C(2) - C(3) - O(3)	107.4(8)
C(11) - C(12) - O(4)	129.0(11)
C(9) - C(8) - O(5)	105.7(8)
C(10) - C(1) - C(2)	120.6(9)
C(9) - C(10) - C(1)	116.3(9)
C(4) - C(3) - C(2)	109.5(9)
C(15) - C(4) - C(3)	115.9(10)
C(6) - C(5) - C(4)	127.7(12)
C(8) - C(7) - C(6)	110.6(8)
C(11) - C(7) - C(8)	109.5(8)
C(12) - C(11) - C(7)	106.7(9)
C(10) - C(9) - C(8)	113.0(8)
C(13) - C(11) - C(12)	123.6(11)
C(5') - C(2') - C(1')	112.2(15)
C(4') - C(3') - C(2')	129.0(23)
C(5A) - C(6A) - O(1A)	108.6(9)
O(4A) - C(12A) - O(1A)	119.6(8)
C(1A') - O(2A) - C(9A)	116.0(8)
C(10A) - C(9A) - O(2A)	109.8(9)
C(2A') - C(1A') - O(2A)	108.7(10)
C(4A) - C(3A) - O(3A)	111.7(10)
C(7A) - C(8A) - O(5A)	108.8(8)
C(2A') - C(1A') - O(6A)	128.9(12)
C(3A) - C(2A) - C(1A)	109.8(8)
C(14A) - C(10A) - C(1A)	127.6(9)
C(5A) - C(4A) - C(3A)	121.6(11)
C(15A) - C(4A) - C(5A)	119.6(11)

Valency Angles (°) (Non

C(5)	C(6)	O(1)	108.4(9)
O(4)	C(12)	O(1)	119.8(10)
C(1')	O(2)	C(9)	113.4(9)
C(10)	C(9)	O(2)	106.5(9)
C(2')	C(1')	O(2)	112.2(12)
C(4)	C(3)	O(3)	111.8(11)
C(7)	C(8)	O(5)	111.1(8)
C(2')	C(1')	O(6)	121.0(13)
C(3)	C(2)	C(1)	108.6(8)
C(14)	C(10)	C(1)	128.0(9)
C(5)	C(4)	C(3)	122.5(12)
C(15)	C(4)	C(5)	121.3(12)
C(7)	C(6)	C(5)	110.2(8)
C(11)	C(7)	C(6)	103.6(8)
C(9)	C(8)	C(7)	102.9(8)
C(13)	C(11)	C(7)	129.6(10)
C(14)	C(10)	C(9)	115.5(9)
C(3')	C(2')	C(1')	123.3(19)
C(5')	C(2')	C(3')	124.0(21)
C(12A)	O(1A)	C(6A)	109.6(7)
C(7A)	C(6A)	O(1A)	107.6(7)
C(11A)	C(12A)	O(1A)	109.7(8)
C(8A)	C(9A)	O(2A)	100.3(8)
O(6A)	C(1A')	O(2A)	122.3(13)
C(2A)	C(3A)	O(3A)	109.9(7)
C(11A)	C(12A)	O(4A)	130.6(9)
C(9A)	C(8A)	O(5A)	105.4(7)
C(10A)	C(1A)	C(2A)	119.9(9)
C(9A)	C(10A)	C(1A)	115.5(9)
C(4A)	C(3A)	C(2A)	108.1(8)
C(15A)	C(4A)	C(3A)	118.8(9)
C(6A)	C(5A)	C(4A)	125.5(11)

Hydrogen Atoms) with e.s.d.'s

TABLE 11.5 (cont.)

C(7A) - C(6A) - C(5A)	108.5(8)	C(8A) - C(7A) - C(6A)	109.8(8)
C(11A)- C(7A) - C(6A)	99.9(7)	C(11A)- C(7A) - C(8A)	113.1(8)
C(9A) - C(8A) - C(7A)	108.5(8)	C(12A)- C(11A)- C(7A)	108.7(8)
C(13A)- C(11A)- C(7A)	126.9(9)	C(10A)- C(9A) - C(8A)	113.3(8)
C(14A)- C(10A)- C(9A)	116.5(9)	C(13A)- C(11A)- C(12A)	124.3(9)
C(3A')- C(2A')- C(1A')	113.2(16)	C(5A')- C(2A')- C(1A')	119.1(15)
C(5A')- C(2A')- C(3A')	127.7(18)	C(4A')- C(3A')- C(2A')	132.9(22)

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 11.6

C(12)	O(1)	C(6)	C(5)	102.9(10)	C(12)	O(1)	C(6)	C(7)	-15.5(11)
C(6)	O(1)	C(12)	O(4)	-172.6(11)	C(6)	O(1)	C(12)	C(11)	7.0(12)
C(1')	O(2)	C(9)	C(8)	122.4(9)	C(1')	O(2)	C(9)	C(10)	-118.9(10)
C(9)	O(2)	C(1')	O(6)	3.9(17)	C(9)	O(2)	C(1')	C(2')	178.1(11)
C(10)	C(1)	C(2)	C(3)	-80.7(12)	C(2)	C(1)	C(10)	C(9)	169.1(9)
C(2)	C(1)	C(10)	C(14)	-6.7(17)	C(1)	C(2)	C(3)	O(3)	179.9(9)
C(1)	C(2)	C(3)	C(4)	-58.5(12)	O(3)	C(3)	C(4)	C(5)	-148.7(11)
O(3)	C(3)	C(4)	C(15)	37.2(14)	C(2)	C(3)	C(4)	C(5)	92.4(13)
C(2)	C(3)	C(4)	C(15)	-81.8(13)	C(3)	C(4)	C(5)	C(6)	3.7(19)
C(15)	C(4)	C(5)	C(6)	177.6(11)	C(4)	C(5)	C(6)	O(1)	126.2(12)
C(4)	C(5)	C(6)	C(7)	-118.0(13)	O(1)	C(6)	C(7)	C(8)	-100.1(9)
O(1)	C(6)	C(7)	C(11)	17.1(10)	C(5)	C(6)	C(7)	C(8)	142.8(9)
C(5)	C(6)	C(7)	C(11)	-100.0(9)	C(6)	C(7)	C(8)	O(5)	151.0(8)
C(6)	C(7)	C(8)	C(9)	-96.4(9)	C(11)	C(7)	C(8)	O(5)	37.5(11)
C(11)	C(7)	C(8)	C(9)	150.1(8)	C(6)	C(7)	C(11)	C(12)	-13.3(11)
C(6)	C(7)	C(11)	C(13)	164.3(13)	C(8)	C(7)	C(11)	C(12)	104.7(10)
C(8)	C(7)	C(11)	C(13)	-77.7(15)	O(5)	C(8)	C(9)	O(2)	-59.6(9)
O(5)	C(8)	C(9)	C(10)	-173.5(8)	C(7)	C(8)	C(9)	O(2)	-176.1(7)
C(7)	C(8)	C(9)	C(10)	69.9(10)	O(2)	C(9)	C(10)	C(1)	149.0(9)
O(2)	C(9)	C(10)	C(14)	-34.6(12)	C(8)	C(9)	C(10)	C(1)	-99.8(11)
C(8)	C(9)	C(10)	C(14)	76.6(11)	C(7)	C(11)	C(12)	O(1)	4.3(13)
C(7)	C(11)	C(12)	O(4)	-176.1(12)	C(13)	C(11)	C(12)	O(1)	-173.4(12)
C(13)	C(11)	C(12)	O(4)	6.2(21)	O(2)	C(1')	C(2')	C(3')	46.5(24)
O(2)	C(1')	C(2')	C(5')	-141.6(14)	O(6)	C(1')	C(2')	C(3')	-138.9(19)
O(6)	C(1')	C(2')	C(5')	32.9(21)	C(1')	C(2')	C(3')	C(4')	-2.2(35)
C(5')	C(2')	C(3')	C(4')	-173.1(20)	C(12A)	O(1A)	C(6A)	C(5A)	100.0(9)
C(12A)	O(1A)	C(6A)	C(7A)	-17.2(10)	C(6A)	O(1A)	C(12A)	O(4A)	-177.3(9)
C(6A)	O(1A)	C(12A)	C(11A)	5.5(11)	C(1A')	O(2A)	C(9A)	C(8A)	161.4(9)
C(1A')	O(2A)	C(9A)	C(10A)	-79.1(11)	C(9A)	O(2A)	C(1A')	O(6A)	1.4(16)
C(9A)	O(2A)	C(1A')	C(2A')	-174.5(10)	C(10A)	C(1A)	C(2A)	C(3A)	-72.7(13)
C(2A)	C(1A)	C(10A)	C(9A)	170.3(9)	C(2A)	C(1A)	C(10A)	C(14A)	-3.5(17)
C(1A)	C(2A)	C(3A)	O(3A)	177.3(9)	C(1A)	C(2A)	C(3A)	C(4A)	-60.7(11)

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

TABLE 11.6 (cont.)

O(3A)	C(3A)	C(4A)	C(5A)	-145.8(10)	O(3A)	C(3A)	C(4A)	C(15A)	35.7(14)
C(2A)	C(3A)	C(4A)	C(5A)	93.3(12)	C(2A)	C(3A)	C(4A)	C(15A)	-85.2(12)
C(3A)	C(4A)	C(5A)	C(6A)	0.7(17)	C(15A)	C(4A)	C(5A)	C(6A)	179.2(11)
C(4A)	C(5A)	C(6A)	O(1A)	121.6(11)	C(4A)	C(5A)	C(6A)	C(7A)	-121.7(12)
O(1A)	C(6A)	C(7A)	C(8A)	-98.0(9)	O(1A)	C(6A)	C(7A)	C(11A)	21.0(9)
C(5A)	C(6A)	C(7A)	C(8A)	144.7(8)	C(5A)	C(6A)	C(7A)	C(11A)	-96.3(9)
C(6A)	C(7A)	C(8A)	O(5A)	158.0(8)	C(6A)	C(7A)	C(8A)	C(9A)	-87.8(9)
C(11A)	C(7A)	C(8A)	O(5A)	47.4(11)	C(11A)	C(7A)	C(8A)	C(9A)	161.6(8)
C(6A)	C(7A)	C(11A)	C(12A)	-17.8(10)	C(6A)	C(7A)	C(11A)	C(13A)	158.6(11)
C(8A)	C(7A)	C(11A)	C(12A)	98.8(10)	C(8A)	C(7A)	C(11A)	C(13A)	-84.8(13)
O(5A)	C(8A)	C(9A)	O(2A)	-63.8(9)	O(5A)	C(8A)	C(9A)	C(10A)	179.3(8)
C(7A)	C(8A)	C(9A)	O(2A)	179.9(7)	C(7A)	C(8A)	C(9A)	C(10A)	63.0(10)
O(2A)	C(9A)	C(10A)	C(1A)	147.7(9)	O(2A)	C(9A)	C(10A)	C(14A)	-37.8(12)
C(8A)	C(9A)	C(10A)	C(1A)	-101.1(11)	C(8A)	C(9A)	C(10A)	C(14A)	73.4(12)
C(7A)	C(11A)	C(12A)	O(1A)	9.0(11)	C(7A)	C(11A)	C(12A)	O(4A)	-167.8(11)
C(13A)	C(11A)	C(12A)	O(1A)	-167.5(10)	C(13A)	C(11A)	C(12A)	O(4A)	15.7(19)
O(2A)	C(1A')	C(2A')	C(3A')	-150.5(14)	O(2A)	C(1A')	C(2A')	C(5A')	27.6(19)
O(6A)	C(1A')	C(2A')	C(3A')	33.9(23)	O(6A)	C(1A')	C(2A')	C(5A')	-147.9(16)
C(1A')	C(2A')	C(3A')	C(4A')	4.6(33)	C(5A')	C(2A')	C(3A')	C(4A')	-173.4(23)

Torsion Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

side chain as shown in Fig. 11.5, this too was resolved as shown in Fig. 11.6. These problems were not detrimental to the analysis.

Structural Analysis 11.4.2

Zoapatanolide B is a 1,5 cis-trans diene [129], and it adopts a twisted boat-chair conformation [89]. The central cyclodecadiene ring adopts the minimum energy conformation for this system [89]. The lactone ring adopts a half chair [130], and is trans fused to the cyclodecadiene ring at C(6) and C(7). The half chair conformation is the less stable conformation for lactone rings, but can be explained by the presence of the methylene group.

In the crystalline state molecules 1 and 2 differ in the arrangement of the side chain. This may be seen from the torsion angles around the side chain. The variation elsewhere in the molecule is minimum. This arrangement could be due to crystal packing forces.

Many similar structures to zoapatanolide B have been studied, and although the most stable form of this molecule is the cis, trans arrangement, the majority of these structures are trans-trans cyclodecadienes. The other structures, examples of which are, two derivatives of Melnerin [131], Neurolenin A and B [132], Tamaulipin A [133] and a sesquiterpene lactone from Eupatorium mikanioides, all show a trans fused lactone ring as in zoapatanolide B.

Molecular Mechanics 11.4.3

The molecules of zoapatanolide B are shown to be stable. There was very little movement of the coordinates during the minimisation process. However the side chains were found to have the same arrangement after minimisation, supporting the theory that crystal packing forces cause the differences in the crystal. This is shown in Table 11.7



FIG. 11.5

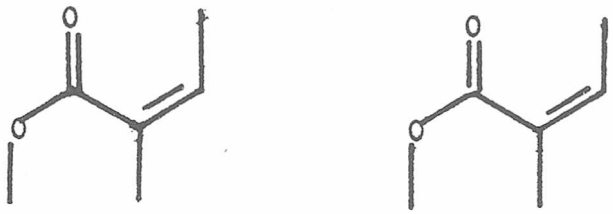


FIG. 11.6

TABLE 11.7

Torsion Angle	Molecule 1 Pre-minimisation	Molecule 1 Post-minimisation	Molecule 2 Pre-minimisation	Molecule 2 Post-minimisation
O(2)-C(9)-C(10)-C(1)	149.9	146.0	148.2	145.6
C(7)-C(8)-C(9)-O(2)	-176.2	-177.4	-179.9	-177.5
O(5)-C(8)-C(9)-O(2)	- 57.7	- 57.3	- 60.9	- 57.46
O(2)-C(9)-C(10)-C(14)	- 33.8	- 37.9	- 37.4	- 38.4
C(1')-O(2)-C(9)-C(10)	-119.8	- 73.3	- 75.9	- 67.9
C(1')-O(2)-C(9)-C(8)	119.8	164.6	161.4	169.7
C(9)-O(2)-C(1')-C(2')	178.0	-179.2	-174.6	178.6
C(9)-O(2)-C(1')-O(6)	4.0	0.5	1.4	- 1.9
O(2)-C(1')-C(2')-C(3')	49.2	- 0.9	151.2	-179.5
O(2)-C(1')-C(2')-C(5')	-139.3	179.1	27	0.43
C(1')-C(2')-C(3')-C(4')	- 2.2	- 0.42	4.3	0.31
C(5')-C(2')-C(3')-C(4')	-173.2	179.4	-173.7	-179.7

CHAPTER TWELVE

THE LEUCOACETATE OF VENTILATONE A - A DISORDERED STRUCTURE

The Leucoacetate of Ventilatone A - a Disordered Structure

Introduction 12.1

This substance (Fig. 12.1) is a derivative of the lactone Ventilatone A (Fig. 12.2). Ventilatone A is not a crystalline substance, so a derivative was formed in order to carry out an X-ray analysis. The derivative was thought to have the structure shown in Fig. 12.1 but the stereochemistry at C(9) had still to be determined.

Experimental 12.2

A small but regularly shaped crystal was chosen. Data were collected as outlined in Chapter 2, the intensities of 3167 independent reflexions being measured, scanning to $2\theta \leq 50^{\circ}$. A fragment was obtained from an E map computed for 312 reflexions with $|E|_{min} > 1.5$, and with an elevated temperature factor of 4.50 (raised from 4.0). This partial structure was entered as an oriented fragment to 'MULTAN' [72], the same fragment being reproduced with some additional atoms. The coordinates of this fragment were input to 'SHELX' [73] to produce a starting R value of 46%. All non-hydrogen atoms were visible from the difference map, and their coordinates included as part of the molecule. Anisotropic temperature factors were introduced for all non-hydrogen atoms, but refinement would not proceed below an R value of 9%. The space group was changed from $P\bar{1}$ to P1. Anisotropic temperature factors were introduced for all the non-hydrogen atoms but several spurious electron density peaks that could not be accounted for by hydrogens were present around C(8) and C(9). The C(8) - C(9) bond in molecule 2 was also showing considerable double bond character. It was decided that the structure was disordered. Several attempts

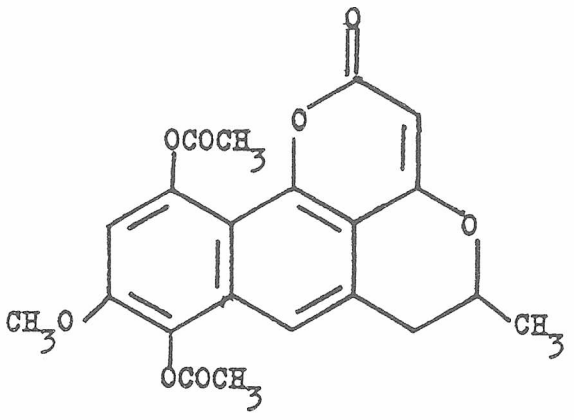


FIG. 12.1

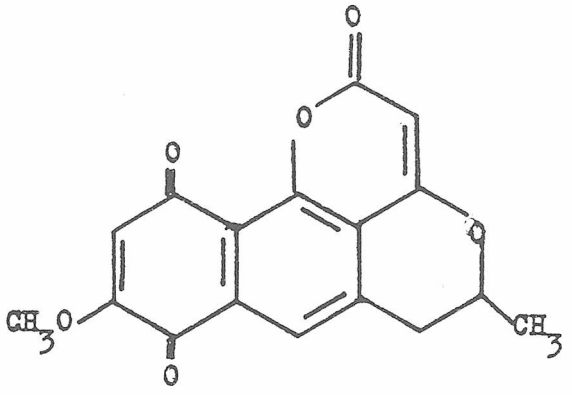


FIG. 12.2

were made to determine the pattern of disorder, but this was found to be impossible. The ^{1}Hnmr was obtained for a second time, but it still showed no evidence of a double bond at the C(8) - C(9) position in the molecule. The ^{1}Hnmr result was accepted as being correct, the hydrogen atom positions being calculated to conform to the ^{1}Hnmr results. The positions of the hydrogen atoms around C(8) and C(9) in both molecules were fixed as rigidly as possible.

Unit weights were used throughout.

Results 12.3

Crystallographic Data 12.3.1

$C_{22}H_{20}O_7$, Mr = 396.396, Dc = 1.196gcm^{-3}, triclinic, P1, Z = 2, a = 8.475(17)Å, b = 9.866(19)Å, c = 11.524(24)Å, α = 97.92(16)°, β = 96.08(16)°, γ = 84.88(16°), U = 936.69Å^3, μ(MoKα) = 0.68cm^{-1}, F(000) = 416.00, Final R value = 6.66% for 2689 unique reflexions ($I > 3\sigma(I)$), R = 6.66%, maximum height on difference map = 0.267 eÅ^{-3} minimum height on difference map = -0.298 eÅ^{-3}, maximum shift/error ratio = 0.213.

Structural Data 12.3.2

The structure of the leucoacetate of ventilatone A is shown in Fig. 12.1. The atomic arrangement is shown in Fig. 12.3 and Fig. 12.4 shows a stereoscopic view of crystal packing. Tables 12.1 - 12.5 list the structural parameters. The ^{1}Hnmr is shown in Fig. 12.5

Discussion 12.4

X-ray Analysis 12.4.1

As seen for previous planar structures, it was necessary to increase the temperature factor in order to effect a solution. However very few other problems were encountered apart from those

O(3)
C(2)
C(3)
C(18)
O(5)
O(7)

Molecule 1

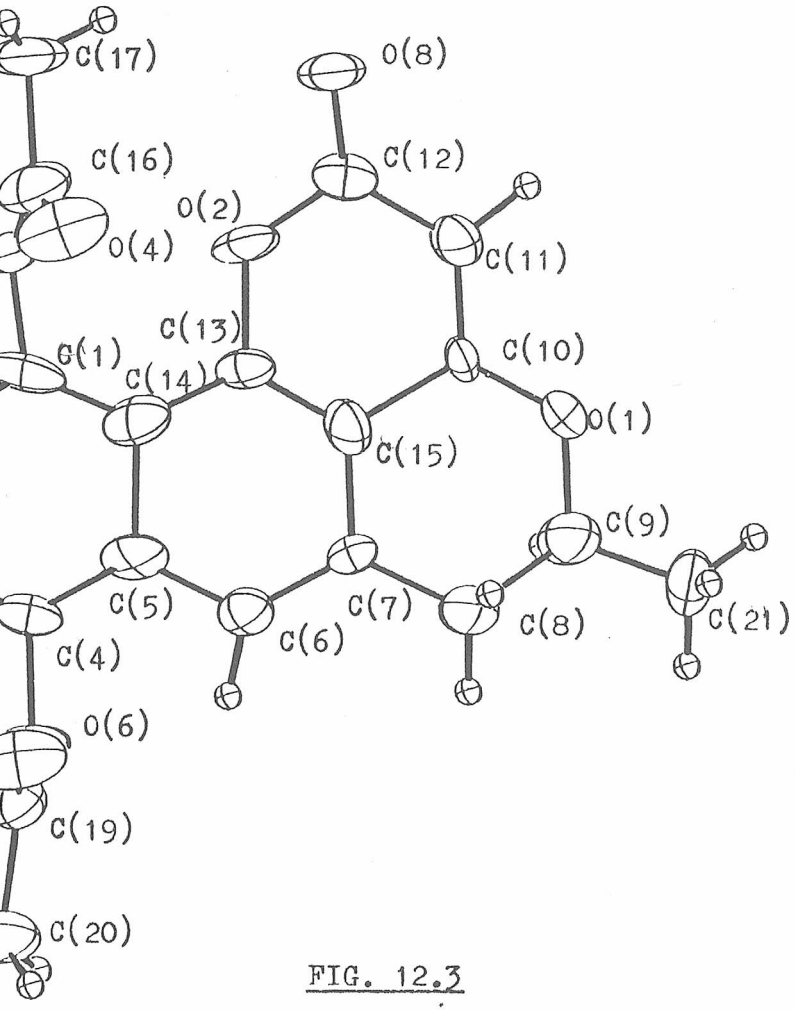


FIG. 12.3

C(2')
C(18')
C(3')
O(5')
O(7')

Molecule 2

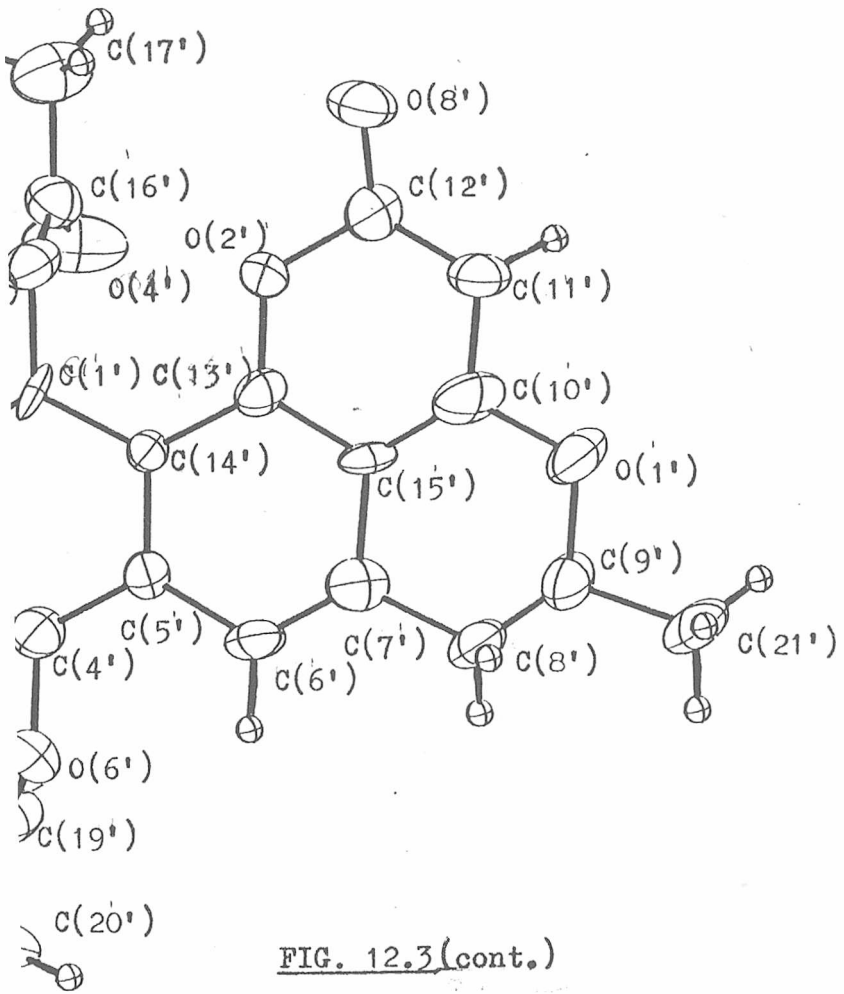
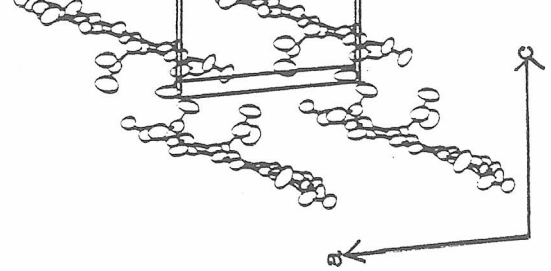


FIG. 12.3(cont.)


a

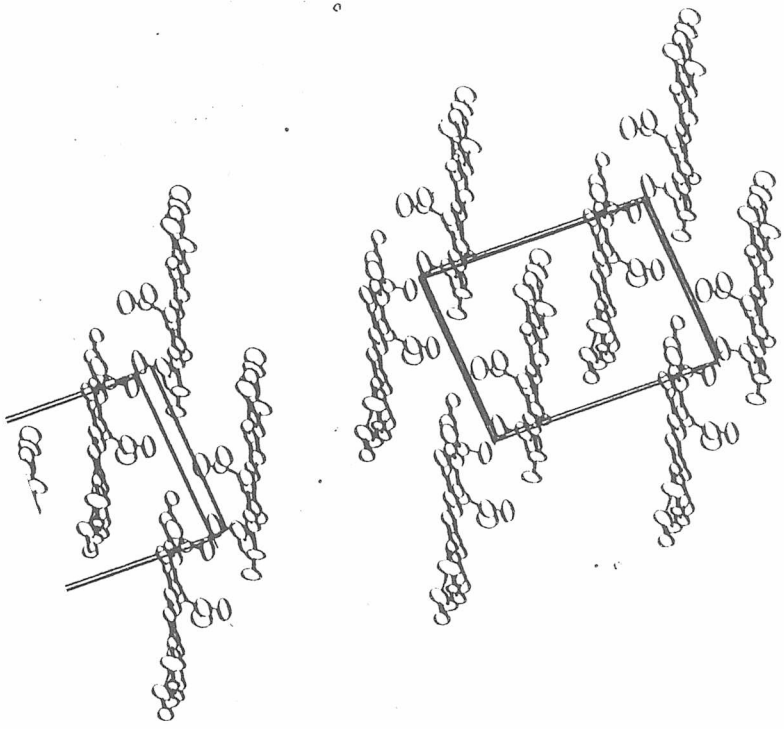


FIG. 12.4

Atom
O(1)
O(2)
O(3)
O(4)
O(5)
O(6)
O(7)
O(8)
C(1)
C(2)
C(3)
C(4)
C(5)
C(6)
C(7)
C(8)
C(9)
C(10)
C(11)
C(12)
C(13)
C(14)
C(15)
C(16)
C(17)
C(18)
C(19)
C(20)
C(21)

Fract

TABLE 12.1

x	y	z
8085(5)	1409(5)	4624(5)
5273(7)	-1732(5)	3267(4)
2516(6)	-2583(5)	2175(4)
4052(9)	-2581(6)	0664(5)
-1690(7)	0843(6)	1469(5)
0110(7)	2879(5)	2388(4)
0648(9)	3038(6)	0609(5)
7158(7)	-3278(5)	3728(5)
2063(10)	-1194(8)	2230(6)
0501(11)	-0948(8)	1767(7)
-0188(10)	0475(8)	1850(6)
0772(9)	1462(7)	2334(6)
2352(10)	1252(7)	2706(6)
3362(9)	2295(7)	3177(6)
4944(8)	2096(6)	3552(6)
6041(13)	3170(9)	3983(11)
7363(12)	2778(8)	4900(8)
7194(7)	0394(7)	4180(6)
7770(10)	-0927(8)	4131(7)
6794(9)	-2074(8)	3697(6)
4631(9)	-0364(7)	3213(5)
3099(11)	-0230(8)	2715(7)
5528(9)	0680(8)	3619(6)
3559(12)	-3173(8)	1390(8)
4088(13)	-4639(8)	1557(9)
-2740(10)	-0089(9)	0863(7)
0053(9)	3498(7)	1431(7)
-1010(13)	4869(9)	1682(8)
8629(10)	3737(9)	5214(7)

ional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 1

TABLE 12.1(cont.)

H(2)	-0254(71)	-1631(62)	1362(60)
H(6)	2569(74)	3114(46)	3318(66)
H(8A)	5447(100)	4042(59)	4315(77)
H(8B)	6738(103)	3092(106)	3307(60)
H(9)	6667(96)	2726(99)	5544(62)
H(11)	8887(40)	-1251(76)	4431(63)
H(17A)	4324(80)	-5086(83)	0755(30)
H(17B)	3172(61)	-5055(83)	1811(57)
H(17C)	5060(51)	-4737(88)	2119(47)
H(18A)	-3172(82)	-0636(65)	1421(55)
H(18B)	-3664(61)	0438(73)	0468(58)
H(18C)	-2162(83)	-0718(64)	0268(47)
H(20A)	-0642(86)	5529(71)	1209(57)
H(20B)	-1083(96)	5348(94)	2528(40)
H(20C)	-2119(49)	4615(88)	1383(62)
H(21A)	8031(81)	4629(49)	5476(57)
H(21B)	9220(79)	3839(79)	4525(42)
H(21C)	9437(68)	3486(78)	5873(43)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 1

O(1')
O(2')
O(3')
O(4')
O(5')
O(6')
O(7')
O(8')
C(1')
C(2')
C(3')
C(4')
C(5')
C(6')
C(7')
C(8')
C(9')
C(10')
C(11')
C(12')
C(13')
C(14')
C(15')
C(16')
C(17')
C(18')
C(19')
C(20')
C(21')

Fractional

TABLE 12.1(cont.)

-8068(8)	-1405(5)	-4655(5)
-5201(6)	1713(5)	-3219(4)
-2465(7)	2643(5)	-2162(5)
-3925(10)	2569(7)	-0658(6)
1759(7)	-0887(6)	-1408(5)
-0076(6)	-2860(5)	-2394(4)
-0570(10)	-3056(6)	-0500(5)
-7138(8)	3296(6)	-3717(6)
-1921(8)	1199(7)	-2144(6)
-0399(9)	0881(9)	-1752(6)
0218(9)	-0474(9)	-1768(6)
-0803(9)	-1512(7)	-2221(7)
-2357(8)	-1236(7)	-2762(5)
-3334(10)	-2347(7)	-3263(7)
-4793(10)	-2077(8)	-3779(6)
-5934(13)	-3158(8)	-4329(10)
-7503(12)	-2806(9)	-4346(8)
-7049(12)	-0380(8)	-4165(7)
-7735(10)	0965(7)	-4251(7)
-6761(11)	2035(8)	-3780(6)
-4595(9)	0406(7)	-3218(6)
-2980(8)	0138(6)	-2693(5)
-5557(9)	-0699(6)	-3719(5)
-3477(10)	3184(8)	-1344(7)
-3957(14)	4659(9)	-1544(8)
2752(11)	0199(11)	-0802(9)
0081(12)	-3540(9)	-1381(8)
0963(14)	-4855(10)	-1556(9)
-8739(13)	-3744(8)	-5076(9)

Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 2

TABLE 12.1(cont.)

H(2')	0351(58)	1612(46)	-1772(52)
H(6')	-2906(67)	-3337(29)	-3250(52)
H(8'A)	-5863(114)	-3971(64)	-3890(73)
H(8'B)	-5614(113)	-3281(101)	-5152(37)
H(9')	-8028(105)	-2723(100)	-3590(47)
H(11')	-8919(26)	1067(63)	-4361(52)
H(17A)	-4998(41)	5045(80)	-1266(55)
H(17B)	-3983(78)	4744(82)	-2410(21)
H(17C)	-3066(56)	5218(76)	-1129(51)
H(18A)	3850(47)	-0268(72)	-0787(61)
H(18B)	2397(84)	0466(75)	-0018(33)
H(18C)	2688(86)	0989(58)	-1271(57)
H(20A)	1222(80)	-5254(77)	-0802(35)
H(20B)	1994(50)	-4750(85)	-1894(54)
H(20C)	0318(75)	-5494(70)	-2134(46)
H(21A)	-8463(84)	-4692(41)	-4858(63)
H(21B)	-8446(83)	-3690(75)	-5881(37)
H(21C)	-9895(38)	-3477(76)	-5017(65)

Fractional Atomic Coordinates ($x10^4$) with e.s.d.'s

Molecule 2

TABLE 12.2

Anisotropic Temperature

Molecule 1

Atom	U_{11}	U_{22}
O(1)	23(2)	36(3)
O(2)	81(4)	17(2)
O(3)	59(3)	22(2)
O(4)	119(6)	48(3)
O(5)	61(4)	42(3)
O(6)	90(4)	25(3)
O(7)	119(6)	49(4)
O(8)	64(4)	22(3)
C(1)	70(6)	42(4)
C(2)	64(5)	28(4)
C(3)	56(5)	38(4)
C(4)	45(4)	33(4)
C(5)	68(6)	31(4)
C(6)	48(4)	31(4)
C(7)	37(4)	19(3)
C(8)	73(8)	35(5)
C(9)	72(6)	36(4)
C(10)	17(3)	30(3)
C(11)	44(5)	41(4)
C(12)	42(4)	36(4)
C(13)	53(5)	23(3)
C(14)	71(6)	31(4)
C(15)	34(4)	46(4)
C(16)	89(7)	30(4)
C(17)	94(8)	25(4)
C(18)	54(5)	59(5)
C(19)	50(5)	32(4)
C(20)	88(7)	42(5)
C(21)	45(5)	51(5)

U_{33}	U_{12}	U_{13}	U_{23}
59(3)	4(2)	-2(2)	2(2)
40(3)	2(2)	-13(3)	7(2)
42(3)	1(2)	-6(3)	1(2)
39(3)	-2(3)	27(3)	5(3)
54(3)	11(3)	-6(3)	7(3)
31(3)	19(3)	-8(3)	-2(2)
44(3)	13(4)	35(4)	6(3)
56(3)	10(2)	1(3)	3(2)
27(4)	31(4)	10(4)	16(3)
47(4)	11(4)	9(4)	2(3)
33(4)	9(4)	5(4)	15(3)
28(3)	16(3)	19(3)	5(3)
35(4)	3(4)	-4(4)	23(3)
32(4)	0(3)	-3(3)	9(3)
29(3)	-3(3)	5(3)	6(3)
113(9)	3(5)	-47(6)	6(5)
57(5)	0(4)	-22(5)	6(4)
37(4)	-7(3)	-8(3)	19(3)
41(4)	4(3)	-15(3)	2(3)
39(4)	13(3)	20(3)	-6(3)
22(3)	5(3)	-1(3)	7(3)
35(4)	2(4)	6(4)	-6(3)
34(4)	-1(3)	-9(3)	4(3)
50(5)	0(4)	-3(5)	-5(4)
82(7)	16(5)	-12(6)	4(4)
40(4)	4(4)	1(4)	13(4)
38(4)	-4(3)	0(4)	-2(3)
57(5)	10(5)	-3(5)	15(4)
49(5)	-19(4)	-9(4)	10(4)

Factor Coefficients ($x10^3$) with e.s.d.'s

TABLE 12.2(cont.)

H(2)	46(13)
H(6)	46(13)
H(8A)	80
H(8B)	80
H(9)	80
H(11)	46(13)
H(17A)	79(9)
H(17B)	79(9)
H(17C)	79(9)
H(18A)	79(9)
H(18B)	79(9)
H(18C)	79(9)
H(20A)	79(9)
H(20B)	79(9)
H(20C)	79(9)
H(21A)	79(9)
H(21B)	79(9)
H(21C)	79(9)

Anisotropic Temperature Coefficients ($x10^3$) with e.s.d.'s

Molecule 1

O(1')	99(5)
O(2')	29(3)
O(3')	60(4)
O(4')	137(7)
O(5')	40(3)
O(6')	33(3)
O(7')	156(7)
O(8')	85(5)
C(1')	36(4)
C(2')	43(4)
C(3')	42(4)
C(4')	51(5)
C(5')	29(3)
C(6')	56(5)
C(7')	66(5)
C(8')	82(7)
C(9')	67(6)
C(10')	86(7)
C(11')	55(5)
C(12')	72(6)
C(13')	45(4)
C(14')	30(3)
C(15')	53(4)
C(16')	46(5)
C(17')	105(8)
C(18')	48(5)
C(19')	91(7)
C(20')	103(8)
C(21')	85(7)

Anisotropic

TABLE 12.2(cont.)

29(3)	69(4)	-20(3)	-46(4)	15(3)
31(3)	46(3)	3(2)	1(2)	-2(2)
29(3)	46(3)	-9(2)	-2(3)	6(2)
40(4)	70(5)	32(4)	29(5)	22(4)
66(4)	68(4)	-14(3)	-25(3)	-2(3)
43(3)	38(3)	4(2)	4(2)	7(2)
52(4)	33(3)	18(4)	13(4)	8(3)
39(3)	80(5)	18(3)	-31(4)	1(3)
29(3)	33(4)	-26(3)	-3(3)	-2(3)
53(5)	33(4)	-28(4)	-14(3)	1(3)
54(5)	35(4)	-10(4)	-12(3)	-3(3)
35(4)	39(4)	-4(4)	-10(3)	11(3)
35(4)	23(3)	1(3)	5(3)	-10(3)
22(4)	53(5)	6(3)	-2(4)	-5(3)
40(4)	26(4)	4(4)	-5(4)	5(3)
24(4)	90(7)	-10(4)	-41(6)	4(4)
37(4)	60(5)	-15(4)	-30(5)	14(4)
35(4)	41(4)	3(4)	6(4)	-14(3)
29(4)	51(5)	6(4)	-1(4)	10(3)
41(5)	33(4)	-7(4)	-26(4)	12(3)
29(4)	27(3)	-4(3)	1(3)	-1(3)
23(3)	24(3)	-4(3)	-6(3)	7(2)
14(3)	23(3)	3(3)	5(3)	7(2)
42(4)	41(4)	3(4)	-1(4)	5(4)
49(5)	49(5)	0(5)	7(5)	5(4)
76(7)	71(6)	-17(5)	-27(5)	-11(5)
38(5)	47(5)	-5(5)	-12(5)	15(4)
62(6)	63(6)	42(6)	18(6)	18(5)
28(4)	85(7)	-19(4)	-13(6)	9(4)

Temperature Factor Coefficients ($x10^3$) with e.s.d.'s

Molecule 2

TABLE 12.2(cont.)

H(2')	46(9)
H(6')	46(9)
H(8'A)	80
H(8'B)	80
H(9')	80
H(11')	46(9)
H(17'A)	79(9)
H(17'B)	79(9)
H(17'C)	79(9)
H(18'A)	79(9)
H(18'B)	79(9)
H(18'C)	79(9)
H(20'A)	79(9)
H(20'B)	79(9)
H(20'C)	79(9)
H(21'A)	79(9)
H(21'B)	79(9)
H(21'C)	79(9)

Anisotropic Temperature Factor Coefficients ($\times 10^3$) with e.s.d.'s

Molecule 2

TABLE 12.3

O(1)	C(9)	1.429(9)	C(3)	C(4)	1.341(10)
O(1)	C(10)	1.314(8)	C(4)	C(5)	1.368(11)
O(2)	C(12)	1.361(9)	C(5)	C(6)	1.403(11)
O(2)	C(13)	1.404(8)	C(5)	C(14)	1.528(10)
O(3)	C(1)	1.371(9)	C(6)	C(7)	1.370(10)
O(3)	C(16)	1.366(11)	C(7)	C(8)	1.464(12)
O(4)	C(16)	1.208(12)	C(7)	C(15)	1.436(9)
O(5)	C(3)	1.336(10)	C(8)	C(9)	1.517(15)
O(5)	C(18)	1.395(10)	C(9)	C(21)	1.471(13)
O(6)	C(4)	1.442(8)	C(10)	C(11)	1.334(10)
O(6)	C(19)	1.323(9)	C(10)	C(15)	1.508(9)
O(7)	C(19)	1.139(10)	C(11)	C(12)	1.453(11)
O(8)	C(12)	1.193(9)	C(13)	C(14)	1.366(11)
C(1)	C(2)	1.387(12)	C(13)	C(15)	1.330(10)
C(1)	C(14)	1.367(11)	C(16)	C(17)	1.494(12)
C(2)	C(3)	1.453(10)	C(19)	C(20)	1.556(11)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

Molecule 1

TABLE 12.3(cont.)

O(1') - C(9')	1.486(10)	C(3') - C(4')	1.401(11)
O(1') - C(10')	1.401(11)	C(4') - C(5')	1.416(10)
O(2') - C(12')	1.437(9)	C(5') - C(6')	1.443(10)
O(2') - C(13')	1.333(8)	C(5') - C(14')	1.392(9)
O(3') - C(1')	1.446(8)	C(6') - C(7')	1.335(11)
O(3') - C(16')	1.363(10)	C(7') - C(8')	1.523(13)
O(4') - C(16')	1.168(12)	C(7') - C(15')	1.436(9)
O(5') - C(3')	1.370(9)	C(8') - C(9')	1.342(15)
O(5') - C(18')	1.467(11)	C(9') - C(21')	1.554(13)
O(6') - C(4')	1.400(8)	C(10')- C(11')	1.400(11)
O(6') - C(19')	1.409(11)	C(10')- C(15')	1.340(12)
O(7') - C(19')	1.224(11)	C(11')- C(12')	1.404(11)
O(8') - C(12')	1.239(9)	C(13')- C(14')	1.453(9)
C(1') - C(2')	1.345(10)	C(13')- C(15')	1.431(9)
C(1') - C(14')	1.464(9)	C(16')- C(17')	1.504(12)
C(2') - C(3')	1.377(11)	C(19')- C(20')	1.428(13)

Bond Lengths (Å) (Non Hydrogen Atoms) with e.s.d.'s

Molecule 2

TABLE 12.4

C(10) - O(1) - C(9)	119.8(6)	C(8) - C(9) - O(1)	113.2(7)
C(21) - C(9) - O(1)	108.5(7)	C(11) - C(10) - O(1)	121.1(6)
C(15) - C(10) - O(1)	121.3(6)	C(13) - O(2) - C(12)	123.9(6)
O(8) - C(12) - O(2)	116.5(7)	C(11) - C(12) - O(2)	116.4(6)
C(14) - C(13) - O(2)	115.4(6)	C(15) - C(13) - O(2)	119.4(7)
C(16) - O(3) - C(1)	119.4(6)	C(2) - C(1) - O(3)	112.1(7)
C(14) - C(1) - O(3)	120.6(7)	O(4) - C(16) - O(3)	124.8(7)
C(17) - C(16) - O(3)	112.3(7)	C(17) - C(16) - O(4)	122.9(9)
C(18) - O(5) - C(3)	123.6(6)	C(2) - C(3) - O(5)	124.1(7)
C(4) - C(3) - O(5)	119.2(7)	C(19) - O(6) - C(4)	118.1(6)
C(3) - C(4) - O(6)	117.0(7)	C(5) - C(4) - O(6)	117.0(6)
O(7) - C(19) - O(6)	124.0(7)	C(20) - C(19) - O(6)	105.6(6)
C(20) - C(19) - O(7)	130.3(7)	C(11) - C(12) - O(8)	127.0(7)
C(14) - C(1) - C(2)	127.3(7)	C(3) - C(2) - C(1)	118.4(7)
C(5) - C(14) - C(1)	112.2(7)	C(13) - C(14) - C(1)	131.7(7)
C(4) - C(3) - C(2)	116.7(7)	C(5) - C(4) - C(3)	125.8(7)
C(6) - C(5) - C(4)	125.5(7)	C(14) - C(5) - C(4)	119.2(7)
C(14) - C(5) - C(6)	115.1(7)	C(7) - C(6) - C(5)	126.0(7)
C(13) - C(14) - C(5)	116.1(7)	C(8) - C(7) - C(6)	126.9(7)
C(15) - C(7) - C(6)	115.4(6)	C(15) - C(7) - C(8)	117.5(7)
C(9) - C(8) - C(7)	115.3(8)	C(10) - C(15) - C(7)	118.4(6)
C(13) - C(15) - C(7)	121.5(7)	C(21) - C(9) - C(8)	116.7(7)
C(15) - C(10) - C(11)	117.4(6)	C(12) - C(11) - C(10)	122.4(7)
C(13) - C(15) - C(10)	120.1(7)	C(15) - C(13) - C(14)	125.3(7)

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

Molecule 1

TABLE 12.4(cont.)

C(10')- O(1') - C(9')	113.2(7)	C(8') - C(9') - O(1')	117.0(8)
C(21')- C(9') - O(1')	102.7(7)	C(11')- C(10')- O(1')	112.8(8)
C(15')- C(10')- O(1')	121.8(7)	C(13')- O(2') - C(12')	121.3(6)
O(8') - C(12')- O(2')	113.3(7)	C(11')- C(12')- O(2')	120.1(7)
C(14')- C(13')- O(2')	119.1(6)	C(15')- C(13')- O(2')	119.2(6)
C(16')- O(3') - C(1')	115.7(6)	C(2') - C(1') - O(3')	118.6(6)
C(14')- C(1') - O(3')	118.7(6)	O(4') - C(16')- O(3')	124.9(8)
C(17')- C(16')- O(3')	107.4(7)	C(17')- C(16')- O(4')	127.6(8)
C(18')- O(5') - C(3')	116.6(7)	C(2') - C(3') - O(5')	125.3(7)
C(4') - C(3') - O(5')	117.5(7)	C(19')- O(6') - C(4')	113.6(6)
C(3') - C(4') - O(6')	115.5(7)	C(5') - C(4') - O(6')	119.1(6)
O(7') - C(19')- O(6')	121.7(8)	C(20')- C(19')- O(6')	112.1(8)
C(20')- C(19')- O(7')	126.1(8)	C(11')- C(12')- O(8')	126.4(8)
C(14')- C(1') - C(2')	122.2(6)	C(3') - C(2') - C(1')	121.6(7)
C(5') - C(14')- C(1')	117.0(6)	C(13')- C(14')- C(1')	125.4(6)
C(4') - C(3') - C(2')	117.2(7)	C(5') - C(4') - C(3')	123.4(7)
C(6') - C(5') - C(4')	121.2(6)	C(14')- C(5') - C(4')	118.0(6)
C(14')- C(5') - C(6')	120.5(6)	C(7') - C(6') - C(5')	120.8(7)
C(13')- C(14')- C(5')	117.6(6)	C(8') - C(7') - C(6')	125.5(7)
C(15')- C(7') - C(6')	122.4(7)	C(15')- C(7') - C(8')	111.5(7)
C(9') - C(8') - C(7')	118.2(8)	C(10')- C(15')- C(7')	124.8(7)
C(13')- C(15')- C(7')	116.5(7)	C(21')- C(9') - C(8')	121.4(8)
C(15')- C(10')- C(11')	125.3(8)	C(12')- C(11')- C(10')	115.3(8)
C(13')- C(15')- C(10')	118.6(6)	C(15')- C(13')- C(14')	121.6(6)

Valency Angles (°) (Non Hydrogen Atoms) with e.s.d.'s

Molecule 2

TABLE 12.5

C(10) - O(1) - C(9) - C(8)	-40.6(10)
C(9) - O(1) - C(10) - C(11)	-166.5(7)
C(13) - O(2) - C(12) - O(8)	177.3(6)
C(12) - O(2) - C(13) - C(14)	177.8(7)
C(16) - O(3) - C(1) - C(2)	108.2(8)
C(1) - O(3) - C(16) - O(4)	-5.3(12)
C(18) - O(5) - C(3) - C(2)	-5.2(12)
C(19) - O(6) - C(4) - C(3)	-81.2(8)
C(4) - O(6) - C(19) - O(7)	-8.9(11)
O(3) - C(1) - C(2) - C(3)	175.2(7)
O(3) - C(1) - C(14) - C(5)	-179.1(6)
C(2) - C(1) - C(14) - C(5)	-0.1(12)
C(1) - C(2) - C(3) - O(5)	-178.0(7)
O(5) - C(3) - C(4) - O(6)	-1.2(10)
C(2) - C(3) - C(4) - O(6)	178.2(6)
O(6) - C(4) - C(5) - C(6)	2.7(11)
C(3) - C(4) - C(5) - C(6)	177.8(8)
C(4) - C(5) - C(6) - C(7)	180.0(7)
C(4) - C(5) - C(14) - C(1)	5.5(10)
C(6) - C(5) - C(14) - C(1)	-179.1(7)
C(5) - C(6) - C(7) - C(8)	176.2(8)
C(6) - C(7) - C(8) - C(9)	148.8(8)
C(6) - C(7) - C(15) - C(10)	-171.8(6)
C(8) - C(7) - C(15) - C(10)	3.0(10)
C(7) - C(8) - C(9) - O(1)	43.6(11)
O(1) - C(10) - C(11) - C(12)	177.0(7)
O(1) - C(10) - C(15) - C(7)	1.6(10)
C(11) - C(10) - C(15) - C(7)	-173.6(7)
C(10) - C(11) - C(12) - O(2)	4.1(11)
O(2) - C(13) - C(14) - C(1)	-1.5(13)
C(15) - C(13) - C(14) - C(1)	177.5(8)
O(2) - C(13) - C(15) - C(7)	178.1(6)
C(14) - C(13) - C(15) - C(7)	-0.8(12)

Torsion Angles (°) (Non

C(10)	- O(1) -	C(9) -	C(21)	-171.8(6)
C(9)	- O(1) -	C(10) -	C(15)	18.5(9)
C(13)	- O(2) -	C(12) -	C(11)	0.8(10)
C(12)	- O(2) -	C(13) -	C(15)	-1.2(10)
C(16)	- O(3) -	C(1) -	C(14)	-72.7(10)
C(1)	- O(3) -	C(16) -	C(17)	172.4(7)
C(18)	- O(5) -	C(3) -	C(4)	174.3(7)
C(19)	- O(6) -	C(4) -	C(5)	94.4(8)
C(4)	- O(6) -	C(19) -	C(20)	168.8(6)
C(14)	- C(1) -	C(2) -	C(3)	-3.9(13)
O(3)	- C(1) -	C(14) -	C(13)	-0.7(14)
C(2)	- C(1) -	C(14) -	C(13)	178.3(8)
C(1)	- C(2) -	C(3) -	C(4)	2.6(11)
O(5)	- C(3) -	C(4) -	C(5)	-176.4(7)
C(2)	- C(3) -	C(4) -	C(5)	3.1(12)
O(6)	- C(4) -	C(5) -	C(14)	177.5(6)
C(3)	- C(4) -	C(5) -	C(14)	-7.3(12)
C(14)	- C(5) -	C(6) -	C(7)	4.9(11)
C(4)	- C(5) -	C(14) -	C(13)	-173.2(7)
C(6)	- C(5) -	C(14) -	C(13)	2.2(10)
C(5)	- C(6) -	C(7) -	C(15)	-9.6(11)
C(15)	- C(7) -	C(8) -	C(9)	-25.3(12)
C(6)	- C(7) -	C(15) -	C(13)	7.5(10)
C(8)	- C(7) -	C(15) -	C(13)	-177.8(8)
C(7)	- C(8) -	C(9) -	C(21)	170.6(8)
C(15)	- C(10) -	C(11) -	C(12)	-7.8(11)
O(1)	- C(10) -	C(15) -	C(13)	-177.7(6)
C(11)	- C(10) -	C(15) -	C(13)	7.2(10)
C(10)	- C(11) -	C(12) -	O(8)	-172.0(8)
O(2)	- C(13) -	C(14) -	C(5)	177.0(6)
C(15)	- C(13) -	C(14) -	C(5)	-4.1(11)
O(2)	- C(13) -	C(15) -	C(10)	-2.7(10)
C(14)	- C(13) -	C(15) -	C(10)	178.4(7)

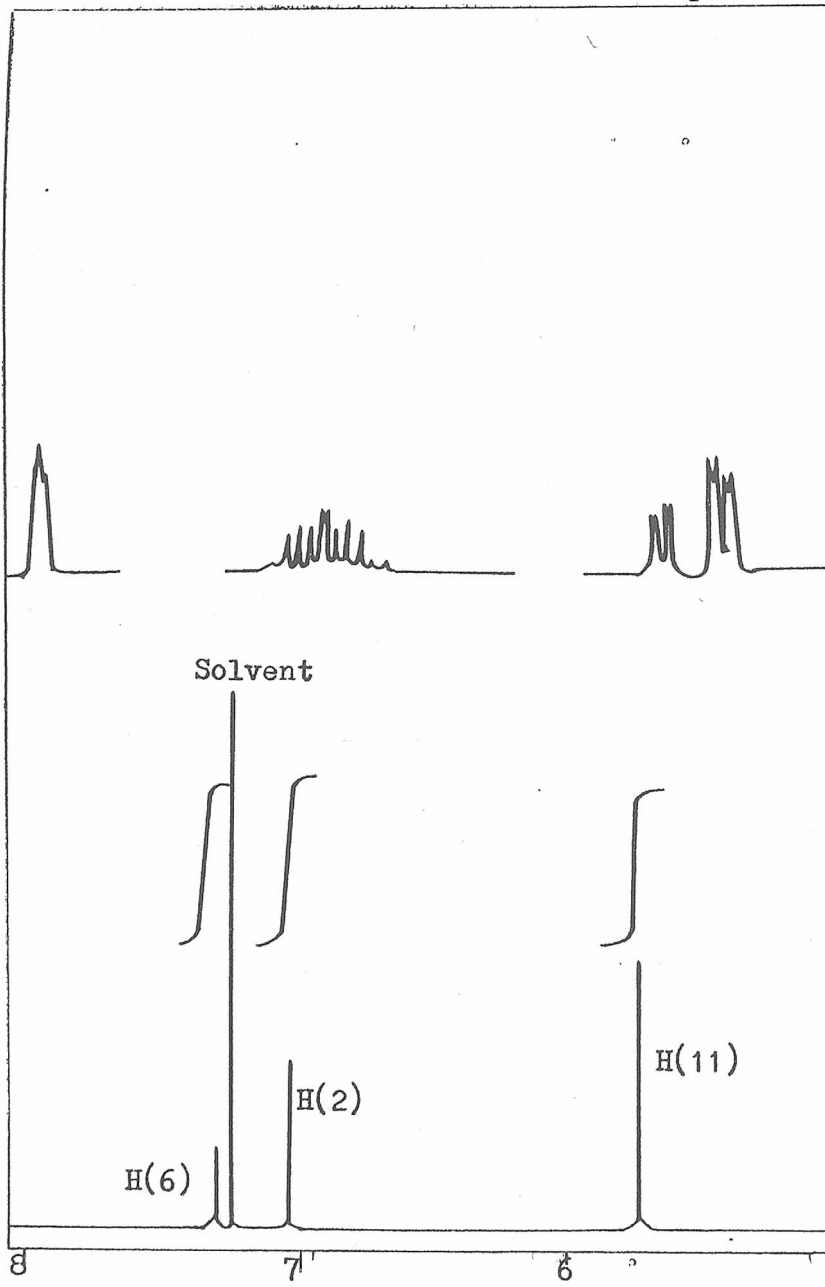
Hydrogen Atoms) with e.s.d.'s

TABLE 12.5(cont.)

C(10')- O(1') - C(9') - C(8')	-39.5(11)	C(10')- O(1') - C(9') - C(21')	-175.2(7)
C(9') - O(1') - C(10')- C(11')	-167.6(7)	C(9') - O(1') - C(10')- C(15')	15.3(11)
C(13')- O(2') - C(12')- O(8')	-179.0(6)	C(13')- O(2') - C(12')- C(11')	5.6(10)
C(12')- O(2') - C(13')- C(14')	176.8(6)	C(12')- O(2') - C(13')- C(15')	-4.1(9)
C(16')- O(3') - C(1') - C(2')	-108.5(8)	C(16')- O(3') - C(1') - C(14')	79.4(8)
C(1') - O(3') - C(16')- O(4')	-0.6(11)	C(1') - O(3') - C(16')- C(17')	-177.4(6)
C(18')- O(5') - C(3') - C(2')	9.4(11)	C(18')- O(5') - C(3') - C(4')	-172.6(7)
C(19')- O(6') - C(4') - C(3')	85.7(8)	C(19')- O(6') - C(4') - C(5')	-109.8(8)
C(4') - O(6') - O(19')- O(7')	9.9(11)	C(4') - O(6') - C(19')- C(20')	-174.3(7)
O(3') - C(1') - C(2') - C(3')	-176.6(7)	C(14')- C(1') - C(2') - C(3')	-4.8(11)
O(3') - C(1') - C(14')- C(5')	173.8(6)	O(3') - C(1') - C(14')- C(13')	-3.2(10)
C(2') - C(1') - C(14')- C(5')	2.0(10)	C(2') - C(1') - C(14')- C(13')	-175.0(7)
C(1') - C(2') - C(3') - O(5')	178.6(7)	C(1') - C(2') - C(3') - C(4')	0.6(12)
O(5') - C(3') - C(4') - O(6')	-7.8(10)	O(5') - C(3') - C(4') - C(5')	-171.6(7)
C(2') - C(3') - C(4') - O(6')	170.4(7)	C(2') - C(3') - C(4') - C(5')	6.6(11)
O(6') - C(4') - C(5') - C(6')	13.8(10)	O(6') - C(4') - C(5') - C(14')	-172.5(6)
C(3') - C(4') - C(5') - C(6')	177.1(7)	C(3') - C(4') - C(5') - C(14')	-9.3(11)
C(4') - C(5') - C(6') - C(7')	-179.5(7)	C(14')- C(5') - C(6') - C(7')	7.0(11)
C(4') - C(5') - C(14')- C(1')	4.8(9)	C(4') - C(5') - C(14')- C(13')	-178.1(6)
C(6') - C(5') - C(14')- C(1')	178.4(6)	C(6') - C(5') - C(14')- C(13')	-4.4(9)
C(5') - C(6') - C(7') - C(8')	-179.6(8)	C(5') - C(6') - C(7') - C(15')	-9.2(12)
C(6') - C(7') - C(8') - C(9')	151.4(9)	C(15')- C(7') - C(8') - C(9')	-19.9(12)
C(6') - C(7') - C(15')- C(10')	-176.2(8)	C(6') - C(7') - C(15')- C(13')	8.5(11)
C(8') - C(7') - C(15')- C(10')	-4.6(11)	C(8') - C(7') - C(15')- C(13')	-179.9(7)
C(7') - C(8') - C(9') - O(1')	42.2(12)	C(7') - C(8') - C(9') - C(21')	169.2(8)
O(1') - C(10')- C(11')- C(12')	-179.6(7)	C(15')- C(10')- C(11')- C(12')	-2.6(13)
O(1') - C(10')- C(15')- C(7')	5.7(12)	O(1') - C(10')- C(15')- C(13')	-179.1(7)
C(11')- C(10')- C(15')- C(7')	-171.1(8)	C(11')- C(10')- C(15')- C(13')	4.1(12)
C(10')- C(11')- C(12')- O(2')	-2.2(11)	C(10')- C(11')- C(12')- O(8')	-177.0(8)
O(2') - C(13')- C(14')- C(1')	0.2(10)	O(2') - C(13')- C(14')- C(5')	-176.7(6)
C(15')- C(13')- C(14')- C(1')	-178.9(6)	C(15')- C(13')- C(14')- C(5')	4.1(9)
O(2') - C(13')- C(15')- C(7')	175.0(6)	O(2') - C(13')- C(15')- C(10')	-0.6(10)
C(14') - C(13') - C(15') - C(7')	-5.9(10)	C(14') - C(13') - C(15') - C(10')	178.5 (7)

Torsion Angles (°) (Non Hydrogen Atoms)with e.s.d.'s

^{1}H nmr Spectrum



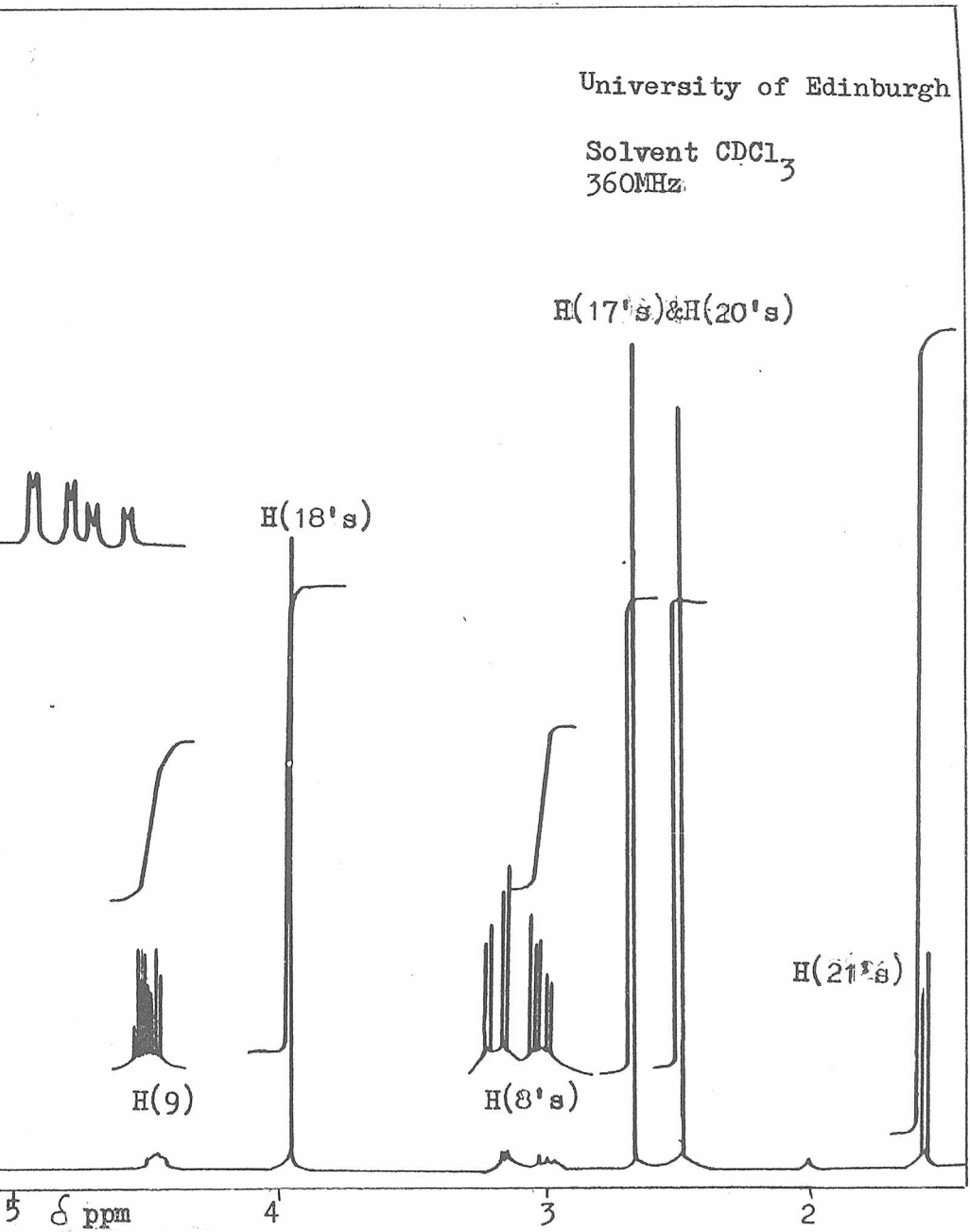

University of Edinburgh
Solvent CDCl3
360MHz
H(17's)&H(20's)
H(18's)
H(9)
H(8's)
H(21's)
5
δ ppm
4
3
2

FIG. 12.5

associated with the disorder. As refinement would not proceed beyond an R value of 9%, it was decided to change the space group from $P\bar{1}$ to P1. This decision was proved to be correct. as the structure is slightly optically active and could not possibly exist in a centrosymmetric space group. This however did not solve the problem of spurious electron density. Further problems were encountered in that one of the molecules appeared to have a single bond between C(8) and C(9) and the other a double bond between these two atoms. On the assumption that a mistake had been made during the analysis, it was repeated without recourse to the procedure used previously. The results were the same, and therefore reinforced the theory of disorder. It was found impossible to determine an occupancy factor for the disorder, although there were several electron density peaks in geometrically sensible positions. It was therefore decided to continue the analysis with this discrepancy.

The end product of this analysis is very unsatisfactory, and it is doubtful whether the results may be regarded as correct with respect to the stereochemistry. The esd's associated with the structural parameters are also high, and it would probably be better to repeat the analysis using a different derivative. It is possible that an improvement could be made on the results by refinement of all the atoms, including the hydrogens visible on the difference map in an attempt to determine the hydrogen atom positions more accurately.

Structural 12.4.2

The leucoacetate of ventilatone A is a very planar structure, most of the torsion angles being close to 0° or 180°. The methyl groups in both molecules are in the β orientation, although in molecule 1 the hydrogen position appears to be slightly distorted. The

oxymethyl groups in both molecules adopt the same orientation. The acetylene groups at C(1) and C(4) are different. In molecule 1 they are α and in molecule 2 they are β .

If the ^{1}Hnmr results had not been available for this structure, the analysis would have been completed with a single bond at C(8) - C(9) in one molecule and a double bond in the other. As may be seen from the ^{1}Hnmr, all the hydrogens are assigned and there is no evidence for the presence of a double bond. Although no data is available for the solubility of this substance in the deuterated chloroform used in the ^{1}Hnmr, it is possible that it is sparingly soluble (780 scans were required to obtain the ^{1}Hnmr spectrum indicating the use of a very weak solution). If the structure with the double bond between C(8) and C(9) were less soluble due to change in polarity, it is possible as an undissolved fraction, it was filtered off prior to the ^{1}Hnmr scan. There is a small peak which could respond to a methyl attached to a sp^2 carbon at δ 2.0 and it is possible that the peak of the C(8) hydrogen is too small to be observed.

In Apomorphine hydrochloride hydrate [134] which is shown in Fig. 12.6, a similar situation as with this structure was found to exist. Here there are also two molecules per assymetric unit. It is found that there is a marked difference in ring A of the two molecules, and that single bonds are shorter than normal. Double bond character is also noted in the valency angles. This structure was however solved successfully and shows no disorder. Other similar structures such as N,O - Diacetyl-4-hydroxynormantenine [135], Leucoxine [136], Isoboldine [136] and Acetyl-1-o-acetyl-6 Launelliptine [137] do not manifest this behaviour, therefore the

above is probably only a coincidence and not a property of this type of structure.

Molecular Mechanics 12.4.3

A satisfactory result was not obtained using molecular mechanics calculations - the energy rose after an initial reduction. This was probably due to the molecule not conforming to accepted geometries. No further calculations were carried out.

CHAPTER THIRTEEN

UNSOLVED STRUCTURES PLUMBASEYLANONE AND BRYAQUINONE

Unsolved Structures Plumbazeylanone and Bryaquinone

Plumbazeylanone 13.1

Introduction 13.1.1

Plumbazeylanone is thought to be a trimeric structure, which has been isolated from plumbago zeylanica [138]. It is thought to be derived from plumbagin [138].

After isolation various spectral analysis were carried out, and these suggested the structure shown in Fig. 13.1.1. The molecular formula is thought to be $C_{34}H_{24}O_9$

Experimental 13.1.2

The data were collected according to the procedure outlined in Chapter 2. 2349 independent reflexions were measured. The space group was chosen as C 2 , although it could also have been Cm or C2/m . The following procedures were then followed.

a. Data prepared for use in 'MULTAN 80' [72]. The E values exhibited a hypercentric distribution and the E map produced nothing.

b. A model of the structure was built up using the programme 'MODEL' [76], and the coordinates input to 'MULTAN', [72] but no fragment was obtained.

c. Data prepared for use in 'MITHRIL' [41] default values being used, and a fragment was produced. This fragment was input as a randomly oriented group, but produced nothing more.

d. Hard instruction used in 'MITHRIL' [41] and the fragment shown in Fig. 13.1.2 was visible from the E map. This again was input as a randomly oriented group, but produced nothing.

e. Very hard instruction used in 'MITHRIL', [41] but no geometrically sensible fragment was produced.

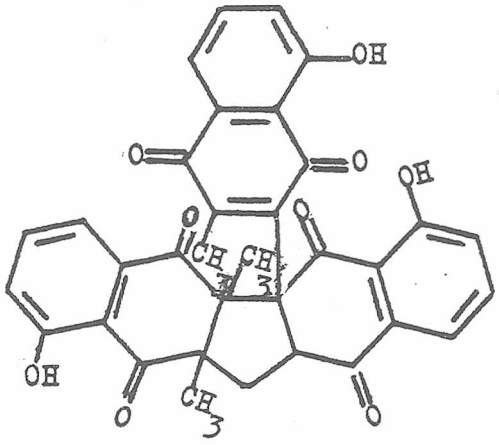


FIG.13.1.1

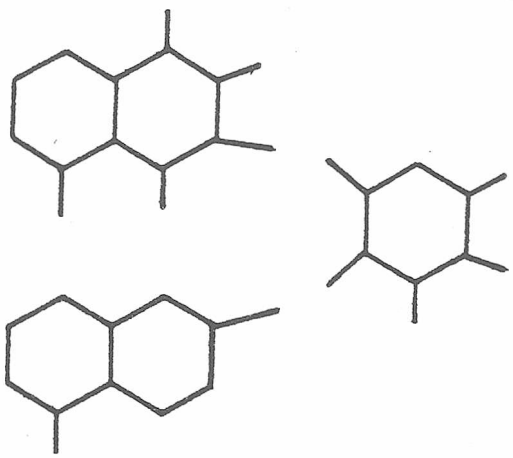


FIG. 13.1.2

f. The limit on the maximum value of |E|'s was set to 3.5 and the number of reflexions for which quartets were generated restricted to 100. A similar fragment to that in Fig. 13.1.2 was visible, but this did not give a satisfactory solution.

g. Karle recycling of the fragment as shown in Fig. 13.1.2 was tried. As the same fragment was reproduced, this was input to 'SHELX' [73] for calculation of a difference map, but this was unsuccessful.

No further improvement on the structure as shown in Fig.13.1.2 was ever found, but many other procedures were tried before the analysis was abandoned.

i. The origin fixing reflexions were weighted heavily on the side of the hol's as was the whole convergence map. therefore these reflexions were excluded from the map.

j. The statistically weighted tangent formula was implemented.

k. MAGEX routine was used with various numbers of primary reflexions.

l. Rantan was used with 100 phase sets.

m. Temperature factor of 5.0 introduced.

n. A totally unrelated molecule was introduced using the group command.

o. The temperature factor was lowered from 4.5 to 3.5.

p. The contents of the unit cell were doubled.

All the above were repeated using the space groups Cm and C2/m . Data were collected from two different crystals, but neither of these data sets furnished a solution.

Results 13.1.3

Monoclinic, C 2 , Z = 4, a = 12.620(6)Å, b = 17.159(9)Å, c = 14.483(14)Å, β = 95.89(6)°, U = 3119(4)Å^3, Dc = 1.35gcm^{-3}.

The ^{1}Hnmr for plumbazeylanone is shown in Fig. 13.1.3.

Discussion 13.1.4

Considerable difficulty was experienced in the analysis of plumbazeylanone, and as yet the structure is unsolved. The first problem was the space group. The systematic absences indicated the three space groups C 2, C2/m and Cm so C 2 was chosen simply because it is the most common. However, as explained none of these space groups proved successful in the structure elucidation.

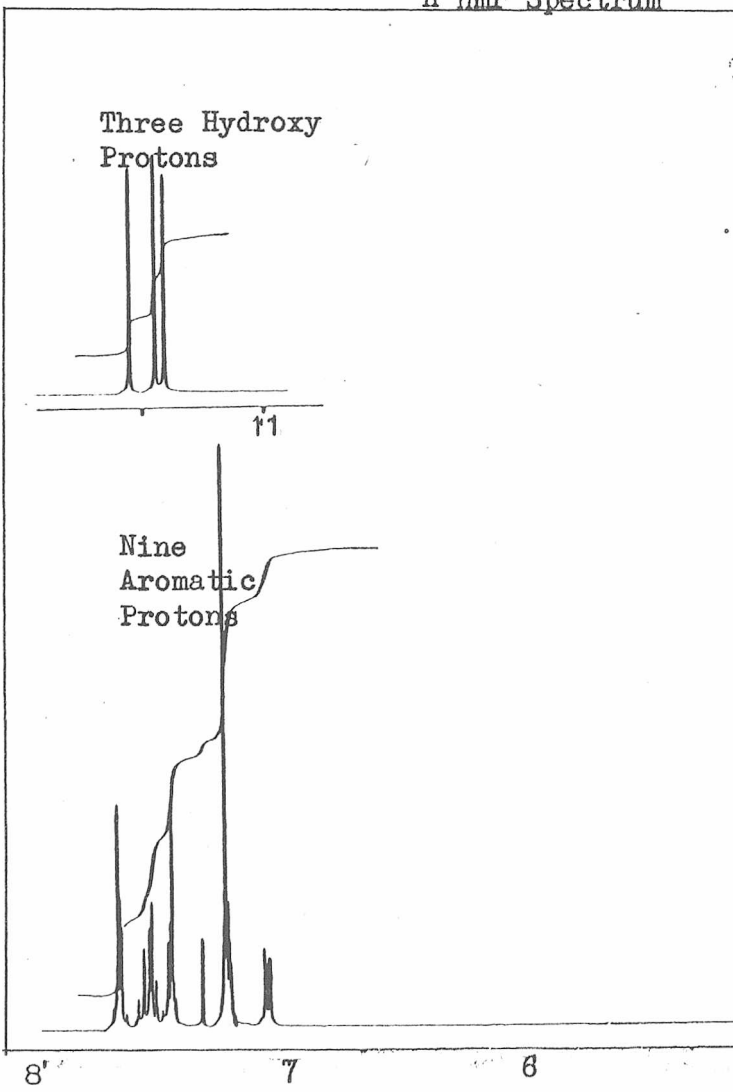
The E values were found to exhibit a hypercentric distribution. If the proposed structure is correct, then the molecule is fairly symmetrical, and this could again be a problem of pseudosymmetry, causing the intensity distribution to tend towards that of a centric crystal.

A model of the structure was used, so that molecular scattering factors could be implemented, but this made no difference. It must be remembered that the postulated structure is possibly incorrect, so input of a model based on this need have little effect.

An interesting fragment was obtained from 'MITHRIL'.[41] This fragment was input as a randomly oriented fragment in order to determine if it was correctly positioned.

Introducing a HARD instruction to the NORMAL routine, allows a new set of default values to be used for difficult structures. More triplets are generated, and quartets are also used. No reflexions are rejected by converge and more than one E map is produced. Quartets have been used successfully before in situations such as these[139], so there was the possibility that they would work now. It has also been found in the past that the E map with the

^{1}H nmr Spectrum



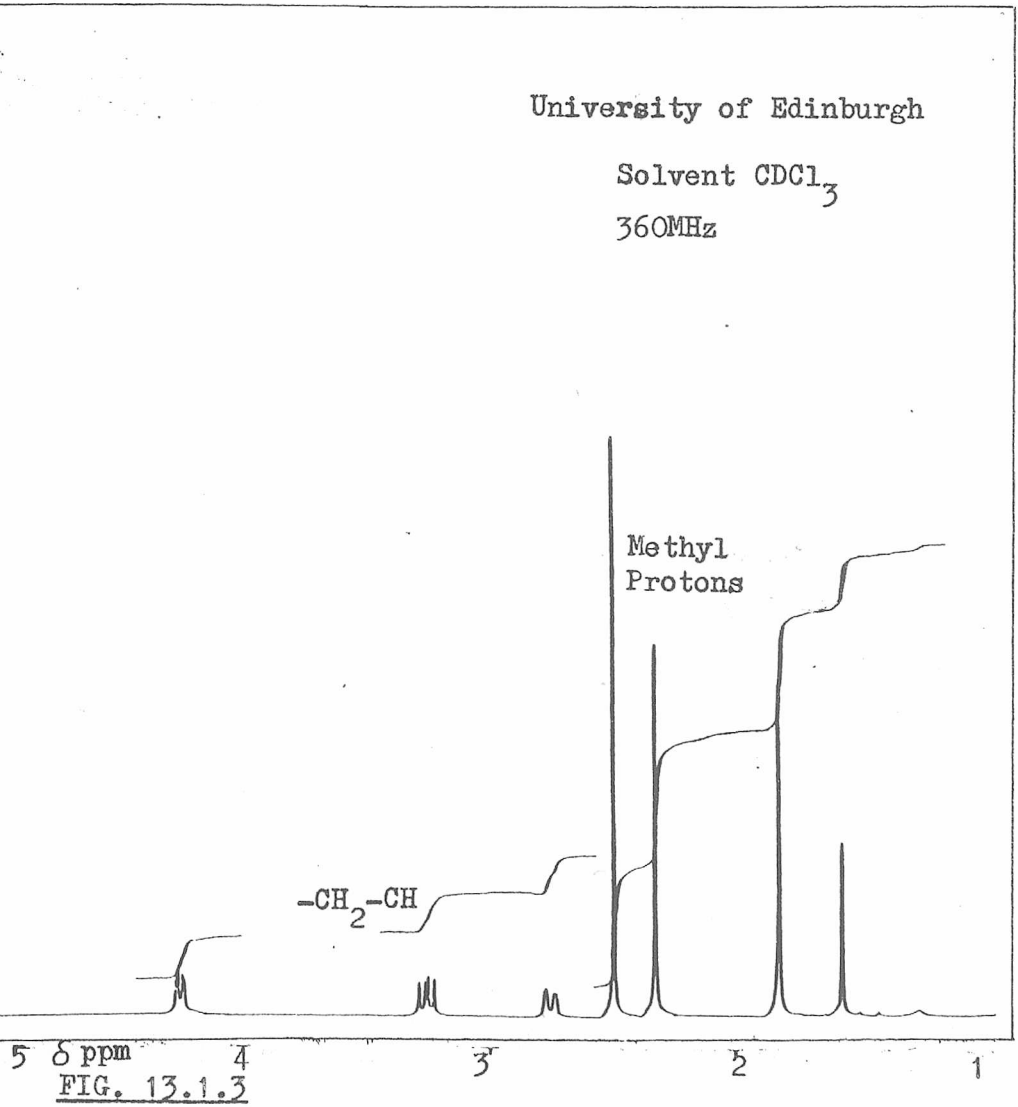

University of Edinburgh
Solvent $CDCl_3$
360MHz
Methyl
Protons
$-CH_2-CH$
5 δ ppm
4
3
2
1

FIG. 13.1.3

most favourable figures of merit does not necessarily forward a solution, therefore the production of more than one E map is desirable. However, in using the HARD instruction. it should be noted that all reflexions are used, and this could result in weak links in the phasing process. As stated a fragment was visible on the E map, but this was misplaced.

The VERY HARD instruction was used, as it employs positive quartets which may sometimes be useful. However, their use is still somewhat experimental and they were not helpful here.

Some of the E magnitudes were greater than five, and as very large E magnitudes can prevent direct methods from being successful, these were removed. The number of quartets used was restricted, as three phases which may only be approximate are used to generate a fourth, and therefore these are more likely to propagate errors than are triplets. Again a fragment which was misplaced was produced.

As the fragment of Fig. 13.1.3 was the closest to the proposed structure, Karle recycling was tried. Here if a partial structure is known it is possible that by application of the tangent formula a whole structure may be obtained [140].

It was thought that the programme in using hol reflexions extensively may have been jeopardising the phasing procedure. as $hOl_1 + hOl_2$ sum to give a third reflexion with a phase of which if incorrect, is out by a factor of 180°.

MAGEX was implemented as it is a method by which phases can be allocated to a larger set of starting reflexions than in 'MULTAN 80'[35]. It allows phases and the relationships between them to be considered simultaneously instead of stepwise, and thereby bypasses possible

weak links. MAGEX has been implemented for many structures where 'MULTAN'[72] has been unsuccessful.

RANTAN[40] overcomes the problems of phase expansion by assigning only random values with low weights to a number of phases and then refining them. This as in MAGEX does away with the stepwise permutation of phases and the need for a large starting set. This too has been employed successfully where 'MULTAN'[72] has failed.

Final attempts were made to change the phasing pathway by raising the temperature factor and input of an unrelated molecule. However these too were unsuccessful, and the analysis was becoming considerably time consuming. Although there were many things that could be tried, time would not permit, and the substance is currently being crystallised from a different solvent. in an attempt to obtain a more suitable crystal, and therefore a better data set.

It is thought that the crystal is disordered in some way. making analysis difficult. The structure as proposed has however been submitted for publication[138] without the confirmation of X-ray results and on the basis of the ^{1}Hnmr assignments shown in Fig.13.1.3.

Bryaquinone 13.2

Introduction 13.2.1

Bryaquinone is thought to have the structure as shown in Fig. 13.2.1. It is a red crystalline substance, the crystals being needle shaped. The molecular formula is $C_{19}H_{13}O_{8}$.

Experimental 13.2.2

The data were collected according to the procedure outlined in Chapter 2 for independent reflexions. The direct methods programme 'MULTAN'[72] was implemented, the E map being computed for 148 reflexions $|E|_{min} > 1.5$. It was then discovered that

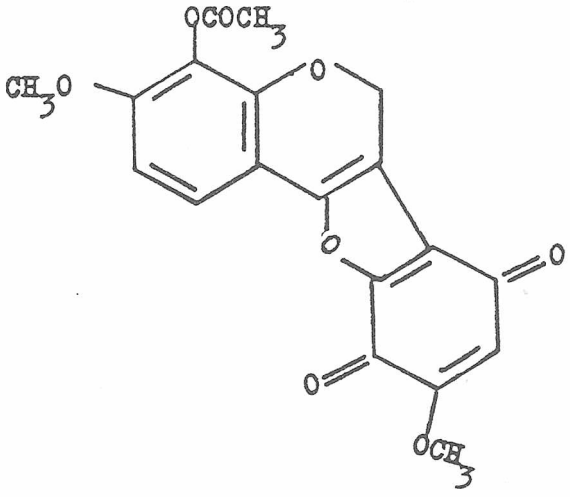


FIG. 13.2.1

a minimum of 308 reflexions were required, so the minimum E value was set to 1.0. The temperature factor was increased to 4.8 from 3.5 and the fragment shown in Fig. 13.2.2 was produced. The coordinates of this fragment were input to 'MULTAN'[72] as an oriented fragment. This gave the fragment shown in Fig. 13.2.3 and the figures of merit associated with it designated it as a correct fragment. This fragment was input as an oriented fragment but no further atoms were visible from the E map. The fragment retained its original coordinates. The coordinates of this fragment were input to 'SHELX'[73] for the calculation of a difference map, however this did not refine. The statistically weighted tangent formula was implemented and the fragment shown in Fig. 13.2.4 was obtained. This was also input as an oriented fragment, and was reproduced with the same coordinates. When the coordinates were used in least squares they did not refine. A model of bryaquinone was built up using the programme 'MODEL'[76] and this input to 'MULTAN'.[72] however nothing was produced. The probability for acceptance of the Σ_1 relationships was increased from 0.95 to 2.00 so that no Σ_1 relationships were used. This was unsuccessful. All phase relationships were used by setting Kmin equal to zero. This did not aid the structure analysis. The space group was changed to $P\bar{1}$, but no solution was found.

Results 13.2.3

Triclinic, P1, Z = 1, a = 4.200(6), b = 8.760(28), c = 16.426(33), $\alpha = 103.8(2)^\circ$, $\beta = 122.5(1)^\circ$, $\gamma = 91.8(2)^\circ$
$U = 491.50\ \text{Å}^3$, $Dc = 1.265\text{gcm}^{-1}$.

Discussion 13.2.4

Bryaquinone presents the very interesting case where the figures of merit for a particular phase set indicated that the

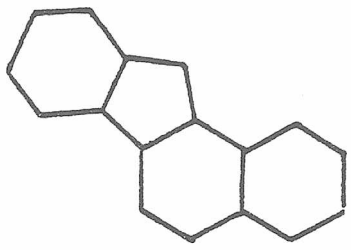


FIG. 13.2.2

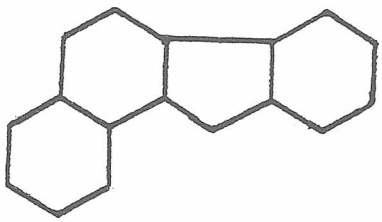


FIG. 13.2.3

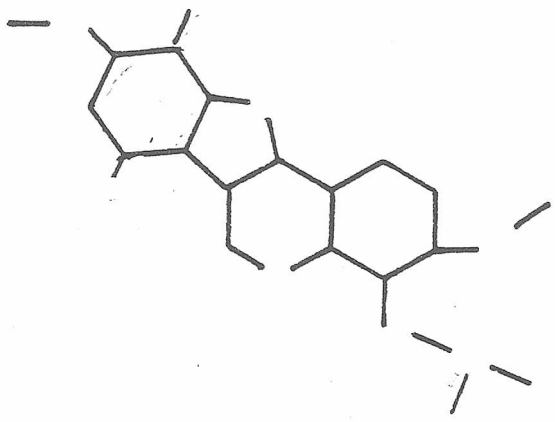


FIG. 13.2.4

solution was correct, but gave a structure that would not refine. As may be seen from Fig. 13.2.3 this fragment resembles closely the proposed structure. The figures of merit for the correct solution output by 'MULTAN' [72] were

ABS FOM	PSI (ZERO)	RESID
1.1121	1.097	14.77

It is not known why this phase set did not produce a solution. It was noted that one of the axes was very small. However doubling its value did not produce a solution.

It is thought that obtaining more crystals and recollecting the data for bryaquinone may be the only answer to this problem.

CHAPTER FOURTEEN

NATURAL PRODUCTS: METHODS OF ANALYSIS, A CRITICAL SURVEY

Natural Products: Methods of Analysis, a Critical Survey

Introduction 14.1

As stated previously, and as illustrated by this work, the structures of natural products are extremely varied, and often very complex. They can also vary widely in nature, e.g. oils, crystalline solids, amorphous solids etc. and for this reason, not one method can be deemed to be the most suitable for natural products. As will be shown in this survey, the most fruitful analysis usually involves two or more techniques.

Historical 14.2

Natural products have been studied for centuries, but the first detailed structural work appears around the beginning of the 20th century. Before the advent of spectroscopy, the only technique available for structural analysis was that of chemical investigation, i.e. by observation of physical properties and the reactions of a substance. The path followed was very similar in all cases and involved melting point, boiling point, density, refractive index, solubility in various solvents, qualitative elemental analysis, reaction to chemical reagents, formation of derivatives, degredation and finally total synthesis. This is obviously a very laborious and time consuming process, and can be seen in use in 1905, when attempts were made to determine the ring structure of the morphine type alkaloids [141,142,143]. Some contention between investigators as to the position of the nitrogen containing ring [145,146] is evident. Although the results of these investigations were close to the accepted structures, it is still shown to be a rather ambiguous method. In 1918 total synthesis was used as a method for the structural analysis of Guiaiene [147]. This method

left no doubt as to the actual structure. The same method was used in 1985 for the structure elucidation of Aristolindiquinone [148]. A complicated synthetic method was used, but was simplified by the identification of intermediate substances by spectroscopic methods. The problems of stereochemistry and configuration are shown to have been solved for the Cinchona alkaloids in 1944 [149], by a process of degradation. Progress is shown to have been slow, as early investigations are reported in 1922 [150].

There are many examples of the structures of natural products being solved in this way, but in general chemical methods are not used alone for structural analysis nowadays. After the second world war, it became more common for spectroscopic methods to be used in structural investigations.

Modern Methods of Analysis 14.3

If any of the modern structural papers are consulted it is obvious that ultraviolet, infrared, mass and nuclear magnetic resonance spectroscopy play a vital role in the studies. It is becoming more and more evident that nmr is a very general technique, and that it continues to grow in sophistication and therefore use.

Ultraviolet Spectroscopy 14.3.1

Ultraviolet spectroscopy is used mainly in the preliminary stages of an analysis. It is useful for determining relationships between functional groups, and for predicting the number of substituents in a ring system. Ultraviolet spectra therefore contain relatively little structural information, and their application to stereochemical investigations is virtually nil. However, ultraviolet is still used in the overall spectroscopic

investigation of a structure, as may be shown in the paper by Fukuyama et al [157].

Infrared Spectroscopy 14.3.2

Infrared is a slightly more versatile spectroscopic method, in that it is useful for the identification of functional groups [152]. Infrared spectroscopy is also of value in the study of diastereoisomers [12]. It has also contributed much in the conformational studies of molecules [153]. Information about hydrogen bonding is also available from infrared spectra.

Chiroptical Spectroscopic Methods 14.3.3

Optical rotatory dispersion and circular dichroism, are used in the measurement of cotton effects. The sign and amplitude of the cotton effect are related to the dissymmetry of the molecule, and therefore this information is invaluable in the study of absolute configurations and conformations [154].

Nuclear Magnetic Resonance 14.3.4

nmr studies have progressed considerably with the advent of Fourier techniques, as only small sample sizes are necessary in order to obtain a spectrum. Only minute quantities of natural products are usually isolated, but with continuous wave nmr, concentration was a problem. Various techniques have been reported as having been used in the study of natural products.

a. Conventional ^{1}Hnmr and ^{13}Cnmr

These have both been used successfully for a very diverse range of natural products. Some of the structural types are terpenes [155], antibiotics [156], steroids [157], proteins [158], alkaloids [159], β-diketones [160], guaianolides [161], and many others. The main

information obtained from nmr is on the basis of chemical shift and coupling constants. A great deal about both the structure and the stereochemistry of a system may be learned from nmr. It is possible to distinguish cis and trans isomers [162]. Conformationsl analysis has however to be carried out at low temperatures, so that exchange between conformation is at a slow enough speed to measure. The determination of coupling constants is useful in the assignment of configuration. The use of lanthide shift reagents also aids in the determination of configuration and conformation [163,164]. In C^{13} spectra, the difference in chemical shift observed with diastereoisomeric molecules is more easily observed than in ^{1}Hnmr. Hydrogen bonding can be determined from nmr spectra.

b. Non-conventional techniques

Recent developments in nmr techniques have led to an increase in the number of methods available for the study of natural products. These are Nuclear overhauser enhancements [165,166], Two dimensional techniques [167,168], Difference spectroscopy [169,170] and Relaxation methods [171]. These methods have a much more general applicability to structural studies.

Selective radiation of a proton can either produce a small or large nuclear overhauser enhancement of another proton depending on the relationship between the two, allowing detailed structural information to be obtained. Most useful information from this is obtained when the enhancement level is 1% or less, which cannot be observed normally. This problem in solved by difference spectra [165] where a control spectrum without NOE is subtracted from the enhanced spectrum, allowing only the changes to be recorded. Difference spectroscopy can also be used to enhance the information obtained by

spin decoupling [165]. Here buried multiplets are more easily identified for both homonuclear and heteronuclear coupling. Noe difference spectroscopy has become a powerful tool for assigning the proton spectra of complex natural products such as steroids and alkaloids, in addition to the proteins for which it was originally developed. The most important aspect of it is the ability to reveal hidden multiplets. It is possible to determine conformations by this method. Often however, information is built up from a series of spectra.

Relaxation rates are also a valuable tool in structure determination, as different protons relax at different rates, e.g. methine protons relax more slowly than methylene protons in rings B and C of 1-Dehydrotestosterone [165]. However this method is really only applicable to rigid molecules for the solving of geometrical problems [172]. However when both the mobility and geometry of molecules are known, then relaxation may be used to assign signals to specific protons [172].

Two dimensional nmr produces a spectrum with chemical shift information along one frequency axis, and coupling constant information along the other axis. It becomes possible by this to measure chemical shifts and coupling constants in a region of the spectrum which normally has so many overlapping multiplets that analysis is impossible. Two dimensional nmr has been successfully used in the structure determination of Talaromycin B [173].

In attempting to determine buried connections, two other techniques have been used in the study of natural products, those of COSY [174] and INADEQUATE. COSY is useful in that it can be adjusted to emphasize long range coupling connectivities. It has

been used in the structural analysis of Cytochalasin B [175].

INADEQUATE is used to give all carbon-carbon connectivities in a molecule. Proton-Proton methods are currently being developed. INADEQUATE has been used successfully for the fungal metabolite oxyrapentyn [176].

A strategy for the solving of the structures of natural products has been outlined by Hall and Sanders [165], which involves preliminary conventional ^{1}H and ^{13}Cnmr, followed by relaxation spectra, then measurement of decoupling difference spectra, then measurement of Noe difference spectra, and finally measurement of 2DJ spectra. This strategy has been used successfully for Vinblastine alkaloids [177] and enkephalin amongst others [178].

Mass Spectroscopy 14.3.5

The mass spectrum helps to determine the structure of a new compound in several different ways. It can give an exact molecular weight, it can give a molecular formula, and can indicate the presence in a molecule of certain structural units. However, although mass spectrometry is a powerful tool for the elucidation of structures, as exhibited for naturally occuring alkyl porphyrins [179], it is not generally used without reference to other spectral methods. This may been seen for the structures of Hypoestestatins 1 and 2 which were determined on the basis of mass spectra and ^{1}Hnmr [180].

Although this is the situation at present, it is possible that in the future positive ion chemical ionisation mass spectrometry [181] may play a more active role in the structural elucidation of natural products.

X-Ray Diffraction Analysis 14.3.6

X-ray diffraction analysis is the only method of structural

analysis which provides a complete picture of a molecule in respect to both overall structure and geometry. This method has been used extensively for a wide range of natural products.

X-ray diffraction became more applicable to natural products with the advent of direct methods [7], for reasons stated earlier. As direct methods became more sophisticated, with the wide use of quartets, quintets, random phasing, magic integers etc., the number of natural products for which the X-ray structure will be obtained, will increase.

X-ray analysis provides information on the stereochemistry and conformation (solid state) of the substance under study. Hydrogen bonding can also be detected.

Again however, X-ray analysis is rarely used on its own as a structural tool, as spectral and chemical analysis are usually carried out prior to the X-ray analysis of a molecule in order to give an overall picture of the structure and its chemical formula. This aids in the subsequent structure determination.

Molecular Mechanics 14.3.7

Empirical force field calculations are useful in this context only in conformational studies, and for predicting the orientation that two parts of a molecule may adopt relative to one another.

Molecular mechanics calculations have been used extensively in the study of the conformations of natural products [182]. They may be used too for determining geometries of postulated structures, and for conformational interconversion pathways. They may help in structural studies by indicating which conformation a structure is likely to adopt.

Other Methods 14.3.8

It is possible to examine small organic molecules by neutron

diffraction[183]. More accurate hydrogen atom positions may be obtained by this method, but it requires larger crystals.

Discussion 14.4

Most structural papers cite two or more of the aforementioned techniques. As seen, only X-ray analysis can give most of the information required about a structure, and even here with small organic molecules the absolute configuration may not be determined.

For some molecules the only method which will satisfactorily determine the structure is X-ray diffraction analysis, e.g. halogenated terpenes[184], where mass fragmentation patterns are unreliable and nmr results are ambiguous. However the only disadvantage of X-ray analysis is that a suitable crystal has to be found, which is not always easy. With most X-ray structure analysis, the molecular formula, and at least a skeletal structure is known beforehand, and these are obtained from mass spectra and nmr.

If a complete geometrical analysis is required, then again X-ray analysis is the method of choice, as it is the only method which affords accurate bond lengths, valency angles and torsion angles. With this information it is also possible to obtain stereochemical and conformational data. As with the other methods though, there are structures which the X-ray analysis will not solve.

Another advantage with X-ray analysis is that no similar structure need have been studied previously in order for the experiment to be successful. With spectral analysis, knowledge of chemical shifts, wavelengths of absorption etc. are necessary. Therefore again, if it is possible to do an X-ray analysis, this is the method of choice. Of course it is necessary to conduct circular dichroism experiments

in order to determine the absolute configuration.

As stated previously X-ray techniques are increasing in sophistication all the time. Most diffractometers are under computer control, which makes them easy to operate with the minimum of training. After the collection of data, there are computer packages available for the automatic solution of crystal structures, and these too can be used with the minimum of user intervention. This makes X-ray analysis a method for everyone, not just skilled operators. Also an X-ray structure may be solved successfully in a few days. It should be remembered that X-ray results only apply to the substance in the solid state and depending on what the substance is, the free molecule may be more interesting. However, it is usually found by comparison with molecular mechanics results that molecules in crystals adopt minimum energy conformations and would not be very different in the free state.

Nmr experiments can give detailed structural results, but not geometrical results. It has been shown that some of the newer methods give results that previously would only have been obtained by X-ray analysis [185]. However, usually only conventional methods are readily available, as the instrumentation required is very expensive and complex for the more recent techniques. Even a high resolution spectrum which would allow identification of individual multiplets is experimentally and computationally very expensive in time.

Nmr also requires skilled operators as even a simple NOE experiment requires careful selection of irradiation position and intensity, prolonged signal averaging and considerable data manipulation to obtain quality spectra [186]. Also the data that

is required for one compound is quite considerable and takes some time to acquire. The final structure is often pieced together like a jigsaw. However, final results are often in very good agreement with the X-ray results as is demonstrated for Rifamycin S [187]. It is still unlikely that the more sophisticated nmr techniques will ever supplant traditional methods, because of the amount of data that has to be acquired.

Nmr is rarely used on its own as a structural tool, as other spectra are usually also run, and the combined result used in the structural elucidation.

Total synthesis although still used to a limited extent for structural analysis, is greatly simplified by the use of spectral analysis.

For conformational analysis, although nmr has been used successfully, molecular mechanics provides a very quick and accurate alternative. The only difficulty with molecular mechanics is that it has not been developed fully for all types of molecules.

Conclusion 14.5

X-ray analysis provides the most complete method of structural analysis for natural products, assuming that a suitable crystal is available. X-ray studies are aided by the preliminary use of spectral analysis.

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APPENDIX A

Structure Factor Lists

SESQUITERPENE LACTONE

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	0	988	-1007	4	8	0	292	-283	4
4	0	0	372	-357	6	8	0	66	-70	6
6	0	0	390	-401	1	9	0	467	463	-9
8	0	0	885	881	3	9	0	135	-139	-7
10	0	0	292	-298	5	9	0	114	96	-5
1	1	0	1271	-1354	7	9	0	413	415	-3
3	1	0	343	-352	0	10	0	79	-58	-1
5	1	0	818	797	2	10	0	203	181	1
7	1	0	52	5	4	10	0	163	179	3
9	1	0	143	-153	6	10	0	144	-149	5
0	2	0	1070	1170	1	11	0	274	-255	7
2	2	0	726	-722	3	11	0	217	-206	9
4	2	0	947	-900	5	11	0	273	-279	-8
6	2	0	90	-83	7	11	0	137	134	-6
10	2	0	207	-216	0	12	0	168	-171	-4
1	3	0	218	233	2	12	0	139	159	-2
5	3	0	125	-136	4	12	0	121	121	2
9	3	0	512	521	6	12	0	146	-142	4
0	4	0	612	646	3	13	0	189	203	6
2	4	0	82	-99	5	13	0	113	112	8
4	4	0	1339	1289	2	14	0	103	-123	-9
6	4	0	633	-642	1	15	0	57	-67	-7
8	4	0	80	78	-9	1	1	295	-288	-5
1	5	0	435	-452	-7	1	1	202	-187	-3
5	5	0	245	-226	-5	1	1	332	-351	-1
7	5	0	157	166	-3	1	1	165	-194	3
9	5	0	235	230	1	1	1	711	-771	5
0	6	0	380	377	3	1	1	496	-488	7
2	6	0	748	718	7	1	1	73	-76	9
6	6	0	291	-287	9	1	1	211	-215	-8
8	6	0	64	-58	-8	2	1	218	223	-6
1	7	0	282	-285	-6	2	1	196	204	-4
3	7	0	452	-444	-4	2	1	660	-641	-2
5	7	0	84	72	-2	2	1	451	-464	0
7	7	0	269	266	0	2	1	1104	1183	2
2	8	0	58	70	2	2	1	691	-706	4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	1		132	139	-6	6	1	286	289	-1	11	1	54	40
2	1		500	513	-9	7	1	144	-153	1	11	1	247	268
3	1		221	231	-7	7	1	242	-241	3	11	1	231	-228
3	1		95	-78	-5	7	1	223	215	5	11	1	76	59
3	1		430	431	-3	7	1	659	628	-4	12	1	354	359
3	1		148	135	-1	7	1	547	-547	-2	12	1	106	-111
3	1		777	802	1	7	1	276	278	0	12	1	254	-260
3	1		179	-206	3	7	1	76	60	2	12	1	151	-135
3	1		158	148	5	7	1	263	-267	4	12	1	584	596
3	1		445	435	7	7	1	490	-485	-5	13	1	347	-348
3	1		316	-308	-8	8	1	85	63	-3	13	1	325	327
3	1		86	-104	-6	8	1	96	-105	-1	13	1	174	177
4	1		165	-157	-4	8	1	235	237	1	13	1	73	95
4	1		91	-92	0	8	1	400	395	3	13	1	306	-317
4	1		199	173	2	8	1	302	-275	-4	14	1	114	-96
4	1		173	187	6	8	1	168	174	-2	14	1	112	68
4	1		790	-785	8	8	1	228	229	0	14	1	514	-546
4	1		63	-50	-7	9	1	185	196	2	14	1	292	287
4	1		276	264	-5	9	1	217	211	-1	15	1	65	-11
4	1		197	-196	-3	9	1	198	194	1	15	1	62	50
5	1		217	221	-1	9	1	564	-569	-10	0	2	229	-224
5	1		248	-250	1	9	1	478	486	-8	0	2	145	141
5	1		72	-72	3	9	1	95	114	-6	0	2	247	-244
5	1		332	321	5	9	1	350	354	-4	0	2	542	-558
5	1		188	-180	7	9	1	375	-394	-2	0	2	227	259
5	1		301	299	-8	10	1	253	-248	0	0	2	125	-130
5	1		53	-22	-6	10	1	126	-122	2	0	2	726	723
5	1		309	-313	-4	10	1	411	-400	4	0	2	366	358
5	1		217	233	-2	10	1	259	271	6	0	2	71	-57
6	1		168	-178	0	10	1	474	-488	8	0	2	258	257
6	1		61	-83	2	10	1	185	-192	-9	1	2	98	-95
6	1		280	262	4	10	1	296	-302	-7	1	2	69	-11
6	1		580	-572	6	10	1	311	304	-5	1	2	158	154
6	1		817	-831	-7	11	1	304	307	-3	1	2	816	817
6	1		87	-53	-5	11	1	285	-296	1	1	2	727	-760
6	1		355	340	-3	11	1	273	270	3	1	2	194	-195

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
9	1	2	201	-231	0	6	2	133	-124
-10	2	2	432	-429	2	6	2	632	-619
-8	2	2	395	383	4	6	2	477	480
-6	2	2	783	-754	6	6	2	129	132
-4	2	2	1259	1271	8	6	2	80	-98
-2	2	2	872	-881	-9	7	2	261	280
0	2	2	723	777	-7	7	2	86	82
2	2	2	1514	-1513	1	7	2	70	-81
4	2	2	842	824	5	7	2	61	-86
6	2	2	504	-520	7	7	2	314	-329
8	2	2	493	483	-8	8	2	241	-236
-9	3	2	171	174	-4	8	2	269	265
-7	3	2	126	120	-2	8	2	99	-88
-5	3	2	142	-130	0	8	2	153	155
-1	3	2	126	-158	2	8	2	325	-307
3	3	2	187	197	6	8	2	225	223
7	3	2	226	-221	-7	9	2	102	-81
9	3	2	273	-283	-3	9	2	77	-99
-10	4	2	90	-121	1	9	2	195	222
-8	4	2	275	-280	5	9	2	186	187
-6	4	2	288	-299	-8	10	2	105	-106
-4	4	2	234	228	-6	10	2	235	235
-2	4	2	530	530	-4	10	2	58	-44
0	4	2	548	-578	-2	10	2	358	-358
2	4	2	132	-123	0	10	2	310	314
4	4	2	365	361	2	10	2	95	99
6	4	2	306	314	4	10	2	228	-228
8	4	2	77	103	6	10	2	102	111
-9	5	2	209	211	-7	11	2	45	-43
-3	5	2	241	243	-5	11	2	161	166
1	5	2	69	-67	-3	11	2	115	-115
7	5	2	290	-290	-1	11	2	60	59
-8	6	2	111	-119	1	11	2	49	27
-6	6	2	394	-386	3	11	2	218	-209
-4	6	2	583	574	5	11	2	94	-62
-2	6	2	84	65	-6	12	2	59	-14

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-4	12	2	172	172	-6	4	3	78	-75	6	8	3	92	99
-2	12	2	157	-157	-4	4	3	718	-714	-7	9	3	309	298
0	12	2	98	118	2	4	3	208	195	-5	9	3	94	101
2	12	2	148	-157	4	4	3	63	-58	-3	9	3	412	389
4	12	2	51	61	6	4	3	130	-130	-1	9	3	432	-420
-5	13	2	164	-161	-9	5	3	274	-262	1	9	3	169	182
-3	13	2	105	-103	-7	5	3	113	-110	3	9	3	210	207
1	13	2	161	172	-5	5	3	289	281	5	9	3	173	189
3	13	2	162	141	1	5	3	149	148	-8	10	3	202	209
-4	14	2	100	140	3	5	3	62	-16	-6	10	3	235	-238
-2	14	2	184	187	5	5	3	317	-324	-4	10	3	295	-298
0	14	2	138	-162	7	5	3	300	301	-2	10	3	446	-434
2	14	2	152	-156	-10	6	3	49	20	0	10	3	184	205
-1	15	2	57	14	-8	6	3	243	244	2	10	3	366	-363
-5	1	3	277	-262	-6	6	3	288	281	4	10	3	124	-143
-3	1	3	1107	-1136	-4	6	3	162	-151	6	10	3	224	-215
1	1	3	264	-278	-2	6	3	569	-568	-5	11	3	200	-200
3	1	3	112	-107	0	6	3	532	-532	-3	11	3	257	248
5	1	3	309	-304	2	6	3	285	282	1	11	3	260	257
7	1	3	284	-287	4	6	3	134	-141	3	11	3	301	-307
-8	2	3	339	348	6	6	3	165	-169	5	11	3	315	311
-6	2	3	68	32	-9	7	3	407	-405	-6	12	3	557	550
-4	2	3	453	-460	-7	7	3	184	-175	-4	12	3	97	-83
-2	2	3	1313	1331	-3	7	3	226	229	-2	12	3	208	-204
0	2	3	400	-427	-1	7	3	560	-560	0	12	3	86	-96
2	2	3	775	-779	1	7	3	621	627	2	12	3	365	376
4	2	3	147	151	3	7	3	189	173	4	12	3	84	-109
6	2	3	317	317	5	7	3	226	-221	-5	13	3	299	-288
-9	3	3	271	-278	7	7	3	183	-179	-3	13	3	79	83
-7	3	3	474	471	-8	8	3	184	177	-1	13	3	176	167
-1	3	3	556	567	-6	8	3	58	63	1	13	3	298	295
1	3	3	418	392	-4	8	3	242	-220	3	13	3	343	-345
3	3	3	301	292	-2	8	3	461	458	-4	14	3	206	199
7	3	3	200	191	0	8	3	70	-70	-2	14	3	496	-497
-10	4	3	111	-109	2	8	3	212	209	0	14	3	59	36
-8	4	3	253	238	4	8	3	138	-137	-10	0	4	610	-615

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-8	0	4	263	281	2	4	4	1031	-1041
-6	0	4	265	265	4	4	4	563	552
-4	0	4	610	615	-9	5	4	154	-161
-2	0	4	5586	-6158	-7	5	4	199	196
0	0	4	815	810	-3	5	4	311	304
2	0	4	227	202	-1	5	4	397	396
4	0	4	275	272	1	5	4	174	165
6	0	4	781	-793	3	5	4	215	184
8	0	4	356	355	5	5	4	161	-167
-11	1	4	121	124	7	5	4	225	-232
-7	1	4	535	-523	-10	6	4	54	32
-5	1	4	87	81	-8	6	4	234	243
-3	1	4	1082	1083	-4	6	4	470	-460
-1	1	4	855	866	-2	6	4	312	-315
1	1	4	328	331	0	6	4	537	-552
3	1	4	739	-733	2	6	4	99	-108
5	1	4	65	52	4	6	4	248	230
7	1	4	117	110	6	6	4	84	55
-10	2	4	61	-31	-9	7	4	253	-242
-8	2	4	186	170	-7	7	4	60	-20
-6	2	4	512	502	-5	7	4	310	299
-4	2	4	351	363	-3	7	4	277	283
-2	2	4	989	-1005	-1	7	4	59	71
0	2	4	530	558	1	7	4	387	389
2	2	4	755	760	3	7	4	137	-131
4	2	4	85	66	5	7	4	209	-202
8	2	4	188	192	-8	8	4	74	73
-7	3	4	153	152	-6	8	4	211	191
-5	3	4	77	-100	-4	8	4	181	-173
-1	3	4	176	-165	-2	8	4	82	-16
1	3	4	71	28	0	8	4	62	-24
7	3	4	501	-505	2	8	4	347	350
-10	4	4	53	-32	4	8	4	69	46
-8	4	4	395	394	6	8	4	58	11
-6	4	4	847	-823	-9	9	4	326	-330
-2	4	4	337	-332	-7	9	4	73	-91

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-5	9	4	185	171	3	1	5	60	-51	-2	6	5	372	364
-3	9	4	390	-389	5	1	5	113	132	0	6	5	86	76
-1	9	4	287	-292	7	1	5	133	127	2	6	5	219	-218
1	9	4	76	75	-10	2	5	66	-85	4	6	5	240	-248
3	9	4	51	-41	-8	2	5	144	-156	-9	7	5	157	158
5	9	4	394	-400	-6	2	5	238	257	-7	7	5	139	-158
-8	10	4	104	112	-4	2	5	202	197	-5	7	5	316	-326
-6	10	4	134	-132	-2	2	5	573	-597	-3	7	5	311	293
-4	10	4	107	-86	0	2	5	476	509	-1	7	5	236	-224
-2	10	4	59	62	2	2	5	82	-81	1	7	5	122	-112
0	10	4	183	-181	4	2	5	464	-448	3	7	5	254	248
2	10	4	200	-195	-11	3	5	205	-192	5	7	5	421	430
4	10	4	166	163	-9	3	5	77	84	-6	8	5	88	-78
-7	11	4	268	264	-7	3	5	296	-285	-4	8	5	52	42
-5	11	4	102	76	-5	3	5	141	166	-2	8	5	124	-96
-3	11	4	203	197	-3	3	5	562	-550	0	8	5	176	167
-1	11	4	185	191	-1	3	5	187	197	4	8	5	178	-183
1	11	4	287	283	1	3	5	100	-81	-9	9	5	138	-135
3	11	4	185	181	3	3	5	150	-168	-7	9	5	145	-157
-6	12	4	73	-73	5	3	5	192	184	-5	9	5	123	-124
-4	12	4	81	-96	7	3	5	88	96	-3	9	5	414	411
-2	12	4	106	122	-10	4	5	135	146	-1	9	5	362	-357
0	12	4	146	-189	0	4	5	398	390	1	9	5	54	-79
2	12	4	94	-117	2	4	5	56	32	3	9	5	248	-258
4	12	4	157	153	4	4	5	243	-244	5	9	5	336	327
-5	13	4	99	-122	6	4	5	222	230	-8	10	5	81	98
-3	13	4	99	-93	-9	5	5	114	133	-6	10	5	240	239
-1	13	4	65	22	-5	5	5	229	-239	-4	10	5	198	-187
1	13	4	214	-216	-3	5	5	110	124	-2	10	5	319	312
-4	14	4	98	89	-1	5	5	113	-107	0	10	5	88	90
-2	14	4	92	-62	1	5	5	116	-128	2	10	5	210	203
0	14	4	139	133	5	5	5	248	265	4	10	5	253	-270
-11	1	5	178	184	-10	6	5	79	106	-7	11	5	177	183
-9	1	5	87	83	-8	6	5	68	56	-5	11	5	210	-205
-7	1	5	240	239	-6	6	5	148	-147	-3	11	5	58	-29
1	1	5	339	354	-4	6	5	269	271	-1	11	5	225	-220

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	11	5	141	158	-3	3	6	131	160
-6	12	5	217	-209	-1	3	6	60	-46
-2	12	5	313	303	1	3	6	90	-82
2	12	5	416	-426	5	3	6	165	159
-5	13	5	230	-234	-10	4	6	178	168
-3	13	5	148	-141	-8	4	6	167	162
-1	13	5	80	-74	-6	4	6	236	-226
1	13	5	231	237	-4	4	6	203	-206
-2	14	5	424	411	-2	4	6	245	247
-10	0	6	179	-174	0	4	6	75	97
-8	0	6	171	161	2	4	6	270	-272
-6	0	6	175	146	4	4	6	225	-222
-4	0	6	158	-178	6	4	6	98	-88
-2	0	6	79	-139	-9	5	6	55	-9
0	0	6	272	-251	-5	5	6	133	-143
2	0	6	302	-298	-1	5	6	79	-94
6	0	6	249	-253	3	5	6	61	17
-11	1	6	84	92	5	5	6	284	281
-5	1	6	342	-360	-10	6	6	129	129
-3	1	6	169	182	-8	6	6	186	176
-1	1	6	192	206	-6	6	6	285	-289
1	1	6	124	156	-4	6	6	100	-98
5	1	6	65	70	-2	6	6	174	166
-10	2	6	136	-153	0	6	6	334	345
-8	2	6	385	387	2	6	6	322	-322
-6	2	6	437	-438	4	6	6	174	-183
-4	2	6	492	520	-9	7	6	62	-71
-2	2	6	367	-369	-7	7	6	66	30
0	2	6	697	716	-5	7	6	64	40
2	2	6	440	-439	-3	7	6	58	79
4	2	6	395	382	-1	7	6	57	71
6	2	6	354	-362	1	7	6	76	-54
-11	3	6	93	-89	5	7	6	300	290
-9	3	6	104	-119	-6	8	6	197	-178
-7	3	6	89	79	-4	8	6	62	50
-5	3	6	107	-60	-2	8	6	126	-121

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	8	6	226	240	4	2	7	282	-271	-5	9	7	217	-216
4	8	6	128	-133	-9	3	7	285	-293	-3	9	7	222	211
-5	9	6	46	-27	-3	3	7	206	-194	-1	9	7	142	-140
-1	9	6	155	-152	-1	3	7	281	-282	1	9	7	155	-146
3	9	6	216	-210	1	3	7	152	-160	3	9	7	116	-128
-8	10	6	195	-177	3	3	7	98	89	-8	10	7	124	114
-4	10	6	223	231	5	3	7	103	-109	-6	10	7	277	264
-2	10	6	154	-147	-10	4	7	165	-165	-4	10	7	240	241
0	10	6	91	-111	-8	4	7	81	75	-2	10	7	91	-58
2	10	6	166	155	-6	4	7	354	347	0	10	7	181	189
-7	11	6	61	-60	-2	4	7	149	130	2	10	7	116	140
-5	11	6	100	104	4	4	7	97	95	-7	11	7	105	122
-3	11	6	88	-96	-9	5	7	119	107	-5	11	7	179	-181
1	11	6	157	163	-7	5	7	191	-177	-3	11	7	50	-63
3	11	6	77	101	-5	5	7	60	-68	-1	11	7	151	-156
-6	12	6	153	-141	-3	5	7	91	-87	1	11	7	195	206
-4	12	6	149	152	3	5	7	265	247	-6	12	7	51	7
-2	12	6	52	-58	5	5	7	272	-275	-4	12	7	170	173
0	12	6	95	94	-10	6	7	176	-167	0	12	7	255	-261
2	12	6	104	-85	-8	6	7	175	-172	-3	13	7	113	-117
-3	13	6	50	12	-6	6	7	193	191	-10	0	8	128	-134
-1	13	6	162	-169	-4	6	7	155	156	-8	0	8	196	-194
-11	1	7	60	20	-2	6	7	283	284	-6	0	8	186	-174
-9	1	7	63	55	0	6	7	231	-230	-4	0	8	2100	2249
-7	1	7	111	110	2	6	7	115	132	-2	0	8	339	-341
-5	1	7	606	610	4	6	7	108	90	0	0	8	166	-164
-1	1	7	144	159	-9	7	7	95	108	2	0	8	105	-104
1	1	7	56	-60	-5	7	7	110	-100	4	0	8	486	466
3	1	7	265	255	-3	7	7	299	306	-9	1	8	253	273
5	1	7	196	194	-1	7	7	312	-308	-5	1	8	611	-608
-10	2	7	155	-146	1	7	7	116	-113	-3	1	8	380	-379
-6	2	7	152	151	3	7	7	147	136	-1	1	8	153	-131
-4	2	7	765	-769	-6	8	7	107	106	1	1	8	410	427
-2	2	7	237	232	-4	8	7	269	-270	3	1	8	88	-106
0	2	7	426	439	2	8	7	87	93	5	1	8	76	-36
2	2	7	105	-96	-7	9	7	83	-71	-10	2	8	147	-144

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-8	2	8	189	-195	-8	8	8	97	-99
-6	2	8	129	-145	-6	8	8	174	170
-4	2	8	599	597	-2	8	8	87	54
-2	2	8	250	-231	0	8	8	261	-258
0	2	8	303	-328	2	8	8	60	-25
2	2	8	90	-73	-7	9	8	105	-101
4	2	8	70	36	-5	9	8	171	174
-9	3	8	82	-82	-3	9	8	139	112
-7	3	8	74	66	-2	10	8	130	119
-1	3	8	53	-12	0	10	8	168	144
1	3	8	128	104	-5	11	8	79	-79
3	3	8	48	-51	-3	11	8	125	-129
-10	4	8	164	-158	-1	11	8	241	-218
-8	4	8	321	318	-4	12	8	58	-60
-4	4	8	51	84	-2	12	8	173	152
0	4	8	459	462	-9	1	9	63	-95
2	4	8	291	-279	-3	1	9	87	101
4	4	8	55	-76	-1	1	9	127	-128
-9	5	8	98	-114	3	1	9	80	-80
-5	5	8	170	-191	-10	2	9	81	93
-3	5	8	188	-194	-6	2	9	117	-121
-1	5	8	208	-207	-4	2	9	195	194
1	5	8	98	-103	-2	2	9	207	-196
3	5	8	106	112	2	2	9	254	253
-10	6	8	152	-150	-9	3	9	130	125
-6	6	8	220	228	-7	3	9	90	-111
-4	6	8	179	179	-5	3	9	227	223
-2	6	8	249	254	-3	3	9	60	-74
0	6	8	79	111	3	3	9	108	-86
2	6	8	141	-124	-2	4	9	155	-146
4	6	8	45	-29	2	4	9	158	173
-7	7	8	162	-161	-7	5	9	100	103
-5	7	8	145	-141	-3	5	9	67	50
-1	7	8	225	-211	-1	5	9	79	59
1	7	8	98	104	3	5	9	168	-156
3	7	8	171	140	-8	6	9	65	65

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	6	9	123	-112	-2	2	10	245	-239	-1	3	11	74	97
-4	6	9	97	-89	0	2	10	163	148	1	3	11	88	-118
-2	6	9	127	-91	2	2	10	174	-171	-8	4	11	164	-161
0	6	9	133	136	-9	3	10	51	-24	-6	4	11	62	-19
2	6	9	179	168	-7	3	10	71	65	-4	4	11	101	-99
-9	7	9	78	73	-5	3	10	79	-92	0	4	11	51	59
-7	7	9	134	134	-8	4	10	96	112	-7	5	11	78	49
-5	7	9	127	-112	-2	4	10	60	-39	-5	5	11	93	60
-3	7	9	123	119	0	4	10	121	140	-1	5	11	60	30
-1	7	9	82	89	2	4	10	141	117	-8	6	11	126	-128
1	7	9	135	-128	-3	5	10	47	84	-4	6	11	137	-122
-4	8	9	51	-22	-1	5	10	81	-44	-2	6	11	116	111
-2	8	9	97	-92	1	5	10	61	-51	0	6	11	117	-74
-5	9	9	185	-185	-8	6	10	106	117	-5	7	11	127	-116
-3	9	9	190	174	-6	6	10	96	85	-3	7	11	124	97
-1	9	9	74	58	-4	6	10	130	-111	-6	8	11	133	115
1	9	9	114	103	-2	6	10	124	-129	-4	8	11	87	17
-6	10	9	99	75	0	6	10	127	133	-2	8	11	59	-25
-4	10	9	155	-144	-5	7	10	53	-65	-6	0	12	801	-812
-2	10	9	51	-39	-1	7	10	72	36	-4	0	12	103	87
0	10	9	60	-69	1	7	10	49	-34	-2	0	12	126	123
-3	11	9	172	151	-6	8	10	64	-20	-7	1	12	297	291
-10	0	10	108	-93	-4	8	10	93	63	-5	1	12	169	186
-8	0	10	44	-21	-2	8	10	153	-143	-3	1	12	70	37
-6	0	10	74	96	-3	9	10	107	52	-1	1	12	212	-209
-2	0	10	53	70	-7	1	11	284	-284	-6	2	12	286	-292
0	0	10	203	207	-5	1	11	58	-7	-4	2	12	94	98
2	0	10	50	18	-1	1	11	52	57	-2	2	12	128	129
-7	1	10	142	150	1	1	11	176	-156	-1	3	12	69	-76
-5	1	10	112	-108	-6	2	11	345	352	-8	4	12	65	-51
-3	1	10	78	-58	-4	2	11	142	-135	-4	4	12	53	-22
-1	1	10	91	-58	-2	2	11	191	-189	-2	4	12	173	-152
-10	2	10	150	-170	0	2	11	87	68	-7	5	12	73	90
-8	2	10	101	91	-7	3	11	70	-57	-5	5	12	74	75
-6	2	10	138	-142	-5	3	11	55	77	-3	5	12	118	148
-4	2	10	62	78	-3	3	11	145	150	-6	6	12	79	-68

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-4	6	12	134	-118	-5	1	13	75	-52	-6	2	13	46	-38	-4	2	13	65	63					

MICROCLADALLENE B

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	0	0	359	353	3	13	0	131	58
4	0	0	196	229	1	14	0	118	130
1	1	0	881	855	2	14	0	103	9
2	1	0	874	835	1	15	0	119	26
3	1	0	121	38	0	16	0	96	115
4	1	0	244	251	1	0	1	999	990
0	2	0	409	404	2	0	1	122	113
1	2	0	906	926	3	0	1	561	561
2	2	0	1397	1292	5	0	1	175	141
1	3	0	484	481	0	1	1	307	282
2	3	0	154	126	1	1	1	669	636
4	3	0	273	315	2	1	1	452	437
0	4	0	2249	2354	3	1	1	178	158
1	4	0	839	806	5	1	1	144	55
3	4	0	174	195	0	2	1	672	659
4	4	0	139	79	1	2	1	1574	1552
1	5	0	124	147	2	2	1	137	138
2	5	0	231	282	3	2	1	338	301
3	5	0	290	305	0	3	1	827	847
4	5	0	208	189	1	3	1	365	369
0	6	0	497	510	2	3	1	630	610
1	6	0	174	143	3	3	1	263	273
2	6	0	307	307	0	4	1	114	117
1	7	0	276	276	1	4	1	498	476
2	7	0	314	320	2	4	1	464	463
0	8	0	1038	1039	3	4	1	374	433
1	8	0	528	528	5	4	1	154	97
3	8	0	208	239	0	5	1	1004	1002
1	9	0	286	271	1	5	1	746	770
4	9	0	139	126	2	5	1	237	231
1	10	0	211	222	0	6	1	744	732
2	10	0	240	239	1	6	1	837	845
1	11	0	104	114	2	6	1	116	107
2	11	0	119	139	3	6	1	237	255
0	12	0	252	246	0	7	1	172	174
1	12	0	257	271	1	7	1	418	405

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	7	1	287	295	0	4	2	1648	1699	1	1	3	977	906
3	7	1	221	220	1	4	2	396	401	2	1	3	640	630
5	7	1	146	53	2	4	2	124	113	3	1	3	138	121
0	8	1	386	376	3	4	2	178	195	4	1	3	186	182
1	8	1	175	159	0	5	2	334	336	0	2	3	145	149
2	8	1	240	250	1	5	2	586	616	1	2	3	439	419
3	8	1	193	235	2	5	2	245	228	2	2	3	338	343
1	9	1	435	427	3	5	2	315	342	3	2	3	145	88
4	9	1	171	74	4	5	2	174	187	4	2	3	322	331
0	10	1	449	447	0	6	2	183	187	0	3	3	1319	1320
1	10	1	189	198	1	6	2	468	474	1	3	3	569	549
4	10	1	148	39	2	6	2	567	589	2	3	3	451	449
2	11	1	150	138	3	6	2	151	156	4	3	3	145	113
4	11	1	136	8	0	7	2	625	627	0	4	3	702	671
1	12	1	114	139	1	7	2	169	179	1	4	3	586	591
0	13	1	126	93	2	7	2	400	385	2	4	3	483	472
1	13	1	170	192	5	7	2	160	72	0	5	3	238	258
2	13	1	118	56	0	8	2	407	412	1	5	3	352	361
0	14	1	194	179	1	8	2	428	415	2	5	3	385	377
1	15	1	102	25	2	8	2	138	142	3	5	3	211	206
0	0	2	371	323	0	9	2	161	167	0	6	3	380	388
1	0	2	577	533	1	9	2	315	319	1	6	3	477	484
3	0	2	255	233	2	9	2	230	225	2	6	3	135	125
0	1	2	527	517	3	9	2	222	238	4	6	3	190	219
1	1	2	1410	1414	1	10	2	305	316	0	7	3	1099	1076
2	1	2	360	325	2	10	2	109	120	1	7	3	223	221
3	1	2	689	679	0	11	2	107	104	2	7	3	353	363
4	1	2	156	246	2	11	2	193	200	3	7	3	143	159
0	2	2	365	362	1	12	2	148	163	4	7	3	146	72
1	2	2	1152	1131	2	13	2	168	174	0	8	3	218	226
2	2	2	453	450	1	15	2	125	81	1	8	3	283	289
3	2	2	209	191	1	16	2	101	79	2	8	3	326	350
0	3	2	475	477	1	0	3	359	334	0	9	3	190	176
1	3	2	598	596	2	0	3	531	515	1	9	3	214	233
2	3	2	841	836	4	0	3	219	202	2	9	3	266	254
3	3	2	472	506	0	1	3	719	717	4	9	3	152	96

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K
5	9	3	150	77	1	5	4	682	687	3	2
0	10	3	390	406	2	5	4	168	179	4	2
1	10	3	149	180	3	5	4	270	298	6	2
0	11	3	417	411	0	6	4	663	654	0	3
2	11	3	120	144	1	6	4	822	846	1	3
0	12	3	267	261	2	6	4	341	344	2	3
2	12	3	132	136	0	7	4	295	304	3	3
3	12	3	154	99	2	7	4	166	187	0	4
0	14	3	131	137	3	7	4	266	282	1	4
0	15	3	155	155	0	8	4	121	128	2	4
0	16	3	155	104	1	8	4	227	235	3	4
0	0	4	142	127	2	8	4	214	204	0	5
1	0	4	1097	1097	3	8	4	153	171	1	5
2	0	4	368	370	1	9	4	264	271	2	5
3	0	4	362	360	2	9	4	180	182	2	6
4	0	4	133	96	0	10	4	298	289	3	6
0	1	4	870	863	1	10	4	265	245	4	6
1	1	4	694	681	2	10	4	291	286	0	7
2	1	4	231	210	0	11	4	226	194	1	7
3	1	4	419	387	1	11	4	102	113	0	8
6	1	4	188	25	1	12	4	208	229	1	8
0	2	4	1074	1073	0	13	4	94	85	2	8
1	2	4	1497	1518	1	13	4	135	130	1	9
2	2	4	246	235	1	14	4	129	93	2	9
4	2	4	136	122	0	15	4	97	72	3	9
5	2	4	145	90	1	0	5	912	887	1	10
6	2	4	170	2	2	0	5	1056	1029	2	10
0	3	4	639	614	3	0	5	153	116	0	11
1	3	4	357	347	4	0	5	160	181	1	11
2	3	4	245	237	5	0	5	148	30	2	11
3	3	4	383	393	0	1	5	507	493	0	12
5	3	4	160	127	1	1	5	729	710	1	12
1	4	4	646	632	2	1	5	493	492	1	13
2	4	4	444	445	3	1	5	241	209	2	13
3	4	4	321	330	0	2	5	160	163	0	14
0	5	4	130	139	1	2	5	378	383	2	14

L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	362	364	0	15	5	100	113	4	8	6	146	84
5	190	206	0	0	6	635	633	0	9	6	241	235
5	156	44	1	0	6	246	237	1	9	6	260	274
5	454	448	2	0	6	628	623	2	9	6	198	220
5	869	858	3	0	6	187	186	5	9	6	180	31
5	187	198	6	0	6	157	15	0	10	6	188	171
5	148	154	0	1	6	1078	1073	2	10	6	157	158
5	166	165	1	1	6	267	274	0	11	6	326	327
5	663	648	2	1	6	588	574	1	11	6	279	294
5	598	591	3	1	6	283	281	1	13	6	214	208
5	170	158	4	1	6	205	209	0	16	6	123	73
5	103	97	0	2	6	1348	1348	1	0	7	405	390
5	532	531	1	2	6	526	526	2	0	7	463	454
5	455	461	2	2	6	248	231	3	0	7	276	287
5	154	167	3	2	6	241	220	4	0	7	206	185
5	197	244	5	2	6	151	58	0	1	7	357	349
5	174	122	0	3	6	1590	1593	1	1	7	902	900
5	554	561	1	3	6	444	440	2	1	7	226	203
5	430	432	2	3	6	349	344	0	2	7	306	288
5	174	196	4	3	6	298	280	2	2	7	508	499
5	530	516	0	4	6	480	468	3	2	7	385	405
5	240	243	1	4	6	362	350	1	3	7	915	940
5	237	249	2	4	6	636	625	0	4	7	192	213
5	280	280	0	5	6	586	573	1	4	7	523	511
5	195	175	1	5	6	418	422	2	4	7	375	364
5	114	107	2	5	6	525	543	3	4	7	199	232
5	176	191	0	6	6	790	766	4	4	7	141	64
5	469	467	1	6	6	370	381	0	5	7	259	267
5	218	227	2	6	6	243	251	1	5	7	736	748
5	129	153	0	7	6	692	699	2	5	7	165	178
5	193	206	1	7	6	334	322	3	5	7	134	109
5	204	215	2	7	6	161	182	1	6	7	347	344
5	113	83	4	7	6	145	119	2	6	7	272	290
5	157	155	0	8	6	254	249	3	6	7	169	197
5	121	112	1	8	6	106	113	0	7	7	263	285
5	113	27	2	8	6	220	211	1	7	7	461	456

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	7	7	159	201	0	4	8	522	511
4	7	7	179	100	1	4	8	848	857
0	8	7	461	452	2	4	8	340	343
1	8	7	143	137	0	5	8	422	421
2	8	7	138	178	1	5	8	181	190
3	8	7	212	202	2	5	8	320	334
0	9	7	321	325	3	5	8	143	162
1	9	7	426	440	0	6	8	318	309
2	9	7	151	169	1	6	8	186	173
1	10	7	272	283	2	6	8	279	302
1	11	7	188	192	3	6	8	282	311
2	11	7	121	116	0	7	8	492	497
4	11	7	149	78	1	7	8	216	210
0	13	7	255	241	2	7	8	209	220
1	13	7	100	73	0	8	8	127	147
0	14	7	118	85	1	8	8	323	317
0	0	8	575	538	2	8	8	300	290
1	0	8	919	948	0	9	8	97	75
2	0	8	584	587	1	9	8	226	251
3	0	8	286	281	2	9	8	143	168
5	0	8	162	51	0	10	8	134	133
0	1	8	573	596	1	10	8	184	207
1	1	8	280	275	2	10	8	235	229
2	1	8	531	523	3	10	8	126	114
3	1	8	198	208	0	11	8	118	127
4	1	8	224	258	1	11	8	137	120
0	2	8	1236	1186	2	11	8	162	149
1	2	8	475	476	0	12	8	100	65
2	2	8	484	476	2	12	8	171	170
3	2	8	340	386	0	13	8	125	96
4	2	8	139	56	2	14	8	137	64
0	3	8	450	447	3	0	9	146	169
1	3	8	390	388	4	0	9	176	162
2	3	8	663	675	0	1	9	539	532
3	3	8	128	103	1	1	9	836	811
5	3	8	146	53	2	1	9	260	265

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	2	9	123	113	0	0	10	611	593	0	11	10	158	138
1	2	9	387	392	1	0	10	928	955	1	11	10	263	244
2	2	9	614	614	2	0	10	272	249	1	12	10	161	178
3	2	9	187	226	3	0	10	151	184	1	13	10	135	125
0	3	9	146	151	4	0	10	131	90	2	13	10	121	22
1	3	9	577	578	0	1	10	879	869	1	0	11	425	434
2	3	9	366	363	1	1	10	240	245	2	0	11	150	144
0	4	9	305	279	2	1	10	386	392	4	0	11	176	172
1	4	9	331	315	3	1	10	336	335	5	0	11	155	47
3	4	9	189	192	0	2	10	398	400	0	1	11	647	626
4	4	9	205	228	1	2	10	552	554	1	1	11	476	470
0	5	9	762	783	3	2	10	160	171	2	1	11	327	326
1	5	9	354	363	0	3	10	805	777	5	1	11	143	49
2	5	9	282	276	1	3	10	474	473	1	2	11	208	224
3	5	9	176	163	2	3	10	122	145	2	2	11	481	483
0	6	9	295	299	3	3	10	289	302	0	3	11	167	156
1	6	9	374	381	5	3	10	180	58	1	3	11	523	531
2	6	9	450	461	0	4	10	506	515	2	3	11	401	397
0	7	9	128	110	1	4	10	550	562	0	4	11	195	177
1	7	9	425	431	0	5	10	662	662	1	4	11	196	180
2	7	9	239	247	1	5	10	262	276	2	4	11	169	154
1	8	9	114	136	2	5	10	224	226	3	4	11	135	103
3	8	9	143	131	3	5	10	211	221	5	4	11	139	45
0	9	9	438	432	4	5	10	133	100	0	5	11	640	642
1	9	9	172	178	1	6	10	178	190	1	5	11	312	308
2	9	9	118	112	2	6	10	147	145	2	5	11	200	201
0	10	9	160	191	0	7	10	156	159	3	5	11	136	140
1	10	9	170	181	1	7	10	429	431	0	6	11	155	118
2	10	9	135	140	2	7	10	294	311	1	6	11	226	229
1	11	9	129	152	0	8	10	199	208	2	6	11	181	182
2	11	9	141	137	1	8	10	241	233	0	7	11	213	207
0	12	9	122	95	0	9	10	420	422	1	7	11	438	449
0	13	9	246	228	1	9	10	300	303	2	7	11	291	319
1	13	9	138	104	4	9	10	135	45	3	7	11	131	120
0	14	9	103	34	0	10	10	241	250	0	8	11	104	98
1	15	9	101	64	1	10	10	152	164	1	8	11	232	245

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	9	11	600	599	1	7	12	226	251
4	9	11	138	7	4	7	12	160	117
1	10	11	123	130	0	8	12	95	116
2	10	11	162	207	1	8	12	260	272
2	11	11	144	137	2	8	12	207	226
3	11	11	130	22	1	9	12	146	123
0	12	11	132	127	2	9	12	136	138
0	13	11	121	114	3	9	12	128	103
0	15	11	102	98	0	10	12	135	110
1	15	11	154	84	1	10	12	165	150
0	0	12	260	249	1	11	12	106	85
1	0	12	362	398	0	14	12	133	93
2	0	12	279	268	1	14	12	111	87
3	0	12	149	153	1	0	13	466	464
5	0	12	138	47	3	0	13	293	284
0	1	12	562	554	4	0	13	137	117
1	1	12	117	133	0	1	13	135	126
2	1	12	195	178	1	1	13	468	480
3	1	12	231	199	2	1	13	191	200
0	2	12	757	768	3	1	13	107	82
1	2	12	231	237	1	2	13	153	189
2	2	12	195	220	2	2	13	198	195
0	3	12	103	107	3	2	13	135	131
1	3	12	334	346	1	3	13	424	432
2	3	12	260	264	2	3	13	201	184
3	3	12	173	183	0	4	13	352	357
1	4	12	478	497	1	4	13	375	380
2	4	12	291	285	3	4	13	132	167
0	5	12	367	366	0	5	13	207	216
1	5	12	162	162	1	5	13	272	279
2	5	12	224	227	2	5	13	185	184
3	5	12	139	182	1	6	13	156	184
0	6	12	584	581	2	6	13	154	177
1	6	12	247	242	3	6	13	127	120
2	6	12	143	141	0	7	13	104	119
3	6	12	144	175	1	7	13	247	257

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	8	13	442	448	1	8	14	190	197	2	10	15	123	126
1	8	13	151	146	2	8	14	179	186	0	11	15	121	115
4	8	13	140	63	0	9	14	178	183	0	12	15	202	149
0	9	13	102	91	2	9	14	136	149	1	12	15	145	70
1	9	13	111	129	3	9	14	136	77	0	13	15	104	46
2	9	13	165	172	0	10	14	139	133	0	0	16	367	371
0	10	13	111	60	1	10	14	138	132	2	0	16	268	250
1	10	13	142	159	1	11	14	152	138	0	1	16	237	241
2	10	13	117	73	0	13	14	101	25	1	1	16	99	112
1	11	13	205	189	0	14	14	110	15	2	1	16	161	151
0	12	13	192	169	1	0	15	598	598	0	2	16	785	776
0	14	13	97	97	2	0	15	182	200	1	2	16	297	313
2	0	14	387	379	0	1	15	239	253	2	2	16	202	194
3	0	14	142	99	1	1	15	285	281	0	3	16	116	103
0	1	14	349	331	2	1	15	237	241	1	3	16	192	186
1	1	14	289	302	3	1	15	104	91	3	3	16	148	149
2	1	14	277	285	0	2	15	102	148	0	4	16	270	275
3	1	14	171	146	1	2	15	346	358	1	4	16	279	280
0	2	14	776	800	2	2	15	121	101	2	4	16	222	207
1	2	14	98	89	3	2	15	128	155	0	6	16	348	343
0	3	14	351	351	0	3	15	105	139	1	6	16	230	243
1	3	14	257	265	1	3	15	250	260	3	6	16	123	86
2	3	14	128	135	2	3	15	121	165	0	7	16	153	159
3	3	14	158	125	0	4	15	261	251	1	7	16	166	184
0	4	14	110	114	1	4	15	405	419	0	8	16	124	96
1	4	14	144	148	1	5	15	103	111	1	8	16	109	113
2	4	14	306	326	2	5	15	238	262	1	9	16	83	48
5	4	14	180	25	1	6	15	140	162	0	10	16	178	188
0	5	14	295	279	2	6	15	130	114	1	10	16	154	129
1	5	14	109	108	1	7	15	204	195	2	10	16	112	72
2	5	14	291	294	0	8	15	213	226	0	11	16	102	47
3	5	14	120	90	1	8	15	270	276	1	11	16	126	36
0	6	14	527	524	0	9	15	99	88	0	13	16	129	54
1	6	14	190	196	2	9	15	142	144	1	0	17	248	252
0	7	14	118	105	0	10	15	132	115	0	1	17	138	129
1	7	14	214	214	1	10	15	100	87	1	1	17	136	161

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	1	17	243	247	3	1	18	158	199	2
1	2	17	259	280	0	2	18	181	177	0
2	2	17	209	221	1	2	18	117	89	1
0	3	17	263	275	2	2	18	166	133	2
1	3	17	135	126	4	2	18	142	31	0
0	4	17	167	177	0	3	18	257	260	1
1	4	17	358	366	1	3	18	116	122	2
3	4	17	149	44	2	3	18	138	135	2
0	5	17	235	220	3	3	18	155	164	0
2	5	17	161	166	0	4	18	218	206	1
0	6	17	241	223	1	4	18	148	109	1
1	6	17	188	202	0	5	18	188	201	2
2	6	17	132	165	0	6	18	105	94	3
0	7	17	237	240	1	6	18	133	138	0
0	8	17	150	168	1	7	18	97	72	0
1	9	17	98	106	0	8	18	172	150	0
0	10	17	152	135	1	8	18	157	136	3
1	10	17	126	99	0	9	18	125	132	0
0	12	17	155	135	2	10	18	115	51	1
0	0	18	194	194	1	0	19	112	145	2
1	0	18	393	385	4	0	19	154	81	1
3	0	18	106	79	0	1	19	244	214	2
1	1	18	205	217	1	1	19	146	132	

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	19		146	128	0	4	20	314	308	0	0	22	173	138
2	19		181	185	1	4	20	190	180	0	1	22	189	184
2	19		167	179	0	5	20	177	152	3	1	22	108	40
2	19		145	142	3	5	20	136	62	2	2	22	148	121
3	19		109	73	0	7	20	103	74	0	3	22	154	177
3	19		137	154	0	8	20	120	145	2	3	22	119	61
3	19		159	188	2	8	20	113	79	0	4	22	133	119
4	19		104	111	0	9	20	127	97	2	4	22	108	91
5	19		188	183	2	9	20	139	46	0	5	22	168	147
5	19		247	242	1	0	21	99	87	1	6	22	94	114
6	19		144	149	2	0	21	128	117	2	6	22	129	98
6	19		115	146	1	1	21	160	156	2	0	23	114	113
7	19		151	105	1	2	21	216	237	1	1	23	208	182
8	19		93	87	0	3	21	112	63	0	3	23	162	140
10	19		146	126	1	3	21	136	128	1	3	23	130	133
0	20		478	471	1	5	21	121	133	1	5	23	126	127
0	20		122	64	0	6	21	149	176	0	7	23	145	130
1	20		236	220	1	6	21	168	135	0	1	24	200	193
2	20		127	156	0	7	21	107	122	0	3	24	145	154
2	20		137	188	1	7	21	98	124	0	5	24	125	149
3	20		129	114	1	8	21	107	30	1	1	25	120	124
3	20		126	107	0	9	21	94	87	0	3	25	133	101

NIRURINE

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	0	266	245	5	5	0	199	205	1
4	0	0	44	42	6	5	0	67	68	2
6	0	0	204	204	7	5	0	12	1	3
1	1	0	869	816	0	6	0	17	18	4
2	1	0	57	52	1	6	0	136	135	5
3	1	0	346	313	2	6	0	163	174	6
4	1	0	162	163	3	6	0	64	65	7
5	1	0	70	69	4	6	0	65	72	0
6	1	0	113	120	5	6	0	66	67	1
7	1	0	60	58	6	6	0	68	73	2
1	2	0	153	147	1	7	0	58	61	3
2	2	0	176	165	2	7	0	111	108	4
3	2	0	112	106	3	7	0	13	10	5
4	2	0	90	82	4	7	0	130	126	6
5	2	0	51	50	5	7	0	29	31	7
6	2	0	100	102	6	7	0	66	65	0
7	2	0	54	55	0	8	0	89	92	1
1	3	0	500	493	1	8	0	82	80	2
2	3	0	303	283	2	8	0	175	178	3
3	3	0	162	144	3	8	0	37	35	4
4	3	0	230	231	4	8	0	45	46	5
5	3	0	126	127	5	8	0	22	21	6
6	3	0	135	141	1	9	0	106	104	7
7	3	0	53	51	2	9	0	48	46	0
0	4	0	173	173	3	9	0	5	3	1
1	4	0	62	64	0	10	0	36	35	2
2	4	0	43	44	2	10	0	84	76	3
3	4	0	13	6	3	10	0	22	21	4
4	4	0	166	166	1	11	0	16	15	5
5	4	0	38	38	1	0	1	423	379	6
6	4	0	79	77	2	0	1	471	423	7
7	4	0	30	32	3	0	1	77	70	0
1	5	0	298	298	4	0	1	162	157	1
2	5	0	66	68	5	0	1	128	126	2
3	5	0	65	64	6	0	1	140	143	3
4	5	0	207	205	0	1	1	77	81	4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	1	1	260	247	5	5	1	70	71	2	0	2	419	374
1	1	1	235	213	6	5	1	37	37	3	0	2	87	82
1	1	1	270	237	7	5	1	90	89	4	0	2	112	106
1	1	1	147	133	0	6	1	90	89	5	0	2	90	94
1	1	1	60	59	1	6	1	24	23	6	0	2	77	77
1	1	1	64	60	2	6	1	126	130	7	0	2	23	25
1	1	1	88	91	3	6	1	62	63	0	1	2	674	737
2	1	1	382	412	4	6	1	128	126	1	1	2	887	847
2	1	1	341	338	5	6	1	31	30	2	1	2	245	224
2	1	1	154	140	6	6	1	36	34	3	1	2	151	143
2	1	1	119	108	0	7	1	68	66	4	1	2	128	116
2	1	1	97	94	1	7	1	17	18	5	1	2	222	217
2	1	1	154	150	2	7	1	80	83	6	1	2	183	184
2	1	1	127	129	3	7	1	41	39	7	1	2	56	57
2	1	1	33	30	4	7	1	61	58	0	2	2	226	255
3	1	1	212	223	5	7	1	30	28	1	2	2	217	227
3	1	1	361	359	6	7	1	21	23	2	2	2	431	399
3	1	1	267	260	0	8	1	20	16	3	2	2	108	103
3	1	1	183	172	1	8	1	47	46	4	2	2	56	53
3	1	1	112	108	2	8	1	19	17	5	2	2	164	165
3	1	1	100	100	3	8	1	38	38	6	2	2	85	89
3	1	1	21	22	4	8	1	35	31	7	2	2	53	54
3	1	1	43	45	5	8	1	11	13	0	3	2	296	313
4	1	1	122	127	0	9	1	14	5	1	3	2	362	369
4	1	1	42	44	1	9	1	77	79	2	3	2	261	248
4	1	1	183	177	2	9	1	91	93	3	3	2	184	170
4	1	1	59	51	3	9	1	18	19	4	3	2	186	182
4	1	1	124	122	4	9	1	16	11	5	3	2	119	122
4	1	1	122	122	0	10	1	24	22	6	3	2	106	105
4	1	1	39	41	1	10	1	16	8	7	3	2	60	61
4	1	1	15	14	2	10	1	39	36	0	4	2	262	281
5	1	1	264	276	3	10	1	25	27	1	4	2	260	262
5	1	1	199	200	0	11	1	32	26	2	4	2	332	320
5	1	1	117	117	1	11	1	52	51	3	4	2	139	132
5	1	1	63	62	0	0	2	505	567	4	4	2	77	78
5	1	1	37	32	1	0	2	397	383	5	4	2	76	75

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
6	4	2	159	162	1	10	2	102	99	0
7	4	2	106	110	2	10	2	14	14	1
0	5	2	162	165	3	10	2	31	33	2
1	5	2	84	83	0	11	2	5	6	3
2	5	2	135	129	1	11	2	10	1	4
3	5	2	114	115	1	0	3	490	502	5
4	5	2	133	130	2	0	3	108	98	6
5	5	2	132	130	3	0	3	118	111	7
6	5	2	71	71	4	0	3	73	61	0
7	5	2	51	50	5	0	3	10	4	1
0	6	2	242	258	6	0	3	66	66	2
1	6	2	59	62	7	0	3	40	39	3
2	6	2	99	96	0	1	3	74	79	4
3	6	2	74	74	1	1	3	459	454	5
4	6	2	136	134	2	1	3	215	189	6
5	6	2	10	9	3	1	3	264	244	7
6	6	2	80	81	4	1	3	129	120	0
0	7	2	10	2	5	1	3	138	135	1
1	7	2	35	38	6	1	3	140	142	2
2	7	2	63	68	7	1	3	73	76	3
3	7	2	166	167	0	2	3	99	102	4
4	7	2	12	9	1	2	3	49	49	5
5	7	2	71	70	2	2	3	163	150	6
6	7	2	12	19	3	2	3	402	371	0
0	8	2	46	47	4	2	3	145	134	1
1	8	2	98	101	5	2	3	61	63	2
2	8	2	77	80	6	2	3	27	23	3
3	8	2	16	11	7	2	3	82	85	4
4	8	2	46	44	0	3	3	139	142	5
5	8	2	24	28	1	3	3	137	144	6
0	9	2	19	18	2	3	3	322	316	0
1	9	2	59	62	3	3	3	219	207	2
2	9	2	61	58	4	3	3	51	51	3
3	9	2	62	62	5	3	3	99	100	4
4	9	2	11	14	6	3	3	128	130	5
0	10	2	13	10	7	3	3	55	53	0

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	3	34	33	1	9	3	79	81	4	3	4	155	152
4	3	158	160	2	9	3	37	41	5	3	4	51	48
4	3	120	117	3	9	3	39	39	6	3	4	34	31
4	3	93	94	0	10	3	65	65	7	3	4	36	39
4	3	52	51	1	10	3	69	65	0	4	4	124	123
4	3	74	71	2	10	3	82	81	1	4	4	172	174
4	3	54	58	3	10	3	11	6	2	4	4	210	209
4	3	66	66	1	11	3	23	26	3	4	4	108	100
5	3	105	103	0	0	4	236	249	4	4	4	45	44
5	3	159	161	1	0	4	174	173	5	4	4	106	107
5	3	159	157	2	0	4	175	168	6	4	4	53	54
5	3	24	22	3	0	4	346	320	7	4	4	62	64
5	3	145	143	4	0	4	363	352	0	5	4	67	65
5	3	28	26	5	0	4	220	220	1	5	4	360	377
5	3	25	28	6	0	4	23	27	2	5	4	56	59
5	3	35	34	7	0	4	54	56	3	5	4	131	134
6	3	26	23	0	1	4	363	389	4	5	4	81	82
6	3	58	66	1	1	4	382	385	5	5	4	59	60
6	3	42	42	2	1	4	207	194	6	5	4	36	37
6	3	100	100	3	1	4	181	169	0	6	4	68	69
6	3	74	75	4	1	4	193	187	1	6	4	92	89
6	3	73	74	5	1	4	177	174	2	6	4	182	188
6	3	40	45	6	1	4	152	153	3	6	4	147	152
7	3	13	11	7	1	4	61	60	4	6	4	58	58
7	3	46	44	0	2	4	174	194	5	6	4	53	54
7	3	88	89	1	2	4	440	438	6	6	4	32	30
7	3	61	62	2	2	4	190	180	0	7	4	48	51
7	3	72	71	3	2	4	333	300	1	7	4	224	229
7	3	35	35	4	2	4	165	156	2	7	4	14	15
7	3	44	42	5	2	4	204	199	3	7	4	73	76
8	3	59	62	6	2	4	37	39	4	7	4	95	96
8	3	142	147	7	2	4	7	15	5	7	4	32	29
8	3	51	51	0	3	4	90	103	6	7	4	18	17
8	3	46	48	1	3	4	278	275	0	8	4	49	48
8	3	13	19	2	3	4	38	38	1	8	4	56	50
9	3	45	47	3	3	4	104	97	2	8	4	92	96

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C12H13NO3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO
3	8	4	84	87	3	3	5	59	56	3	8	5	100
5	8	4	46	47	4	3	5	68	68	4	8	5	42
1	9	4	31	32	5	3	5	75	74	5	8	5	45
2	9	4	29	29	6	3	5	62	64	0	9	5	79
3	9	4	18	21	7	3	5	21	25	1	9	5	51
4	9	4	13	5	0	4	5	338	350	2	9	5	80
0	10	4	5	6	1	4	5	115	115	3	9	5	49
1	10	4	36	34	2	4	5	198	196	4	9	5	46
2	10	4	29	24	3	4	5	115	110	0	10	5	36
3	10	4	26	25	4	4	5	58	60	1	10	5	53
1	0	5	59	60	5	4	5	11	8	2	10	5	18
2	0	5	214	207	6	4	5	89	89	3	10	5	47
3	0	5	119	118	7	4	5	26	23	0	0	6	16
4	0	5	39	40	0	5	5	33	35	1	0	6	55
5	0	5	8	2	1	5	5	95	98	2	0	6	56
6	0	5	66	67	2	5	5	76	80	3	0	6	71
7	0	5	68	67	3	5	5	79	77	4	0	6	19
0	1	5	230	244	4	5	5	165	165	5	0	6	133
1	1	5	185	181	5	5	5	69	70	6	0	6	17
2	1	5	353	334	6	5	5	8	10	7	0	6	73
3	1	5	84	74	0	6	5	19	19	0	1	6	182
4	1	5	82	75	1	6	5	100	106	1	1	6	187
5	1	5	123	122	2	6	5	59	59	2	1	6	188
6	1	5	80	82	3	6	5	163	157	3	1	6	292
7	1	5	12	17	4	6	5	28	31	4	1	6	109
0	2	5	440	463	5	6	5	5	7	5	1	6	16
1	2	5	409	409	6	6	5	33	33	6	1	6	5
2	2	5	373	360	0	7	5	10	7	7	1	6	50
3	2	5	247	230	1	7	5	117	118	0	2	6	238
4	2	5	91	84	2	7	5	124	125	1	2	6	81
5	2	5	43	45	3	7	5	105	103	2	2	6	214
6	2	5	154	155	4	7	5	111	113	3	2	6	99
7	2	5	28	26	5	7	5	54	54	4	2	6	46
0	3	5	221	234	0	8	5	10	4	5	2	6	102
1	3	5	24	30	1	8	5	20	22	6	2	6	37
2	3	5	239	227	2	8	5	26	21	7	2	6	30

10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
98	0	3	6	137	136	0	8	6	58	59
40	1	3	6	188	182	1	8	6	104	106
45	2	3	6	310	305	2	8	6	60	60
81	3	3	6	112	112	3	8	6	58	55
51	4	3	6	108	103	4	8	6	36	35
77	5	3	6	57	56	5	8	6	50	47
48	6	3	6	23	26	0	9	6	63	64
40	7	3	6	24	27	1	9	6	70	70
32	0	4	6	323	332	2	9	6	27	24
51	1	4	6	8	4	3	9	6	22	18
18	2	4	6	249	255	4	9	6	36	36
44	3	4	6	158	162	0	10	6	5	1
12	4	4	6	11	8	1	10	6	50	49
60	5	4	6	91	85	2	10	6	29	25
58	6	4	6	44	44	1	0	7	78	77
68	7	4	6	32	36	2	0	7	12	16
7	0	5	6	127	126	3	0	7	231	227
137	1	5	6	161	162	4	0	7	38	39
21	2	5	6	155	158	5	0	7	5	9
72	3	5	6	42	42	6	0	7	32	33
181	4	5	6	64	63	7	0	7	80	80
189	5	5	6	10	15	0	1	7	38	40
178	6	5	6	18	10	1	1	7	223	219
272	0	6	6	148	151	2	1	7	227	217
106	1	6	6	120	123	3	1	7	40	41
12	2	6	6	46	44	4	1	7	55	51
8	3	6	6	126	131	5	1	7	59	58
52	4	6	6	22	23	6	1	7	34	35
244	5	6	6	70	69	7	1	7	47	48
82	6	6	6	43	41	0	2	7	273	276
212	0	7	6	60	59	1	2	7	132	130
93	1	7	6	152	160	2	2	7	180	180
45	2	7	6	40	39	3	2	7	131	128
102	3	7	6	16	21	4	2	7	68	68
42	4	7	6	29	29	5	2	7	37	33
31	5	7	6	32	33	6	2	7	35	36

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
7	2	7	59	57	0	8	7	71	73	1
0	3	7	225	235	1	8	7	60	58	2
1	3	7	234	227	2	8	7	72	72	3
2	3	7	181	175	3	8	7	93	92	4
3	3	7	98	98	4	8	7	38	39	5
4	3	7	88	88	0	9	7	27	27	6
5	3	7	80	81	1	9	7	19	18	0
6	3	7	34	36	2	9	7	14	7	1
7	3	7	27	26	3	9	7	53	50	2
0	4	7	66	67	0	10	7	5	0	3
1	4	7	55	52	1	10	7	21	14	4
2	4	7	172	183	2	10	7	46	41	5
3	4	7	71	70	0	0	8	301	325	6
4	4	7	59	61	2	0	8	168	158	0
5	4	7	61	63	3	0	8	4	2	1
6	4	7	18	22	4	0	8	100	100	2
0	5	7	72	73	5	0	8	188	188	3
1	5	7	25	30	6	0	8	104	109	4
2	5	7	103	104	7	0	8	35	35	5
3	5	7	121	120	0	1	8	68	73	6
4	5	7	53	58	1	1	8	125	128	0
5	5	7	88	90	2	1	8	162	158	1
6	5	7	14	16	3	1	8	23	27	2
0	6	7	100	102	4	1	8	92	92	3
1	6	7	117	119	5	1	8	62	66	4
2	6	7	127	132	6	1	8	51	51	5
3	6	7	188	190	7	1	8	82	79	0
4	6	7	55	59	0	2	8	72	75	1
5	6	7	73	74	1	2	8	96	95	2
6	6	7	15	15	2	2	8	59	59	3
0	7	7	34	34	3	2	8	105	106	4
1	7	7	83	82	4	2	8	14	7	5
2	7	7	84	88	5	2	8	60	63	0
3	7	7	47	47	6	2	8	55	58	1
4	7	7	51	49	7	2	8	44	43	2
5	7	7	21	23	0	3	8	31	31	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	8		205	210	4	8	8	69	69	0	4	9	44	41
3	8		38	37	0	9	8	72	73	1	4	9	120	127
3	8		120	120	1	9	8	25	28	2	4	9	151	152
3	8		92	88	2	9	8	34	31	3	4	9	136	133
3	8		108	106	3	9	8	9	11	4	4	9	103	110
3	8		80	81	0	10	8	37	38	5	4	9	32	30
4	8		42	44	1	10	8	29	26	6	4	9	23	22
4	8		17	18	1	0	9	226	228	0	5	9	55	56
4	8		124	122	2	0	9	156	158	1	5	9	99	101
4	8		91	95	3	0	9	118	119	2	5	9	64	63
4	8		142	141	4	0	9	117	120	3	5	9	146	145
4	8		25	24	5	0	9	168	169	4	5	9	65	67
4	8		18	17	6	0	9	38	34	5	5	9	57	63
5	8		50	49	7	0	9	99	97	6	5	9	25	26
5	8		88	88	0	1	9	80	83	0	6	9	49	53
5	8		36	34	1	1	9	112	114	1	6	9	103	107
5	8		170	174	2	1	9	120	114	2	6	9	60	62
5	8		86	87	3	1	9	131	129	3	6	9	48	46
5	8		94	93	4	1	9	121	121	4	6	9	37	39
5	8		31	29	5	1	9	64	68	5	6	9	19	24
6	8		55	59	6	1	9	77	74	0	7	9	65	64
6	8		75	80	7	1	9	54	56	1	7	9	96	98
6	8		66	67	0	2	9	172	175	2	7	9	60	60
6	8		79	79	1	2	9	200	197	3	7	9	13	17
6	8		100	103	2	2	9	185	188	4	7	9	19	22
6	8		48	46	3	2	9	126	123	5	7	9	38	38
7	8		55	58	4	2	9	58	56	0	8	9	26	30
7	8		25	23	5	2	9	133	135	1	8	9	30	28
7	8		77	75	6	2	9	5	5	2	8	9	10	12
7	8		98	100	0	3	9	34	34	3	8	9	13	13
7	8		104	101	1	3	9	52	53	4	8	9	40	39
7	8		61	60	2	3	9	93	89	0	9	9	10	20
8	8		82	82	3	3	9	196	199	1	9	9	28	28
8	8		43	43	4	3	9	56	57	2	9	9	43	42
8	8		20	20	5	3	9	91	96	0	0	10	166	168
8	8		36	37	6	3	9	34	32	1	0	10	61	66

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	10	139	139	4	5	10	135	137	3
3	0	10	108	109	5	5	10	14	11	4
4	0	10	41	44	0	6	10	24	28	5
5	0	10	73	76	1	6	10	75	76	6
6	0	10	18	13	2	6	10	77	74	0
0	1	10	80	89	3	6	10	39	42	1
1	1	10	158	162	4	6	10	47	50	2
2	1	10	56	53	5	6	10	37	36	3
3	1	10	65	62	0	7	10	91	92	4
4	1	10	96	99	1	7	10	43	47	5
5	1	10	49	51	2	7	10	53	53	6
6	1	10	28	30	3	7	10	38	38	0
0	2	10	110	113	4	7	10	58	57	1
1	2	10	99	98	0	8	10	79	75	2
2	2	10	75	69	1	8	10	28	25	3
3	2	10	52	49	2	8	10	15	15	4
4	2	10	53	55	3	8	10	35	33	5
5	2	10	58	60	0	9	10	14	13	0
6	2	10	29	26	1	9	10	45	49	1
0	3	10	20	18	2	9	10	22	17	2
1	3	10	73	74	1	0	11	64	65	3
2	3	10	86	92	2	0	11	109	113	4
3	3	10	96	98	3	0	11	24	24	0
4	3	10	155	160	4	0	11	112	106	1
5	3	10	29	30	5	0	11	115	115	2
6	3	10	9	13	6	0	11	77	80	3
0	4	10	98	101	0	1	11	41	43	4
1	4	10	98	100	1	1	11	157	156	0
2	4	10	125	126	2	1	11	152	148	1
3	4	10	73	77	3	1	11	88	88	2
4	4	10	81	80	4	1	11	274	272	3
6	4	10	25	23	5	1	11	27	26	4
0	5	10	106	115	6	1	11	15	11	0
1	5	10	56	57	0	2	11	70	74	1
2	5	10	114	117	1	2	11	39	44	2
3	5	10	38	36	2	2	11	141	142	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	11		129	126	1	9	11	11	11	3	5	12	44	45
2	11		53	48	0	0	12	68	66	4	5	12	22	23
2	11		49	49	2	0	12	67	68	5	5	12	5	3
2	11		23	27	3	0	12	15	22	0	6	12	47	50
3	11		13	5	4	0	12	45	40	1	6	12	19	21
3	11		64	61	5	0	12	61	63	2	6	12	23	20
3	11		109	111	6	0	12	5	1	3	6	12	37	36
3	11		41	43	0	1	12	134	137	4	6	12	34	30
3	11		111	106	1	1	12	14	23	0	7	12	78	78
3	11		32	36	2	1	12	209	212	1	7	12	27	25
3	11		30	28	3	1	12	76	76	2	7	12	20	23
4	11		203	209	4	1	12	54	59	3	7	12	27	29
4	11		22	19	5	1	12	22	20	0	8	12	18	15
4	11		116	118	6	1	12	21	22	1	8	12	27	27
4	11		41	45	0	2	12	18	20	2	8	12	10	11
4	11		22	22	1	2	12	23	23	1	0	13	54	55
4	11		44	44	2	2	12	38	37	2	0	13	208	210
5	11		38	39	3	2	12	84	87	3	0	13	70	73
5	11		53	56	4	2	12	82	81	4	0	13	72	71
5	11		70	71	5	2	12	60	58	5	0	13	43	41
5	11		32	33	6	2	12	52	52	0	1	13	73	76
5	11		49	52	0	3	12	41	39	1	1	13	68	68
6	11		180	187	1	3	12	82	85	2	1	13	78	82
6	11		85	86	2	3	12	39	39	3	1	13	21	24
6	11		46	46	3	3	12	35	37	4	1	13	76	73
6	11		53	58	4	3	12	13	14	5	1	13	13	8
6	11		17	18	5	3	12	17	15	0	2	13	108	108
7	11		79	81	0	4	12	104	109	1	2	13	114	115
7	11		36	36	1	4	12	9	10	2	2	13	140	141
7	11		94	88	2	4	12	30	30	3	2	13	100	99
7	11		31	30	3	4	12	139	141	4	2	13	64	66
7	11		45	44	4	4	12	44	42	5	2	13	46	45
8	11		49	54	5	4	12	44	42	0	3	13	30	30
8	11		31	32	0	5	12	77	83	1	3	13	98	100
8	11		12	11	1	5	12	68	71	2	3	13	91	90
8	11		29	23	2	5	12	49	47	3	3	13	67	67

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
4	3	13	56	54	2	1	14	53	53	1
5	3	13	19	19	3	1	14	63	60	2
0	4	13	104	106	4	1	14	50	51	3
1	4	13	100	102	5	1	14	47	45	5
2	4	13	69	68	0	2	14	39	44	0
3	4	13	72	71	1	2	14	43	42	1
4	4	13	11	12	2	2	14	13	19	2
5	4	13	12	9	3	2	14	118	118	3
0	5	13	99	100	4	2	14	22	21	4
1	5	13	41	41	0	3	14	26	26	0
2	5	13	73	69	1	3	14	26	31	1
3	5	13	50	49	2	3	14	25	25	2
4	5	13	30	30	3	3	14	58	59	3
0	6	13	5	2	4	3	14	30	34	4
1	6	13	87	87	5	3	14	17	16	1
2	6	13	31	26	0	4	14	10	14	2
3	6	13	24	24	1	4	14	22	29	3
4	6	13	53	47	2	4	14	22	20	4
0	7	13	30	37	3	4	14	54	55	0
1	7	13	14	16	4	4	14	8	15	1
2	7	13	32	32	0	5	14	13	12	2
3	7	13	36	34	1	5	14	27	26	3
0	8	13	5	2	2	5	14	35	36	4
1	8	13	8	11	3	5	14	27	25	0
0	0	14	14	15	4	5	14	13	15	1
1	0	14	18	22	0	6	14	5	8	2
3	0	14	127	128	1	6	14	65	65	3
4	0	14	17	18	3	6	14	5	12	0
5	0	14	24	20	1	7	14	10	8	1
0	1	14	13	20	2	7	14	24	26	2
1	1	14	22	31						

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	15		34	32	0	7	15	9	12	0	1	17	35	35
0	15		83	86	0	0	16	19	17	1	1	17	21	27
0	15		17	19	1	0	16	53	47	2	1	17	60	55
0	15		5	7	2	0	16	84	86	3	1	17	32	29
1	15		12	5	3	0	16	20	23	1	2	17	19	23
1	15		184	181	4	0	16	33	28	2	2	17	23	20
1	15		103	100	0	1	16	26	30	3	2	17	15	16
1	15		11	14	1	1	16	97	102	0	3	17	63	62
1	15		18	11	2	1	16	37	37	1	3	17	40	39
2	15		38	39	3	1	16	38	38	2	3	17	52	51
2	15		29	27	4	1	16	32	27	3	3	17	13	15
2	15		29	31	0	2	16	28	30	0	4	17	23	19
2	15		45	44	1	2	16	27	27	1	4	17	18	9
2	15		19	20	2	2	16	78	79	2	4	17	17	18
3	15		47	49	3	2	16	21	21	1	5	17	24	21
3	15		69	69	0	3	16	53	53	0	0	18	79	79
3	15		23	21	1	3	16	64	65	1	0	18	68	66
3	15		22	22	2	3	16	33	31	2	0	18	66	62
4	15		15	16	3	3	16	61	57	0	1	18	27	23
4	15		20	13	0	4	16	46	46	1	1	18	38	37
4	15		40	37	1	4	16	37	37	2	1	18	15	16
4	15		37	38	2	4	16	11	12	0	2	18	40	41
4	15		28	28	3	4	16	40	38	1	2	18	43	39
5	15		20	20	0	5	16	72	65	2	2	18	36	32
5	15		27	28	1	5	16	15	16	0	3	18	16	18
5	15		57	56	2	5	16	28	23	1	3	18	15	16
5	15		36	30	0	6	16	13	12	0	4	18	16	7
6	15		12	10	1	6	16	28	25	1	0	19	44	43
6	15		34	36	1	0	17	37	38	0	1	19	14	10
6	15		27	22	2	0	17	15	13	1	1	19	17	16

VENTILONE C

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	0	3376	3734	3	7	0	136	27	2
4	0	0	1396	1410	0	8	0	130	123	3
6	0	0	558	580	1	8	0	77	78	4
8	0	0	239	249	2	8	0	79	82	5
3	1	0	90	89	4	8	0	59	61	6
4	1	0	137	125	1	9	0	73	93	7
5	1	0	75	68	2	9	0	53	57	0
6	1	0	100	100	3	9	0	53	63	1
7	1	0	45	52	0	10	0	667	661	2
0	2	0	1177	1194	2	10	0	481	474	3
2	2	0	1076	1098	4	10	0	169	164	4
3	2	0	81	85	2	11	0	96	104	5
4	2	0	576	598	4	11	0	63	62	6
6	2	0	276	278	1	12	0	84	80	0
2	3	0	382	403	2	12	0	64	61	1
3	3	0	135	115	3	12	0	49	57	3
4	3	0	200	221	5	12	0	41	30	5
5	3	0	111	91	2	14	0	56	51	6
6	3	0	89	111	4	14	0	145	140	0
7	3	0	42	45	6	14	0	123	123	1
0	4	0	828	823	0	16	0	555	564	2
2	4	0	666	683	1	16	0	243	234	3
3	4	0	74	77	2	16	0	458	455	4
4	4	0	274	290	3	16	0	163	157	5
1	5	0	101	128	4	16	0	302	294	0
2	5	0	190	186	5	16	0	84	95	1
3	5	0	58	71	1	18	0	68	73	2
4	5	0	59	66	2	18	0	73	74	3
0	6	0	219	206	3	18	0	71	74	0
1	6	0	144	150	4	18	0	91	86	1
2	6	0	153	153	5	18	0	41	29	2
3	6	0	191	195	1	20	0	51	56	3
4	6	0	68	67	2	20	0	43	35	5
6	6	0	53	51	2	21	0	39	29	0
1	7	0	155	95	1	22	0	41	22	1
2	7	0	82	83	0	0	1	413	411	2

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	1	1	520	541	3	6	1	160	153	6	13	1	45	48
0	1	1	81	87	4	6	1	56	56	0	14	1	142	138
0	1	1	297	298	5	6	1	61	65	1	14	1	151	145
0	1	1	90	100	1	7	1	149	165	2	14	1	87	87
0	1	1	119	117	2	7	1	126	117	3	14	1	126	115
0	1	1	57	49	3	7	1	114	126	4	14	1	58	47
1	1	1	525	523	4	7	1	92	93	5	14	1	81	77
1	1	1	164	174	5	7	1	42	44	6	14	1	41	47
1	1	1	421	406	7	7	1	49	36	0	15	1	460	453
1	1	1	98	104	0	8	1	242	239	1	15	1	101	101
1	1	1	279	293	1	8	1	93	100	2	15	1	384	382
1	1	1	64	72	2	8	1	147	150	3	15	1	94	101
1	1	1	173	185	3	8	1	64	78	4	15	1	242	247
2	1	1	236	235	4	8	1	94	101	5	15	1	58	51
2	1	1	678	677	6	8	1	56	60	6	15	1	141	144
2	1	1	197	181	7	8	1	43	47	0	16	1	254	271
2	1	1	48	66	0	9	1	283	264	1	16	1	331	335
2	1	1	46	47	1	9	1	140	149	2	16	1	236	252
3	1	1	246	237	2	9	1	146	140	3	16	1	269	269
3	1	1	271	291	3	9	1	107	101	4	16	1	170	169
3	1	1	213	222	0	10	1	64	56	5	16	1	172	172
3	1	1	178	172	1	10	1	163	155	0	17	1	240	239
3	1	1	83	94	2	10	1	117	122	2	17	1	219	206
3	1	1	70	62	4	10	1	61	71	4	17	1	157	144
4	1	1	97	102	6	10	1	39	30	1	18	1	192	189
4	1	1	365	350	0	11	1	163	151	3	18	1	134	127
4	1	1	73	70	2	11	1	139	133	4	18	1	39	22
4	1	1	98	99	4	11	1	73	71	0	19	1	69	67
5	1	1	353	348	0	12	1	155	159	1	19	1	52	70
5	1	1	273	291	1	12	1	190	186	3	19	1	67	62
5	1	1	153	134	2	12	1	97	94	0	20	1	69	77
5	1	1	121	128	3	12	1	78	74	1	20	1	58	72
5	1	1	63	61	4	12	1	51	58	2	20	1	66	58
6	1	1	149	143	6	12	1	47	38	1	21	1	81	79
6	1	1	83	77	0	13	1	73	74	3	21	1	60	57
6	1	1	160	169	1	13	1	107	101	0	22	1	46	48

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	22	1	74	68	4	7	2	88	95
0	0	2	250	250	5	7	2	105	101
1	0	2	200	192	6	7	2	43	50
2	0	2	100	89	7	7	2	61	61
3	0	2	149	130	1	8	2	773	728
0	1	2	411	405	2	8	2	63	43
2	1	2	292	276	3	8	2	472	448
3	1	2	145	154	5	8	2	234	225
4	1	2	132	136	7	8	2	106	108
5	1	2	45	55	0	9	2	301	293
0	2	2	152	155	1	9	2	451	434
1	2	2	616	620	2	9	2	277	266
2	2	2	173	182	3	9	2	246	238
3	2	2	211	216	4	9	2	164	162
4	2	2	100	101	5	9	2	116	117
5	2	2	47	53	6	9	2	57	53
0	3	2	257	261	7	9	2	59	63
1	3	2	513	522	0	10	2	89	79
2	3	2	329	332	1	10	2	312	291
3	3	2	98	98	2	10	2	92	83
4	3	2	185	174	3	10	2	175	174
6	3	2	61	62	5	10	2	89	101
1	4	2	101	124	7	10	2	57	61
2	4	2	72	59	0	11	2	190	192
1	5	2	251	233	1	11	2	136	135
2	5	2	121	113	2	11	2	144	149
3	5	2	74	74	3	11	2	64	64
4	5	2	58	63	4	11	2	83	75
1	6	2	292	273	0	12	2	72	70
3	6	2	202	187	1	12	2	290	282
5	6	2	111	121	2	12	2	77	78
7	6	2	72	75	3	12	2	161	157
0	7	2	467	440	5	12	2	51	53
1	7	2	429	407	0	13	2	44	37
2	7	2	271	257	1	13	2	154	151
3	7	2	231	213	3	13	2	125	123

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	13	2	41	55	0	2	3	100	103	0	9	3	161	153
6	13	2	48	48	1	2	3	93	99	1	9	3	497	466
0	14	2	89	93	2	2	3	69	75	2	9	3	69	66
1	14	2	137	136	4	2	3	53	36	3	9	3	389	372
2	14	2	66	73	5	2	3	44	43	5	9	3	197	190
3	14	2	108	104	1	3	3	637	623	7	9	3	91	86
5	14	2	44	43	2	3	3	159	152	0	10	3	343	316
6	14	2	52	57	3	3	3	229	234	1	10	3	78	75
3	15	2	82	81	4	3	3	77	92	2	10	3	276	277
5	15	2	69	60	5	3	3	64	59	4	10	3	128	130
4	16	2	46	29	0	4	3	307	314	0	11	3	83	83
0	17	2	44	38	1	4	3	67	88	1	11	3	76	85
1	17	2	157	155	2	4	3	233	220	2	11	3	60	54
3	17	2	90	88	4	4	3	95	97	3	11	3	84	83
5	17	2	56	55	5	4	3	45	32	5	11	3	44	37
0	18	2	82	83	6	4	3	59	58	0	12	3	98	96
1	18	2	46	54	0	5	3	165	150	1	12	3	77	89
2	18	2	79	73	1	5	3	523	521	2	12	3	64	77
3	18	2	59	55	3	5	3	289	282	3	12	3	58	46
4	18	2	43	53	5	5	3	89	87	0	13	3	69	66
1	19	2	78	76	0	6	3	73	72	1	13	3	177	182
4	19	2	55	59	1	6	3	240	238	2	13	3	69	70
0	20	2	47	49	2	6	3	69	65	3	13	3	116	122
2	20	2	47	50	3	6	3	123	122	4	13	3	57	54
3	20	2	38	28	4	6	3	48	47	5	13	3	42	31
0	21	2	85	92	5	6	3	61	60	0	14	3	224	219
2	21	2	68	55	0	7	3	231	240	1	14	3	56	61
3	21	2	36	36	1	7	3	695	649	2	14	3	189	179
1	22	2	51	55	2	7	3	74	80	3	14	3	59	54
0	0	3	844	838	3	7	3	423	397	4	14	3	100	101
1	0	3	119	116	4	7	3	45	30	1	15	3	101	99
2	0	3	634	621	5	7	3	201	207	0	16	3	45	36
3	0	3	81	84	7	7	3	90	94	1	16	3	108	112
4	0	3	278	278	0	8	3	134	130	2	16	3	60	70
6	0	3	83	83	2	8	3	145	145	3	16	3	74	84
1	1	3	125	114	4	8	3	82	77	4	16	3	44	49

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C15H1207

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO
5	16	3	66	79	4	4	4	47	40	1	12	4	137
1	17	3	347	357	5	4	4	70	60	2	12	4	90
2	17	3	44	34	6	4	4	37	39	3	12	4	87
3	17	3	212	212	0	5	4	211	199	4	12	4	43
1	19	3	119	118	1	5	4	195	198	0	13	4	149
3	19	3	117	117	2	5	4	108	103	1	13	4	71
1	20	3	59	52	3	5	4	67	64	2	13	4	134
1	21	3	91	88	4	5	4	51	50	3	13	4	66
0	0	4	667	672	1	6	4	73	70	0	14	4	58
1	0	4	75	81	3	6	4	102	115	1	14	4	53
2	0	4	422	426	5	6	4	65	70	2	14	4	42
3	0	4	69	66	0	7	4	163	167	3	14	4	44
4	0	4	196	200	2	7	4	98	95	0	15	4	302
6	0	4	122	116	4	7	4	45	47	1	15	4	84
7	0	4	42	14	6	7	4	44	37	2	15	4	269
0	1	4	466	460	0	8	4	67	73	3	15	4	72
1	1	4	380	386	1	8	4	54	54	4	15	4	160
2	1	4	349	337	2	8	4	57	54	0	16	4	253
3	1	4	253	265	3	8	4	48	57	2	16	4	198
4	1	4	214	207	5	8	4	46	32	4	16	4	106
5	1	4	102	95	0	9	4	144	136	5	16	4	58
6	1	4	122	118	1	9	4	125	122	0	17	4	160
7	1	4	39	34	2	9	4	137	124	2	17	4	131
1	2	4	575	561	4	9	4	89	83	4	17	4	67
2	2	4	105	88	1	10	4	130	125	0	18	4	54
3	2	4	283	281	2	10	4	88	82	2	18	4	72
4	2	4	103	99	3	10	4	102	111	3	18	4	45
5	2	4	119	135	4	10	4	54	51	4	18	4	66
6	2	4	58	65	5	10	4	45	54	0	19	4	57
7	2	4	51	59	0	11	4	164	160	2	19	4	56
0	3	4	103	115	1	11	4	178	175	0	21	4	79
1	3	4	279	260	2	11	4	126	126	0	0	5	379
2	3	4	55	64	3	11	4	92	89	1	0	5	58
3	3	4	92	97	4	11	4	51	53	2	0	5	223
1	4	4	201	197	5	11	4	47	45	3	0	5	82
3	4	4	137	135	0	12	4	83	81	4	0	5	102

10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
137	5	0	5	57	56	5	6	5	55	68
85	6	0	5	68	64	6	6	5	36	38
78	0	1	5	618	579	0	7	5	87	88
46	2	1	5	471	461	1	7	5	95	89
150	3	1	5	51	53	3	7	5	57	59
74	4	1	5	256	253	4	7	5	45	34
133	5	1	5	51	36	6	7	5	36	26
65	6	1	5	140	135	0	8	5	107	111
56	0	2	5	154	147	1	8	5	60	62
52	1	2	5	199	203	2	8	5	49	50
32	2	2	5	166	162	3	8	5	66	63
41	3	2	5	188	191	0	9	5	214	224
326	4	2	5	107	117	1	9	5	54	61
86	5	2	5	93	90	2	9	5	180	192
277	6	2	5	71	65	3	9	5	58	64
74	7	2	5	39	30	4	9	5	116	123
159	1	3	5	181	176	6	9	5	61	66
271	2	3	5	84	76	1	10	5	99	101
204	3	3	5	103	104	4	10	5	39	34
107	4	3	5	68	67	0	11	5	227	229
41	5	3	5	55	51	1	11	5	116	115
167	0	4	5	126	111	2	11	5	160	171
127	1	4	5	228	218	3	11	5	64	70
86	2	4	5	75	71	4	11	5	60	66
65	3	4	5	75	82	5	11	5	51	44
73	4	4	5	45	54	0	12	5	149	162
34	6	4	5	41	43	1	12	5	46	41
71	0	5	5	114	102	2	12	5	100	95
56	1	5	5	166	160	3	12	5	42	50
40	2	5	5	50	53	5	12	5	43	34
73	3	5	5	80	71	0	13	5	162	170
382	0	6	5	77	77	2	13	5	155	152
57	1	6	5	75	72	4	13	5	111	102
218	2	6	5	74	76	0	14	5	87	87
77	3	6	5	44	53	1	14	5	66	59
97	4	6	5	54	60	2	14	5	111	111

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	14	5	71	74	0	6	6	200	204
4	14	5	98	99	1	6	6	72	78
5	14	5	66	60	2	6	6	152	158
0	15	5	80	88	4	6	6	88	95
1	15	5	121	138	5	6	6	40	41
2	15	5	97	105	0	7	6	60	59
3	15	5	105	101	1	7	6	405	429
4	15	5	94	98	2	7	6	45	49
1	16	5	104	99	3	7	6	316	317
3	16	5	69	81	4	7	6	54	46
1	17	5	144	137	5	7	6	196	198
3	17	5	99	92	6	7	6	49	43
4	17	5	47	45	0	8	6	209	214
0	18	5	97	95	2	8	6	159	167
1	18	5	49	35	3	8	6	42	50
2	18	5	70	59	4	8	6	100	114
0	0	6	535	531	6	8	6	65	67
2	0	6	307	297	0	9	6	105	104
3	0	6	48	49	1	9	6	445	442
4	0	6	82	77	2	9	6	105	111
1	1	6	63	68	3	9	6	328	328
4	1	6	50	53	4	9	6	90	93
6	1	6	54	40	5	9	6	221	208
2	2	6	69	76	6	9	6	60	52
4	2	6	45	59	0	10	6	432	442
0	3	6	140	142	1	10	6	111	119
1	3	6	266	256	2	10	6	383	386
2	3	6	83	89	3	10	6	64	57
3	3	6	175	172	4	10	6	242	238
5	3	6	71	75	5	10	6	38	29
0	4	6	178	173	1	11	6	71	81
1	4	6	81	82	2	11	6	45	62
2	4	6	131	145	3	11	6	84	86
1	5	6	235	237	4	11	6	45	45
3	5	6	132	125	5	11	6	79	73
5	5	6	53	46	0	12	6	47	45

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	13	6	61	62	4	4	7	73	70	0	11	7	108	108
1	13	6	182	178	5	4	7	46	41	2	11	7	98	97
2	13	6	49	37	6	4	7	54	49	3	11	7	51	42
3	13	6	103	106	0	5	7	218	218	4	11	7	58	61
5	13	6	44	48	2	5	7	175	165	5	11	7	56	46
0	14	6	196	190	4	5	7	102	96	1	12	7	141	144
2	14	6	147	138	5	5	7	54	55	2	12	7	63	64
4	14	6	75	82	6	5	7	60	67	3	12	7	85	84
1	15	6	99	104	0	6	7	165	170	4	12	7	62	67
3	15	6	85	88	1	6	7	231	229	1	13	7	142	141
0	16	6	62	74	2	6	7	150	156	2	13	7	47	39
2	16	6	86	94	3	6	7	177	182	3	13	7	67	72
0	17	6	42	46	4	6	7	95	95	0	14	7	94	94
1	17	6	84	81	5	6	7	146	152	1	14	7	79	81
2	17	6	41	31	6	6	7	64	53	2	14	7	57	66
3	17	6	43	69	0	7	7	179	178	3	14	7	60	59
1	19	6	67	61	1	7	7	359	352	0	15	7	39	27
0	0	7	122	133	2	7	7	176	177	1	16	7	36	32
2	0	7	60	68	3	7	7	272	269	2	16	7	47	38
5	0	7	40	27	4	7	7	124	131	0	1	8	104	101
0	1	7	77	84	5	7	7	181	174	1	1	8	42	45
1	1	7	197	198	0	8	7	57	56	2	1	8	72	64
2	1	7	71	69	1	8	7	584	605	3	1	8	78	79
3	1	7	87	95	2	8	7	61	61	4	1	8	45	36
4	1	7	60	58	3	8	7	452	463	5	1	8	43	46
6	1	7	53	31	5	8	7	275	267	1	2	8	118	127
0	2	7	65	68	1	9	7	400	399	3	2	8	84	82
1	2	7	260	273	3	9	7	316	308	5	2	8	56	53
3	2	7	150	143	4	9	7	60	45	0	3	8	70	66
5	2	7	46	51	5	9	7	189	183	1	3	8	56	49
2	3	7	59	57	0	10	7	158	156	2	3	8	44	42
3	3	7	43	39	1	10	7	139	137	4	3	8	46	38
4	3	7	78	67	2	10	7	134	123	0	4	8	42	52
0	4	7	110	110	3	10	7	127	129	2	4	8	48	45
2	4	7	96	96	4	10	7	78	78	2	5	8	58	63
3	4	7	63	62	5	10	7	103	103	4	5	8	49	67

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	6	8	58	62	0	10	8	52	60	2
1	6	8	199	202	1	10	8	52	52	4
2	6	8	68	76	2	10	8	66	68	0
3	6	8	148	143	4	10	8	38	41	1
4	6	8	58	52	0	11	8	92	101	2
5	6	8	90	83	2	11	8	71	71	4
0	7	8	194	197	3	11	8	60	59	0
2	7	8	141	141	1	12	8	52	53	2
3	7	8	51	47	3	12	8	44	48	4
4	7	8	78	75	0	13	8	53	52	1
0	8	8	97	109	1	13	8	46	47	3
1	8	8	102	92	2	13	8	48	37	0
2	8	8	119	116	3	13	8	42	45	2
3	8	8	108	97	0	14	8	56	39	4
4	8	8	91	86	1	14	8	68	65	3
5	8	8	90	94	0	15	8	54	57	1
0	9	8	53	67	2	15	8	45	49	3
1	9	8	62	60	0	16	8	61	60	1
2	9	8	78	73	1	16	8	109	98	3
4	9	8	53	58	0	0	9	499	531	0

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	9		427	452	1	9	9	47	48	0	6	10	81	75
0	9		274	287	3	9	9	53	50	1	6	10	44	54
1	9		52	41	0	10	9	160	162	2	6	10	78	67
1	9		72	88	2	10	9	130	128	0	7	10	54	57
1	9		69	66	3	10	9	43	37	1	7	10	50	45
1	9		62	54	0	12	9	63	63	2	7	10	65	58
2	9		173	186	2	12	9	49	47	0	8	10	62	59
2	9		145	154	1	13	9	78	71	1	8	10	107	101
2	9		97	93	2	13	9	42	37	2	8	10	47	46
3	9		38	33	0	14	9	44	63	0	9	10	69	73
3	9		48	58	0	0	10	94	102	1	9	10	83	74
4	9		244	250	2	0	10	66	71	2	9	10	63	67
4	9		189	191	0	3	10	68	67	0	10	10	68	76
4	9		94	88	1	3	10	45	48	0	11	10	37	37
5	9		52	45	2	3	10	44	48	1	1	11	58	46
6	9		47	45	3	3	10	41	37	2	1	11	42	21
6	9		37	19	1	4	10	40	42	2	2	11	37	17
7	9		93	93	3	4	10	41	52	1	4	11	34	16
7	9		68	62	0	5	10	60	58	0	5	11	55	53
8	9		48	39	2	5	10	52	38	1	6	11	122	116

ARISTOLINDIQUINONE

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	0	0	687	-704	-3	0	1	282	-293
4	0	0	90	84	-1	0	1	131	146
1	1	0	96	-121	1	0	1	209	-209
3	1	0	246	257	-5	1	1	63	72
4	1	0	72	68	-3	1	1	313	330
5	1	0	80	-57	-1	1	1	160	-150
0	2	0	512	-493	0	1	1	186	-153
2	2	0	174	-181	1	1	1	466	-489
3	2	0	119	120	2	1	1	219	-212
1	3	0	361	-334	3	1	1	70	-52
2	3	0	69	-75	5	1	1	139	-152
5	3	0	45	-32	-4	2	1	63	57
0	4	0	455	-443	-3	2	1	98	103
1	4	0	108	102	-1	2	1	137	157
2	4	0	90	-89	0	2	1	322	-310
2	5	0	239	-216	2	2	1	118	118
5	5	0	67	-41	-2	3	1	206	193
0	6	0	115	118	-1	3	1	112	105
1	6	0	321	313	0	3	1	236	-205
2	6	0	169	167	2	3	1	300	296
3	6	0	354	-359	-2	4	1	173	-174
1	7	0	96	95	-1	4	1	106	124
2	7	0	100	95	1	4	1	178	180
3	7	0	49	36	-5	5	1	98	-105
0	8	0	69	83	-4	5	1	50	43
1	8	0	106	102	-2	5	1	188	176
3	8	0	99	95	-1	5	1	75	-50
1	9	0	87	-104	0	5	1	166	-171
0	10	0	58	59	1	5	1	88	-79
3	10	0	46	37	2	5	1	392	379
1	11	0	68	-67	3	5	1	130	139
2	11	0	132	-130	4	5	1	224	-198
0	12	0	81	79	-5	6	1	122	103
2	12	0	97	-87	-4	6	1	62	54
1	13	0	63	62	-3	6	1	410	-408
-5	0	1	70	-61	-1	6	1	270	239

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	6	1	193	161	0	1	2	645	604	1	7	2	52	-29
1	6	1	101	-102	2	1	2	490	-466	2	7	2	153	154
3	6	1	263	-273	3	1	2	110	96	3	7	2	74	-73
4	6	1	63	-64	-3	2	2	159	158	-4	8	2	70	-76
-3	7	1	176	-174	-2	2	2	158	-152	0	8	2	197	-180
-2	7	1	153	-149	-1	2	2	946	-954	1	8	2	63	-71
-1	7	1	177	166	0	2	2	248	-250	2	8	2	208	218
0	7	1	380	-357	2	2	2	79	66	3	8	2	67	61
1	7	1	62	-70	-2	3	2	142	-144	-3	9	2	107	104
3	7	1	72	-71	-1	3	2	96	86	-1	9	2	79	-94
-4	8	1	48	40	0	3	2	160	163	3	9	2	48	-32
-3	8	1	89	90	1	3	2	120	-118	-2	10	2	51	39
0	8	1	106	-91	2	3	2	77	-79	1	10	2	53	-71
2	8	1	78	76	3	3	2	64	-46	3	10	2	68	56
3	8	1	89	94	-4	4	2	151	138	-3	11	2	54	43
4	8	1	67	-51	-1	4	2	136	125	-2	11	2	84	92
-1	9	1	120	123	0	4	2	281	-285	-1	11	2	125	-138
0	9	1	86	94	1	4	2	301	283	1	11	2	153	149
1	9	1	60	-55	4	4	2	115	-110	-1	12	2	72	-72
-3	10	1	47	52	-5	5	2	64	-47	1	12	2	84	84
3	10	1	80	73	-4	5	2	51	-55	0	13	2	69	84
-1	11	1	120	130	-3	5	2	160	-163	-3	0	3	72	-55
0	11	1	53	63	-1	5	2	463	435	-1	0	3	687	713
1	11	1	176	-170	0	5	2	244	242	1	0	3	129	146
3	11	1	51	22	2	5	2	207	-196	3	0	3	204	-195
-2	12	1	108	-107	3	5	2	62	64	-5	1	3	51	21
1	12	1	113	-101	4	5	2	148	145	-3	1	3	116	132
2	12	1	101	94	-4	6	2	63	-57	-2	1	3	212	206
-1	13	1	77	-80	-3	6	2	129	137	0	1	3	199	197
0	13	1	59	59	-1	6	2	116	-110	1	1	3	387	404
1	13	1	68	71	0	6	2	79	73	2	1	3	445	-419
0	0	2	966	906	2	6	2	87	-80	-3	2	3	191	207
2	0	2	375	-372	3	6	2	142	-140	-2	2	3	292	291
4	0	2	103	99	4	6	2	62	63	-1	2	3	1119	-1149
-3	1	2	156	153	-3	7	2	72	-72	0	2	3	150	145
-1	1	2	304	-338	0	7	2	175	-165	1	2	3	456	462

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	2	3	114	116	3	8	3	50	25	2
-4	3	3	119	-135	4	8	3	101	-96	3
-3	3	3	87	79	-1	9	3	185	170	4
-2	3	3	77	-71	1	9	3	114	115	-4
-1	3	3	96	103	3	9	3	47	-36	-3
0	3	3	170	171	-1	10	3	77	-85	-2
1	3	3	406	397	0	10	3	95	-89	-1
2	3	3	143	127	1	10	3	191	198	0
3	3	3	53	-52	-2	11	3	83	-90	2
4	3	3	217	227	1	11	3	110	-106	3
-4	4	3	283	-283	1	12	3	111	-107	4
-2	4	3	104	89	2	12	3	55	-69	-4
0	4	3	220	-228	-1	13	3	52	-29	-1
2	4	3	221	-221	0	13	3	96	100	0
4	4	3	146	147	1	13	3	50	59	1
-2	5	3	72	75	-4	0	4	71	-76	-5
-1	5	3	354	-325	-2	0	4	258	-246	-4
0	5	3	85	-81	0	0	4	130	132	-3
1	5	3	269	-260	2	0	4	249	246	-2
2	5	3	69	64	-3	1	4	91	-97	-1
3	5	3	49	-48	-1	1	4	630	670	3
4	5	3	68	-68	0	1	4	458	-431	4
-4	6	3	137	131	1	1	4	355	-346	-3
-3	6	3	60	50	2	1	4	273	-256	-2
0	6	3	97	-96	3	1	4	98	106	-1
1	6	3	65	-82	4	1	4	55	-62	0
4	6	3	133	-137	-4	2	4	63	60	2
-3	7	3	177	-159	-3	2	4	133	147	3
-2	7	3	84	74	-2	2	4	73	-73	-3
-1	7	3	139	136	-1	2	4	1440	-1515	-1
1	7	3	57	-48	1	2	4	391	375	0
4	7	3	61	-23	3	2	4	94	-102	1
-4	8	3	55	66	-4	3	4	174	-175	2
-2	8	3	75	79	-2	3	4	119	114	4
0	8	3	156	-143	0	3	4	186	173	-3
2	8	3	409	418	1	3	4	463	-458	-2

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	4		94	-111	-1	9	4	91	85	3	4	5	155	-165
3	4		75	80	0	9	4	123	112	4	4	5	90	92
3	4		95	110	1	9	4	124	-120	-4	5	5	109	-114
4	4		323	307	-3	10	4	66	51	-3	5	5	62	-52
4	4		130	139	0	10	4	63	50	-2	5	5	119	104
4	4		279	-293	1	10	4	178	-180	1	5	5	235	-239
4	4		139	146	2	10	4	124	-132	4	5	5	78	67
4	4		217	188	3	10	4	85	85	-5	6	5	50	69
4	4		76	-79	-2	11	4	102	107	-1	6	5	66	75
4	4		63	75	0	11	4	59	37	0	6	5	105	-109
4	4		203	-204	1	12	4	92	90	1	6	5	65	83
5	4		108	103	0	13	4	49	42	3	6	5	59	-63
5	4		203	198	-3	0	5	140	138	-2	7	5	106	103
5	4		270	266	1	0	5	152	146	-1	7	5	110	113
5	4		102	91	-3	1	5	58	42	2	7	5	68	-62
6	4		46	-26	-2	1	5	114	116	3	7	5	57	57
6	4		78	-82	-1	1	5	253	262	-3	8	5	57	-64
6	4		85	72	0	1	5	222	-213	-1	8	5	130	-123
6	4		60	-50	1	1	5	166	186	0	8	5	83	83
6	4		202	193	5	1	5	47	34	2	8	5	195	197
6	4		56	52	-5	2	5	49	-49	3	8	5	56	67
6	4		65	58	-4	2	5	146	142	-2	9	5	98	122
7	4		92	-94	-2	2	5	94	-108	-1	9	5	139	132
7	4		206	-214	-1	2	5	388	-402	0	9	5	173	-186
7	4		56	-40	0	2	5	133	-121	1	9	5	110	-100
7	4		103	103	1	2	5	218	226	2	9	5	118	109
7	4		158	-164	-1	3	5	443	-462	3	9	5	50	12
7	4		76	68	1	3	5	817	805	-2	10	5	63	47
8	4		72	-64	3	3	5	269	-277	-1	10	5	103	100
8	4		58	-58	4	3	5	97	113	0	10	5	146	-138
8	4		69	-73	-5	4	5	58	47	-2	11	5	75	-69
8	4		60	-67	-4	4	5	238	-231	1	11	5	47	41
8	4		279	280	-2	4	5	379	367	0	12	5	60	-49
8	4		83	-73	-1	4	5	148	150	2	12	5	44	-17
9	4		100	101	0	4	5	225	-217	-2	0	6	321	-313
9	4		187	-209	1	4	5	104	98	2	0	6	95	-101

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-1	1	6	95	102	2	7	6	73	-72
0	1	6	157	-139	0	8	6	174	-172
1	1	6	271	-267	2	8	6	61	56
2	1	6	115	-119	3	8	6	53	-21
3	1	6	76	-83	-4	9	6	114	95
-5	2	6	173	160	-3	9	6	98	105
-4	2	6	91	89	-2	9	6	76	-79
-2	2	6	186	-186	0	9	6	195	193
-1	2	6	133	-141	1	9	6	56	50
0	2	6	94	101	-2	10	6	81	-80
1	2	6	168	169	-1	10	6	72	59
2	2	6	123	113	0	10	6	245	246
3	2	6	288	-303	1	10	6	190	-195
4	2	6	64	-64	2	10	6	59	-69
-4	3	6	84	-94	3	10	6	66	69
-2	3	6	323	330	-2	11	6	56	37
0	3	6	121	122	-1	11	6	55	-30
1	3	6	238	215	0	11	6	56	-50
3	3	6	183	197	-2	12	6	61	-45
-5	4	6	84	-90	-1	12	6	46	13
-4	4	6	62	-57	-3	0	7	159	149
-2	4	6	456	470	-1	0	7	143	130
-1	4	6	66	72	-4	1	7	75	60
1	4	6	143	-133	0	1	7	844	774
3	4	6	153	160	1	1	7	116	132
4	4	6	75	71	2	1	7	180	-185
-2	5	6	161	-165	3	1	7	77	88
-1	5	6	77	-68	-5	2	7	180	-171
0	5	6	62	64	-3	2	7	159	166
3	5	6	120	-123	-2	2	7	156	-167
-4	6	6	64	-51	-1	2	7	192	-205
0	6	6	152	-160	1	2	7	216	-217
3	6	6	66	62	2	2	7	96	82
-3	7	6	75	-77	3	2	7	249	256
-2	7	6	140	-142	4	2	7	71	66
-1	7	6	96	-107	-5	3	7	101	-92

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-2	3	7	464	-477	2	0	8	165	-167	-2	7	8	54	-53
-1	3	7	75	-66	-5	1	8	52	-55	-1	7	8	128	141
0	3	7	254	-237	0	1	8	295	-266	-2	8	8	62	-68
1	3	7	310	298	1	1	8	72	-62	-1	8	8	171	173
2	3	7	88	-93	2	1	8	145	151	0	8	8	183	-191
-5	4	7	49	43	-5	2	8	176	166	1	8	8	89	-91
-4	4	7	74	-74	-3	2	8	82	-70	2	8	8	101	100
-3	4	7	59	-55	0	2	8	193	-178	-4	9	8	53	48
-2	4	7	440	459	1	2	8	162	-173	-3	9	8	109	120
-1	4	7	124	-117	2	2	8	197	189	1	9	8	77	77
0	4	7	297	-301	3	2	8	188	-193	-2	10	8	64	61
3	4	7	150	-159	-5	3	8	97	102	-1	10	8	61	65
-5	5	7	64	56	-3	3	8	100	-90	0	10	8	68	64
-3	5	7	93	112	-2	3	8	240	-242	1	10	8	170	-181
-2	5	7	117	115	-1	3	8	302	321	-2	11	8	67	-67
-1	5	7	167	-173	0	3	8	346	327	1	11	8	51	-59
2	5	7	61	69	1	3	8	116	118	2	11	8	49	44
3	5	7	158	-168	2	3	8	68	-62	-2	12	8	68	-68
-2	6	7	63	-62	4	3	8	60	-66	1	12	8	53	29
1	6	7	146	160	-5	4	8	115	-112	-1	0	9	218	-203
-3	7	7	79	84	-2	4	8	413	433	3	0	9	50	51
-2	7	7	55	67	-1	4	8	75	88	-5	1	9	65	54
-1	7	7	64	-63	0	4	8	275	-279	-3	1	9	200	-195
0	7	7	121	125	2	4	8	168	176	-2	1	9	102	-102
-4	8	7	59	-69	3	4	8	125	130	-1	1	9	172	-162
-2	8	7	77	-78	4	4	8	70	-51	0	1	9	522	485
-1	8	7	78	-78	-5	5	8	74	65	1	1	9	94	-97
0	8	7	268	278	-3	5	8	198	-203	2	1	9	217	-224
1	8	7	100	103	-2	5	8	57	62	4	1	9	56	58
-3	9	7	78	-89	-1	5	8	94	99	-3	2	9	106	106
1	9	7	116	123	0	5	8	127	-112	-1	2	9	188	-188
-2	10	7	63	70	1	5	8	65	-55	0	2	9	176	180
0	10	7	123	-126	2	5	8	72	75	1	2	9	84	84
1	10	7	288	-300	3	5	8	141	-148	2	2	9	256	-267
-2	11	7	55	-69	-2	6	8	60	-62	4	2	9	64	59
-2	0	8	235	-227	-3	7	8	157	-168	-3	3	9	89	86

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-2	3	9	138	-132	-3	1	10	108	115
1	3	9	66	86	-2	1	10	75	83
3	3	9	66	68	0	1	10	78	87
-5	4	9	63	-46	2	1	10	146	145
-3	4	9	114	116	-3	2	10	130	129
-2	4	9	123	129	-1	2	10	94	95
1	4	9	86	-93	2	2	10	236	249
-3	5	9	63	52	3	2	10	51	47
-2	5	9	223	231	-4	3	10	61	-47
0	5	9	305	-282	-3	3	10	84	-88
1	5	9	108	113	-1	3	10	58	-53
3	5	9	78	-83	0	3	10	74	73
-5	6	9	59	47	1	3	10	57	67
-3	6	9	177	-180	4	3	10	47	36
-1	6	9	179	178	-5	4	10	59	-58
2	6	9	88	82	-3	4	10	117	112
-4	7	9	88	-85	1	4	10	75	-70
-3	7	9	82	93	2	4	10	116	120
-1	7	9	198	-208	-1	5	10	72	-71
2	7	9	86	94	0	5	10	210	-202
3	7	9	46	-53	2	5	10	65	-58
-3	8	9	91	94	-5	6	10	64	46
-1	8	9	203	-203	-3	6	10	187	-184
1	8	9	62	64	-1	6	10	168	-165
-3	9	9	50	-54	-4	7	10	67	71
-1	9	9	53	54	-2	7	10	49	39
1	9	9	86	87	-1	7	10	64	62
2	9	9	64	-77	1	7	10	53	-61
-2	10	9	65	72	2	7	10	64	53
-1	10	9	70	-60	3	7	10	48	-41
0	10	9	105	-99	-1	8	10	273	275
0	12	9	49	73	-3	9	10	55	50
-4	0	10	73	-64	0	9	10	47	33
0	0	10	176	177	1	9	10	55	47
2	0	10	647	-646	2	9	10	57	57
4	0	10	122	106	0	10	10	46	-38

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	11	10	84	-83	-4	7	11	70	-58	-2	5	12	78	81
-1	12	10	66	-65	-3	7	11	50	-53	-1	5	12	68	-56
0	12	10	132	140	-1	7	11	83	87	0	5	12	80	-76
-1	0	11	260	245	0	7	11	79	90	2	5	12	45	-43
1	0	11	159	-150	1	7	11	95	-100	-4	6	12	128	-137
3	0	11	127	126	2	7	11	121	138	-3	6	12	115	-123
-3	1	11	112	-108	-4	8	11	57	-34	-2	6	12	166	176
-2	1	11	68	-82	-3	8	11	61	47	0	6	12	87	-80
-1	1	11	510	-485	-2	8	11	49	-37	2	6	12	54	-53
0	1	11	79	78	-1	8	11	137	-135	-4	7	12	84	97
1	1	11	141	140	0	8	11	68	-53	-3	7	12	96	-106
2	1	11	84	88	1	9	11	63	-59	-1	7	12	55	45
3	1	11	52	47	2	9	11	43	14	1	7	12	49	49
-2	2	11	72	-84	-3	10	11	74	71	2	7	12	108	123
0	2	11	52	-49	-1	10	11	53	-41	-2	8	12	61	44
2	2	11	151	-151	-2	11	11	45	16	-1	8	12	55	63
-4	3	11	67	82	0	11	11	82	-72	0	8	12	77	-78
-1	3	11	143	-146	-4	0	12	87	-81	-1	9	12	51	-35
1	3	11	80	92	-2	0	12	87	116	0	11	12	134	-125
3	3	11	63	-58	0	0	12	153	-157	-1	0	13	240	227
-2	4	11	85	86	2	0	12	178	-184	1	0	13	556	-569
0	4	11	165	170	-4	1	12	125	-112	3	0	13	90	92
1	4	11	61	-63	-1	1	12	74	-64	-5	1	13	46	18
-4	5	11	90	99	1	1	12	146	155	-4	1	13	64	-39
-3	5	11	215	221	2	1	12	65	-69	-2	1	13	109	-119
-2	5	11	123	-133	3	1	12	59	-69	-1	1	13	147	-137
0	5	11	83	-71	1	2	12	205	214	1	1	13	85	100
1	5	11	67	84	3	2	12	69	-48	3	1	13	52	-58
2	5	11	64	-63	-2	3	12	60	64	-1	2	13	52	-44
-3	6	11	182	-193	-1	3	12	83	-78	0	2	13	76	-79
-2	6	11	98	-103	1	3	12	55	72	1	2	13	158	168
-1	6	11	367	377	2	4	12	51	55	-4	3	13	66	66
0	6	11	84	97	3	4	12	70	-76	-1	3	13	118	-93
1	6	11	105	-117	-5	5	12	54	35	0	3	13	116	113
2	6	11	70	-51	-4	5	12	49	-64	-4	4	13	62	-48
3	6	11	49	42	-3	5	12	103	108	-2	4	13	181	196

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
1	4	13	62	64	-1	3	14	72	-63	-2
-4	5	13	56	42	0	3	14	79	-78	0
-2	5	13	78	-71	-2	4	14	213	-226	2
-1	5	13	68	-56	0	4	14	50	47	-3
1	5	13	59	77	0	5	14	54	-47	-2
-2	6	13	223	-235	-4	6	14	65	-52	-1
-1	6	13	64	-75	-3	6	14	48	-10	0
0	6	13	102	113	-2	6	14	187	189	-2
2	6	13	89	-84	1	6	14	73	-76	-1
-4	7	13	67	-62	-4	7	14	63	-66	0
-1	7	13	115	101	-1	7	14	137	131	2
1	7	13	70	-51	1	7	14	71	77	-3
-4	8	13	86	89	-2	8	14	51	-24	-2
-3	8	13	56	34	-1	9	14	61	-53	0
-2	8	13	53	-54	0	9	14	57	57	-2
-1	8	13	46	-41	1	9	14	69	-66	1
-2	10	13	57	41	-2	10	14	58	-37	0
-4	0	14	92	97	0	10	14	45	-11	2
-2	0	14	109	105	-3	0	15	56	-54	-2
0	0	14	65	72	-1	0	15	59	-60	-1
2	0	14	47	41	1	0	15	161	-160	0
-4	1	14	92	-74	-2	1	15	93	-102	-3
-3	1	14	66	84	1	1	15	109	-120	-2
-2	1	14	94	-93	-5	2	15	60	27	0
2	1	14	126	127	-2	2	15	124	-136	2
-2	2	14	64	-59	-1	2	15	90	93	-3
-1	2	14	67	-58	0	2	15	142	134	-2
1	2	14	319	329	1	2	15	89	97	0
3	2	14	58	-63	-3	3	15	59	-64	-3
-2	3	14	91	85						

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	15		102	109	0	4	16	95	-100	1	3	18	88	-79
3	15		61	-64	1	5	16	46	45	-3	4	18	144	141
3	15		47	40	0	7	16	44	-15	-1	4	18	84	-80
4	15		101	-94	1	7	16	56	56	0	4	18	182	-175
4	15		137	133	-1	8	16	46	-34	1	4	18	69	35
4	15		83	83	-1	9	16	64	-74	-1	5	18	67	70
4	15		91	-98	-1	0	17	46	-28	-1	7	18	70	66
5	15		102	116	0	1	17	50	-33	-3	0	19	55	-47
5	15		63	59	2	1	17	63	-54	-1	0	19	54	43
5	15		114	-127	-4	2	17	52	20	-4	1	19	56	-46
5	15		46	31	-3	2	17	103	97	-1	1	19	65	58
6	15		55	-65	0	2	17	233	245	0	1	19	45	-40
6	15		86	-88	-3	3	17	56	66	-1	2	19	54	-36
6	15		49	56	0	3	17	75	72	1	2	19	55	44
8	15		86	-78	1	3	17	49	-18	-3	3	19	74	67
8	15		63	70	-3	4	17	139	-137	-2	3	19	46	67
0	16		75	74	-2	4	17	46	-13	-1	3	19	74	-62
0	16		81	-80	-1	4	17	88	85	-3	4	19	50	36
1	16		49	-41	0	4	17	144	-142	-1	4	19	109	-102
1	16		80	-93	-4	5	17	64	28	0	4	19	73	-66
1	16		48	25	-2	6	17	47	-25	-1	5	19	52	36
2	16		89	-97	1	6	17	60	-61	0	5	19	45	-36
2	16		73	-74	0	0	18	81	84	-3	1	20	51	22
2	16		162	163	0	1	18	54	-30	-3	3	20	50	24
2	16		93	-96	1	1	18	54	44	-2	3	20	51	26
3	16		84	87	-4	2	18	65	44	-1	3	20	60	61
3	16		82	77	-3	2	18	161	-161	-1	4	20	134	-117
3	16		117	-124	0	2	18	59	66	-1	0	21	62	69
4	16		124	142	0	3	18	52	66	-1	1	21	59	41

ANCISTROTECTORINE

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
1	0	0	347	320	0	4	0	142	134	6
2	0	0	973	970	1	4	0	100	98	7
3	0	0	421	427	2	4	0	248	245	8
4	0	0	311	316	3	4	0	103	101	9
6	0	0	193	185	4	4	0	176	173	10
7	0	0	213	213	5	4	0	98	99	-10
8	0	0	161	171	6	4	0	76	99	-9
9	0	0	22	26	7	4	0	91	105	-8
1	1	0	718	728	8	4	0	45	50	-7
2	1	0	703	703	2	5	0	126	137	-6
3	1	0	267	268	3	5	0	170	180	-5
4	1	0	193	200	4	5	0	78	91	-4
5	1	0	154	138	5	5	0	243	251	-3
6	1	0	192	197	6	5	0	158	156	-2
7	1	0	87	96	7	5	0	22	12	-1
8	1	0	52	46	0	6	0	60	66	0
9	1	0	55	42	1	6	0	99	100	1
10	1	0	77	39	2	6	0	43	40	2
0	2	0	337	333	3	6	0	83	77	3
1	2	0	342	347	4	6	0	44	33	4
2	2	0	378	371	-10	0	1	23	21	5
3	2	0	52	56	-9	0	1	150	138	6
4	2	0	212	202	-8	0	1	84	81	8
5	2	0	117	127	-7	0	1	81	79	9
7	2	0	86	88	-6	0	1	240	229	10
8	2	0	60	67	-5	0	1	72	74	-9
9	2	0	101	80	-4	0	1	42	63	-8
10	2	0	63	36	-3	0	1	243	238	-7
1	3	0	210	194	-2	0	1	128	127	-6
2	3	0	218	218	-1	0	1	454	446	-5
3	3	0	324	316	0	0	1	159	123	-4
4	3	0	104	105	1	0	1	434	435	-3
5	3	0	72	65	2	0	1	50	23	-2
6	3	0	128	112	3	0	1	239	246	-1
7	3	0	61	70	4	0	1	101	101	0
8	3	0	45	32	5	0	1	124	114	1

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	1	1	59	44	2	2	1	381	386	3	4	1	116	139
0	1	1	35	22	3	2	1	151	160	4	4	1	72	65
0	1	1	105	91	4	2	1	31	64	5	4	1	69	80
0	1	1	54	62	5	2	1	81	83	6	4	1	106	102
0	1	1	107	101	6	2	1	62	53	7	4	1	71	83
1	1	1	57	29	7	2	1	74	76	8	4	1	41	67
1	1	1	79	67	8	2	1	63	48	-6	5	1	155	167
1	1	1	183	182	9	2	1	34	32	-5	5	1	133	129
1	1	1	251	245	-9	3	1	47	46	-4	5	1	222	238
1	1	1	109	96	-8	3	1	113	113	-3	5	1	69	57
1	1	1	232	236	-7	3	1	60	36	-2	5	1	21	39
1	1	1	81	84	-6	3	1	49	45	-1	5	1	106	97
1	1	1	508	511	-5	3	1	73	83	0	5	1	66	64
1	1	1	732	742	-4	3	1	130	151	1	5	1	61	52
1	1	1	759	780	-3	3	1	409	406	2	5	1	102	106
1	1	1	178	165	-2	3	1	94	83	3	5	1	81	86
1	1	1	582	610	-1	3	1	164	153	4	5	1	112	100
1	1	1	471	467	0	3	1	133	138	5	5	1	83	95
1	1	1	482	483	1	3	1	216	208	6	5	1	65	53
1	1	1	86	69	2	3	1	107	94	-5	6	1	104	96
1	1	1	161	158	3	3	1	157	152	-4	6	1	63	68
1	1	1	95	82	4	3	1	53	59	-3	6	1	129	117
1	1	1	21	15	5	3	1	84	88	-2	6	1	109	85
1	1	1	48	62	6	3	1	21	57	-1	6	1	96	95
1	1	1	61	73	7	3	1	21	34	0	6	1	72	45
2	1	1	52	55	9	3	1	91	73	1	6	1	70	63
2	1	1	51	63	-8	4	1	109	106	3	6	1	53	43
2	1	1	90	96	-6	4	1	100	111	4	6	1	22	30
2	1	1	71	55	-5	4	1	125	114	-11	0	2	84	69
2	1	1	154	152	-4	4	1	223	214	-10	0	2	102	103
2	1	1	394	387	-3	4	1	91	75	-8	0	2	117	125
2	1	1	393	408	-2	4	1	248	244	-7	0	2	36	28
2	1	1	320	314	-1	4	1	31	29	-6	0	2	284	287
2	1	1	187	182	0	4	1	218	194	-5	0	2	127	113
2	1	1	146	144	1	4	1	109	103	-4	0	2	79	99
2	1	1	313	301	2	4	1	108	105	-3	0	2	372	373

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-2	0	2	87	91	-1	2	2	114	115	-2
-1	0	2	322	333	0	2	2	191	184	-1
0	0	2	133	134	1	2	2	274	279	0
1	0	2	729	712	2	2	2	179	174	1
2	0	2	117	111	3	2	2	376	385	2
3	0	2	327	322	4	2	2	167	183	4
4	0	2	155	163	5	2	2	142	128	5
5	0	2	42	30	6	2	2	48	53	6
6	0	2	43	36	7	2	2	21	13	7
8	0	2	114	122	8	2	2	67	84	-7
-10	1	2	33	50	9	2	2	22	25	-6
-9	1	2	48	40	-10	3	2	99	98	-5
-8	1	2	80	71	-9	3	2	90	121	-3
-7	1	2	87	80	-8	3	2	47	37	-1
-6	1	2	275	278	-7	3	2	90	91	1
-5	1	2	96	103	-6	3	2	173	175	2
-4	1	2	215	226	-5	3	2	309	313	3
-3	1	2	312	316	-4	3	2	180	187	4
-2	1	2	525	537	-3	3	2	146	129	5
-1	1	2	304	329	-2	3	2	166	146	6
0	1	2	144	146	-1	3	2	252	247	-5
1	1	2	658	662	0	3	2	107	107	-4
2	1	2	854	855	1	3	2	258	248	-3
3	1	2	372	361	2	3	2	91	93	-2
4	1	2	114	108	3	3	2	60	58	-1
5	1	2	183	196	4	3	2	118	109	0
6	1	2	184	203	5	3	2	62	60	1
7	1	2	68	68	7	3	2	114	125	3
9	1	2	77	60	8	3	2	96	89	-11
-10	2	2	44	48	-9	4	2	40	51	-10
-9	2	2	110	98	-8	4	2	57	70	-9
-7	2	2	90	86	-7	4	2	41	26	-8
-5	2	2	188	178	-6	4	2	91	87	-7
-4	2	2	244	265	-5	4	2	86	71	-6
-3	2	2	311	322	-4	4	2	130	136	-5
-2	2	2	320	323	-3	4	2	84	101	-4

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	2	106	112	-3	0	3	217	230	-6	2	3	138	124
4	2	227	244	-2	0	3	352	366	-5	2	3	246	234
4	2	96	110	-1	0	3	142	127	-4	2	3	206	210
4	2	85	84	0	0	3	152	147	-3	2	3	163	159
4	2	119	130	1	0	3	261	255	-2	2	3	188	184
4	2	91	96	2	0	3	115	119	-1	2	3	380	379
4	2	105	96	3	0	3	57	55	0	2	3	66	72
4	2	127	125	4	0	3	283	273	1	2	3	163	157
4	2	127	120	5	0	3	60	41	2	2	3	175	171
5	2	22	16	6	0	3	149	157	3	2	3	269	279
5	2	88	88	7	0	3	71	80	4	2	3	217	216
5	2	95	101	8	0	3	78	89	5	2	3	85	98
5	2	100	117	-11	1	3	46	54	6	2	3	71	79
5	2	76	76	-10	1	3	48	60	7	2	3	90	90
5	2	51	47	-9	1	3	44	50	8	2	3	59	46
5	2	20	17	-8	1	3	41	40	9	2	3	23	38
5	2	47	53	-7	1	3	191	188	-10	3	3	58	51
5	2	35	50	-6	1	3	84	58	-9	3	3	66	40
5	2	54	45	-5	1	3	195	189	-8	3	3	22	42
5	2	75	84	-4	1	3	56	54	-7	3	3	113	124
6	2	69	67	-3	1	3	478	475	-6	3	3	46	38
6	2	94	104	-2	1	3	470	462	-5	3	3	107	109
6	2	92	85	-1	1	3	56	60	-4	3	3	140	115
6	2	56	26	0	1	3	196	210	-3	3	3	118	111
6	2	114	115	1	1	3	551	558	-2	3	3	79	84
6	2	42	54	2	1	3	295	305	-1	3	3	226	212
6	2	65	54	3	1	3	249	257	0	3	3	148	145
6	2	23	37	4	1	3	119	121	1	3	3	199	196
0	3	72	30	5	1	3	42	57	3	3	3	32	32
0	3	60	39	6	1	3	125	138	4	3	3	143	160
0	3	22	15	7	1	3	21	33	6	3	3	86	97
0	3	69	57	8	1	3	121	123	7	3	3	61	76
0	3	104	101	9	1	3	156	153	8	3	3	82	90
0	3	90	92	-9	2	3	85	63	-8	4	3	64	58
0	3	161	151	-8	2	3	109	96	-7	4	3	90	97
0	3	93	97	-7	2	3	159	146	-6	4	3	51	55

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-5	4	3	77	72	-7	0	4	49	30	-7
-4	4	3	73	55	-6	0	4	42	40	-6
-3	4	3	84	75	-4	0	4	131	125	-5
-2	4	3	112	108	-3	0	4	129	139	-4
-1	4	3	183	190	-2	0	4	314	321	-3
0	4	3	106	106	-1	0	4	106	116	-2
1	4	3	62	54	0	0	4	410	401	-1
2	4	3	137	114	1	0	4	195	188	0
3	4	3	42	24	2	0	4	191	179	1
4	4	3	43	32	3	0	4	197	200	2
5	4	3	93	95	4	0	4	95	97	3
6	4	3	103	117	5	0	4	115	112	4
7	4	3	89	88	6	0	4	35	32	5
-7	5	3	22	15	8	0	4	159	173	6
-6	5	3	38	25	9	0	4	94	94	7
-5	5	3	43	47	-11	1	4	85	69	8
-4	5	3	44	57	-9	1	4	88	80	-10
-3	5	3	122	120	-8	1	4	114	103	-7
-2	5	3	44	24	-7	1	4	80	85	-6
-1	5	3	42	28	-6	1	4	71	65	-5
0	5	3	87	92	-5	1	4	112	112	-4
1	5	3	35	33	-4	1	4	423	436	-3
2	5	3	79	47	-3	1	4	84	75	-2
3	5	3	50	49	-2	1	4	158	167	-1
4	5	3	60	50	-1	1	4	279	251	0
5	5	3	79	101	0	1	4	248	229	1
-5	6	3	75	60	1	1	4	105	97	2
-4	6	3	103	99	2	1	4	159	164	4
-3	6	3	23	58	3	1	4	160	155	5
-2	6	3	178	176	4	1	4	216	206	6
-1	6	3	47	35	6	1	4	82	91	7
1	6	3	45	24	7	1	4	88	100	-9
2	6	3	63	62	8	1	4	88	107	-8
-11	0	4	88	51	-10	2	4	67	66	-6
-10	0	4	50	36	-9	2	4	53	31	-5
-9	0	4	58	50	-8	2	4	51	68	-4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	4		111	117	-3	4	4	150	156	1	0	5	184	173
2	4		213	211	-2	4	4	50	74	2	0	5	296	295
2	4		138	137	-1	4	4	45	36	3	0	5	290	288
2	4		178	184	0	4	4	202	199	4	0	5	95	96
2	4		92	99	1	4	4	65	57	5	0	5	51	50
2	4		36	51	2	4	4	70	84	6	0	5	21	2
2	4		65	66	3	4	4	87	84	7	0	5	59	37
2	4		79	70	5	4	4	91	99	8	0	5	109	105
2	4		192	182	6	4	4	49	64	-11	1	5	55	42
2	4		334	323	-7	5	4	22	27	-10	1	5	65	59
2	4		119	121	-6	5	4	55	40	-9	1	5	22	47
2	4		367	361	-5	5	4	57	43	-8	1	5	53	65
2	4		150	143	-4	5	4	82	80	-7	1	5	63	77
2	4		198	192	-3	5	4	65	78	-6	1	5	106	118
2	4		163	160	-2	5	4	166	160	-5	1	5	190	196
2	4		46	58	0	5	4	21	15	-4	1	5	168	158
3	4		84	78	1	5	4	97	91	-3	1	5	54	65
3	4		90	74	2	5	4	65	61	-2	1	5	69	79
3	4		87	73	3	5	4	22	45	-1	1	5	326	323
3	4		209	191	4	5	4	67	55	0	1	5	162	145
3	4		173	163	5	5	4	44	58	1	1	5	234	223
3	4		97	94	-4	6	4	42	41	2	1	5	138	129
3	4		173	167	-3	6	4	58	64	3	1	5	272	261
3	4		138	132	-2	6	4	58	53	4	1	5	230	225
3	4		120	115	-1	6	4	80	73	5	1	5	92	97
3	4		69	49	0	6	4	104	105	6	1	5	48	70
3	4		112	126	1	6	4	57	59	7	1	5	110	110
3	4		102	85	-11	0	5	51	49	8	1	5	91	74
3	4		142	158	-10	0	5	116	108	-9	2	5	39	38
3	4		54	55	-9	0	5	39	22	-8	2	5	22	44
3	4		89	107	-7	0	5	100	89	-7	2	5	144	143
4	4		81	76	-6	0	5	87	90	-6	2	5	121	106
4	4		64	53	-3	0	5	272	280	-5	2	5	107	92
4	4		65	52	-2	0	5	145	161	-4	2	5	175	181
4	4		116	120	-1	0	5	367	365	-3	2	5	71	63
4	4		43	29	0	0	5	393	398	-2	2	5	258	263

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-1	2	5	277	275	3	4	5	89	82	6
0	2	5	330	329	4	4	5	118	118	7
1	2	5	329	333	5	4	5	102	112	-11
2	2	5	150	157	6	4	5	54	57	-10
3	2	5	320	320	-7	5	5	67	72	-9
4	2	5	55	44	-5	5	5	92	99	-8
5	2	5	79	63	-4	5	5	44	46	-7
6	2	5	156	163	-3	5	5	131	123	-6
7	2	5	97	107	-2	5	5	57	66	-5
-10	3	5	50	33	-1	5	5	67	76	-4
-9	3	5	48	29	0	5	5	22	44	-3
-8	3	5	21	21	1	5	5	55	66	-2
-7	3	5	174	154	2	5	5	40	45	-1
-6	3	5	47	66	3	5	5	47	67	0
-5	3	5	96	83	4	5	5	125	121	1
-4	3	5	79	84	-4	6	5	120	121	2
-3	3	5	141	139	-3	6	5	77	64	3
-2	3	5	267	267	-2	6	5	88	68	4
-1	3	5	86	80	0	6	5	71	55	5
0	3	5	192	193	1	6	5	65	79	6
1	3	5	67	72	-11	0	6	117	126	7
2	3	5	96	103	-10	0	6	103	109	-9
3	3	5	50	62	-9	0	6	41	29	-8
4	3	5	95	109	-8	0	6	39	31	-7
5	3	5	309	310	-7	0	6	58	54	-6
6	3	5	236	244	-6	0	6	57	62	-5
7	3	5	97	113	-5	0	6	193	173	-4
-8	4	5	69	73	-4	0	6	80	66	-3
-6	4	5	96	87	-3	0	6	21	6	-2
-4	4	5	77	79	-2	0	6	380	394	-1
-3	4	5	43	44	-1	0	6	202	216	0
-2	4	5	127	114	0	0	6	106	117	1
-1	4	5	50	78	1	0	6	98	82	2
0	4	5	105	104	2	0	6	399	382	3
1	4	5	78	86	3	0	6	100	117	4
2	4	5	65	64	4	0	6	89	104	5

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	6	60	59	6	2	6	48	29	-4	5	6	127	140
0	6	75	76	7	2	6	35	15	-3	5	6	118	122
1	6	67	61	-10	3	6	48	45	-2	5	6	45	48
1	6	109	88	-9	3	6	69	46	-1	5	6	104	112
1	6	84	105	-8	3	6	103	92	0	5	6	88	92
1	6	55	49	-6	3	6	72	82	1	5	6	78	81
1	6	119	123	-5	3	6	72	75	2	5	6	157	146
1	6	86	89	-4	3	6	157	146	3	5	6	114	103
1	6	182	184	-3	3	6	186	177	-3	6	6	58	42
1	6	515	500	-2	3	6	121	108	-2	6	6	23	41
1	6	380	378	-1	3	6	203	203	-1	6	6	68	50
1	6	252	241	0	3	6	20	17	0	6	6	50	27
1	6	104	101	1	3	6	60	37	-11	0	7	340	356
1	6	233	229	2	3	6	21	42	-10	0	7	111	127
1	6	213	206	3	3	6	52	41	-9	0	7	223	198
1	6	194	195	4	3	6	21	25	-8	0	7	81	80
1	6	149	168	5	3	6	36	57	-7	0	7	50	50
1	6	84	71	6	3	6	55	69	-6	0	7	75	73
1	6	66	69	-9	4	6	87	96	-5	0	7	315	311
1	6	34	43	-8	4	6	95	101	-4	0	7	191	179
1	6	77	65	-7	4	6	116	124	-3	0	7	411	418
2	6	51	56	-6	4	6	123	127	-1	0	7	257	251
2	6	103	101	-5	4	6	108	113	0	0	7	206	196
2	6	121	94	-4	4	6	111	110	1	0	7	239	245
2	6	79	86	-3	4	6	178	177	2	0	7	45	16
2	6	62	56	-2	4	6	117	116	4	0	7	87	57
2	6	148	151	-1	4	6	149	136	5	0	7	88	67
2	6	189	187	0	4	6	217	213	6	0	7	100	87
2	6	208	203	1	4	6	51	39	7	0	7	48	30
2	6	164	152	2	4	6	113	106	-11	1	7	59	51
2	6	121	95	3	4	6	143	137	-10	1	7	114	113
2	6	165	155	4	4	6	71	65	-9	1	7	145	153
2	6	134	121	5	4	6	91	100	-8	1	7	122	125
2	6	129	146	-7	5	6	128	128	-7	1	7	91	107
2	6	118	116	-6	5	6	161	153	-6	1	7	172	164
2	6	53	57	-5	5	6	197	201	-5	1	7	95	90

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-4	1	7	237	222	-1	3	7	142	135	1
-3	1	7	183	187	1	3	7	110	109	3
-2	1	7	150	152	2	3	7	51	35	4
-1	1	7	57	43	3	3	7	92	87	5
0	1	7	80	93	4	3	7	85	88	-10
1	1	7	74	77	5	3	7	46	68	-9
2	1	7	166	161	-8	4	7	76	69	-8
4	1	7	162	170	-7	4	7	190	199	-7
5	1	7	79	84	-6	4	7	188	195	-6
6	1	7	68	57	-5	4	7	139	140	-5
7	1	7	75	66	-4	4	7	140	138	-4
-10	2	7	72	53	-3	4	7	53	68	-3
-9	2	7	96	98	-2	4	7	73	90	-2
-8	2	7	105	101	-1	4	7	142	141	-1
-7	2	7	128	136	1	4	7	45	49	0
-6	2	7	123	117	2	4	7	53	42	1
-5	2	7	21	29	3	4	7	49	60	2
-4	2	7	179	171	4	4	7	64	33	3
-3	2	7	122	133	-6	5	7	261	244	4
-2	2	7	138	133	-5	5	7	191	194	6
-1	2	7	166	151	-4	5	7	62	65	-10
0	2	7	132	119	-3	5	7	42	46	-9
1	2	7	86	75	-2	5	7	111	105	-7
2	2	7	93	94	0	5	7	59	48	-6
3	2	7	21	27	1	5	7	114	119	-5
4	2	7	73	81	2	5	7	33	25	-4
6	2	7	76	60	-11	0	8	119	128	-3
-10	3	7	35	29	-10	0	8	186	196	-2
-9	3	7	81	63	-8	0	8	75	67	-1
-8	3	7	173	180	-7	0	8	21	21	0
-7	3	7	132	117	-6	0	8	240	234	1
-6	3	7	106	97	-5	0	8	47	49	2
-5	3	7	58	70	-4	0	8	37	38	3
-4	3	7	82	96	-3	0	8	127	120	4
-3	3	7	73	86	-2	0	8	241	244	5
-2	3	7	64	41	-1	0	8	180	179	-9

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	8	44	55	-8	3	8	156	161	-3	0	9	83	72
0	8	60	56	-7	3	8	54	53	-2	0	9	116	104
0	8	106	108	-6	3	8	71	64	-1	0	9	63	22
0	8	93	97	-5	3	8	77	61	0	0	9	217	220
1	8	205	206	-3	3	8	118	119	1	0	9	112	105
1	8	106	108	-2	3	8	103	113	2	0	9	58	52
1	8	143	152	-1	3	8	130	140	4	0	9	69	43
1	8	153	143	0	3	8	83	87	5	0	9	60	57
1	8	109	123	1	3	8	80	64	-10	1	9	23	35
1	8	90	77	2	3	8	84	89	-9	1	9	170	171
1	8	45	62	3	3	8	89	83	-8	1	9	80	95
1	8	111	117	4	3	8	51	52	-6	1	9	89	94
1	8	223	221	5	3	8	42	24	-5	1	9	169	175
1	8	52	51	-8	4	8	45	57	-4	1	9	54	46
1	8	121	121	-7	4	8	46	51	-3	1	9	47	50
1	8	49	57	-6	4	8	77	73	-2	1	9	67	79
1	8	85	60	-5	4	8	113	103	-1	1	9	104	90
1	8	79	79	-4	4	8	102	115	0	1	9	176	181
1	8	106	102	-3	4	8	96	100	2	1	9	97	77
1	8	68	82	-2	4	8	89	93	3	1	9	56	49
2	8	105	110	-1	4	8	64	77	4	1	9	39	23
2	8	169	178	1	4	8	88	79	5	1	9	23	22
2	8	117	113	3	4	8	96	94	-10	2	9	80	80
2	8	100	98	-5	5	8	73	76	-9	2	9	100	111
2	8	58	35	-3	5	8	22	31	-8	2	9	60	68
2	8	94	85	-2	5	8	32	27	-7	2	9	167	162
2	8	66	43	-1	5	8	115	110	-6	2	9	107	107
2	8	66	91	0	5	8	74	53	-5	2	9	72	52
2	8	96	82	1	5	8	100	91	-4	2	9	44	26
2	8	58	43	-10	0	9	43	29	-3	2	9	99	103
2	8	57	48	-9	0	9	92	65	-2	2	9	126	142
2	8	99	122	-8	0	9	88	78	-1	2	9	74	75
2	8	57	59	-7	0	9	75	101	0	2	9	61	69
2	8	48	37	-6	0	9	71	51	1	2	9	116	107
2	8	85	60	-5	0	9	69	70	2	2	9	89	76
3	8	99	106	-4	0	9	73	78	3	2	9	118	127

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
4	2	9	37	44	3	0	10	73	44	1
-9	3	9	44	45	4	0	10	73	72	2
-8	3	9	32	39	-10	1	10	56	47	-7
-7	3	9	33	39	-9	1	10	92	94	-6
-6	3	9	22	35	-8	1	10	113	114	-5
-5	3	9	21	32	-7	1	10	74	57	-4
-4	3	9	138	154	-6	1	10	50	68	-3
-3	3	9	124	120	-5	1	10	86	69	-1
-2	3	9	151	149	-4	1	10	21	39	0
-1	3	9	94	85	-2	1	10	169	157	1
0	3	9	21	25	-1	1	10	101	96	-9
1	3	9	47	48	0	1	10	114	127	-8
2	3	9	43	35	1	1	10	56	38	-7
3	3	9	48	44	2	1	10	84	63	-6
-7	4	9	44	23	3	1	10	44	67	-5
-6	4	9	69	68	4	1	10	64	65	-4
-5	4	9	145	138	-9	2	10	104	101	-3
-4	4	9	152	163	-8	2	10	131	127	-2
-3	4	9	178	196	-7	2	10	165	157	-1
-2	4	9	73	62	-6	2	10	76	77	0
0	4	9	33	30	-5	2	10	60	40	1
1	4	9	57	61	-3	2	10	80	72	2
2	4	9	53	72	-2	2	10	89	94	3
-5	5	9	71	65	-1	2	10	71	72	-8
-4	5	9	63	41	0	2	10	62	64	-6
-2	5	9	41	40	1	2	10	89	53	-5
-1	5	9	52	39	2	2	10	22	39	-4
-9	0	10	57	44	-8	3	10	23	10	-2
-7	0	10	108	98	-7	3	10	100	106	-1
-6	0	10	57	69	-6	3	10	62	71	0
-5	0	10	90	103	-5	3	10	60	63	1
-4	0	10	42	59	-4	3	10	121	139	2
-1	0	10	96	111	-3	3	10	151	149	3
0	0	10	138	124	-2	3	10	150	147	-9
1	0	10	89	75	-1	3	10	96	94	-8
2	0	10	58	39	0	3	10	99	86	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	10		88	98	-6	2	11	111	107	-4	1	12	89	82
3	10		120	131	-5	2	11	105	95	-3	1	12	95	86
4	10		40	29	-4	2	11	120	111	-2	1	12	162	176
4	10		92	92	-3	2	11	49	44	-1	1	12	166	175
4	10		102	83	-2	2	11	114	128	0	1	12	131	126
4	10		80	83	-1	2	11	210	210	1	1	12	79	83
4	10		22	32	0	2	11	193	205	-8	2	12	105	114
4	10		49	49	1	2	11	235	227	-7	2	12	52	58
4	10		22	18	2	2	11	124	129	-5	2	12	22	33
4	10		94	68	-8	3	11	56	47	-4	2	12	156	158
0	11		59	41	-6	3	11	35	64	-2	2	12	97	90
0	11		100	76	-5	3	11	79	67	-1	2	12	198	209
0	11		31	17	-4	3	11	140	149	0	2	12	166	158
0	11		85	86	-3	3	11	148	144	1	2	12	80	71
0	11		21	15	-2	3	11	115	111	-6	3	12	216	216
0	11		21	43	-1	3	11	148	136	-5	3	12	160	168
0	11		129	135	0	3	11	130	132	-4	3	12	87	67
0	11		116	117	1	3	11	77	86	-3	3	12	139	132
0	11		130	109	-5	4	11	45	39	-2	3	12	71	58
0	11		132	138	-4	4	11	22	33	-1	3	12	23	39
0	11		52	43	-3	4	11	38	59	-8	0	13	59	40
0	11		114	69	-2	4	11	70	69	-7	0	13	67	59
0	11		97	99	-1	4	11	97	73	-6	0	13	36	25
1	11		87	73	-9	0	12	23	19	-5	0	13	116	124
1	11		47	28	-8	0	12	23	36	-4	0	13	22	19
1	11		56	44	-5	0	12	59	45	-3	0	13	181	186
1	11		84	61	-4	0	12	22	9	0	0	13	23	28
1	11		127	129	-3	0	12	234	256	-7	1	13	55	90
1	11		180	180	-2	0	12	221	225	-6	1	13	108	122
1	11		128	123	-1	0	12	52	44	-5	1	13	92	124
1	11		76	80	1	0	12	22	14	-4	1	13	114	133
1	11		22	15	2	0	12	56	41	-3	1	13	67	80
1	11		22	19	-8	1	12	133	112	-2	1	13	40	54
2	11		42	36	-7	1	12	55	58	-1	1	13	60	31
2	11		102	109	-6	1	12	22	23	0	1	13	86	76
2	11		105	111	-5	1	12	41	34	-6	2	13	67	49

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-5	2	13	23	44	-3	2	13	47	46	-5	0	14	23	28	-4	0	14	40	30	-3	0	14	35	30
-4	2	13	77	68	-2	2	13	55	54															

PERCHORORUBIAN

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	0	2793	2524	4	7	0	1175	-1127	11
4	0	0	997	903	6	7	0	339	344	0
6	0	0	262	-229	8	7	0	535	-526	1
8	0	0	776	-691	10	7	0	67	-84	2
10	0	0	326	317	0	8	0	795	764	3
2	1	0	178	-126	2	8	0	232	-222	4
4	1	0	1291	-1138	4	8	0	310	-301	5
6	1	0	353	333	6	8	0	272	-262	6
8	1	0	1168	-1058	8	8	0	106	112	7
10	1	0	262	279	10	8	0	208	196	8
0	2	0	2664	-2458	2	9	0	415	-415	9
2	2	0	1029	-913	4	9	0	308	-312	10
4	2	0	3060	-2749	6	9	0	97	-80	11
8	2	0	246	-235	8	9	0	234	-218	1
10	2	0	501	-443	10	9	0	104	-12	2
2	3	0	991	-884	8	10	0	119	-98	3
4	3	0	433	410	10	10	0	236	228	4
6	3	0	580	-546	2	11	0	71	74	5
8	3	0	72	45	4	11	0	344	341	6
10	3	0	145	134	6	11	0	209	-213	7
2	4	0	1290	1217	10	11	0	98	-107	11
4	4	0	414	393	0	12	0	876	-885	0
6	4	0	1421	1316	2	12	0	342	-337	1
8	4	0	1538	1442	4	12	0	118	-97	2
10	4	0	826	-789	8	12	0	115	108	3
2	5	0	597	-568	4	13	0	180	186	4
4	5	0	583	549	0	14	0	308	-303	5
6	5	0	827	-794	4	14	0	146	158	6
8	5	0	409	-391	2	15	0	83	84	7
10	5	0	440	-417	4	15	0	144	-136	8
0	6	0	459	435	3	1	1	430	-383	9
2	6	0	329	-326	4	1	1	817	769	10
4	6	0	290	-306	5	1	1	428	387	11
6	6	0	327	-297	6	1	1	1070	1001	1
8	6	0	369	-351	8	1	1	277	-246	2
2	7	0	189	180	9	1	1	252	-221	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	1	1	154	129	4	5	1	1053	995	7	9	1	73	-80
2	1	1	137	-83	5	5	1	76	-87	8	9	1	72	-107
2	1	1	255	-232	6	5	1	827	-795	9	9	1	97	60
2	1	1	489	-486	7	5	1	125	-171	10	9	1	183	-154
2	1	1	2312	2544	8	5	1	131	-123	0	10	1	437	-423
2	1	1	434	381	9	5	1	131	-137	1	10	1	118	-98
2	1	1	632	573	10	5	1	287	-275	2	10	1	163	157
2	1	1	99	77	11	5	1	228	-209	3	10	1	368	366
2	1	1	291	-299	0	6	1	233	-227	4	10	1	235	224
2	1	1	450	-425	1	6	1	843	796	5	10	1	136	-142
2	1	1	199	176	3	6	1	1284	-1225	7	10	1	209	-209
2	1	1	335	296	4	6	1	916	857	8	10	1	152	-153
2	1	1	396	-370	5	6	1	82	-83	9	10	1	95	78
3	1	1	577	524	6	6	1	313	-298	10	10	1	72	-92
3	1	1	97	-95	7	6	1	769	751	1	11	1	266	259
3	1	1	350	330	8	6	1	389	-390	2	11	1	204	-194
3	1	1	865	927	10	6	1	66	39	3	11	1	287	-280
3	1	1	244	228	1	7	1	200	-181	5	11	1	77	78
3	1	1	840	818	2	7	1	144	135	6	11	1	313	-300
3	1	1	102	92	4	7	1	229	-198	7	11	1	251	-237
3	1	1	285	-253	5	7	1	312	-283	8	11	1	198	199
4	1	1	125	124	6	7	1	759	742	9	11	1	77	79
4	1	1	229	-229	7	7	1	549	536	10	11	1	120	-84
4	1	1	1503	1355	8	7	1	99	-72	0	12	1	319	295
4	1	1	409	-350	10	7	1	115	142	1	12	1	230	245
4	1	1	826	776	0	8	1	222	-194	2	12	1	265	-270
4	1	1	939	-907	1	8	1	69	-60	3	12	1	145	147
4	1	1	136	-108	2	8	1	848	811	4	12	1	246	239
4	1	1	795	-752	3	8	1	257	-251	5	12	1	132	113
4	1	1	159	-110	4	8	1	384	372	6	12	1	87	93
4	1	1	131	-121	5	8	1	694	-663	7	12	1	77	-61
4	1	1	255	-222	10	8	1	78	-60	8	12	1	145	-118
4	1	1	189	-167	1	9	1	101	-95	10	12	1	175	168
5	1	1	145	107	3	9	1	391	-384	1	13	1	174	-178
5	1	1	597	-558	4	9	1	643	-624	3	13	1	120	118
5	1	1	199	196	5	9	1	270	267	4	13	1	203	-205

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
5	13	1	226	-220	3	2	2	199	-204	10
6	13	1	91	-127	4	2	2	571	582	11
7	13	1	163	188	6	2	2	121	115	0
8	13	1	176	-153	7	2	2	618	583	1
0	14	1	151	-139	8	2	2	185	192	2
3	14	1	171	-174	9	2	2	88	-91	3
5	14	1	153	-166	10	2	2	401	368	4
1	15	1	150	-146	11	2	2	450	431	6
2	15	1	64	-20	1	3	2	1761	1593	7
4	15	1	78	-37	2	3	2	226	225	8
5	15	1	84	-41	3	3	2	254	-228	9
0	16	1	103	-121	4	3	2	442	-476	10
1	16	1	68	36	5	3	2	333	342	1
0	0	2	551	-587	6	3	2	222	-211	2
1	0	2	152	-98	7	3	2	690	-637	3
2	0	2	364	327	8	3	2	72	-57	5
3	0	2	929	821	9	3	2	78	-63	7
4	0	2	458	-463	11	3	2	140	135	8
6	0	2	637	-630	0	4	2	3054	-2781	10
7	0	2	544	499	1	4	2	554	511	0
8	0	2	644	-619	2	4	2	96	-110	1
9	0	2	558	531	5	4	2	626	648	2
10	0	2	313	279	6	4	2	219	241	3
11	0	2	496	-470	7	4	2	128	94	4
1	1	2	1805	2622	8	4	2	121	-96	5
2	1	2	558	559	9	4	2	406	399	6
3	1	2	2036	-1943	10	4	2	238	-203	7
4	1	2	921	832	11	4	2	211	-178	8
5	1	2	781	735	1	5	2	204	192	9
7	1	2	1192	-1077	2	5	2	950	882	10
8	1	2	546	511	3	5	2	484	478	1
9	1	2	493	463	5	5	2	655	-652	2
10	1	2	145	131	6	5	2	214	206	3
0	2	2	208	-179	7	5	2	687	805	4
1	2	2	699	665	8	5	2	713	724	5
2	2	2	1223	-1171	9	5	2	522	-518	6

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	2	89	88	7	9	2	109	-86	6	14	2	77	72
5	2	214	-191	8	9	2	214	216	7	14	2	125	-133
6	2	238	217	10	9	2	241	239	1	15	2	80	-60
6	2	1097	-1008	0	10	2	686	682	4	15	2	93	-135
6	2	212	217	1	10	2	109	127	0	16	2	118	101
6	2	1083	1004	2	10	2	479	483	1	1	3	245	236
6	2	451	423	3	10	2	305	-312	2	1	3	1007	1049
6	2	191	178	5	10	2	85	-79	3	1	3	1534	-1436
6	2	173	-193	8	10	2	77	-44	4	1	3	1779	-1674
6	2	208	210	9	10	2	212	-200	5	1	3	1021	-949
6	2	202	234	10	10	2	196	-193	6	1	3	673	643
6	2	204	-221	1	11	2	515	517	7	1	3	636	584
7	2	414	388	2	11	2	70	29	9	1	3	78	55
7	2	569	550	3	11	2	84	-46	10	1	3	147	119
7	2	416	400	4	11	2	78	45	0	2	3	1453	1328
7	2	208	193	5	11	2	143	126	1	2	3	695	627
7	2	177	175	6	11	2	272	-264	2	2	3	783	762
7	2	365	360	8	11	2	160	152	3	2	3	1597	1820
7	2	115	-142	0	12	2	94	-101	4	2	3	462	-466
8	2	503	481	2	12	2	75	-50	5	2	3	929	849
8	2	356	327	3	12	2	97	106	6	2	3	318	313
8	2	474	-461	6	12	2	91	84	7	2	3	94	70
8	2	634	-611	7	12	2	185	-183	8	2	3	253	-229
8	2	264	-262	8	12	2	78	57	1	3	3	241	198
8	2	443	-414	10	12	2	74	-81	2	3	3	598	-564
8	2	122	-124	2	13	2	147	173	3	3	3	1607	-1598
8	2	91	-81	3	13	2	126	-117	4	3	3	530	568
8	2	294	-279	4	13	2	140	139	5	3	3	325	339
8	2	69	-67	5	13	2	72	-68	6	3	3	1233	-1218
8	2	250	282	6	13	2	79	-97	7	3	3	210	184
9	2	369	348	8	13	2	81	52	8	3	3	590	558
9	2	425	-419	9	13	2	111	-109	9	3	3	403	376
9	2	224	-225	0	14	2	152	166	11	3	3	207	181
9	2	494	-489	1	14	2	271	-275	0	4	3	1163	1019
9	2	173	-183	4	14	2	80	62	1	4	3	889	835
9	2	118	112	5	14	2	86	-98	2	4	3	1357	-1285

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	4	3	399	-368	2	8	3	129	111
4	4	3	551	-538	3	8	3	102	117
5	4	3	259	320	4	8	3	832	-821
7	4	3	221	246	5	8	3	355	339
8	4	3	240	-238	6	8	3	256	-254
9	4	3	312	-297	8	8	3	115	135
11	4	3	221	217	10	8	3	342	-357
1	5	3	106	-90	1	9	3	292	281
2	5	3	417	387	2	9	3	176	-182
3	5	3	470	-459	3	9	3	86	-77
4	5	3	971	936	4	9	3	470	-455
5	5	3	788	-776	5	9	3	223	-215
6	5	3	597	-619	6	9	3	141	151
7	5	3	370	432	7	9	3	91	-88
8	5	3	135	-112	9	9	3	66	20
9	5	3	247	-264	10	9	3	115	102
10	5	3	83	-41	0	10	3	663	643
11	5	3	66	-65	1	10	3	381	368
0	6	3	175	177	2	10	3	261	250
1	6	3	429	-410	3	10	3	192	202
3	6	3	966	909	4	10	3	93	95
5	6	3	346	354	5	10	3	267	265
6	6	3	472	481	6	10	3	216	218
7	6	3	241	246	7	10	3	175	155
8	6	3	520	592	9	10	3	97	-113
9	6	3	365	446	1	11	3	224	226
1	7	3	397	371	2	11	3	100	-105
2	7	3	210	202	3	11	3	504	489
4	7	3	186	-167	4	11	3	107	120
5	7	3	447	432	5	11	3	145	-142
6	7	3	589	576	6	11	3	100	-107
8	7	3	172	-178	9	11	3	148	-164
9	7	3	75	97	10	11	3	80	-85
10	7	3	123	150	0	12	3	186	168
0	8	3	467	-447	1	12	3	73	-85
1	8	3	73	101	2	12	3	520	-520

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	12	3	196	184	6	1	4	691	-638	8	4	4	175	-176
4	12	3	122	-133	7	1	4	115	112	9	4	4	427	-436
6	12	3	64	12	8	1	4	152	-134	10	4	4	281	270
7	12	3	75	60	9	1	4	330	318	11	4	4	153	154
8	12	3	97	-52	10	1	4	623	-571	1	5	4	914	-844
9	12	3	83	66	11	1	4	222	202	2	5	4	237	-224
10	12	3	123	125	0	2	4	637	599	3	5	4	693	636
3	13	3	72	43	1	2	4	655	-612	4	5	4	401	-394
4	13	3	324	314	2	2	4	1624	-1664	5	5	4	127	-151
5	13	3	206	203	3	2	4	819	-900	6	5	4	328	359
7	13	3	98	-97	4	2	4	1362	-1363	7	5	4	122	-155
8	13	3	84	-83	5	2	4	192	-166	8	5	4	306	-336
9	13	3	69	46	7	2	4	996	-928	9	5	4	200	-214
0	14	3	169	164	8	2	4	290	270	10	5	4	88	-80
2	14	3	135	161	9	2	4	266	-244	11	5	4	181	160
3	14	3	178	-169	10	2	4	94	-73	2	6	4	114	-109
5	14	3	140	-156	11	2	4	284	-280	3	6	4	217	-207
7	14	3	96	-100	1	3	4	481	-441	4	6	4	470	-459
3	15	3	85	97	2	3	4	576	549	5	6	4	140	159
0	0	4	964	-1069	3	3	4	1464	-1477	6	6	4	97	99
1	0	4	717	-717	4	3	4	213	233	7	6	4	133	-139
2	0	4	2075	1945	5	3	4	309	348	8	6	4	76	-42
3	0	4	874	-818	6	3	4	515	510	10	6	4	207	-228
4	0	4	457	-407	7	3	4	564	-543	1	7	4	1308	-1280
5	0	4	189	-148	8	3	4	144	127	2	7	4	242	-229
6	0	4	974	902	9	3	4	458	420	3	7	4	544	533
7	0	4	319	296	10	3	4	261	249	4	7	4	779	762
8	0	4	640	600	11	3	4	331	301	5	7	4	167	-150
9	0	4	801	-778	0	4	4	2261	2079	6	7	4	154	144
10	0	4	734	-675	1	4	4	711	-659	7	7	4	572	602
11	0	4	675	621	2	4	4	545	-515	8	7	4	258	255
1	1	4	804	-857	3	4	4	415	395	10	7	4	65	13
2	1	4	404	-409	4	4	4	246	244	0	8	4	453	-424
3	1	4	153	146	5	4	4	533	550	1	8	4	608	-576
4	1	4	148	-121	6	4	4	374	428	2	8	4	264	246
5	1	4	323	294	7	4	4	255	-271	3	8	4	138	138

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
5	8	4	95	-73	4	14	4	90	75	8
7	8	4	155	160	2	15	4	76	-74	9
8	8	4	221	226	5	15	4	140	130	10
10	8	4	64	-21	1	1	5	175	-184	11
1	9	4	657	-637	2	1	5	1382	-1459	1
2	9	4	198	-190	3	1	5	847	834	2
3	9	4	502	-486	4	1	5	1021	-978	3
5	9	4	375	371	5	1	5	572	537	4
6	9	4	135	-142	6	1	5	275	269	5
7	9	4	418	-401	7	1	5	376	-356	6
8	9	4	106	118	8	1	5	398	-372	7
9	9	4	389	382	9	1	5	172	162	8
0	10	4	98	57	10	1	5	87	-100	9
1	10	4	784	748	11	1	5	226	-216	10
2	10	4	160	132	0	2	5	748	-689	0
3	10	4	74	-63	1	2	5	2498	2434	1
4	10	4	110	93	3	2	5	1720	-1940	2
5	10	4	387	391	4	2	5	653	-687	3
6	10	4	193	-182	5	2	5	400	-396	4
7	10	4	415	415	6	2	5	383	-370	5
9	10	4	72	-63	7	2	5	513	497	6
1	11	4	166	172	9	2	5	616	-586	7
4	11	4	228	-225	11	2	5	461	420	8
6	11	4	221	211	2	3	5	839	-787	9
8	11	4	71	-49	3	3	5	1188	1237	10
9	11	4	88	-90	4	3	5	593	646	1
3	12	4	96	95	5	3	5	357	-401	2
7	12	4	168	144	6	3	5	481	-511	3
10	12	4	98	91	7	3	5	181	181	4
2	13	4	257	254	9	3	5	613	-570	5
3	13	4	86	58	11	3	5	235	220	6
5	13	4	74	-67	0	4	5	1244	1105	8
6	13	4	117	101	1	4	5	623	-573	9
1	14	4	131	-124	3	4	5	212	-199	10
2	14	4	68	54	4	4	5	469	487	0
3	14	4	175	164	5	4	5	996	-1101	1

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	5	204	-198	2	8	5	177	160	4	12	5	73	-52
4	5	294	301	4	8	5	305	303	5	12	5	218	-205
4	5	379	373	5	8	5	214	-228	6	12	5	83	49
4	5	253	-246	6	8	5	160	167	7	12	5	156	152
5	5	415	395	7	8	5	90	-111	2	13	5	86	96
5	5	241	-226	8	8	5	105	92	3	13	5	233	-234
5	5	404	-381	9	8	5	70	85	4	13	5	162	153
5	5	239	-257	1	9	5	219	-211	5	13	5	103	96
5	5	878	916	2	9	5	151	-148	8	13	5	112	118
5	5	114	117	3	9	5	93	77	1	14	5	65	35
5	5	451	-544	4	9	5	127	-145	3	14	5	199	195
5	5	442	-501	5	9	5	517	-524	2	15	5	136	128
5	5	111	-134	7	9	5	301	298	3	15	5	189	-188
5	5	201	-192	8	9	5	90	87	4	15	5	76	80
6	5	1873	-1709	9	9	5	89	45	5	15	5	63	60
6	5	77	-91	10	9	5	85	-111	0	0	6	203	-217
6	5	947	-883	0	10	5	290	299	1	0	6	409	411
6	5	486	471	1	10	5	235	240	2	0	6	320	-291
6	5	645	-617	2	10	5	226	-214	4	0	6	859	-786
6	5	586	-613	3	10	5	324	-334	5	0	6	72	44
6	5	112	-121	4	10	5	253	-253	6	0	6	732	-684
6	5	272	-312	5	10	5	372	-388	7	0	6	156	145
6	5	285	334	6	10	5	274	-277	8	0	6	89	96
6	5	197	237	8	10	5	185	-184	9	0	6	861	792
6	5	110	134	9	10	5	73	77	10	0	6	737	-694
7	5	386	-347	10	10	5	77	108	11	0	6	167	-156
7	5	95	100	2	11	5	244	247	1	1	6	476	-505
7	5	328	-309	3	11	5	72	-71	2	1	6	642	658
7	5	148	157	5	11	5	192	-183	3	1	6	179	-170
7	5	375	387	6	11	5	139	144	4	1	6	293	-296
7	5	192	209	7	11	5	89	88	6	1	6	672	631
7	5	200	212	8	11	5	109	78	7	1	6	217	200
7	5	148	180	9	11	5	89	-60	9	1	6	265	-236
7	5	96	124	10	11	5	97	85	10	1	6	125	109
8	5	314	-312	1	12	5	169	174	0	2	6	1136	1068
8	5	262	258	3	12	5	80	-29	1	2	6	108	-92

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	2	6	2417	2641	3	6	6	123	139
4	2	6	591	607	4	6	6	114	88
5	2	6	420	-406	5	6	6	94	89
6	2	6	519	504	6	6	6	342	353
7	2	6	294	293	7	6	6	96	116
8	2	6	157	139	8	6	6	214	-258
9	2	6	111	85	10	6	6	244	258
1	3	6	904	848	1	7	6	622	590
2	3	6	441	414	2	7	6	92	67
3	3	6	171	179	3	7	6	505	-487
4	3	6	656	-742	4	7	6	365	-371
5	3	6	439	-472	5	7	6	334	326
6	3	6	697	-703	6	7	6	425	-435
7	3	6	545	547	7	7	6	114	102
9	3	6	111	-85	8	7	6	81	-72
10	3	6	346	-334	9	7	6	63	-84
0	4	6	2585	2334	0	8	6	1111	-1053
1	4	6	771	730	2	8	6	223	223
2	4	6	1695	-1608	3	8	6	538	-546
3	4	6	737	691	4	8	6	428	-420
4	4	6	374	-365	5	8	6	235	-247
6	4	6	410	-486	7	8	6	207	-207
7	4	6	127	-148	8	8	6	322	349
8	4	6	378	-413	10	8	6	444	-487
10	4	6	160	153	1	9	6	213	209
1	5	6	826	768	2	9	6	310	-300
2	5	6	152	148	3	9	6	138	-149
3	5	6	684	-659	4	9	6	191	194
4	5	6	431	-430	5	9	6	201	-196
6	5	6	303	-338	6	9	6	623	-632
7	5	6	89	-62	7	9	6	361	378
8	5	6	378	-430	8	9	6	158	162
11	5	6	130	-143	9	9	6	165	-162
0	6	6	861	832	10	9	6	116	-140
1	6	6	117	119	0	10	6	118	-105
2	6	6	521	479	1	10	6	372	-351

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	10	6	322	-315	4	1	7	2291	2334	8	4	7	72	93
3	10	6	216	218	5	1	7	171	-151	9	4	7	240	-262
4	10	6	129	123	6	1	7	207	-182	10	4	7	602	-608
8	10	6	158	138	7	1	7	395	376	11	4	7	64	-30
9	10	6	96	105	8	1	7	586	546	1	5	7	653	598
10	10	6	100	119	10	1	7	88	-77	2	5	7	71	-56
1	11	6	124	117	11	1	7	336	-308	3	5	7	424	-448
2	11	6	419	424	0	2	7	1059	1012	4	5	7	126	144
3	11	6	142	-140	1	2	7	155	167	5	5	7	127	128
4	11	6	311	-315	2	2	7	539	-545	6	5	7	217	260
5	11	6	113	-88	3	2	7	1618	-1765	7	5	7	87	-101
6	11	6	115	125	4	2	7	255	287	8	5	7	159	195
8	11	6	195	-189	5	2	7	218	-201	9	5	7	196	-216
9	11	6	62	32	6	2	7	213	-195	10	5	7	73	69
1	12	6	64	-49	7	2	7	362	361	11	5	7	134	-131
2	12	6	286	-301	9	2	7	137	-117	0	6	7	682	672
3	12	6	73	-65	10	2	7	225	229	1	6	7	535	-509
4	12	6	148	-149	11	2	7	403	381	3	6	7	418	421
9	12	6	107	-127	1	3	7	340	314	4	6	7	442	441
10	12	6	110	125	2	3	7	747	763	5	6	7	491	513
1	13	6	135	121	3	3	7	750	-763	6	6	7	393	413
2	13	6	117	104	4	3	7	901	-1006	7	6	7	77	-107
3	13	6	109	-104	5	3	7	196	-197	8	6	7	323	-362
4	13	6	176	-150	6	3	7	1061	1111	10	6	7	126	-116
5	13	6	73	-70	7	3	7	744	763	1	7	7	468	451
6	13	6	100	124	8	3	7	640	-645	2	7	7	177	-175
8	13	6	78	-79	9	3	7	95	-82	3	7	7	169	-183
0	14	6	163	-156	10	3	7	308	293	4	7	7	186	175
1	14	6	96	91	0	4	7	170	-190	6	7	7	313	-322
2	14	6	77	-21	1	4	7	169	-165	8	7	7	101	-142
4	14	6	141	-116	2	4	7	1721	1625	9	7	7	143	174
2	15	6	71	-41	3	4	7	425	386	10	7	7	193	-234
4	15	6	160	177	4	4	7	109	-163	0	8	7	639	629
1	1	7	984	1030	5	4	7	75	96	1	8	7	160	-160
2	1	7	171	161	6	4	7	83	-102	2	8	7	244	-237
3	1	7	719	-727	7	4	7	144	-165	3	8	7	319	-309

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
4	8	7	413	422	6	13	7	71	35	1
5	8	7	68	-27	7	13	7	197	-203	2
6	8	7	281	302	4	14	7	73	27	3
7	8	7	222	-237	6	14	7	63	51	4
8	8	7	245	-251	1	15	7	91	-59	5
9	8	7	155	-168	0	0	8	1848	-1971	6
10	8	7	117	-56	1	0	8	1399	-1431	7
1	9	7	431	-430	2	0	8	728	721	8
2	9	7	173	-172	3	0	8	514	-509	9
3	9	7	179	198	5	0	8	1472	1423	10
4	9	7	242	254	6	0	8	854	836	11
5	9	7	175	-159	7	0	8	187	178	0
6	9	7	86	60	8	0	8	730	687	2
7	9	7	184	192	9	0	8	445	419	3
0	10	7	108	90	10	0	8	287	277	4
2	10	7	321	307	11	0	8	117	84	5
3	10	7	179	-185	1	1	8	187	221	6
5	10	7	142	149	2	1	8	230	-231	7
7	10	7	175	182	3	1	8	911	949	8
9	10	7	89	-94	4	1	8	270	275	9
10	10	7	76	-98	5	1	8	165	-153	10
1	11	7	68	51	6	1	8	515	-503	1
2	11	7	374	-378	7	1	8	790	743	2
3	11	7	170	174	8	1	8	421	414	3
4	11	7	225	-240	9	1	8	71	58	5
5	11	7	122	-122	0	2	8	920	-844	6
6	11	7	165	-177	1	2	8	1175	-1160	7
8	11	7	134	-138	2	2	8	2143	-2328	9
9	11	7	91	97	3	2	8	269	-298	10
0	12	7	131	-146	4	2	8	182	-197	0
2	12	7	96	83	5	2	8	675	680	1
3	12	7	77	-50	6	2	8	278	-287	2
4	12	7	83	-87	8	2	8	241	-243	3
5	12	7	106	-86	9	2	8	371	-343	4
2	13	7	129	-138	10	2	8	72	-58	5
4	13	7	361	-349	11	2	8	130	106	6

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	8	1	1203	-1119	8	6	8	142	177	1	12	8	159	172
3	8	1	187	-190	9	6	8	300	-363	2	12	8	111	108
3	8	1	966	1027	10	6	8	180	-200	5	12	8	187	-190
3	8	1	88	-87	1	7	8	248	232	6	12	8	100	-97
3	8	1	662	-748	3	7	8	245	-252	7	12	8	128	-133
3	8	1	696	727	4	7	8	630	649	8	12	8	119	-109
3	8	1	166	166	5	7	8	341	360	9	12	8	71	-66
3	8	1	258	241	6	7	8	272	286	3	13	8	100	-58
3	8	1	272	-252	7	7	8	654	-717	6	13	8	111	-114
3	8	1	190	181	8	7	8	456	498	7	13	8	84	-71
3	8	1	107	-112	10	7	8	75	75	8	13	8	68	64
4	8	1	301	272	0	8	8	246	245	0	14	8	332	310
4	8	1	1022	950	3	8	8	461	-452	1	14	8	317	292
4	8	1	331	320	4	8	8	280	277	2	14	8	141	127
4	8	1	164	167	6	8	8	105	88	3	14	8	144	-135
4	8	1	510	574	8	8	8	129	-156	1	15	8	79	75
4	8	1	327	366	10	8	8	140	170	1	1	9	755	-801
4	8	1	358	393	2	9	8	227	223	2	1	9	331	-338
4	8	1	101	-109	3	9	8	575	589	3	1	9	215	227
4	8	1	158	-155	4	9	8	328	-337	4	1	9	675	650
4	8	1	144	131	5	9	8	120	-108	5	1	9	77	-97
5	8	1	985	908	6	9	8	203	213	6	1	9	650	-643
5	8	1	543	511	7	9	8	241	236	10	1	9	192	-179
5	8	1	183	193	9	9	8	315	-331	11	1	9	276	261
5	8	1	451	481	0	10	8	220	210	0	2	9	119	107
5	8	1	78	99	1	10	8	597	-577	1	2	9	834	-848
5	8	1	420	-512	2	10	8	308	301	3	2	9	1383	1463
5	8	1	254	268	3	10	8	186	182	4	2	9	546	-579
5	8	1	105	-93	5	10	8	103	-123	5	2	9	131	-130
6	8	1	589	-552	8	10	8	62	38	6	2	9	696	-686
6	8	1	149	152	9	10	8	225	242	8	2	9	148	143
6	8	1	360	-350	10	10	8	167	-169	9	2	9	531	509
6	8	1	206	-192	1	11	8	137	-140	11	2	9	431	-391
6	8	1	227	-232	3	11	8	181	180	1	3	9	700	-660
6	8	1	220	220	8	11	8	180	163	2	3	9	751	-703
6	8	1	218	-218	0	12	8	95	108	3	3	9	891	-927

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	3	9	937	-1010	10	6	9	162	-181
5	3	9	114	124	1	7	9	195	-186
6	3	9	260	275	3	7	9	112	-110
7	3	9	665	-703	4	7	9	638	654
9	3	9	523	501	5	7	9	187	-192
10	3	9	255	247	6	7	9	262	-268
11	3	9	179	167	7	7	9	83	-49
0	4	9	459	428	8	7	9	242	261
1	4	9	1366	1284	9	7	9	123	-139
3	4	9	211	212	10	7	9	141	-159
4	4	9	151	-171	0	8	9	1260	1183
5	4	9	127	167	1	8	9	436	429
6	4	9	292	314	2	8	9	168	-176
7	4	9	197	225	3	8	9	361	352
8	4	9	356	390	5	8	9	128	-159
10	4	9	169	155	6	8	9	229	234
11	4	9	71	63	7	8	9	146	145
2	5	9	438	409	9	8	9	145	163
3	5	9	616	623	10	8	9	150	203
4	5	9	795	-851	1	9	9	444	450
5	5	9	391	-430	4	9	9	303	303
6	5	9	256	311	5	9	9	410	406
7	5	9	314	351	6	9	9	192	-177
8	5	9	182	-231	7	9	9	99	-105
10	5	9	75	61	9	9	9	123	-117
11	5	9	95	87	10	9	9	89	84
0	6	9	875	-808	0	10	9	134	-137
1	6	9	679	630	1	10	9	252	252
2	6	9	663	-626	2	10	9	290	-289
3	6	9	288	292	3	10	9	367	386
4	6	9	601	-596	4	10	9	323	-311
5	6	9	116	-118	7	10	9	102	113
6	6	9	134	138	10	10	9	100	-91
7	6	9	295	348	1	11	9	119	102
8	6	9	107	-109	2	11	9	69	102
9	6	9	229	254	6	11	9	355	360

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
10	11	9	101	108	1	2	10	129	126	9	5	10	124	144
2	12	9	409	402	2	2	10	208	258	10	5	10	66	-74
3	12	9	175	152	3	2	10	278	283	11	5	10	73	74
4	12	9	139	-146	4	2	10	667	-674	0	6	10	219	213
7	12	9	87	-77	5	2	10	434	-441	1	6	10	870	833
8	12	9	257	242	6	2	10	241	272	5	6	10	150	161
9	12	9	128	140	7	2	10	493	-489	6	6	10	123	145
1	13	9	145	163	8	2	10	206	201	7	6	10	73	-49
0	14	9	166	171	9	2	10	479	480	8	6	10	206	233
2	14	9	83	-84	11	2	10	182	-202	9	6	10	109	-156
3	14	9	96	-87	1	3	10	541	-504	2	7	10	259	257
6	14	9	86	-30	2	3	10	619	-622	3	7	10	410	-424
1	15	9	149	135	3	3	10	585	-582	4	7	10	256	-274
2	15	9	67	53	4	3	10	70	-83	5	7	10	272	-302
3	15	9	162	146	5	3	10	202	248	6	7	10	131	148
0	0	10	2725	2796	6	3	10	446	-471	7	7	10	362	379
1	0	10	216	227	7	3	10	474	506	8	7	10	274	-299
2	0	10	1347	-1375	8	3	10	309	-296	9	7	10	179	-222
3	0	10	561	-556	9	3	10	119	83	10	7	10	65	-95
5	0	10	503	-492	0	4	10	1242	-1130	0	8	10	552	530
6	0	10	73	47	1	4	10	388	-380	1	8	10	613	-587
7	0	10	258	-241	2	4	10	369	362	2	8	10	480	-488
8	0	10	500	-491	3	4	10	225	-219	3	8	10	410	410
9	0	10	682	-668	4	4	10	478	-530	4	8	10	100	-116
10	0	10	224	192	5	4	10	595	-654	5	8	10	265	312
11	0	10	79	93	6	4	10	79	86	6	8	10	177	-184
1	1	10	856	-918	7	4	10	303	-320	8	8	10	88	-100
2	1	10	109	-175	8	4	10	373	414	9	8	10	72	98
3	1	10	396	384	9	4	10	333	-342	10	8	10	168	182
5	1	10	853	-882	10	4	10	472	-491	1	9	10	239	-255
6	1	10	593	564	1	5	10	257	-272	3	9	10	381	-376
7	1	10	623	600	3	5	10	960	-993	4	9	10	124	-122
8	1	10	235	219	4	5	10	466	481	5	9	10	153	171
10	1	10	393	373	5	5	10	228	250	6	9	10	208	212
11	1	10	85	75	7	5	10	78	-58	7	9	10	71	70
0	2	10	1019	977	8	5	10	234	260	8	9	10	203	-213

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	10	10	85	90	5	1	11	720	716	10
2	10	10	118	118	6	1	11	364	343	11
3	10	10	141	-116	7	1	11	246	239	1
5	10	10	260	261	8	1	11	515	-490	2
6	10	10	129	148	9	1	11	400	-377	3
7	10	10	121	98	11	1	11	121	-140	5
9	10	10	107	94	0	2	11	205	-180	6
10	10	10	71	62	1	2	11	679	-707	7
1	11	10	220	-196	2	2	11	286	305	8
2	11	10	232	-235	3	2	11	831	855	9
3	11	10	162	-163	4	2	11	75	-60	11
4	11	10	247	256	5	2	11	78	92	1
7	11	10	355	359	7	2	11	463	-471	2
8	11	10	178	186	8	2	11	88	104	3
9	11	10	86	77	10	2	11	91	73	4
0	12	10	333	-349	11	2	11	145	-141	5
5	12	10	194	198	1	3	11	923	903	7
6	12	10	76	-26	2	3	11	675	-695	9
7	12	10	139	107	3	3	11	497	523	1
1	13	10	70	41	4	3	11	526	553	2
2	13	10	125	-109	5	3	11	121	-120	3
4	13	10	109	134	7	3	11	124	122	4
5	13	10	99	-112	8	3	11	72	-93	5
6	13	10	151	-145	9	3	11	139	-135	7
7	13	10	86	-69	10	3	11	252	-266	8
8	13	10	73	36	11	3	11	209	-200	9
0	14	10	63	-21	0	4	11	243	-225	0
4	14	10	128	138	1	4	11	1099	-1078	1
5	14	10	122	132	2	4	11	607	-600	2
6	14	10	62	-21	3	4	11	561	-555	3
1	15	10	245	242	4	4	11	82	-78	4
2	15	10	98	76	5	4	11	219	-235	5
1	1	11	137	360	6	4	11	207	-250	9
2	1	11	866	-891	7	4	11	432	-486	1
3	1	11	357	-388	8	4	11	137	-173	3
4	1	11	270	-260	9	4	11	101	-99	4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	11		203	188	6	9	11	148	162	8	0	12	563	-545
4	11		273	-292	7	9	11	222	-230	9	0	12	424	407
5	11		146	134	9	9	11	145	146	10	0	12	389	381
5	11		499	-491	10	9	11	109	-122	1	1	12	1327	1341
5	11		241	-238	1	10	11	328	-317	4	1	12	389	-395
5	11		165	-194	3	10	11	184	197	5	1	12	1191	1195
5	11		110	134	4	10	11	96	97	6	1	12	376	363
5	11		110	-134	7	10	11	186	-197	7	1	12	468	-459
5	11		114	-123	9	10	11	105	122	8	1	12	543	-532
5	11		108	131	10	10	11	77	75	9	1	12	283	245
5	11		239	-242	1	11	11	67	-58	0	2	12	1245	-1172
6	11		634	610	3	11	11	160	-166	1	2	12	348	-345
6	11		181	181	5	11	11	332	330	2	2	12	270	299
6	11		1106	-1135	6	11	11	171	157	3	2	12	339	-357
6	11		276	278	7	11	11	165	-170	4	2	12	462	-473
6	11		87	62	8	11	11	115	112	5	2	12	502	509
6	11		280	339	9	11	11	144	152	6	2	12	560	-579
6	11		235	-267	0	12	11	75	-16	7	2	12	370	375
7	11		302	-298	3	12	11	171	-168	8	2	12	409	-398
7	11		279	-287	5	12	11	125	101	9	2	12	465	-458
7	11		409	428	6	12	11	90	-70	11	2	12	122	158
7	11		127	-134	8	12	11	81	-65	1	3	12	196	187
7	11		125	-106	9	12	11	65	-38	2	3	12	160	170
7	11		448	495	1	13	11	277	-277	3	3	12	478	-482
7	11		160	-203	5	13	11	345	-348	4	3	12	129	145
7	11		77	-94	6	13	11	71	-53	5	3	12	176	-189
8	11		889	-847	7	13	11	130	135	6	3	12	343	-379
8	11		402	-395	2	14	11	84	-103	7	3	12	514	-545
8	11		244	-242	3	15	11	69	59	8	3	12	309	305
8	11		321	-351	0	0	12	3444	3563	10	3	12	94	100
8	11		164	-152	1	0	12	736	-713	0	4	12	391	-379
8	11		333	-370	2	0	12	434	390	1	4	12	109	105
8	11		252	-293	4	0	12	1579	1576	2	4	12	970	960
9	11		227	-223	5	0	12	569	540	4	4	12	593	619
9	11		418	-421	6	0	12	143	121	5	4	12	521	576
9	11		87	109	7	0	12	292	268	6	4	12	199	176

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
7	4	12	95	-97	3	8	12	150	-155
8	4	12	384	413	5	8	12	111	-99
9	4	12	153	174	7	8	12	68	64
10	4	12	246	-256	8	8	12	84	-92
11	4	12	66	50	1	9	12	180	182
1	5	12	398	397	2	9	12	192	178
2	5	12	311	-293	4	9	12	109	-126
3	5	12	708	726	6	9	12	232	-243
4	5	12	340	-346	7	9	12	80	-39
5	5	12	174	-196	0	10	12	74	41
6	5	12	597	-664	1	10	12	155	157
7	5	12	314	347	2	10	12	85	-43
8	5	12	69	80	3	10	12	176	-195
9	5	12	144	-166	5	10	12	239	-263
10	5	12	115	-133	7	10	12	89	-94
11	5	12	111	89	8	10	12	184	-178
0	6	12	373	-337	9	10	12	134	-136
1	6	12	871	-843	1	11	12	218	208
2	6	12	417	-407	3	11	12	80	-89
3	6	12	129	149	5	11	12	140	147
4	6	12	158	169	6	11	12	242	-243
5	6	12	156	-159	8	11	12	133	118
6	6	12	143	-127	9	11	12	77	71
7	6	12	106	-109	0	12	12	380	-363
8	6	12	143	-153	1	12	12	69	88
9	6	12	78	92	2	12	12	249	-248
10	6	12	148	164	4	12	12	106	-86
2	7	12	703	685	5	12	12	78	-87
3	7	12	208	225	6	12	12	162	173
4	7	12	720	-733	8	12	12	145	160
6	7	12	67	-84	1	13	12	96	-55
7	7	12	84	-101	3	13	12	229	-212
8	7	12	191	-202	5	13	12	149	-125
9	7	12	89	-82	6	13	12	72	-70
0	8	12	466	455	7	13	12	95	32
1	8	12	310	297	0	14	12	152	-151

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	14	12	78	-44	1	5	13	83	71	8	9	13	205	-210
3	14	12	152	146	2	5	13	371	-380	10	9	13	150	-184
5	14	12	114	-99	3	5	13	150	147	0	10	13	145	151
2	1	13	716	755	4	5	13	719	773	3	10	13	97	92
4	1	13	234	-232	5	5	13	228	-230	4	10	13	243	-255
5	1	13	106	-100	6	5	13	725	-786	6	10	13	102	105
6	1	13	630	628	7	5	13	80	73	7	10	13	172	-171
8	1	13	186	193	8	5	13	169	193	8	10	13	102	104
10	1	13	163	148	10	5	13	104	-130	9	10	13	79	-100
0	2	13	850	811	0	6	13	1113	1058	10	10	13	106	-100
1	2	13	79	50	1	6	13	117	130	2	11	13	231	-244
2	2	13	166	-218	4	6	13	90	-44	6	11	13	277	-286
3	2	13	496	474	5	6	13	132	-147	8	11	13	116	121
4	2	13	142	-143	7	6	13	88	86	9	11	13	107	-77
5	2	13	218	215	9	6	13	107	93	0	12	13	643	621
6	2	13	327	350	10	6	13	191	-205	2	12	13	284	-276
8	2	13	353	-355	1	7	13	211	205	3	12	13	87	61
9	2	13	211	183	2	7	13	341	347	4	12	13	129	107
11	2	13	148	-147	3	7	13	149	-136	5	12	13	108	108
2	3	13	777	787	4	7	13	190	-198	6	12	13	132	115
3	3	13	148	-178	6	7	13	453	483	7	12	13	65	31
4	3	13	692	721	8	7	13	158	-181	8	12	13	107	-123
6	3	13	141	-159	10	7	13	293	322	2	13	13	133	-152
8	3	13	209	209	0	8	13	135	109	4	13	13	119	-121
9	3	13	129	126	1	8	13	114	124	5	13	13	79	-8
10	3	13	141	126	2	8	13	579	564	7	13	13	89	54
0	4	13	736	681	3	8	13	107	112	0	14	13	77	-75
1	4	13	443	427	6	8	13	275	291	3	14	13	169	-182
2	4	13	271	284	8	8	13	116	109	4	14	13	122	-98
3	4	13	179	195	9	8	13	103	114	5	14	13	118	-122
5	4	13	76	-63	10	8	13	369	-422	0	0	14	1568	-1650
6	4	13	313	346	1	9	13	162	148	1	0	14	468	-430
7	4	13	66	90	2	9	13	101	-120	2	0	14	453	419
8	4	13	75	41	4	9	13	425	-439	3	0	14	284	-251
9	4	13	90	-53	5	9	13	168	168	4	0	14	1418	-1400
10	4	13	234	-247	6	9	13	112	105	5	0	14	595	-575

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
7	0	14	125	114	6	4	14	71	67
8	0	14	119	124	7	4	14	126	-149
10	0	14	472	-429	9	4	14	90	-102
11	0	14	83	96	10	4	14	136	142
3	1	14	1007	-1076	11	4	14	78	-27
4	1	14	341	375	1	5	14	328	-317
5	1	14	206	221	2	5	14	295	321
6	1	14	471	-463	4	5	14	372	384
7	1	14	628	-620	5	5	14	457	-516
8	1	14	439	440	6	5	14	679	721
9	1	14	103	88	7	5	14	310	320
0	2	14	509	509	9	5	14	372	-425
1	2	14	135	155	10	5	14	70	102
2	2	14	962	-989	11	5	14	66	-36
4	2	14	212	240	0	6	14	576	552
5	2	14	524	555	1	6	14	747	-723
6	2	14	502	548	2	6	14	398	397
7	2	14	157	158	3	6	14	73	83
8	2	14	393	402	5	6	14	112	138
9	2	14	101	-110	7	6	14	72	-52
10	2	14	88	58	8	6	14	149	146
11	2	14	125	77	9	6	14	163	209
1	3	14	937	961	10	6	14	172	-189
2	3	14	357	-352	2	7	14	527	-521
3	3	14	218	-229	3	7	14	335	357
5	3	14	509	544	4	7	14	547	561
6	3	14	552	383	5	7	14	117	143
7	3	14	160	-172	6	7	14	112	109
8	3	14	118	-119	7	7	14	239	261
9	3	14	143	154	8	7	14	129	154
11	3	14	172	155	0	8	14	176	-192
0	4	14	297	-272	1	8	14	69	-15
2	4	14	712	-723	3	8	14	138	-152
3	4	14	248	240	8	8	14	84	-88
4	4	14	418	-450	9	8	14	86	-108
5	4	14	422	430	10	8	14	137	134

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	9	14	119	-117	9	1	15	81	92	7	5	15	107	-138
3	9	14	257	-262	10	1	15	71	-57	8	5	15	277	-293
5	9	14	97	96	1	2	15	582	573	9	5	15	132	-137
6	9	14	237	236	2	2	15	315	333	10	5	15	63	32
8	9	14	83	57	3	2	15	169	-196	0	6	15	1020	-951
0	10	14	92	97	4	2	15	234	-243	1	6	15	530	-513
1	10	14	250	233	5	2	15	516	536	2	6	15	102	-95
2	10	14	296	304	6	2	15	245	-244	3	6	15	735	750
3	10	14	204	-192	7	2	15	627	645	4	6	15	81	-57
4	10	14	110	121	8	2	15	98	-104	5	6	15	127	162
5	10	14	128	151	11	2	15	88	89	7	6	15	87	103
7	10	14	139	156	2	3	15	943	-978	8	6	15	261	278
8	10	14	95	80	4	3	15	631	662	9	6	15	255	286
9	10	14	154	-183	5	3	15	209	247	10	6	15	99	94
10	10	14	198	-207	6	3	15	284	-334	1	7	15	291	303
1	11	14	332	336	7	3	15	111	84	2	7	15	73	-69
4	11	14	75	93	9	3	15	81	89	5	7	15	248	275
5	11	14	66	6	10	3	15	161	-162	6	7	15	238	248
6	11	14	107	90	11	3	15	123	119	7	7	15	142	-155
7	11	14	96	-101	0	4	15	100	104	9	7	15	294	323
0	12	14	137	129	1	4	15	272	268	10	7	15	77	-75
1	12	14	132	-134	2	4	15	633	-649	0	8	15	355	-339
2	12	14	196	175	3	4	15	278	-286	1	8	15	198	189
4	12	14	188	178	4	4	15	73	96	3	8	15	193	188
6	12	14	86	-105	5	4	15	184	-209	4	8	15	92	-118
5	13	14	61	-32	7	4	15	66	-79	5	8	15	179	184
6	13	14	160	154	8	4	15	121	-129	6	8	15	224	-228
7	13	14	86	-15	9	4	15	125	-142	7	8	15	95	-121
0	14	14	158	141	10	4	15	339	357	9	8	15	97	106
1	14	14	139	-134	11	4	15	127	120	1	9	15	215	-217
1	1	15	316	-495	1	5	15	537	-540	2	9	15	94	79
2	1	15	876	-912	2	5	15	146	-142	3	9	15	143	162
4	1	15	667	-716	3	5	15	293	-306	5	9	15	85	-133
5	1	15	283	-303	4	5	15	116	116	6	9	15	118	143
6	1	15	381	401	5	5	15	147	-159	7	9	15	85	-99
8	1	15	440	-415	6	5	15	139	167	8	9	15	175	177

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
9	9	15	150	-173	10	0	16	419	-407	3
0	10	15	217	211	11	0	16	410	391	4
1	10	15	288	273	3	1	16	531	557	5
4	10	15	64	88	4	1	16	320	-317	6
5	10	15	135	125	5	1	16	312	-317	7
7	10	15	204	210	6	1	16	140	-145	8
8	10	15	147	-149	7	1	16	275	278	9
9	10	15	88	-66	10	1	16	248	-245	1
10	10	15	105	110	11	1	16	194	183	2
1	11	15	104	92	0	2	16	945	-921	4
2	11	15	147	143	1	2	16	194	-217	7
3	11	15	362	367	5	2	16	745	-789	8
5	11	15	124	-146	6	2	16	202	-220	9
7	11	15	118	126	7	2	16	742	-774	1
8	11	15	100	94	9	2	16	148	-138	2
9	11	15	96	-107	10	2	16	98	101	3
2	12	15	75	-68	11	2	16	310	-307	4
3	12	15	116	134	1	3	16	552	-565	5
7	12	15	104	116	2	3	16	276	298	6
8	12	15	73	-92	3	3	16	668	-689	7
2	13	15	162	190	4	3	16	266	-280	8
3	13	15	89	-92	5	3	16	127	-122	9
4	13	15	227	224	8	3	16	209	225	10
5	13	15	168	169	11	3	16	96	77	0
0	14	15	88	100	0	4	16	1281	1281	1
2	14	15	76	72	1	4	16	219	-229	2
4	14	15	108	79	2	4	16	431	-443	3
0	0	16	397	-423	3	4	16	178	180	8
1	0	16	183	183	4	4	16	374	380	10
2	0	16	1207	1194	5	4	16	85	112	1
3	0	16	269	-246	6	4	16	213	-186	3
4	0	16	525	521	8	4	16	223	-219	4
5	0	16	312	314	10	4	16	422	434	5
6	0	16	159	-150	11	4	16	152	126	6
7	0	16	335	333	1	5	16	175	-198	7
8	0	16	119	118	2	5	16	306	300	8

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	16		156	-150	9	9	16	126	136	4	2	17	280	-292
5	16		330	-379	0	10	16	93	-73	5	2	17	192	-215
5	16		270	281	1	10	16	167	159	6	2	17	310	-335
5	16		136	-166	2	10	16	185	-186	7	2	17	271	301
5	16		70	82	4	10	16	94	89	8	2	17	111	-123
5	16		227	-241	5	10	16	110	117	9	2	17	332	-334
5	16		171	171	6	10	16	74	84	10	2	17	83	-77
6	16		485	467	7	10	16	180	200	11	2	17	346	330
6	16		146	153	9	10	16	142	154	1	3	17	133	-144
6	16		180	-184	2	11	16	282	297	2	3	17	113	112
6	16		136	-154	3	11	16	186	-198	4	3	17	247	-274
6	16		264	-307	4	11	16	147	-156	5	3	17	416	-428
6	16		127	-143	5	11	16	62	-43	6	3	17	233	258
7	16		179	-163	6	11	16	185	197	7	3	17	335	353
7	16		166	164	7	11	16	222	219	9	3	17	297	-306
7	16		207	-220	8	11	16	105	-80	10	3	17	114	134
7	16		97	121	9	11	16	62	-46	0	4	17	434	427
7	16		241	-259	2	12	16	144	-146	1	4	17	788	-774
7	16		274	-287	3	12	16	168	194	2	4	17	394	421
7	16		154	189	4	12	16	118	-98	3	4	17	224	-228
7	16		114	113	1	13	16	62	72	5	4	17	383	-402
7	16		144	-131	2	13	16	109	94	6	4	17	313	329
7	16		66	35	3	13	16	70	11	7	4	17	121	-134
8	16		792	-760	6	13	16	101	122	8	4	17	173	199
8	16		229	-215.	0	14	16	70	76	9	4	17	104	96
8	16		80	69	3	14	16	100	79	10	4	17	185	-186
8	16		67	70	4	1	17	277	298	11	4	17	146	-154
8	16		253	285	5	1	17	285	300	1	5	17	950	920
8	16		184	-210	6	1	17	442	-444	3	5	17	76	-113
9	16		227	-233	8	1	17	113	92	4	5	17	315	-330
9	16		377	-391	9	1	17	71	-24	5	5	17	499	541
9	16		127	117	10	1	17	71	-61	7	5	17	227	-226
9	16		153	161	11	1	17	297	-285	8	5	17	215	-253
9	16		403	-427	0	2	17	392	384	0	6	17	479	-465
9	16		93	39	1	2	17	614	629	1	6	17	136	-135
9	16		84	125	3	2	17	1127	-1182	2	6	17	604	-606

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
3	6	17	165	177	5	11	17	123	-113	9
4	6	17	339	-355	6	11	17	97	94	11
5	6	17	86	73	7	11	17	82	-85	1
6	6	17	234	258	8	11	17	122	-117	2
7	6	17	360	-382	0	12	17	168	-151	3
9	6	17	87	-122	2	12	17	68	74	4
10	6	17	104	-109	3	12	17	128	-119	5
1	7	17	413	-403	4	12	17	135	-158	6
2	7	17	83	-101	5	12	17	67	63	7
3	7	17	385	-390	6	12	17	83	48	9
4	7	17	195	209	7	12	17	168	157	10
5	7	17	104	-98	1	14	17	81	-66	11
10	7	17	127	-115	0	0	18	398	-396	0
0	8	17	719	703	1	0	18	689	-660	3
1	8	17	185	-168	2	0	18	269	-250	4
3	8	17	179	-179	3	0	18	381	-390	5
6	8	17	192	198	4	0	18	174	173	6
7	8	17	167	-169	5	0	18	453	463	7
9	8	17	155	-158	6	0	18	174	166	8
1	9	17	114	-107	7	0	18	130	-134	9
2	9	17	204	-206	9	0	18	401	407	11
3	9	17	229	224	10	0	18	476	-458	1
4	9	17	123	109	11	0	18	86	-48	2
5	9	17	155	-153	2	1	18	82	106	3
6	9	17	165	-169	4	1	18	237	262	6
7	9	17	340	358	5	1	18	187	-202	7
9	9	17	143	153	6	1	18	597	604	8
1	10	17	197	-180	7	1	18	137	148	0
2	10	17	227	-215	9	1	18	180	-154	1
3	10	17	364	-370	0	2	18	386	381	2
4	10	17	141	-161	1	2	18	381	-380	3
5	10	17	93	-119	2	2	18	396	411	4
8	10	17	82	-46	5	2	18	490	502	5
1	11	17	203	-200	6	2	18	159	170	7
2	11	17	197	-188	7	2	18	298	307	9
3	11	17	141	-165	8	2	18	151	158	10

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	18		137	-170	1	7	18	560	548	4	12	18	93	-80
2	18		180	186	4	7	18	179	180	5	12	18	128	-129
3	18		125	129	5	7	18	361	381	7	12	18	145	-152
3	18		186	-173	7	7	18	305	-343	5	13	18	76	9
3	18		904	936	8	7	18	216	-225	0	14	18	124	-105
3	18		221	-234	9	7	18	87	95	1	1	19	371	-408
3	18		133	-176	0	8	18	590	-569	3	1	19	153	234
3	18		171	-177	3	8	18	554	-551	4	1	19	1153	1200
3	18		397	391	5	8	18	188	-200	8	1	19	141	139
3	18		141	-137	7	8	18	167	-169	10	1	19	101	-114
3	18		91	-110	8	8	18	116	114	11	1	19	66	45
3	18		94	-60	9	8	18	110	-131	0	2	19	264	270
4	18		629	616	10	8	18	90	-102	1	2	19	220	-218
4	18		164	197	2	9	18	213	-225	3	2	19	348	361
4	18		282	-296	3	9	18	206	192	4	2	19	168	187
4	18		368	375	5	9	18	233	-256	6	2	19	241	-249
4	18		198	-215	6	9	18	118	-122	7	2	19	119	142
4	18		145	150	7	9	18	184	199	9	2	19	274	271
4	18		248	-269	8	9	18	104	-124	10	2	19	83	73
4	18		148	-159	9	9	18	150	-160	11	2	19	115	-88
4	18		161	-157	10	9	18	78	-95	1	3	19	209	-206
5	18		387	400	1	10	18	430	-409	2	3	19	100	133
5	18		550	-553	2	10	18	67	79	4	3	19	530	-558
5	18		160	-183	3	10	18	243	253	6	3	19	653	659
5	18		70	-15	4	10	18	244	243	7	3	19	276	-286
5	18		418	-430	6	10	18	103	-77	8	3	19	283	-280
5	18		86	-92	9	10	18	143	142	9	3	19	236	261
6	18		291	282	2	11	18	88	-83	10	3	19	300	293
6	18		120	-124	3	11	18	160	175	11	3	19	233	224
6	18		206	217	4	11	18	171	-172	0	4	19	309	-295
6	18		174	-188	6	11	18	95	115	1	4	19	179	170
6	18		178	213	7	11	18	73	-61	2	4	19	696	709
6	18		185	178	0	12	18	85	-93	3	4	19	196	-208
6	18		152	148	1	12	18	68	87	5	4	19	160	-168
6	18		83	-85	2	12	18	92	-133	8	4	19	64	77
6	18		82	72	3	12	18	91	-79	10	4	19	243	-268

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
1	5	19	81	100	6	9	19	120	-115	0
2	5	19	197	-215	7	9	19	69	-46	2
3	5	19	299	294	8	9	19	97	-108	5
4	5	19	258	-277	2	10	19	228	231	7
5	5	19	280	-303	5	10	19	126	-130	10
6	5	19	150	163	6	10	19	72	-37	11
7	5	19	93	-105	7	10	19	67	109	1
8	5	19	124	136	9	10	19	136	159	2
10	5	19	86	-76	1	11	19	107	110	3
0	6	19	149	154	2	11	19	75	-44	5
1	6	19	367	371	3	11	19	99	88	7
2	6	19	86	-63	4	11	19	168	-173	9
3	6	19	522	551	5	11	19	123	129	10
4	6	19	107	99	6	11	19	149	-156	11
5	6	19	236	265	8	11	19	73	-26	0
8	6	19	118	-120	0	12	19	139	-135	1
9	6	19	147	174	4	12	19	75	-31	2
10	6	19	91	86	5	12	19	64	-69	3
2	7	19	160	167	3	13	19	100	-94	4
3	7	19	197	197	4	13	19	207	-208	7
4	7	19	328	347	0	0	20	547	-529	8
5	7	19	155	143	1	0	20	172	188	1
6	7	19	77	-111	2	0	20	208	-215	2
7	7	19	78	-44	3	0	20	292	290	4
8	7	19	133	133	4	0	20	223	227	5
0	8	19	614	586	6	0	20	101	113	8
1	8	19	219	215	7	0	20	110	117	9
3	8	19	119	123	9	0	20	246	253	10
4	8	19	289	305	10	0	20	301	272	0
5	8	19	84	-79	11	0	20	252	239	1
6	8	19	68	88	3	1	20	776	890	3
8	8	19	257	-261	5	1	20	433	448	4
9	8	19	113	-144	7	1	20	681	692	6
2	9	19	87	87	8	1	20	374	349	7
3	9	19	81	64	10	1	20	120	95	8
4	9	19	117	93	11	1	20	98	-95	10

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	20		359	-338	1	7	20	153	129	2	13	20	158	-151
2	20		780	-779	2	7	20	237	245	3	13	20	108	-115
2	20		224	-215	3	7	20	407	-407	2	1	21	98	-155
2	20		316	-321	4	7	20	413	413	4	1	21	128	-183
2	20		129	112	5	7	20	75	-79	6	1	21	121	-134
2	20		172	-186	6	7	20	240	235	7	1	21	107	132
3	20		851	-866	7	7	20	69	-41	8	1	21	292	-307
3	20		183	-198	8	7	20	155	151	9	1	21	194	-205
3	20		377	-472	0	8	20	280	258	0	2	21	576	-568
3	20		341	-343	1	8	20	165	-151	1	2	21	862	-841
3	20		82	106	2	8	20	331	-326	3	2	21	309	302
3	20		86	-108	4	8	20	220	235	4	2	21	185	-183
3	20		144	141	6	8	20	160	186	6	2	21	71	79
3	20		172	-174	7	8	20	76	-87	7	2	21	183	-176
4	20		520	-517	10	8	20	174	193	8	2	21	186	204
4	20		297	288	2	9	20	133	131	9	2	21	185	194
4	20		556	562	3	9	20	96	-101	10	2	21	93	-88
4	20		352	360	4	9	20	270	-274	11	2	21	245	-242
4	20		93	-113	6	9	20	191	196	1	3	21	423	-428
4	20		175	157	7	9	20	194	211	2	3	21	367	-358
4	20		66	53	8	9	20	129	-136	3	3	21	593	-598
5	20		657	646	9	9	20	111	-106	6	3	21	119	-129
5	20		255	286	0	10	20	149	168	8	3	21	147	134
5	20		159	190	2	10	20	117	137	9	3	21	313	315
5	20		267	279	3	10	20	204	214	11	3	21	73	-61
5	20		220	221	4	10	20	95	-102	0	4	21	358	361
5	20		80	44	5	10	20	152	186	2	4	21	223	-227
5	20		74	-48	6	10	20	93	98	5	4	21	376	384
6	20		89	-56	1	11	20	67	53	8	4	21	82	-92
6	20		191	195	2	11	20	130	-137	10	4	21	111	111
6	20		124	-148	3	11	20	113	94	11	4	21	140	153
6	20		143	-152	4	11	20	217	214	1	5	21	297	302
6	20		209	-189	7	11	20	130	153	2	5	21	257	-267
6	20		124	136	0	12	20	199	193	3	5	21	344	356
6	20		114	98	2	12	20	179	196	4	5	21	396	-409
6	20		183	-196	1	13	20	78	59	5	5	21	139	-149

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
6	5	21	208	218	0	10	21	83	69	2
7	5	21	180	174	1	10	21	203	-200	5
9	5	21	63	-24	4	10	21	81	-106	6
10	5	21	76	-48	5	10	21	128	107	8
0	6	21	278	-260	6	10	21	97	-96	9
1	6	21	109	114	2	11	21	117	118	10
2	6	21	252	-272	3	11	21	61	-67	0
3	6	21	269	-296	4	11	21	79	83	1
4	6	21	381	-394	6	11	21	231	235	2
5	6	21	183	181	7	11	21	97	-77	3
7	6	21	122	122	0	12	21	142	-135	5
8	6	21	166	177	1	12	21	66	-21	6
9	6	21	337	-338	3	12	21	105	77	7
1	7	21	242	-238	5	12	21	172	179	8
2	7	21	109	-116	2	13	21	118	-101	9
3	7	21	81	-96	0	0	22	1171	1123	10
4	7	21	212	229	1	0	22	100	-106	1
5	7	21	368	-392	2	0	22	595	-595	2
6	7	21	70	-34	3	0	22	168	-141	3
8	7	21	97	69	4	0	22	120	111	5
10	7	21	66	76	5	0	22	174	164	6
1	8	21	100	-122	6	0	22	129	-130	7
2	8	21	145	-132	7	0	22	234	-227	8
4	8	21	68	-54	8	0	22	287	-289	0
5	8	21	144	144	9	0	22	562	-539	4
6	8	21	97	80	10	0	22	355	338	5
7	8	21	120	128	1	1	22	528	-542	6
8	8	21	62	59	6	1	22	402	407	7
9	8	21	85	63	8	1	22	73	-41	10
10	8	21	105	82	9	1	22	93	70	2
1	9	21	73	61	10	1	22	147	164	3
4	9	21	234	260	0	2	22	140	-117	4
5	9	21	357	371	1	2	22	204	-207	5
6	9	21	92	76	2	2	22	538	535	6
7	9	21	81	-74	8	2	22	68	-91	7
8	9	21	135	-157	11	2	22	89	85	8

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	22		355	-356	1	8	22	145	-151	2	3	23	216	-231
3	22		406	401	2	8	22	154	-142	3	3	23	440	477
3	22		256	-259	3	8	22	132	135	6	3	23	192	-221
3	22		70	-50	4	8	22	110	103	7	3	23	185	186
3	22		124	132	7	8	22	71	-62	8	3	23	241	223
3	22		67	-60	1	9	22	207	-224	10	3	23	79	-12
4	22		365	-362	3	9	22	70	-75	0	4	23	145	148
4	22		351	-366	7	9	22	210	-206	1	4	23	115	-123
4	22		315	307	8	9	22	103	-81	3	4	23	110	135
4	22		494	-508	9	9	22	71	78	4	4	23	69	97
4	22		91	-95	0	10	22	146	-143	5	4	23	208	-197
4	22		211	-215	1	10	22	261	260	7	4	23	144	-143
4	22		226	-224	2	10	22	128	-107	11	4	23	106	-86
4	22		154	153	1	11	22	165	-155	1	5	23	105	-113
4	22		180	-163	3	11	22	60	-53	2	5	23	86	-61
4	22		201	-222	4	11	22	142	136	4	5	23	288	297
5	22		299	-294	5	11	22	135	136	6	5	23	273	-273
5	22		165	-181	6	11	22	84	-53	7	5	23	270	-274
5	22		103	-65	0	12	22	157	-170	8	5	23	120	125
5	22		164	-165	1	12	22	93	95	0	6	23	238	224
5	22		339	-353	3	12	22	73	20	1	6	23	326	316
5	22		174	-168	4	12	22	64	39	3	6	23	232	-239
5	22		119	-95	2	1	23	455	439	4	6	23	68	76
6	22		186	-194	3	1	23	185	246	5	6	23	72	-102
6	22		93	108	5	1	23	134	144	6	6	23	87	-1
6	22		67	96	6	1	23	222	226	7	6	23	76	77
6	22		76	-64	7	1	23	328	-332	8	6	23	85	-25
6	22		110	102	8	1	23	202	-206	9	6	23	136	-138
6	22		190	184	9	1	23	93	-100	1	7	23	195	200
7	22		224	222	0	2	23	61	-78	2	7	23	98	87
7	22		163	159	3	2	23	148	-172	3	7	23	211	197
7	22		267	-280	5	2	23	178	-198	4	7	23	207	-229
7	22		243	-258	6	2	23	215	215	6	7	23	112	122
7	22		115	132	7	2	23	237	-229	7	7	23	93	105
7	22		156	138	8	2	23	139	-139	8	7	23	172	-163
7	22		128	-137	1	3	23	79	-50	0	8	23	73	-43

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	8	23	242	249	7	1	24	127	-134	3
3	8	23	130	-136	9	1	24	262	257	6
4	8	23	218	-230	1	2	24	245	228	7
5	8	23	317	-328	3	2	24	95	-114	1
7	8	23	68	-59	9	2	24	180	-190	2
8	8	23	67	71	11	2	24	89	78	4
1	9	23	263	-272	1	3	24	101	94	8
2	9	23	84	81	3	3	24	123	-236	3
3	9	23	263	-263	4	3	24	70	89	6
4	9	23	167	-189	7	3	24	246	-259	1
5	9	23	65	-3	9	3	24	191	-186	2
6	9	23	96	-93	0	4	24	73	59	3
7	9	23	93	-51	5	4	24	247	241	4
1	10	23	113	-120	6	4	24	92	63	5
2	10	23	66	-85	7	4	24	255	234	0
5	10	23	109	-113	9	4	24	306	305	6
6	10	23	100	109	1	5	24	391	388	7
7	10	23	185	-166	2	5	24	210	-219	1
1	11	23	77	74	3	5	24	280	288	3
4	11	23	74	46	5	5	24	141	-145	5
5	11	23	172	153	7	5	24	394	385	6
0	12	23	80	92	9	5	24	178	-168	9
1	12	23	76	75	10	5	24	83	27	1
2	12	23	237	-228	0	6	24	94	70	2
1	0	24	111	112	1	6	24	419	-423	3
2	0	24	107	97	2	6	24	67	-98	4
3	0	24	587	587	3	6	24	161	158	10
4	0	24	66	-68	5	6	24	79	-61	11
5	0	24	181	190	7	6	24	166	-170	0
6	0	24	81	67	1	7	24	74	57	1
7	0	24	272	270	2	7	24	137	-137	2
9	0	24	288	274	4	7	24	114	-113	5
1	1	24	627	601	5	7	24	78	-88	7
4	1	24	183	195	0	8	24	182	188	10
5	1	24	588	575	1	8	24	153	166	2
6	1	24	122	151	2	8	24	144	159	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
8	24		139	-138	4	5	25	389	390	1	0	26	80	95
8	24		85	115	5	5	25	74	79	2	0	26	323	299
8	24		63	-15	7	5	25	206	206	4	0	26	93	-70
9	24		340	337	8	5	25	118	116	5	0	26	67	-47
9	24		87	-116	0	6	25	102	106	8	0	26	168	151
9	24		70	76	1	6	25	337	-324	9	0	26	221	-221
9	24		89	-95	2	6	25	129	139	10	0	26	141	-140
10	24		115	-116	3	6	25	212	215	11	0	26	68	45
10	24		115	-119	6	6	25	158	-152	1	1	26	500	-494
11	24		89	67	7	6	25	122	-129	3	1	26	366	-367
11	24		87	-88	1	7	25	68	-90	4	1	26	169	197
11	24		87	-78	2	7	25	76	-39	5	1	26	187	-288
11	24		122	105	3	7	25	133	-124	6	1	26	144	-178
11	24		110	77	4	7	25	141	-132	7	1	26	211	-209
12	24		105	96	5	7	25	80	79	8	1	26	178	161
1	25		321	316	6	7	25	136	131	9	1	26	141	-140
1	25		75	46	7	7	25	247	-238	10	1	26	143	-141
2	25		204	201	8	7	25	116	-122	2	2	26	210	-207
2	25		68	76	2	8	25	217	207	7	2	26	63	-79
2	25		226	225	4	8	25	65	23	8	2	26	77	77
2	25		146	169	5	8	25	321	314	10	2	26	165	153
2	25		92	-95	6	8	25	59	-63	1	3	26	372	380
3	25		178	-204	8	8	25	84	65	2	3	26	236	238
3	25		182	-205	1	9	25	339	325	3	3	26	163	223
3	25		122	-243	3	9	25	261	249	5	3	26	291	299
3	25		172	167	6	9	25	99	101	6	3	26	126	102
3	25		62	-5	7	9	25	61	-98	8	3	26	136	-138
3	25		84	91	0	10	25	89	-57	1	4	26	93	-116
4	25		158	168	2	10	25	72	53	2	4	26	271	-270
4	25		105	100	3	10	25	102	-86	4	4	26	193	188
4	25		98	104	4	10	25	62	70	5	4	26	87	-92
4	25		237	248	1	11	25	111	-112	6	4	26	150	-134
4	25		230	254	2	11	25	81	-13	8	4	26	280	-260
4	25		64	-41	3	11	25	156	187	9	4	26	107	-56
5	25		115	-133	4	11	25	81	55	10	4	26	207	203
5	25		139	159	0	0	26	1392	-1287	1	5	26	497	-495

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	5	26	159	148	4	3	27	106	80	2
3	5	26	118	-150	6	3	27	216	-205	3
5	5	26	139	-163	0	4	27	107	120	4
6	5	26	266	253	2	4	27	145	-142	5
8	5	26	131	114	5	4	27	118	-112	7
0	6	26	202	-182	6	4	27	80	40	8
2	6	26	100	67	7	4	27	108	-94	9
5	6	26	115	120	9	4	27	63	-60	10
6	6	26	100	101	1	5	27	159	-157	3
7	6	26	69	85	2	5	27	125	123	7
1	7	26	115	-86	3	5	27	185	-187	8
2	7	26	125	-123	4	5	27	169	-145	1
3	7	26	151	152	5	5	27	183	186	2
4	7	26	228	244	6	5	27	226	221	5
6	7	26	94	-72	8	5	27	70	-56	9
8	7	26	195	189	0	6	27	209	-202	1
0	8	26	236	-219	1	6	27	141	-156	3
3	8	26	79	76	2	6	27	98	-92	5
4	8	26	72	45	3	6	27	207	197	7
2	9	26	104	118	4	6	27	298	-274	8
4	9	26	66	72	8	6	27	134	132	9
5	9	26	74	52	2	7	27	63	-49	0
1	10	26	186	176	6	7	27	148	-147	2
2	11	26	99	103	8	7	27	68	81	3
4	1	27	175	-293	1	8	27	79	-57	5
6	1	27	204	-214	2	8	27	192	-173	8
7	1	27	90	-120	3	8	27	92	-79	1
0	2	27	163	-166	6	8	27	117	84	2
1	2	27	301	298	7	8	27	105	-93	5
3	2	27	228	-237	4	9	27	83	89	7
4	2	27	104	-92	5	9	27	149	-174	8
7	2	27	205	209	0	10	27	226	201	2
8	2	27	87	110	1	10	27	65	71	3
9	2	27	97	-72	2	10	27	127	-119	1
10	2	27	73	-77	4	10	27	117	-115	2
2	3	27	77	59	0	0	28	183	235	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	28		130	106	4	7	28	100	-102	5	6	29	107	81
0	28		156	-140	0	8	28	311	-299	7	6	29	85	-91
0	28		67	-8	3	8	28	71	-4	1	7	29	265	-232
0	28		172	172	4	8	28	92	-63	3	7	29	123	-130
0	28		82	44	5	8	28	81	76	5	7	29	178	-179
0	28		91	77	1	9	28	61	-40	1	8	29	76	-7
0	28		172	144	2	9	28	165	-161	3	8	29	84	-48
0	28		137	-134	4	9	28	77	100	4	8	29	117	95
1	28		385	382	1	10	28	77	-88	1	9	29	83	48
1	28		290	273	2	10	28	64	-64	3	9	29	253	240
1	28		77	-77	1	1	29	117	110	1	0	30	139	-117
2	28		145	-136	2	1	29	77	-97	4	0	30	149	111
2	28		240	250	4	1	29	382	356	5	0	30	73	-62
2	28		221	-211	5	1	29	70	62	6	0	30	84	-115
2	28		123	-110	8	1	29	61	70	7	0	30	97	-101
3	28		239	-202	9	1	29	61	65	9	0	30	77	79
3	28		139	-157	0	2	29	174	169	5	1	30	131	-157
3	28		297	-295	3	2	29	201	-209	7	1	30	76	-29
3	28		78	78	4	2	29	94	85	9	1	30	67	-51
3	28		75	-92	6	2	29	114	-105	0	2	30	203	-188
3	28		103	-95	8	2	29	68	-45	4	2	30	107	123
4	28		414	375	4	3	29	194	-190	5	2	30	193	181
4	28		158	-159	6	3	29	266	245	7	2	30	141	140
4	28		101	88	8	3	29	127	-145	1	3	30	80	50
4	28		80	68	9	3	29	157	-146	2	3	30	68	40
4	28		100	-83	0	4	29	246	-232	3	3	30	343	362
5	28		195	190	1	4	29	170	-143	7	3	30	74	80
5	28		161	-172	2	4	29	241	206	0	4	30	236	-207
5	28		154	186	3	4	29	102	72	1	4	30	131	122
5	28		73	-108	5	4	29	127	-116	2	4	30	123	135
5	28		147	-131	7	4	29	171	-143	3	4	30	75	44
6	28		67	48	8	4	29	62	55	2	5	30	96	77
6	28		258	-244	1	5	29	193	189	3	5	30	103	-118
7	28		66	-105	3	5	29	247	233	4	5	30	110	133
7	28		241	-223	5	5	29	328	308	0	6	30	70	74
7	28		221	-229	4	6	29	107	134	1	6	30	109	71

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	6	30	81	95	5	4	31	68	82	7
3	6	30	70	81	1	5	31	75	-91	0
4	6	30	123	119	4	5	31	146	-122	7
6	6	30	92	-84	5	5	31	187	-184	1
7	6	30	97	68	6	5	31	94	-54	3
1	7	30	270	247	3	6	31	65	47	7
4	7	30	63	-57	5	6	31	150	134	0
5	7	30	150	132	3	7	31	92	77	3
0	8	30	67	66	4	7	31	179	152	4
3	8	30	113	-116	0	8	31	293	247	6
1	9	30	63	37	0	0	32	191	-180	1
1	2	31	237	-235	2	0	32	382	-339	5
3	2	31	303	287	3	0	32	69	13	0
6	2	31	62	-43	5	0	32	64	65	2
5	3	31	84	101	7	0	32	76	81	1
6	3	31	206	182	1	1	32	224	220	2
1	4	31	116	100	3	1	32	310	290	0
2	4	31	99	100	4	1	32	81	130	0

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	32		126	137	3	2	33	70	87	0	2	34	89	-104
2	32		213	185	1	3	33	157	-160	1	3	34	212	201
2	32		156	-148	2	3	33	110	-108	5	3	34	179	141
3	32		382	-342	6	3	33	232	-187	4	4	34	71	86
3	32		292	-270	2	4	33	190	-169	2	5	34	112	-111
3	32		109	-109	5	4	33	73	53	1	6	34	87	-63
4	32		323	-296	1	5	33	76	48	1	2	35	70	76
4	32		66	-62	3	5	33	131	121	2	2	35	97	-108
4	32		83	-43	0	6	33	118	-109	4	3	35	113	90
4	32		120	-105	1	6	33	112	95	0	4	35	131	144
5	32		63	62	3	6	33	147	-124	1	4	35	68	-46
5	32		121	107	1	7	33	199	-164	2	4	35	125	122
6	32		153	-129	0	0	34	426	377	1	5	35	73	10
6	32		78	-21	1	0	34	75	34	0	0	36	246	-214
7	32		118	-98	5	0	34	96	-71	3	0	36	172	162
7	32		60	47	1	1	34	191	-221	1	1	36	112	153
8	32		66	62	3	1	34	228	-214	2	1	36	133	110
2	33		68	-96	6	1	34	126	107	2	3	36	65	12

ZOAPATANOLIDE B

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	0	0	121	148	7	4	0	100	95
2	0	0	1112	1052	1	5	0	326	339
3	0	0	267	274	2	5	0	210	215
4	0	0	136	133	3	5	0	75	76
5	0	0	26	13	4	5	0	128	125
6	0	0	192	191	5	5	0	42	39
7	0	0	73	75	6	5	0	42	42
1	1	0	483	462	7	5	0	99	104
2	1	0	579	551	0	6	0	189	184
3	1	0	139	130	1	6	0	228	228
4	1	0	236	222	2	6	0	93	90
5	1	0	87	91	3	6	0	253	260
6	1	0	72	74	4	6	0	123	119
7	1	0	92	93	5	6	0	212	210
0	2	0	1363	1341	6	6	0	139	136
1	2	0	253	263	7	6	0	120	121
2	2	0	278	275	1	7	0	200	189
3	2	0	88	91	2	7	0	352	356
4	2	0	205	205	3	7	0	61	65
5	2	0	228	230	4	7	0	134	138
6	2	0	135	137	5	7	0	85	87
7	2	0	90	90	6	7	0	76	74
1	3	0	308	327	7	7	0	63	60
2	3	0	570	580	0	8	0	140	138
3	3	0	313	321	1	8	0	174	170
4	3	0	322	319	2	8	0	283	286
5	3	0	111	116	3	8	0	150	157
6	3	0	172	173	4	8	0	231	228
7	3	0	99	93	5	8	0	41	31
0	4	0	736	722	6	8	0	121	129
1	4	0	401	416	1	9	0	330	320
2	4	0	664	654	2	9	0	98	99
3	4	0	152	157	3	9	0	87	85
4	4	0	124	122	4	9	0	20	7
5	4	0	125	131	5	9	0	61	70
6	4	0	112	115	6	9	0	52	50

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	10	0	257	248	-4	0	1	20	6	0	2	1	233	249
1	10	0	81	84	-3	0	1	1012	996	1	2	1	220	213
2	10	0	99	97	-2	0	1	152	176	2	2	1	484	472
3	10	0	61	62	-1	0	1	1675	1593	3	2	1	113	112
4	10	0	94	102	0	0	1	36	24	4	2	1	229	228
5	10	0	184	186	1	0	1	760	728	5	2	1	77	76
6	10	0	126	121	2	0	1	696	622	6	2	1	20	20
1	11	0	45	38	3	0	1	481	455	7	2	1	196	194
2	11	0	219	217	4	0	1	26	20	-8	3	1	54	54
3	11	0	29	20	5	0	1	34	25	-7	3	1	54	54
4	11	0	134	126	6	0	1	8	3	-6	3	1	63	63
5	11	0	105	109	7	0	1	334	333	-5	3	1	31	31
0	12	0	240	242	-8	1	1	70	66	-4	3	1	120	113
1	12	0	115	109	-7	1	1	130	126	-3	3	1	47	41
2	12	0	102	106	-6	1	1	72	77	-2	3	1	60	74
3	12	0	114	111	-5	1	1	27	29	-1	3	1	822	807
4	12	0	21	29	-4	1	1	133	127	0	3	1	606	657
5	12	0	130	130	-3	1	1	204	203	1	3	1	523	531
1	13	0	78	85	-2	1	1	196	216	2	3	1	386	383
2	13	0	123	117	-1	1	1	341	331	3	3	1	591	563
3	13	0	94	90	0	1	1	132	154	4	3	1	195	200
4	13	0	97	100	1	1	1	436	402	5	3	1	150	148
0	14	0	125	127	2	1	1	205	210	6	3	1	66	76
1	14	0	46	43	3	1	1	50	47	7	3	1	150	154
2	14	0	78	74	4	1	1	25	30	-8	4	1	31	36
3	14	0	61	58	5	1	1	48	41	-7	4	1	155	157
1	15	0	35	37	6	1	1	117	115	-6	4	1	124	123
2	15	0	83	87	7	1	1	180	177	-5	4	1	176	177
3	15	0	27	16	-8	2	1	81	80	-4	4	1	325	331
0	16	0	108	106	-7	2	1	92	93	-3	4	1	435	444
1	16	0	14	27	-6	2	1	8	9	-2	4	1	169	169
2	16	0	24	32	-5	2	1	216	212	-1	4	1	245	265
-8	0	1	30	22	-4	2	1	247	238	0	4	1	376	384
-7	0	1	16	19	-3	2	1	275	284	1	4	1	429	423
-6	0	1	65	68	-2	2	1	266	272	2	4	1	199	196
-5	0	1	125	122	-1	2	1	92	78	3	4	1	527	531

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	4	1	221	220	-4	7	1	66	66
5	4	1	151	155	-3	7	1	178	185
6	4	1	89	98	-2	7	1	55	60
7	4	1	95	97	-1	7	1	237	246
-7	5	1	88	81	0	7	1	277	298
-6	5	1	230	233	1	7	1	360	361
-5	5	1	74	63	2	7	1	183	183
-4	5	1	218	221	3	7	1	249	248
-3	5	1	265	272	4	7	1	83	80
-2	5	1	334	343	5	7	1	68	69
-1	5	1	441	432	6	7	1	15	13
0	5	1	405	414	-7	8	1	110	111
1	5	1	262	249	-6	8	1	64	68
2	5	1	78	73	-5	8	1	197	196
3	5	1	282	277	-4	8	1	29	32
4	5	1	147	145	-3	8	1	104	103
5	5	1	114	109	-2	8	1	133	133
6	5	1	62	58	-1	8	1	365	355
7	5	1	95	104	0	8	1	51	63
-7	6	1	107	106	1	8	1	785	773
-6	6	1	115	119	2	8	1	30	24
-5	6	1	197	199	3	8	1	61	61
-4	6	1	92	88	4	8	1	74	74
-3	6	1	168	179	5	8	1	102	93
-2	6	1	52	51	6	8	1	95	93
-1	6	1	246	242	-6	9	1	152	151
0	6	1	125	122	-5	9	1	135	140
1	6	1	307	306	-4	9	1	57	56
2	6	1	246	252	-3	9	1	162	159
3	6	1	160	155	-2	9	1	142	135
4	6	1	119	124	-1	9	1	197	198
5	6	1	186	186	0	9	1	299	304
6	6	1	67	68	1	9	1	108	101
-7	7	1	73	68	2	9	1	37	32
-6	7	1	24	18	3	9	1	36	39
-5	7	1	100	109	4	9	1	70	72

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	9	1	149	155	-2	13	1	29	31	0	0	2	203	183
6	9	1	60	64	-1	13	1	164	165	1	0	2	233	257
-6	10	1	80	82	0	13	1	67	64	2	0	2	671	637
-5	10	1	163	169	1	13	1	126	130	3	0	2	185	181
-4	10	1	129	121	2	13	1	60	66	4	0	2	105	102
-3	10	1	94	95	3	13	1	125	123	5	0	2	46	52
-2	10	1	40	43	-4	14	1	47	47	6	0	2	83	87
-1	10	1	98	104	-3	14	1	63	58	7	0	2	28	35
1	10	1	102	99	-2	14	1	21	14	-8	1	2	231	224
2	10	1	202	210	-1	14	1	101	103	-7	1	2	56	60
3	10	1	74	71	0	14	1	48	44	-6	1	2	185	182
4	10	1	83	84	1	14	1	68	73	-5	1	2	174	171
5	10	1	35	54	2	14	1	35	24	-4	1	2	220	221
-6	11	1	101	98	3	14	1	53	56	-3	1	2	124	140
-5	11	1	107	113	-3	15	1	111	111	-2	1	2	440	479
-4	11	1	80	74	-2	15	1	21	8	-1	1	2	159	157
-3	11	1	73	67	-1	15	1	143	139	0	1	2	998	961
-2	11	1	31	30	0	15	1	104	97	1	1	2	251	238
-1	11	1	80	80	1	15	1	71	72	2	1	2	456	436
0	11	1	62	55	2	15	1	95	98	3	1	2	258	264
1	11	1	319	316	3	15	1	86	86	4	1	2	175	173
2	11	1	26	45	-3	16	1	9	6	5	1	2	58	56
3	11	1	125	123	-2	16	1	38	31	6	1	2	102	96
4	11	1	44	49	-1	16	1	72	75	7	1	2	63	74
5	11	1	130	135	0	16	1	79	80	-8	2	2	82	83
-5	12	1	42	31	1	16	1	75	73	-7	2	2	232	232
-4	12	1	80	76	2	16	1	78	79	-6	2	2	114	117
-3	12	1	24	19	3	16	1	32	34	-5	2	2	118	123
-2	12	1	92	93	-8	0	2	69	72	-4	2	2	357	339
-1	12	1	234	236	-7	0	2	110	109	-3	2	2	361	391
1	12	1	79	81	-6	0	2	43	36	-2	2	2	35	38
2	12	1	171	177	-5	0	2	199	193	-1	2	2	533	557
3	12	1	129	136	-4	0	2	583	562	0	2	2	477	473
4	12	1	89	91	-3	0	2	161	146	1	2	2	196	176
-4	13	1	74	75	-2	0	2	338	338	2	2	2	100	94
-3	13	1	113	112	-1	0	2	300	292	3	2	2	327	323

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	2	2	136	132	-6	5	2	293	293
5	2	2	25	13	-5	5	2	100	104
6	2	2	71	76	-4	5	2	270	263
7	2	2	54	55	-3	5	2	201	201
-8	3	2	157	158	-2	5	2	173	172
-7	3	2	64	74	-1	5	2	164	172
-6	3	2	182	178	0	5	2	236	262
-5	3	2	44	41	1	5	2	269	261
-4	3	2	71	73	2	5	2	521	510
-3	3	2	116	111	3	5	2	153	157
-2	3	2	721	749	4	5	2	199	203
-1	3	2	293	297	5	5	2	154	150
0	3	2	716	702	6	5	2	167	164
1	3	2	492	455	-8	6	2	19	26
2	3	2	482	456	-7	6	2	39	44
3	3	2	110	111	-6	6	2	167	169
4	3	2	113	115	-5	6	2	39	39
5	3	2	97	97	-4	6	2	98	98
6	3	2	190	189	-3	6	2	70	75
-8	4	2	69	62	-2	6	2	212	215
-7	4	2	41	31	-1	6	2	279	293
-6	4	2	129	128	0	6	2	26	31
-5	4	2	74	63	1	6	2	219	208
-4	4	2	125	123	2	6	2	324	333
-3	4	2	334	365	3	6	2	77	80
-2	4	2	455	486	4	6	2	129	125
-1	4	2	422	445	5	6	2	57	59
0	4	2	292	290	6	6	2	61	56
1	4	2	211	210	-7	7	2	98	96
2	4	2	609	626	-6	7	2	202	202
3	4	2	293	297	-5	7	2	46	44
4	4	2	169	165	-4	7	2	177	173
5	4	2	57	50	-3	7	2	124	125
6	4	2	137	137	-2	7	2	51	42
-8	5	2	84	86	-1	7	2	146	146
-7	5	2	102	104	0	7	2	149	152

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	7	2	377	365	-2	10	2	112	112	2	13	2	65	64
2	7	2	277	277	-1	10	2	39	39	3	13	2	133	132
3	7	2	121	132	0	10	2	127	128	-4	14	2	101	97
4	7	2	94	91	1	10	2	138	137	-3	14	2	129	128
5	7	2	138	140	2	10	2	140	136	-2	14	2	93	101
6	7	2	106	110	3	10	2	81	79	-1	14	2	42	44
-7	8	2	74	71	4	10	2	16	9	1	14	2	71	77
-6	8	2	95	92	5	10	2	88	86	2	14	2	80	82
-5	8	2	120	123	-6	11	2	23	18	3	14	2	71	75
-4	8	2	92	95	-5	11	2	87	98	-3	15	2	51	49
-3	8	2	35	33	-4	11	2	193	194	-2	15	2	120	122
-2	8	2	116	115	-3	11	2	35	45	-1	15	2	94	91
-1	8	2	167	162	-2	11	2	48	53	0	15	2	218	216
0	8	2	215	206	-1	11	2	133	129	1	15	2	98	92
1	8	2	74	62	0	11	2	171	171	2	15	2	76	85
2	8	2	142	149	1	11	2	31	43	3	15	2	63	54
3	8	2	117	109	2	11	2	73	72	-3	16	2	52	46
4	8	2	144	143	3	11	2	162	161	-1	16	2	73	74
5	8	2	94	99	4	11	2	57	61	0	16	2	65	65
-7	9	2	99	101	-5	12	2	65	61	1	16	2	51	57
-6	9	2	109	102	-4	12	2	89	106	2	16	2	52	52
-5	9	2	15	26	-3	12	2	112	117	3	16	2	47	42
-4	9	2	81	84	-2	12	2	177	182	-7	0	3	13	11
-3	9	2	68	79	-1	12	2	63	61	-6	0	3	131	131
-2	9	2	28	24	0	12	2	101	103	-5	0	3	189	183
-1	9	2	52	53	1	12	2	105	105	-4	0	3	34	48
0	9	2	191	187	2	12	2	49	58	-3	0	3	1556	1717
1	9	2	136	131	3	12	2	35	33	-2	0	3	34	36
2	9	2	127	138	4	12	2	62	68	-1	0	3	10	41
3	9	2	101	99	-5	13	2	97	98	0	0	3	590	616
4	9	2	127	130	-4	13	2	176	181	1	0	3	100	90
5	9	2	107	115	-3	13	2	34	31	2	0	3	122	117
-6	10	2	62	65	-2	13	2	195	196	3	0	3	372	373
-5	10	2	84	84	-1	13	2	28	30	4	0	3	174	174
-4	10	2	176	176	0	13	2	131	131	5	0	3	98	102
-3	10	2	80	82	1	13	2	28	32	6	0	3	119	125

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-8	1	3	91	89	-2	3	3	229	232	4
-7	1	3	107	104	-1	3	3	793	731	5
-6	1	3	78	72	0	3	3	339	339	6
-5	1	3	455	440	1	3	3	213	195	-8
-4	1	3	118	114	2	3	3	125	122	-7
-3	1	3	566	623	3	3	3	105	101	-6
-2	1	3	104	116	4	3	3	259	259	-5
-1	1	3	2157	1995	5	3	3	82	83	-4
0	1	3	535	541	6	3	3	68	74	-3
1	1	3	400	382	-8	4	3	55	50	-2
2	1	3	702	665	-7	4	3	110	108	-1
3	1	3	169	168	-6	4	3	86	87	0
4	1	3	192	189	-5	4	3	64	72	1
5	1	3	174	176	-4	4	3	124	126	2
6	1	3	48	48	-3	4	3	193	208	3
-8	2	3	44	40	-2	4	3	85	89	4
-7	2	3	104	105	-1	4	3	241	218	5
-6	2	3	121	118	0	4	3	245	232	-8
-5	2	3	192	185	1	4	3	491	476	-7
-4	2	3	152	146	2	4	3	129	124	-6
-3	2	3	747	779	3	4	3	166	171	-5
-2	2	3	52	48	4	4	3	196	198	-4
-1	2	3	1222	1146	5	4	3	153	154	-3
0	2	3	580	563	6	4	3	60	62	-2
1	2	3	714	724	-8	5	3	26	31	-1
2	2	3	86	78	-7	5	3	199	201	0
3	2	3	303	309	-6	5	3	48	43	1
4	2	3	97	101	-5	5	3	107	106	2
5	2	3	64	57	-4	5	3	286	287	3
6	2	3	30	38	-3	5	3	509	553	4
-8	3	3	121	121	-2	5	3	149	166	5
-7	3	3	129	128	-1	5	3	36	31	-7
-6	3	3	123	124	0	5	3	224	232	-6
-5	3	3	146	145	1	5	3	261	253	-5
-4	3	3	97	100	2	5	3	246	248	-4
-3	3	3	332	367	3	5	3	182	178	-3

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	3	131	139	-2	8	3	51	49	-1	11	3	63	65
5	3	51	56	-1	8	3	386	386	0	11	3	75	75
5	3	92	96	0	8	3	107	106	1	11	3	39	38
6	3	117	115	1	8	3	202	192	2	11	3	104	109
6	3	105	103	2	8	3	106	104	3	11	3	78	73
6	3	47	43	4	8	3	75	69	4	11	3	65	62
6	3	101	103	5	8	3	40	30	-6	12	3	61	62
6	3	245	242	-7	9	3	89	87	-5	12	3	183	182
6	3	78	71	-6	9	3	174	175	-4	12	3	143	132
6	3	92	95	-5	9	3	151	149	-3	12	3	138	133
6	3	212	205	-4	9	3	192	197	-2	12	3	110	110
6	3	109	113	-3	9	3	89	91	-1	12	3	37	34
6	3	292	290	-2	9	3	186	190	0	12	3	96	94
6	3	76	67	-1	9	3	88	85	1	12	3	34	29
6	3	140	138	0	9	3	102	104	2	12	3	31	32
6	3	56	55	1	9	3	123	122	3	12	3	55	61
6	3	35	37	2	9	3	71	71	-5	13	3	142	143
7	3	126	128	3	9	3	109	107	-4	13	3	49	51
7	3	144	139	4	9	3	121	120	-3	13	3	179	178
7	3	30	23	5	9	3	165	169	-2	13	3	53	49
7	3	68	56	-7	10	3	86	88	-1	13	3	47	47
7	3	105	104	-6	10	3	188	193	0	13	3	39	47
7	3	165	165	-5	10	3	70	80	1	13	3	32	35
7	3	207	212	-4	10	3	72	76	2	13	3	119	117
7	3	138	139	-3	10	3	228	220	3	13	3	110	106
7	3	274	263	-2	10	3	65	61	-4	14	3	125	122
7	3	64	62	-1	10	3	102	98	-3	14	3	72	61
7	3	39	49	0	10	3	224	220	-2	14	3	43	46
7	3	148	145	1	10	3	194	196	-1	14	3	89	86
7	3	110	104	2	10	3	100	97	0	14	3	74	68
7	3	87	83	3	10	3	20	17	1	14	3	31	22
8	3	66	66	4	10	3	29	26	2	14	3	9	10
8	3	63	58	-6	11	3	117	115	3	14	3	18	9
8	3	49	54	-5	11	3	83	80	-3	15	3	61	61
8	3	74	64	-3	11	3	115	116	-2	15	3	34	47
8	3	188	197	-2	11	3	144	143	-1	15	3	208	208

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	15	3	35	43	1	1	4	380	376	-8
1	15	3	50	49	2	1	4	63	63	-7
2	15	3	55	46	3	1	4	66	64	-6
3	15	3	23	26	4	1	4	247	251	-5
-3	16	3	42	40	5	1	4	29	35	-4
-1	16	3	59	55	6	1	4	112	110	-3
0	16	3	33	30	-9	2	4	75	80	-2
1	16	3	52	52	-8	2	4	114	116	-1
2	16	3	27	26	-7	2	4	81	88	0
3	16	3	36	21	-6	2	4	160	164	1
-9	0	4	43	49	-5	2	4	368	343	2
-8	0	4	116	116	-4	2	4	351	342	3
-7	0	4	68	71	-3	2	4	187	195	4
-6	0	4	314	301	-2	2	4	572	581	5
-5	0	4	45	42	-1	2	4	243	245	-8
-4	0	4	113	105	0	2	4	383	387	-7
-3	0	4	361	378	1	2	4	250	235	-6
-2	0	4	344	299	2	2	4	206	201	-5
-1	0	4	482	459	3	2	4	142	134	-4
0	0	4	782	773	4	2	4	94	91	-3
1	0	4	486	468	5	2	4	98	101	-2
2	0	4	167	172	-9	3	4	21	11	-1
3	0	4	119	124	-8	3	4	126	130	0
4	0	4	177	174	-7	3	4	55	50	1
5	0	4	93	87	-6	3	4	16	29	2
6	0	4	153	154	-5	3	4	360	353	3
-9	1	4	50	53	-4	3	4	276	256	4
-8	1	4	39	43	-3	3	4	377	415	5
-7	1	4	88	86	-2	3	4	748	740	-8
-6	1	4	94	89	-1	3	4	296	302	-7
-5	1	4	320	305	0	3	4	297	277	-6
-4	1	4	475	464	1	3	4	184	182	-5
-3	1	4	204	225	2	3	4	195	187	-4
-2	1	4	886	877	3	3	4	233	237	-3
-1	1	4	242	232	4	3	4	95	97	-2
0	1	4	132	130	5	3	4	24	23	-1

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	4		35	30	0	6	4	115	108	-4	9	4	143	140
4	4		111	110	1	6	4	149	143	-3	9	4	68	68
4	4		109	112	2	6	4	286	282	-2	9	4	246	244
4	4		117	116	3	6	4	40	48	-1	9	4	83	82
4	4		455	447	4	6	4	76	77	0	9	4	268	275
4	4		181	199	5	6	4	112	112	1	9	4	53	52
4	4		224	228	-8	7	4	110	112	2	9	4	98	98
4	4		239	245	-7	7	4	39	37	3	9	4	53	49
4	4		452	464	-6	7	4	144	142	4	9	4	179	181
4	4		78	87	-5	7	4	26	28	-7	10	4	118	122
4	4		307	287	-4	7	4	156	151	-6	10	4	185	184
4	4		159	159	-3	7	4	135	146	-5	10	4	71	65
4	4		27	32	-2	7	4	211	216	-4	10	4	184	185
4	4		49	49	-1	7	4	191	194	-3	10	4	106	113
5	4		156	161	0	7	4	310	318	-2	10	4	152	144
5	4		134	136	1	7	4	131	127	-1	10	4	105	102
5	4		29	36	2	7	4	44	47	0	10	4	237	241
5	4		15	15	3	7	4	141	144	1	10	4	48	49
5	4		320	301	4	7	4	111	105	2	10	4	145	152
5	4		85	98	5	7	4	70	78	3	10	4	88	87
5	4		429	478	-8	8	4	56	58	4	10	4	105	108
5	4		196	197	-7	8	4	73	76	-6	11	4	65	64
5	4		244	236	-6	8	4	71	72	-5	11	4	50	51
5	4		97	100	-5	8	4	103	110	-4	11	4	67	78
5	4		108	111	-4	8	4	108	108	-3	11	4	60	61
5	4		98	89	-3	8	4	52	63	-2	11	4	57	58
5	4		26	25	-2	8	4	251	260	-1	11	4	122	122
5	4		113	109	-1	8	4	290	282	0	11	4	157	158
6	4		39	36	0	8	4	311	301	1	11	4	56	60
6	4		43	44	1	8	4	111	108	2	11	4	84	89
6	4		75	81	2	8	4	88	88	3	11	4	59	56
6	4		69	68	3	8	4	72	64	-6	12	4	103	101
6	4		308	296	4	8	4	71	69	-5	12	4	57	59
6	4		26	25	-7	9	4	104	101	-4	12	4	160	161
6	4		353	340	-6	9	4	133	132	-3	12	4	81	75
6	4		486	474	-5	9	4	168	168	-2	12	4	179	178

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-1	12	4	51	54	-3	0	5	566	520
0	12	4	109	111	-2	0	5	113	116
1	12	4	81	85	-1	0	5	409	365
2	12	4	29	30	0	0	5	358	358
3	12	4	75	75	1	0	5	196	201
-5	13	4	33	25	2	0	5	216	209
-4	13	4	86	82	3	0	5	30	25
-3	13	4	63	68	4	0	5	60	63
-2	13	4	129	124	5	0	5	112	115
-1	13	4	15	11	-8	1	5	71	71
0	13	4	78	82	-7	1	5	97	94
1	13	4	93	89	-6	1	5	75	77
2	13	4	94	95	-5	1	5	567	537
3	13	4	26	23	-4	1	5	82	85
-4	14	4	55	52	-3	1	5	302	323
-3	14	4	60	60	-2	1	5	418	407
-2	14	4	43	46	-1	1	5	494	466
-1	14	4	76	80	0	1	5	169	169
0	14	4	70	70	1	1	5	413	394
1	14	4	126	130	2	1	5	133	140
2	14	4	39	40	3	1	5	150	147
3	14	4	49	55	4	1	5	43	45
-2	15	4	94	86	5	1	5	116	116
-1	15	4	41	42	-9	2	5	67	64
0	15	4	92	91	-8	2	5	143	147
1	15	4	64	62	-7	2	5	109	110
-3	16	4	73	71	-6	2	5	122	124
-2	16	4	85	84	-5	2	5	390	379
-1	16	4	60	56	-4	2	5	222	216
1	16	4	23	9	-3	2	5	326	343
2	16	4	22	17	-2	2	5	168	158
3	16	4	10	22	-1	2	5	378	361
-9	0	5	47	52	0	2	5	235	234
-8	0	5	88	82	1	2	5	289	279
-6	0	5	156	148	2	2	5	308	302
-5	0	5	182	176	3	2	5	169	170

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	2	5	126	128	-4	5	5	102	107	-8	8	5	64	59
5	2	5	184	189	-3	5	5	34	32	-7	8	5	117	114
-9	3	5	30	23	-2	5	5	105	103	-6	8	5	112	100
-8	3	5	66	67	-1	5	5	396	394	-5	8	5	121	122
-7	3	5	151	152	0	5	5	203	194	-4	8	5	90	88
-6	3	5	120	118	1	5	5	199	193	-3	8	5	127	139
-5	3	5	604	576	2	5	5	144	145	-2	8	5	136	144
-4	3	5	176	171	3	5	5	45	40	-1	8	5	216	219
-3	3	5	265	273	4	5	5	96	100	0	8	5	185	184
-2	3	5	142	137	5	5	5	34	35	1	8	5	91	99
-1	3	5	151	153	-8	6	5	98	95	2	8	5	95	100
0	3	5	40	43	-7	6	5	116	111	3	8	5	69	70
1	3	5	198	197	-6	6	5	37	29	4	8	5	41	41
2	3	5	133	128	-5	6	5	84	83	-7	9	5	41	39
3	3	5	126	130	-4	6	5	128	126	-6	9	5	101	102
4	3	5	55	51	-3	6	5	187	192	-5	9	5	148	148
5	3	5	53	60	-2	6	5	126	124	-4	9	5	49	49
-9	4	5	54	59	-1	6	5	70	73	-3	9	5	49	49
-8	4	5	57	57	0	6	5	116	115	-2	9	5	73	77
-7	4	5	86	84	1	6	5	338	341	-1	9	5	178	178
-6	4	5	55	58	2	6	5	75	78	0	9	5	71	70
-5	4	5	264	267	3	6	5	141	144	1	9	5	143	143
-4	4	5	72	67	4	6	5	102	107	2	9	5	129	131
-3	4	5	357	400	-8	7	5	72	78	3	9	5	44	45
-2	4	5	158	150	-7	7	5	104	106	-7	10	5	129	130
-1	4	5	43	59	-6	7	5	98	94	-6	10	5	81	81
0	4	5	281	270	-5	7	5	58	63	-5	10	5	82	81
1	4	5	370	364	-4	7	5	70	67	-4	10	5	84	75
2	4	5	141	139	-3	7	5	44	31	-3	10	5	171	170
3	4	5	109	111	-2	7	5	104	105	-2	10	5	68	67
4	4	5	35	28	-1	7	5	238	236	-1	10	5	165	153
5	4	5	112	114	0	7	5	187	194	0	10	5	19	12
-8	5	5	49	56	1	7	5	116	115	1	10	5	160	159
-7	5	5	105	106	2	7	5	132	129	2	10	5	74	82
-6	5	5	191	195	3	7	5	102	100	3	10	5	71	80
-5	5	5	53	42	4	7	5	56	54	-6	11	5	91	94

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-5	11	5	135	131	-3	15	5	89	86	1
-4	11	5	54	49	-2	15	5	48	44	2
-3	11	5	54	57	-1	15	5	21	8	3
-2	11	5	170	176	0	15	5	80	83	4
-1	11	5	99	93	2	15	5	30	25	-9
0	11	5	56	59	-3	16	5	49	52	-8
1	11	5	98	103	-2	16	5	23	19	-7
2	11	5	71	82	-1	16	5	44	48	-6
3	11	5	36	43	0	16	5	35	34	-5
-6	12	5	76	74	1	16	5	20	12	-4
-5	12	5	112	110	2	16	5	50	37	-3
-4	12	5	86	83	3	16	5	30	21	-2
-3	12	5	220	227	-9	0	6	9	13	-1
-2	12	5	77	79	-8	0	6	79	83	0
-1	12	5	74	75	-7	0	6	73	69	1
0	12	5	116	117	-6	0	6	186	180	2
1	12	5	135	136	-5	0	6	202	201	3
2	12	5	24	30	-4	0	6	379	354	4
3	12	5	34	39	-3	0	6	343	340	-9
-5	13	5	102	110	-2	0	6	27	9	-8
-4	13	5	103	107	-1	0	6	326	302	-7
-3	13	5	74	77	0	0	6	356	358	-6
-2	13	5	94	98	1	0	6	229	230	-5
-1	13	5	87	93	2	0	6	145	146	-4
0	13	5	161	161	3	0	6	84	86	-3
1	13	5	18	16	4	0	6	17	19	-2
2	13	5	82	83	-9	1	6	87	87	-1
3	13	5	20	28	-8	1	6	49	47	0
-4	14	5	44	46	-7	1	6	49	53	1
-3	14	5	77	81	-6	1	6	73	70	2
-2	14	5	78	72	-5	1	6	93	89	3
-1	14	5	139	141	-4	1	6	251	238	4
0	14	5	191	185	-3	1	6	133	139	-9
1	14	5	60	72	-2	1	6	125	128	-8
2	14	5	60	70	-1	1	6	273	265	-7
3	14	5	55	49	0	1	6	285	283	-6

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	6		152	151	-5	4	6	85	93	4	6	6	126	130
1	6		203	200	-4	4	6	318	306	-8	7	6	54	54
1	6		190	188	-3	4	6	100	105	-7	7	6	86	83
1	6		114	111	-2	4	6	336	330	-5	7	6	39	47
2	6		36	34	-1	4	6	35	32	-4	7	6	215	206
2	6		49	41	0	4	6	209	210	-3	7	6	104	106
2	6		162	158	1	4	6	194	192	-2	7	6	90	89
2	6		306	302	2	4	6	267	276	-1	7	6	137	136
2	6		150	148	3	4	6	47	55	0	7	6	213	214
2	6		275	277	4	4	6	58	57	1	7	6	50	48
2	6		168	167	-9	5	6	59	58	3	7	6	47	58
2	6		231	231	-8	5	6	71	65	-8	8	6	81	77
2	6		270	272	-7	5	6	123	125	-7	8	6	95	105
2	6		210	210	-6	5	6	47	34	-6	8	6	148	144
2	6		197	198	-5	5	6	148	146	-5	8	6	84	84
2	6		305	302	-4	5	6	179	173	-4	8	6	233	232
2	6		62	68	-3	5	6	32	33	-3	8	6	42	39
2	6		75	76	-2	5	6	112	124	-2	8	6	8	17
3	6		35	36	-1	5	6	59	56	-1	8	6	207	203
3	6		82	80	0	5	6	241	239	0	8	6	84	86
3	6		26	18	1	5	6	158	156	1	8	6	240	239
3	6		206	198	2	5	6	45	42	2	8	6	65	64
3	6		89	82	3	5	6	72	80	3	8	6	64	62
3	6		512	476	4	5	6	62	67	-7	9	6	78	82
3	6		154	163	-8	6	6	134	132	-6	9	6	110	108
3	6		359	344	-7	6	6	47	51	-5	9	6	30	29
3	6		292	277	-6	6	6	52	53	-4	9	6	102	107
3	6		245	245	-5	6	6	170	165	-3	9	6	143	146
3	6		295	294	-4	6	6	210	207	-2	9	6	45	52
3	6		164	157	-3	6	6	88	102	-1	9	6	99	102
3	6		112	117	-2	6	6	134	137	0	9	6	162	162
3	6		114	113	-1	6	6	85	88	1	9	6	130	132
4	6		87	87	0	6	6	168	164	2	9	6	142	144
4	6		102	100	1	6	6	147	150	3	9	6	20	29
4	6		51	44	2	6	6	70	74	-7	10	6	62	56
4	6		118	113	3	6	6	33	33	-6	10	6	100	102

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-5	10	6	40	47	2	13	6	23	29	-7
-4	10	6	106	106	3	13	6	45	48	-6
-3	10	6	106	108	-3	14	6	63	62	-5
-2	10	6	77	79	-2	14	6	156	158	-4
-1	10	6	73	71	-1	14	6	63	62	-3
1	10	6	20	22	0	14	6	41	34	-2
2	10	6	75	65	1	14	6	88	82	-1
3	10	6	63	64	3	14	6	35	29	0
-7	11	6	16	12	-3	15	6	62	65	1
-6	11	6	49	54	-2	15	6	87	81	2
-5	11	6	95	93	-1	15	6	48	57	3
-4	11	6	139	136	0	15	6	31	24	4
-3	11	6	132	131	1	15	6	21	22	-9
-2	11	6	94	95	2	15	6	31	21	-8
-1	11	6	47	44	3	15	6	19	20	-7
0	11	6	94	93	-3	16	6	10	19	-6
1	11	6	70	74	-2	16	6	36	43	-5
2	11	6	105	111	-1	16	6	35	27	-4
3	11	6	69	65	0	16	6	56	52	-3
-6	12	6	59	58	1	16	6	33	31	-2
-5	12	6	81	87	2	16	6	29	18	-1
-4	12	6	122	116	3	16	6	24	20	0
-3	12	6	128	134	-9	0	7	19	4	1
-2	12	6	145	144	-8	0	7	64	65	2
-1	12	6	120	120	-7	0	7	62	63	3
0	12	6	46	40	-6	0	7	171	170	4
1	12	6	59	58	-5	0	7	114	111	-9
2	12	6	38	34	-4	0	7	12	3	-8
3	12	6	76	70	-3	0	7	386	391	-7
-5	13	6	66	76	-2	0	7	83	84	-6
-4	13	6	64	67	-1	0	7	38	29	-5
-3	13	6	163	162	1	0	7	123	124	-4
-2	13	6	21	33	2	0	7	27	19	-3
-1	13	6	154	150	3	0	7	105	111	-2
0	13	6	40	40	4	0	7	34	27	-1
1	13	6	47	46	-8	1	7	105	109	0

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	7	7	122	117	1	3	7	68	73	-3	6	7	235	242
1	7	7	187	178	2	3	7	111	113	-2	6	7	84	81
1	7	7	268	268	3	3	7	132	135	-1	6	7	177	176
1	7	7	261	254	4	3	7	36	31	0	6	7	73	73
1	7	7	327	325	-9	4	7	92	94	1	6	7	111	110
1	7	7	381	364	-8	4	7	150	152	2	6	7	22	19
1	7	7	238	234	-7	4	7	132	138	3	6	7	109	116
1	7	7	250	253	-6	4	7	38	39	-8	7	7	87	92
1	7	7	30	27	-5	4	7	50	55	-7	7	7	113	119
1	7	7	131	132	-4	4	7	130	132	-6	7	7	104	103
1	7	7	55	53	-3	4	7	145	149	-5	7	7	87	84
1	7	7	18	13	-2	4	7	226	225	-4	7	7	130	129
2	7	7	102	102	-1	4	7	185	178	-3	7	7	157	164
2	7	7	108	112	0	4	7	244	245	-2	7	7	109	109
2	7	7	57	56	1	4	7	126	126	-1	7	7	226	219
2	7	7	141	138	2	4	7	34	37	0	7	7	31	30
2	7	7	286	272	3	4	7	87	85	1	7	7	159	158
2	7	7	298	290	-9	5	7	3[illegible]	38	2	7	7	106	110
2	7	7	121	119	-8	5	7	32	27	3	7	7	49	39
2	7	7	143	139	-7	5	7	106	106	-8	8	7	34	32
2	7	7	90	77	-6	5	7	74	65	-7	8	7	100	98
2	7	7	187	186	-5	5	7	197	194	-6	8	7	152	153
2	7	7	63	63	-4	5	7	92	93	-5	8	7	53	50
2	7	7	48	49	-3	5	7	94	102	-4	8	7	91	94
2	7	7	110	117	-2	5	7	26	25	-3	8	7	133	135
2	7	7	28	30	-1	5	7	186	183	-2	8	7	41	35
3	7	7	45	50	0	5	7	94	94	-1	8	7	14	20
3	7	7	34	37	1	5	7	23	30	0	8	7	97	98
3	7	7	75	79	2	5	7	105	104	1	8	7	80	78
3	7	7	90	83	3	5	7	173	178	2	8	7	59	56
3	7	7	345	351	-9	6	7	59	67	3	8	7	70	77
3	7	7	195	194	-8	6	7	43	42	-8	9	7	100	100
3	7	7	134	129	-7	6	7	74	72	-7	9	7	58	59
3	7	7	103	106	-6	6	7	25	8	-6	9	7	118	120
3	7	7	139	135	-5	6	7	125	117	-5	9	7	146	142
3	7	7	107	111	-4	6	7	53	53	-4	9	7	70	72

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-3	9	7	17	21	3	12	7	48	42	-4
-2	9	7	98	95	-5	13	7	24	34	-3
-1	9	7	179	181	-4	13	7	114	114	-2
0	9	7	31	29	-3	13	7	90	87	-1
1	9	7	96	97	-2	13	7	61	66	0
2	9	7	53	47	-1	13	7	57	55	1
3	9	7	45	47	0	13	7	36	36	2
-7	10	7	67	66	1	13	7	24	20	3
-6	10	7	124	114	2	13	7	44	35	4
-5	10	7	118	118	3	13	7	19	27	-9
-4	10	7	63	68	-3	14	7	115	109	-8
-3	10	7	108	104	-2	14	7	86	87	-7
-2	10	7	114	123	-1	14	7	21	19	-6
-1	10	7	179	175	0	14	7	39	26	-5
0	10	7	79	73	1	14	7	17	26	-4
1	10	7	79	80	2	14	7	27	20	-3
2	10	7	69	71	3	14	7	44	29	-2
3	10	7	34	38	-3	15	7	37	46	-1
-5	11	7	71	76	-2	15	7	41	40	0
-4	11	7	72	73	-1	15	7	46	40	1
-3	11	7	61	71	0	15	7	37	41	2
-2	11	7	128	130	1	15	7	54	47	3
-1	11	7	110	108	2	15	7	21	22	4
0	11	7	127	137	3	15	7	33	31	-9
1	11	7	72	68	-3	16	7	46	35	-8
2	11	7	34	32	-2	16	7	23	20	-7
3	11	7	25	20	-1	16	7	59	52	-6
-6	12	7	72	68	0	16	7	46	46	-5
-5	12	7	134	131	1	16	7	33	12	-4
-4	12	7	79	84	2	16	7	27	20	-3
-3	12	7	136	134	3	16	7	37	34	-2
-2	12	7	63	60	-9	0	8	50	52	-1
-1	12	7	143	141	-8	0	8	108	102	0
0	12	7	50	54	-7	0	8	99	95	1
1	12	7	84	78	-6	0	8	278	275	2
2	12	7	87	90	-5	0	8	205	201	3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	0	8	216	208	-9	3	8	56	58	1	5	8	120	124
0	0	8	125	121	-8	3	8	53	58	2	5	8	87	91
0	0	8	328	309	-7	3	8	73	67	3	5	8	66	66
0	0	8	13	11	-6	3	8	124	126	-9	6	8	60	55
0	0	8	59	45	-5	3	8	129	129	-8	6	8	93	91
0	0	8	42	50	-4	3	8	175	170	-7	6	8	84	87
0	0	8	140	145	-3	3	8	253	257	-6	6	8	28	32
0	0	8	27	30	-2	3	8	218	212	-5	6	8	73	73
0	0	8	74	75	-1	3	8	127	128	-4	6	8	67	71
1	0	8	71	60	0	3	8	53	50	-3	6	8	13	17
1	0	8	102	109	1	3	8	110	116	-2	6	8	65	73
1	0	8	163	164	2	3	8	95	95	-1	6	8	90	93
1	0	8	450	445	3	3	8	93	91	0	6	8	135	134
1	0	8	294	285	-9	4	8	98	98	1	6	8	83	84
1	0	8	150	150	-8	4	8	66	68	2	6	8	19	14
1	0	8	248	248	-7	4	8	104	99	3	6	8	9	10
1	0	8	498	463	-6	4	8	66	65	-8	7	8	80	63
1	0	8	148	147	-5	4	8	72	82	-7	7	8	120	118
1	0	8	93	96	-4	4	8	197	193	-6	7	8	66	62
1	0	8	98	93	-3	4	8	124	126	-5	7	8	80	76
1	0	8	152	154	-2	4	8	173	172	-4	7	8	57	63
1	0	8	25	26	-1	4	8	61	66	-3	7	8	50	49
1	0	8	100	105	0	4	8	109	115	-2	7	8	143	141
2	0	8	74	72	1	4	8	105	110	-1	7	8	106	102
2	0	8	129	128	2	4	8	112	117	0	7	8	51	53
2	0	8	25	22	3	4	8	68	67	1	7	8	134	132
2	0	8	197	200	-9	5	8	66	62	2	7	8	140	143
2	0	8	148	142	-8	5	8	82	84	3	7	8	94	94
2	0	8	277	273	-7	5	8	32	39	-8	8	8	39	37
2	0	8	184	188	-6	5	8	187	187	-7	8	8	94	99
2	0	8	203	201	-5	5	8	114	119	-6	8	8	147	143
2	0	8	169	162	-4	5	8	55	52	-5	8	8	28	29
2	0	8	247	248	-3	5	8	120	126	-4	8	8	170	167
2	0	8	71	69	-2	5	8	15	3	-3	8	8	65	72
2	0	8	110	112	-1	5	8	59	60	-2	8	8	20	21
2	0	8	48	57	0	5	8	76	80	-1	8	8	20	13

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	8	8	135	136	-2	12	8	150	149	-3
1	8	8	132	136	-1	12	8	122	117	-2
2	8	8	48	44	0	12	8	123	125	-1
-7	9	8	59	59	1	12	8	48	46	0
-6	9	8	51	50	2	12	8	47	50	1
-5	9	8	59	48	3	12	8	43	33	2
-4	9	8	113	113	-4	13	8	86	88	3
-3	9	8	84	86	-3	13	8	57	66	4
-2	9	8	230	239	-2	13	8	17	28	-9
0	9	8	38	38	-1	13	8	30	30	-8
1	9	8	60	63	0	13	8	31	31	-7
2	9	8	74	75	1	13	8	46	39	-6
3	9	8	72	70	2	13	8	17	22	-5
-7	10	8	84	82	3	13	8	10	13	-4
-6	10	8	104	110	-2	14	8	46	37	-3
-5	10	8	60	53	-1	14	8	77	80	-2
-4	10	8	8	19	0	14	8	32	28	-1
-3	10	8	177	174	1	14	8	17	17	0
-2	10	8	121	125	-3	15	8	39	49	1
-1	10	8	41	36	-2	15	8	43	24	2
0	10	8	130	130	-1	15	8	74	67	3
1	10	8	53	55	0	15	8	44	44	4
2	10	8	38	41	1	15	8	61	51	-9
3	10	8	18	21	2	15	8	30	26	-8
-6	11	8	43	48	3	15	8	29	25	-7
-5	11	8	46	43	-3	16	8	37	36	-6
-4	11	8	101	96	-2	16	8	23	8	-5
-3	11	8	104	102	-1	16	8	47	49	-4
-2	11	8	101	105	0	16	8	10	17	-3
-1	11	8	185	185	1	16	8	10	12	-2
0	11	8	86	86	2	16	8	57	48	-1
1	11	8	37	48	-8	0	9	59	58	0
2	11	8	9	17	-7	0	9	242	240	1
-5	12	8	36	35	-6	0	9	39	47	2
-4	12	8	89	87	-5	0	9	192	190	3
-3	12	8	127	126	-4	0	9	41	41	-9

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	9	105	103	-7	3	9	162	168	3	5	9	33	28
0	9	25	24	-6	3	9	110	112	-8	6	9	61	61
0	9	73	72	-5	3	9	111	119	-7	6	9	122	118
0	9	117	117	-4	3	9	83	78	-6	6	9	74	73
0	9	71	72	-3	3	9	97	95	-5	6	9	78	76
0	9	66	76	-2	3	9	24	28	-4	6	9	113	107
0	9	76	82	-1	3	9	63	58	-3	6	9	20	9
0	9	45	50	0	3	9	132	133	-2	6	9	74	71
1	9	132	127	1	3	9	155	158	-1	6	9	156	158
1	9	75	80	2	3	9	102	105	0	6	9	84	82
1	9	218	216	3	3	9	24	29	1	6	9	131	131
1	9	206	204	-9	4	9	58	65	2	6	9	23	20
1	9	81	79	-8	4	9	49	44	3	6	9	31	22
1	9	111	104	-7	4	9	82	84	-8	7	9	85	94
1	9	152	157	-6	4	9	98	97	-7	7	9	77	82
1	9	95	99	-5	4	9	39	45	-6	7	9	80	82
1	9	120	115	-4	4	9	42	48	-5	7	9	136	140
1	9	77	75	-3	4	9	82	83	-4	7	9	24	29
1	9	274	275	-2	4	9	220	220	-2	7	9	138	143
1	9	29	31	-1	4	9	40	44	-1	7	9	86	84
1	9	49	54	0	4	9	128	132	0	7	9	223	225
1	9	50	54	1	4	9	97	92	1	7	9	56	63
2	9	66	65	2	4	9	60	53	2	7	9	67	61
2	9	16	25	3	4	9	22	22	3	7	9	29	29
2	9	55	59	-9	5	9	34	27	-8	8	9	79	74
2	9	71	73	-8	5	9	9	15	-7	8	9	32	26
2	9	183	186	-7	5	9	171	172	-6	8	9	70	68
2	9	219	222	-6	5	9	24	19	-5	8	9	16	17
2	9	107	114	-5	5	9	48	44	-4	8	9	60	61
2	9	110	111	-4	5	9	98	101	-3	8	9	87	100
2	9	186	183	-3	5	9	35	45	-2	8	9	132	139
2	9	125	132	-2	5	9	150	148	-1	8	9	203	199
2	9	90	87	-1	5	9	192	188	0	8	9	94	100
2	9	72	76	0	5	9	29	28	1	8	9	104	110
2	9	30	33	1	5	9	183	185	2	8	9	41	47
3	9	53	56	2	5	9	71	73	3	8	9	18	14

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-7	9	9	25	18	-1	12	9	117	113	-1
-6	9	9	90	89	0	12	9	95	92	0
-5	9	9	127	129	1	12	9	31	21	1
-4	9	9	81	81	2	12	9	19	21	2
-3	9	9	86	87	3	12	9	20	14	3
-2	9	9	71	72	-3	13	9	96	97	4
-1	9	9	54	57	-2	13	9	84	82	-9
0	9	9	144	147	-1	13	9	42	31	-8
1	9	9	62	68	2	13	9	19	15	-7
2	9	9	15	22	3	13	9	18	22	-6
3	9	9	29	18	-3	14	9	37	23	-5
-7	10	9	29	29	-2	14	9	82	90	-4
-6	10	9	33	31	-1	14	9	21	28	-3
-5	10	9	44	51	0	14	9	23	24	-2
-4	10	9	65	70	1	14	9	29	33	-1
-3	10	9	76	76	2	14	9	24	16	0
-2	10	9	80	80	3	14	9	33	28	1
-1	10	9	53	60	-2	15	9	22	20	2
0	10	9	78	75	-1	15	9	56	55	3
1	10	9	44	50	0	15	9	41	28	4
2	10	9	33	33	1	15	9	26	22	-9
3	10	9	35	25	2	15	9	22	14	-8
-6	11	9	25	6	-3	16	9	20	18	-7
-5	11	9	37	36	-2	16	9	50	44	-6
-4	11	9	128	127	0	16	9	15	4	-5
-3	11	9	20	31	1	16	9	17	27	-4
-2	11	9	60	69	2	16	9	11	15	-3
-1	11	9	73	73	3	16	9	33	28	-2
0	11	9	64	74	-9	0	10	52	53	-1
1	11	9	31	16	-8	0	10	34	28	0
2	11	9	21	16	-7	0	10	141	149	1
3	11	9	41	37	-6	0	10	117	115	2
-5	12	9	118	119	-5	0	10	31	39	3
-4	12	9	84	84	-4	0	10	42	34	-9
-3	12	9	75	76	-3	0	10	124	121	-8
-2	12	9	32	45	-2	0	10	118	117	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	10		119	119	-6	3	10	33	32	-7	6	10	96	87
0	10		128	129	-5	3	10	32	40	-6	6	10	77	78
0	10		26	26	-4	3	10	133	140	-4	6	10	84	81
0	10		128	120	-3	3	10	50	48	-3	6	10	63	60
0	10		14	14	-2	3	10	53	54	-2	6	10	179	181
0	10		22	24	-1	3	10	133	134	-1	6	10	73	74
1	10		30	26	0	3	10	163	166	0	6	10	135	141
1	10		54	55	1	3	10	138	142	1	6	10	10	17
1	10		113	111	2	3	10	74	72	2	6	10	58	58
1	10		96	94	3	3	10	18	8	3	6	10	42	24
1	10		96	99	-9	4	10	72	80	-8	7	10	87	84
1	10		117	122	-8	4	10	73	73	-7	7	10	72	76
1	10		61	66	-7	4	10	58	65	-6	7	10	79	85
1	10		122	127	-6	4	10	116	121	-5	7	10	17	29
1	10		87	93	-5	4	10	188	192	-4	7	10	65	66
1	10		98	100	-4	4	10	27	29	-3	7	10	86	99
1	10		9	37	-3	4	10	124	128	-2	7	10	57	60
1	10		38	30	-2	4	10	92	92	-1	7	10	132	135
1	10		34	25	-1	4	10	38	40	0	7	10	54	59
1	10		39	36	0	4	10	118	121	1	7	10	23	22
2	10		39	34	1	4	10	70	70	2	7	10	43	50
2	10		69	65	2	4	10	40	30	3	7	10	33	29
2	10		44	46	3	4	10	28	31	-8	8	10	53	64
2	10		71	69	-9	5	10	47	44	-7	8	10	31	35
2	10		54	66	-8	5	10	59	59	-6	8	10	27	29
2	10		142	143	-7	5	10	81	79	-4	8	10	128	129
2	10		122	125	-6	5	10	85	85	-3	8	10	64	71
2	10		16	9	-5	5	10	112	106	-2	8	10	93	98
2	10		44	42	-4	5	10	42	43	-1	8	10	57	49
2	10		110	108	-3	5	10	65	64	0	8	10	32	39
2	10		32	23	-2	5	10	60	60	1	8	10	64	63
2	10		88	91	-1	5	10	90	96	3	8	10	60	58
2	10		43	38	0	5	10	203	205	-7	9	10	32	29
3	10		64	63	2	5	10	10	18	-6	9	10	46	53
3	10		112	104	3	5	10	17	13	-5	9	10	41	37
3	10		143	145	-8	6	10	40	37	-4	9	10	57	55

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-3	9	10	95	96	3	13	10	37	22	-3
-2	9	10	64	73	-3	14	10	42	54	-2
-1	9	10	78	79	-2	14	10	61	61	-1
0	9	10	33	29	0	14	10	57	54	1
1	9	10	27	39	1	14	10	34	34	2
2	9	10	29	28	2	14	10	31	30	3
3	9	10	35	29	-3	15	10	57	51	-9
-6	10	10	25	25	-2	15	10	19	19	-8
-5	10	10	108	109	-1	15	10	38	35	-7
-4	10	10	81	83	0	15	10	10	15	-6
-3	10	10	105	108	2	15	10	22	22	-5
-2	10	10	65	70	3	15	10	39	20	-4
-1	10	10	53	62	-3	16	10	22	18	-3
0	10	10	72	72	-2	16	10	10	14	-2
1	10	10	25	27	-1	16	10	20	17	-1
2	10	10	37	28	0	16	10	26	13	1
3	10	10	51	48	1	16	10	27	27	2
-5	11	10	102	98	2	16	10	26	25	3
-4	11	10	65	62	3	16	10	29	20	-9
-3	11	10	75	74	-9	0	11	82	82	-8
-2	11	10	83	85	-8	0	11	218	218	-7
-1	11	10	27	23	-7	0	11	358	367	-6
0	11	10	45	46	-6	0	11	43	49	-5
1	11	10	38	44	-5	0	11	84	89	-4
2	11	10	56	54	-4	0	11	8	1	-3
3	11	10	39	39	-3	0	11	14	18	-2
-3	12	10	41	48	-2	0	11	44	36	-1
-2	12	10	53	52	-1	0	11	51	47	1
-1	12	10	74	69	1	0	11	36	45	2
0	12	10	49	38	2	0	11	48	45	3
1	12	10	37	29	-9	1	11	43	47	-9
-3	13	10	48	47	-8	1	11	60	49	-8
-2	13	10	10	10	-7	1	11	143	145	-7
-1	13	10	18	23	-6	1	11	77	81	-6
0	13	10	31	29	-5	1	11	135	134	-5
1	13	10	10	24	-4	1	11	24	18	-4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	11		74	77	-3	4	11	129	140	2	7	11	37	31
1	11		120	124	-2	4	11	59	56	3	7	11	30	16
1	11		104	103	-1	4	11	84	83	-7	8	11	20	13
1	11		60	61	1	4	11	70	67	-6	8	11	53	46
1	11		28	20	2	4	11	27	19	-5	8	11	9	11
1	11		50	43	3	4	11	18	25	-4	8	11	67	62
2	11		52	55	-7	5	11	25	23	-3	8	11	81	87
2	11		91	89	-6	5	11	102	103	-2	8	11	49	57
2	11		189	189	-5	5	11	30	33	-1	8	11	10	29
2	11		118	119	-4	5	11	39	28	1	8	11	10	15
2	11		96	90	-3	5	11	133	147	2	8	11	49	51
2	11		102	102	-2	5	11	90	91	3	8	11	62	59
2	11		135	137	-1	5	11	93	88	-6	9	11	17	20
2	11		142	144	1	5	11	38	38	-5	9	11	47	49
2	11		78	71	2	5	11	58	61	-4	9	11	33	30
2	11		44	40	3	5	11	45	43	-3	9	11	28	28
2	11		16	22	-8	6	11	45	47	-2	9	11	81	85
2	11		10	25	-7	6	11	58	56	-1	9	11	113	108
3	11		66	63	-6	6	11	76	76	1	9	11	19	16
3	11		31	21	-5	6	11	39	40	2	9	11	36	34
3	11		92	95	-4	6	11	56	56	3	9	11	23	5
3	11		112	110	-3	6	11	79	86	-5	10	11	66	60
3	11		152	152	-2	6	11	55	48	-3	10	11	81	83
3	11		43	38	-1	6	11	48	56	-2	10	11	68	69
3	11		64	60	1	6	11	90	83	-1	10	11	107	99
3	11		48	44	2	6	11	38	34	1	10	11	43	41
3	11		34	28	3	6	11	35	36	2	10	11	48	43
3	11		54	51	-8	7	11	75	75	3	10	11	40	38
3	11		73	76	-7	7	11	70	75	-3	11	11	65	71
3	11		43	35	-6	7	11	25	35	-2	11	11	29	25
4	11		97	92	-5	7	11	53	54	-1	11	11	86	76
4	11		71	60	-4	7	11	117	119	1	11	11	24	36
4	11		77	75	-3	7	11	59	64	2	11	11	61	56
4	11		119	118	-2	7	11	70	69	3	11	11	30	27
4	11		55	47	-1	7	11	90	86	-3	12	11	62	59
4	11		17	23	1	7	11	37	28	-2	12	11	47	50

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-1	12	11	23	13	-6	1	12	65	68	-3
2	12	11	38	31	-5	1	12	52	50	-2
-3	13	11	16	20	-4	1	12	79	78	-1
-2	13	11	50	51	-3	1	12	76	80	1
-1	13	11	19	24	-2	1	12	42	47	2
1	13	11	10	18	-1	1	12	40	47	3
2	13	11	34	30	1	1	12	18	18	-8
3	13	11	39	36	2	1	12	61	54	-7
-3	14	11	65	66	3	1	12	11	15	-6
-2	14	11	10	18	-9	2	12	30	23	-5
-1	14	11	61	56	-8	2	12	162	163	-4
1	14	11	29	34	-7	2	12	200	203	-3
2	14	11	27	20	-6	2	12	45	45	-2
3	14	11	28	22	-5	2	12	136	132	-1
-3	15	11	18	24	-4	2	12	88	86	1
-2	15	11	21	15	-3	2	12	64	69	2
1	15	11	36	37	-2	2	12	94	91	3
2	15	11	28	22	-1	2	12	79	76	-7
-3	16	11	20	25	1	2	12	29	30	-6
-2	16	11	32	21	3	2	12	24	32	-5
-1	16	11	26	28	-8	3	12	71	65	-4
1	16	11	46	38	-7	3	12	9	11	-3
3	16	11	18	23	-6	3	12	31	24	-2
-8	0	12	71	72	-5	3	12	57	64	-1
-7	0	12	100	102	-4	3	12	28	32	1
-6	0	12	55	55	-3	3	12	15	25	2
-5	0	12	8	26	-2	3	12	9	17	3
-4	0	12	140	143	-1	3	12	87	84	-7
-2	0	12	80	79	1	3	12	75	68	-6
-1	0	12	38	37	2	3	12	39	39	-5
1	0	12	23	28	3	3	12	11	10	-4
3	0	12	51	50	-8	4	12	90	79	-3
4	0	12	22	15	-7	4	12	36	38	-2
-9	1	12	29	43	-6	4	12	65	58	-1
-8	1	12	42	43	-5	4	12	61	66	1
-7	1	12	67	67	-4	4	12	103	108	2

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	12		82	88	3	7	12	50	36	-1	14	12	10	24
4	12		150	149	-6	8	12	101	103	1	14	12	41	24
4	12		48	44	-5	8	12	26	24	2	14	12	18	17
4	12		54	58	-4	8	12	78	80	3	14	12	36	21
4	12		36	37	-3	8	12	41	40	-3	15	12	10	21
4	12		32	26	-2	8	12	73	70	-2	15	12	10	8
5	12		69	70	-1	8	12	109	109	-1	15	12	25	24
5	12		50	43	2	8	12	24	10	1	15	12	11	19
5	12		79	74	3	8	12	24	13	2	15	12	17	20
5	12		9	8	-3	9	12	24	19	3	15	12	22	4
5	12		76	76	-2	9	12	139	134	-3	16	12	36	33
5	12		82	86	-1	9	12	48	54	-2	16	12	32	31
5	12		56	63	1	9	12	30	32	-1	16	12	11	13
5	12		58	57	2	9	12	22	21	1	16	12	20	11
5	12		71	70	3	9	12	26	18	2	16	12	29	8
5	12		56	51	-3	10	12	20	28	3	16	12	38	24
5	12		17	30	-2	10	12	43	47	-8	0	13	124	125
6	12		9	9	-1	10	12	39	47	-7	0	13	124	123
6	12		79	80	1	10	12	10	13	-6	0	13	32	39
6	12		19	12	2	10	12	22	6	-5	0	13	124	121
6	12		34	24	-3	11	12	45	48	-3	0	13	71	66
6	12		63	79	-2	11	12	63	59	-2	0	13	19	10
6	12		66	66	1	11	12	33	29	-1	0	13	76	70
6	12		122	120	2	11	12	33	32	1	0	13	10	20
6	12		32	27	3	11	12	11	11	2	0	13	35	30
6	12		46	46	-3	12	12	33	29	3	0	13	61	56
6	12		11	10	-2	12	12	49	46	-8	1	13	88	88
7	12		31	19	1	12	12	27	24	-7	1	13	34	41
7	12		63	67	2	12	12	42	28	-6	1	13	47	43
7	12		50	58	-3	13	12	63	63	-5	1	13	132	137
7	12		61	58	-2	13	12	21	23	-4	1	13	47	40
7	12		81	85	-1	13	12	34	31	-3	1	13	38	45
7	12		68	68	1	13	12	30	26	-2	1	13	68	72
7	12		61	73	3	13	12	28	12	-1	1	13	119	115
7	12		10	5	-3	14	12	23	26	1	1	13	23	17
7	12		21	17	-2	14	12	46	44	3	1	13	16	10

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
-8	2	13	92	89	1	5	13	17	18
-7	2	13	26	31	2	5	13	38	19
-6	2	13	54	51	-6	6	13	45	51
-5	2	13	82	82	-4	6	13	70	79
-4	2	13	35	33	-3	6	13	65	78
-3	2	13	32	29	-2	6	13	80	86
-2	2	13	56	53	-1	6	13	39	45
-1	2	13	89	87	1	6	13	10	24
1	2	13	10	30	2	6	13	36	26
2	2	13	27	14	3	6	13	23	6
3	2	13	35	41	-3	7	13	33	43
-8	3	13	22	12	-2	7	13	54	50
-7	3	13	34	31	-1	7	13	55	53
-6	3	13	66	69	2	7	13	24	22
-5	3	13	74	73	3	7	13	29	17
-3	3	13	47	41	-3	8	13	62	70
-2	3	13	49	37	-2	8	13	27	33
-1	3	13	168	168	-1	8	13	58	46
1	3	13	35	23	1	8	13	53	41
-7	4	13	59	58	2	8	13	33	30
-6	4	13	36	41	3	8	13	30	25
-5	4	13	49	56	-3	9	13	68	70
-4	4	13	42	53	-2	9	13	10	7
-3	4	13	79	82	-1	9	13	41	25
-2	4	13	28	25	1	9	13	31	27
-1	4	13	83	93	2	9	13	31	26
1	4	13	10	21	-3	10	13	44	41
2	4	13	22	19	-2	10	13	52	53
3	4	13	29	29	-1	10	13	23	31
-7	5	13	65	60	1	10	13	42	37
-6	5	13	92	86	2	10	13	11	21
-5	5	13	28	37	3	10	13	30	20
-4	5	13	75	77	-3	11	13	64	60
-3	5	13	97	99	-1	11	13	17	25
-2	5	13	80	76	1	11	13	50	42
-1	5	13	40	46	-3	12	13	40	38

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-2	12	13	21	14	-6	2	14	88	89	2	7	14	35	26
-1	12	13	16	9	-5	2	14	53	50	3	7	14	12	19
1	12	13	37	33	-4	2	14	31	39	-3	8	14	30	30
2	12	13	11	23	-3	2	14	61	60	-2	8	14	39	37
3	12	13	18	9	-2	2	14	33	38	-1	8	14	27	22
-3	13	13	27	23	-1	2	14	10	23	1	8	14	36	38
1	13	13	19	2	1	2	14	33	33	2	8	14	32	12
3	13	13	29	3	2	2	14	31	28	3	8	14	24	12
-2	14	13	35	30	3	2	14	45	32	-3	9	14	26	10
1	15	13	28	19	-6	3	14	32	30	-2	9	14	25	33
3	15	13	19	17	-5	3	14	37	39	-1	9	14	37	37
-3	16	13	19	15	-3	3	14	41	37	1	9	14	48	40
-1	16	13	39	31	-2	3	14	38	49	2	9	14	11	24
2	16	13	25	15	-1	3	14	46	38	-3	10	14	53	52
3	16	13	24	10	1	3	14	11	24	-2	10	14	26	22
-6	0	14	59	52	2	3	14	17	36	-1	10	14	23	32
-5	0	14	62	63	3	3	14	32	7	1	10	14	26	26
-4	0	14	26	39	-3	4	14	27	34	2	10	14	31	12
-3	0	14	63	67	-2	4	14	93	95	3	10	14	12	5
-2	0	14	72	67	-1	4	14	14	12	-3	11	14	10	22
-1	0	14	71	66	1	4	14	25	29	-2	11	14	31	31
1	0	14	18	10	2	4	14	56	48	-1	11	14	11	10
2	0	14	76	69	3	4	14	21	25	1	11	14	37	36
3	0	14	23	3	-3	5	14	35	29	2	11	14	36	10
-7	1	14	61	69	-1	5	14	46	46	3	11	14	12	7
-6	1	14	70	72	1	5	14	11	10	-3	12	14	10	9
-5	1	14	102	106	2	5	14	23	18	-1	12	14	36	29
-4	1	14	37	36	3	5	14	30	15	1	12	14	41	36
-3	1	14	91	97	-3	6	14	47	46	2	12	14	25	15
-2	1	14	18	14	-2	6	14	81	81	3	12	14	25	12
-1	1	14	75	74	-1	6	14	63	65	-3	13	14	28	25
1	1	14	21	8	3	6	14	40	20	-2	13	14	38	32
2	1	14	20	21	-3	7	14	20	11	-1	13	14	11	6
3	1	14	25	23	-2	7	14	62	61	1	13	14	27	18
4	1	14	20	12	-1	7	14	64	63	2	13	14	30	11
-7	2	14	80	78	1	7	14	41	24	3	13	14	25	20

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-2	14	14	23	8	-2	1	15	29	21	-1
1	14	14	23	22	-1	1	15	45	51	1
2	14	14	25	4	2	1	15	22	17	2
3	14	14	28	6	3	1	15	34	16	3
-2	15	14	29	18	4	1	15	12	22	-3
2	15	14	27	13	-4	2	15	52	45	-2
3	15	14	28	12	-3	2	15	26	24	-1
-3	16	14	11	16	-2	2	15	10	6	1
-2	16	14	37	23	-1	2	15	52	52	2
-1	16	14	12	14	1	2	15	35	33	3
1	16	14	22	10	2	2	15	35	27	-3
2	16	14	28	14	3	2	15	28	16	-2
-4	0	15	68	65	-3	3	15	63	64	-1
-3	0	15	54	51	-2	3	15	43	21	1
-2	0	15	10	3	-1	3	15	30	33	3
-1	0	15	31	32	1	3	15	47	46	-3
2	0	15	24	3	2	3	15	34	22	-2
4	0	15	12	12	3	3	15	22	16	-1
-4	1	15	40	43	-3	4	15	53	55	1
-3	1	15	60	63	-2	4	15	15	22	3

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	15	31	19	-3	8	15	38	34	-1	12	15	32	29
4	15	59	58	-2	8	15	35	38	1	12	15	20	8
4	15	39	20	-1	8	15	43	28	-3	13	15	11	19
4	15	20	15	1	8	15	11	10	-2	13	15	19	4
5	15	16	21	2	8	15	31	23	1	13	15	30	15
5	15	77	76	3	8	15	12	6	2	13	15	26	14
5	15	31	28	-3	9	15	10	15	3	13	15	22	5
5	15	11	27	-2	9	15	15	19	-2	14	15	18	12
5	15	34	28	1	9	15	42	26	-1	14	15	11	17
5	15	28	10	2	9	15	11	9	1	14	15	23	3
6	15	100	101	-3	10	15	61	60	2	14	15	12	11
6	15	36	36	-2	10	15	34	13	-3	15	15	11	16
6	15	32	27	1	10	15	24	6	-2	15	15	25	16
6	15	33	25	3	10	15	27	6	-1	15	15	12	17
6	15	17	11	-3	11	15	26	20	1	15	15	31	1
7	15	20	21	-2	11	15	31	22	2	15	15	20	14
7	15	65	60	1	11	15	11	8	-3	16	15	27	19
7	15	19	5	2	11	15	20	6	-2	16	15	23	8
7	15	23	21	-3	12	15	20	9	-1	16	15	18	7
7	15	20	14	-2	12	15	31	34					

LEUCOACETATE OF VENTILATONE A

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	0	0	114	127	3	2	0	281	283	2
3	0	0	142	161	4	2	0	117	123	3
4	0	0	364	383	5	2	0	23	32	5
5	0	0	31	37	6	2	0	36	23	6
6	0	0	44	49	7	2	0	194	204	7
7	0	0	34	27	8	2	0	20	24	9
9	0	0	20	12	9	2	0	75	66	-8
10	0	0	23	16	-9	3	0	22	14	-7
-9	1	0	20	35	-7	3	0	31	29	-6
-8	1	0	120	127	-6	3	0	32	31	-5
-7	1	0	38	37	-5	3	0	64	65	-4
-6	1	0	44	41	-4	3	0	99	107	-3
-5	1	0	58	70	-3	3	0	199	209	-2
-4	1	0	582	603	-2	3	0	184	175	-1
-3	1	0	181	188	-1	3	0	29	35	0
-2	1	0	231	229	0	3	0	268	253	1
-1	1	0	386	397	1	3	0	211	217	2
0	1	0	701	682	2	3	0	78	83	4
1	1	0	188	185	3	3	0	746	737	7
2	1	0	263	298	4	3	0	92	84	8
3	1	0	178	180	5	3	0	92	94	9
5	1	0	76	81	6	3	0	63	64	-8
6	1	0	142	134	7	3	0	364	367	-6
7	1	0	76	71	8	3	0	75	73	-5
-9	2	0	23	27	9	3	0	32	27	-4
-8	2	0	128	132	-9	4	0	30	30	-2
-7	2	0	61	55	-8	4	0	62	60	-1
-6	2	0	68	73	-7	4	0	31	31	0
-5	2	0	35	40	-6	4	0	32	41	1
-4	2	0	40	38	-5	4	0	118	123	2
-3	2	0	99	110	-4	4	0	69	68	3
-2	2	0	258	257	-3	4	0	144	136	4
-1	2	0	141	143	-2	4	0	232	227	5
0	2	0	320	300	-1	4	0	520	494	6
1	2	0	68	67	0	4	0	118	116	8
2	2	0	157	174	1	4	0	129	126	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	0	0	203	201	-6	7	0	72	65	4	10	0	35	40
4	0	0	462	456	-5	7	0	77	81	5	10	0	31	32
4	0	0	101	98	-4	7	0	54	61	-1	11	0	18	13
4	0	0	93	91	-2	7	0	34	38	1	11	0	36	38
4	0	0	153	158	-1	7	0	109	108	-4	-11	1	58	54
4	0	0	25	22	0	7	0	30	31	-1	-11	1	41	44
5	0	0	21	28	2	7	0	219	226	0	-11	1	26	36
5	0	0	41	40	3	7	0	43	46	2	-11	1	19	17
5	0	0	170	166	4	7	0	69	67	-5	-10	1	30	32
5	0	0	366	379	6	7	0	48	60	-4	-10	1	46	43
5	0	0	41	45	7	7	0	28	33	-3	-10	1	44	49
5	0	0	73	77	-5	8	0	19	22	-1	-10	1	32	37
5	0	0	185	172	-4	8	0	36	39	1	-10	1	29	24
5	0	0	529	488	-3	8	0	112	121	4	-10	1	43	41
5	0	0	418	408	-2	8	0	63	63	-6	-9	1	28	29
5	0	0	79	71	-1	8	0	22	20	-3	-9	1	105	101
5	0	0	40	46	1	8	0	205	212	-2	-9	1	57	55
5	0	0	32	26	2	8	0	249	251	-1	-9	1	33	32
5	0	0	19	27	3	8	0	197	196	1	-9	1	103	99
5	0	0	28	27	5	8	0	45	37	2	-9	1	50	56
5	0	0	19	18	6	8	0	73	70	3	-9	1	77	81
6	0	0	27	39	-5	9	0	28	31	4	-9	1	22	18
6	0	0	54	53	-4	9	0	26	23	5	-9	1	39	40
6	0	0	204	212	-3	9	0	77	73	-6	-8	1	69	65
6	0	0	80	80	-2	9	0	111	114	-5	-8	1	46	47
6	0	0	28	36	-1	9	0	128	128	-3	-8	1	112	112
6	0	0	61	56	1	9	0	58	64	-2	-8	1	32	32
6	0	0	95	88	2	9	0	93	95	0	-8	1	36	41
6	0	0	118	120	4	9	0	51	51	1	-8	1	27	23
6	0	0	177	179	5	9	0	42	33	2	-8	1	75	69
6	0	0	129	120	-4	10	0	22	27	3	-8	1	107	110
6	0	0	78	75	-2	10	0	20	14	4	-8	1	67	71
6	0	0	32	29	-1	10	0	80	81	6	-8	1	51	57
6	0	0	45	47	1	10	0	41	43	-7	-7	1	19	11
6	0	0	18	35	2	10	0	26	29	-6	-7	1	80	73
7	0	0	19	17	3	10	0	26	22	-5	-7	1	20	20

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-4	-7	1	39	40	-9	-4	1	25	28	-3
-3	-7	1	29	28	-8	-4	1	41	32	-2
-2	-7	1	88	95	-7	-4	1	100	99	-1
-1	-7	1	165	166	-6	-4	1	35	36	0
1	-7	1	28	26	-5	-4	1	79	78	2
3	-7	1	60	56	-4	-4	1	101	97	3
4	-7	1	44	44	-3	-4	1	226	234	5
6	-7	1	31	33	-2	-4	1	79	83	6
-7	-6	1	27	23	-1	-4	1	150	150	7
-6	-6	1	61	54	0	-4	1	386	368	9
-5	-6	1	55	47	2	-4	1	293	272	-10
-3	-6	1	81	86	3	-4	1	74	76	-9
-2	-6	1	72	75	4	-4	1	69	72	-8
-1	-6	1	89	88	5	-4	1	235	241	-7
0	-6	1	80	85	7	-4	1	30	25	-6
1	-6	1	78	81	-9	-3	1	30	35	-5
2	-6	1	69	74	-6	-3	1	74	69	-4
5	-6	1	57	65	-5	-3	1	58	53	-3
6	-6	1	66	66	-4	-3	1	100	92	-2
7	-6	1	78	66	-3	-3	1	66	78	-1
8	-6	1	40	33	-2	-3	1	102	102	0
-9	-5	1	31	27	-1	-3	1	78	82	1
-8	-5	1	21	13	0	-3	1	97	100	2
-7	-5	1	34	35	1	-3	1	226	218	3
-6	-5	1	51	42	2	-3	1	352	342	4
-5	-5	1	98	95	3	-3	1	110	115	5
-4	-5	1	66	69	4	-3	1	63	63	6
-3	-5	1	116	112	5	-3	1	56	61	7
-1	-5	1	35	33	7	-3	1	72	71	8
0	-5	1	180	173	8	-3	1	34	32	-7
1	-5	1	129	125	9	-3	1	18	13	-6
2	-5	1	212	200	-9	-2	1	37	29	-5
3	-5	1	107	105	-7	-2	1	33	30	-4
4	-5	1	182	186	-6	-2	1	62	51	-3
5	-5	1	148	148	-5	-2	1	113	118	-2
7	-5	1	52	51	-4	-2	1	115	120	-1

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-2	1		77	84	0	0	1	305	292	1	2	1	290	279
-2	1		363	390	1	0	1	283	294	2	2	1	429	461
-2	1		260	273	2	0	1	427	460	3	2	1	168	161
-2	1		272	256	3	0	1	220	215	4	2	1	200	194
-2	1		388	388	4	0	1	239	228	5	2	1	22	32
-2	1		187	202	5	0	1	64	58	6	2	1	150	151
-2	1		58	61	6	0	1	117	116	7	2	1	38	41
-2	1		27	24	7	0	1	22	25	8	2	1	27	22
-2	1		20	22	8	0	1	68	58	9	2	1	64	57
-2	1		36	32	9	0	1	37	31	-9	3	1	33	35
-1	1		24	14	-9	1	1	74	81	-8	3	1	45	48
-1	1		28	20	-7	1	1	22	24	-7	3	1	20	23
-1	1		26	31	-6	1	1	34	41	-5	3	1	25	27
-1	1		73	68	-5	1	1	32	18	-4	3	1	75	72
-1	1		51	44	-4	1	1	50	43	-3	3	1	217	209
-1	1		90	92	-3	1	1	83	45	-2	3	1	75	65
-1	1		102	95	-2	1	1	57	53	-1	3	1	129	122
-1	1		76	84	-1	1	1	248	243	0	3	1	46	47
-1	1		63	62	0	1	1	276	263	1	3	1	96	88
-1	1		658	678	1	1	1	130	133	2	3	1	436	439
-1	1		174	169	2	1	1	784	849	3	3	1	67	64
-1	1		495	506	3	1	1	84	79	4	3	1	114	109
-1	1		383	383	4	1	1	118	111	5	3	1	120	112
-1	1		164	170	5	1	1	177	174	6	3	1	265	269
-1	1		209	204	6	1	1	59	60	7	3	1	172	169
-1	1		88	88	7	1	1	67	67	8	3	1	44	39
-1	1		89	86	8	1	1	19	16	-7	4	1	21	24
-1	1		52	48	9	1	1	23	24	-6	4	1	228	224
-1	1		80	88	-9	2	1	44	55	-4	4	1	126	122
0	1		37	34	-7	2	1	28	43	-2	4	1	628	583
0	1		71	80	-5	2	1	49	63	-1	4	1	49	49
0	1		167	187	-4	2	1	105	96	0	4	1	135	128
0	1		34	35	-3	2	1	111	119	1	4	1	178	187
0	1		277	325	-2	2	1	285	276	2	4	1	94	83
0	1		238	245	-1	2	1	266	263	3	4	1	202	193
0	1		560	643	0	2	1	205	211	4	4	1	49	44

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	4	1	134	129	-2	7	1	72	65
6	4	1	23	36	-1	7	1	66	57
7	4	1	82	93	0	7	1	68	73
9	4	1	28	21	1	7	1	383	390
-8	5	1	24	18	4	7	1	34	39
-6	5	1	318	316	5	7	1	199	199
-5	5	1	94	91	7	7	1	28	22
-4	5	1	85	81	-6	8	1	22	17
-3	5	1	107	108	-4	8	1	36	38
-2	5	1	275	261	-3	8	1	187	187
-1	5	1	87	86	-1	8	1	47	48
0	5	1	128	128	0	8	1	30	37
1	5	1	24	23	1	8	1	490	504
2	5	1	117	122	2	8	1	52	51
3	5	1	128	128	3	8	1	43	44
4	5	1	47	47	4	8	1	53	55
5	5	1	66	67	5	8	1	116	117
6	5	1	28	40	-5	9	1	20	19
8	5	1	31	28	-3	9	1	126	117
-8	6	1	24	23	-2	9	1	50	51
-7	6	1	31	23	-1	9	1	19	16
-6	6	1	61	57	0	9	1	29	32
-5	6	1	100	98	1	9	1	139	144
-4	6	1	25	30	2	9	1	28	23
-3	6	1	82	80	3	9	1	42	42
-2	6	1	60	56	4	9	1	32	35
-1	6	1	82	83	-1	10	1	20	27
0	6	1	50	47	-1	10	1	26	23
1	6	1	158	157	1	10	1	92	91
3	6	1	20	16	2	10	1	41	40
4	6	1	57	56	5	10	1	29	24
6	6	1	23	17	-1	11	1	23	24
-7	7	1	19	12	0	11	1	44	41
-6	7	1	67	69	1	11	1	30	37
-5	7	1	48	48	2	11	1	18	18
-3	7	1	31	32	3	11	1	39	37

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-4	-11	2	58	60	1	-7	2	55	49	-7	-4	2	132	133
-2	-11	2	18	19	2	-7	2	69	63	-6	-4	2	21	34
-1	-11	2	20	29	4	-7	2	107	108	-5	-4	2	54	52
1	-11	2	36	32	5	-7	2	47	52	-4	-4	2	42	39
2	-11	2	31	22	6	-7	2	18	25	-3	-4	2	122	128
-4	-10	2	47	48	-7	-6	2	31	32	-2	-4	2	103	107
-3	-10	2	21	23	-5	-6	2	39	23	-1	-4	2	28	27
-1	-10	2	21	18	-4	-6	2	34	32	0	-4	2	72	62
2	-10	2	32	24	-3	-6	2	76	80	1	-4	2	337	321
3	-10	2	40	40	-2	-6	2	72	74	2	-4	2	214	206
-6	-9	2	69	57	-1	-6	2	108	103	3	-4	2	22	10
-5	-9	2	48	42	0	-6	2	105	110	4	-4	2	111	116
-4	-9	2	59	55	1	-6	2	36	34	5	-4	2	140	138
-3	-9	2	54	54	2	-6	2	47	49	6	-4	2	25	26
-2	-9	2	283	290	3	-6	2	31	29	7	-4	2	71	69
-1	-9	2	44	53	5	-6	2	265	267	8	-4	2	65	67
0	-9	2	40	41	6	-6	2	54	49	-8	-3	2	72	65
1	-9	2	32	40	7	-6	2	22	26	-7	-3	2	170	176
2	-9	2	154	144	-9	-5	2	23	22	-6	-3	2	76	64
3	-9	2	19	20	-8	-5	2	28	38	-5	-3	2	54	53
-6	-8	2	128	128	-6	-5	2	42	40	-4	-3	2	50	47
-4	-8	2	119	120	-5	-5	2	47	45	-3	-3	2	269	281
-3	-8	2	73	76	-4	-5	2	64	68	-2	-3	2	103	113
-2	-8	2	370	389	-3	-5	2	98	98	0	-3	2	468	450
-1	-8	2	131	132	-2	-5	2	48	54	1	-3	2	114	113
0	-8	2	46	42	-1	-5	2	42	46	2	-3	2	160	149
1	-8	2	73	74	0	-5	2	192	190	3	-3	2	81	79
2	-8	2	126	126	1	-5	2	640	595	4	-3	2	184	187
3	-8	2	44	44	2	-5	2	30	28	7	-3	2	39	48
4	-8	2	47	48	3	-5	2	50	51	8	-3	2	46	45
6	-8	2	21	32	4	-5	2	63	63	-10	-2	2	31	29
-8	-7	2	50	37	5	-5	2	278	284	-9	-2	2	27	12
-6	-7	2	83	78	6	-5	2	72	62	-7	-2	2	57	66
-4	-7	2	31	26	7	-5	2	32	32	-6	-2	2	69	85
-2	-7	2	168	169	-9	-4	2	37	30	-5	-2	2	25	37
0	-7	2	95	95	-8	-4	2	83	81	-3	-2	2	120	124

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-2	-2	2	183	193	1	0	2	563	559	4
-1	-2	2	583	581	2	0	2	164	165	5
0	-2	2	152	137	3	0	2	219	212	7
1	-2	2	206	199	4	0	2	251	246	-9
2	-2	2	88	91	5	0	2	110	109	-7
3	-2	2	52	58	6	0	2	90	91	-6
6	-2	2	22	26	7	0	2	50	48	-5
7	-2	2	38	42	-9	1	2	19	28	-4
8	-2	2	98	100	-7	1	2	54	49	-3
9	-2	2	42	36	-6	1	2	44	58	-2
-10	-1	2	31	29	-5	1	2	174	189	-1
-8	-1	2	22	40	-4	1	2	66	75	0
-7	-1	2	69	73	-3	1	2	98	106	1
-5	-1	2	38	34	-2	1	2	119	112	2
-4	-1	2	36	44	-1	1	2	694	699	3
-3	-1	2	156	179	0	1	2	221	208	4
-2	-1	2	79	86	1	1	2	150	147	5
-1	-1	2	415	412	2	1	2	478	501	6
0	-1	2	409	404	3	1	2	120	113	8
1	-1	2	255	250	4	1	2	157	146	9
2	-1	2	191	198	5	1	2	47	44	-9
3	-1	2	90	93	6	1	2	98	91	-8
4	-1	2	89	107	7	1	2	42	39	-7
5	-1	2	70	63	8	1	2	81	64	-6
6	-1	2	62	55	-9	2	2	18	17	-5
7	-1	2	37	46	-8	2	2	62	61	-4
8	-1	2	50	55	-7	2	2	59	52	-3
-10	0	2	24	26	-5	2	2	103	105	-2
-9	0	2	29	50	-4	2	2	165	177	-1
-6	0	2	23	34	-3	2	2	115	118	0
-5	0	2	135	138	-2	2	2	126	107	1
-4	0	2	179	222	-1	2	2	65	72	2
-3	0	2	71	90	0	2	2	52	51	3
-2	0	2	593	631	1	2	2	640	629	5
-1	0	2	374	375	2	2	2	66	72	6
0	0	2	442	429	3	2	2	107	115	7

K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	2	325	303	8	4	2	19	23	3	7	2	95	99
2	2	27	17	9	4	2	26	30	4	7	2	89	82
2	2	27	38	-8	5	2	20	20	5	7	2	116	119
3	2	21	24	-7	5	2	76	60	-6	8	2	33	41
3	2	28	35	-6	5	2	81	77	-4	8	2	41	46
3	2	101	103	-5	5	2	25	29	-3	8	2	56	53
3	2	134	150	-4	5	2	35	25	-2	8	2	68	70
3	2	44	46	-3	5	2	63	71	-1	8	2	52	50
3	2	129	116	-2	5	2	213	208	1	8	2	163	169
3	2	73	63	-1	5	2	37	31	2	8	2	109	113
3	2	141	136	0	5	2	230	230	3	8	2	30	29
3	2	46	44	2	5	2	192	188	4	8	2	22	16
3	2	143	130	3	5	2	97	107	5	8	2	34	41
3	2	268	268	4	5	2	162	163	6	8	2	33	30
3	2	76	72	5	5	2	19	17	7	8	2	60	55
3	2	141	139	7	5	2	36	36	-4	9	2	46	46
3	2	131	121	8	5	2	59	54	-3	9	2	24	21
3	2	172	167	-8	6	2	23	14	-2	9	2	76	65
3	2	58	46	-7	6	2	58	53	0	9	2	27	33
3	2	21	24	-5	6	2	52	54	1	9	2	25	26
4	2	25	11	-4	6	2	52	55	2	9	2	25	17
4	2	20	19	-3	6	2	65	63	-1	10	2	23	26
4	2	45	40	-2	6	2	124	119	0	10	2	51	45
4	2	76	72	-1	6	2	170	165	2	10	2	18	11
4	2	42	38	0	6	2	75	69	3	10	2	50	51
4	2	98	107	4	6	2	45	46	4	10	2	33	34
4	2	32	33	6	6	2	29	28	-4	-11	3	29	33
4	2	76	79	7	6	2	37	34	-3	-11	3	31	28
4	2	190	183	8	6	2	17	17	-1	-11	3	20	18
4	2	152	142	-4	7	2	100	98	0	-11	3	45	44
4	2	314	310	-3	7	2	55	66	1	-11	3	20	19
4	2	66	70	-2	7	2	37	32	2	-11	3	46	43
4	2	39	34	-1	7	2	28	21	-5	-10	3	68	57
4	2	65	60	0	7	2	117	122	-3	-10	3	45	48
4	2	125	127	1	7	2	84	88	-2	-10	3	87	93
4	2	52	55	2	7	2	109	114	-1	-10	3	45	42

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	-10	3	34	34	0	-7	3	29	27	-8
1	-10	3	53	54	1	-7	3	118	116	-7
2	-10	3	24	26	2	-7	3	111	108	-6
3	-10	3	20	24	3	-7	3	19	18	-5
4	-10	3	21	21	5	-7	3	65	61	-4
-7	-9	3	51	40	-7	-6	3	40	32	-3
-4	-9	3	96	92	-6	-6	3	52	53	-2
-3	-9	3	132	128	-5	-6	3	41	29	-1
-2	-9	3	170	177	-4	-6	3	32	32	0
0	-9	3	55	49	-3	-6	3	56	60	1
1	-9	3	135	141	-2	-6	3	108	109	2
2	-9	3	164	162	-1	-6	3	60	56	3
3	-9	3	35	41	0	-6	3	125	119	4
4	-9	3	22	26	1	-6	3	134	128	6
5	-9	3	64	57	2	-6	3	41	39	7
-7	-8	3	24	27	3	-6	3	64	65	8
-6	-8	3	50	52	4	-6	3	305	303	-9
-5	-8	3	59	53	5	-6	3	127	130	-8
-3	-8	3	228	228	6	-6	3	36	33	-7
-2	-8	3	274	275	7	-6	3	48	53	-6
-1	-8	3	65	69	-8	-5	3	72	62	-5
0	-8	3	76	79	-7	-5	3	27	20	-4
1	-8	3	71	69	-6	-5	3	45	51	-3
2	-8	3	69	71	-5	-5	3	77	77	-2
3	-8	3	27	43	-4	-5	3	175	175	-1
4	-8	3	45	45	-3	-5	3	80	78	0
5	-8	3	48	52	-2	-5	3	61	60	1
6	-8	3	33	37	-1	-5	3	41	30	2
-8	-7	3	20	23	0	-5	3	385	367	4
-7	-7	3	63	68	1	-5	3	229	213	5
-6	-7	3	44	22	2	-5	3	174	173	7
-5	-7	3	36	28	4	-5	3	313	311	9
-4	-7	3	65	62	5	-5	3	219	229	-10
-3	-7	3	27	36	7	-5	3	54	51	-9
-2	-7	3	144	145	8	-5	3	27	29	-8
-1	-7	3	25	23	-9	-4	3	39	42	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-4	3		199	181	-5	-2	3	56	71	-2	0	3	76	69
-4	3		42	47	-4	-2	3	150	165	-1	0	3	1592	1629
-4	3		67	65	-3	-2	3	134	139	0	0	3	335	325
-4	3		47	50	-2	-2	3	212	209	2	0	3	130	137
-4	3		412	422	-1	-2	3	301	294	3	0	3	429	411
-4	3		184	183	0	-2	3	309	292	4	0	3	107	102
-4	3		56	57	1	-2	3	63	54	5	0	3	95	92
-4	3		76	71	2	-2	3	123	126	6	0	3	23	19
-4	3		290	269	3	-2	3	150	130	7	0	3	24	29
-4	3		121	119	4	-2	3	55	48	-9	1	3	70	107
-4	3		80	81	5	-2	3	43	37	-8	1	3	32	31
-4	3		213	215	6	-2	3	74	81	-7	1	3	80	70
-4	3		115	120	7	-2	3	158	173	-5	1	3	128	135
-4	3		33	36	8	-2	3	27	33	-4	1	3	63	60
-4	3		33	39	9	-2	3	26	20	-3	1	3	192	203
-4	3		18	16	-7	-1	3	84	80	-2	1	3	167	155
-3	3		27	33	-6	-1	3	23	32	-1	1	3	321	311
-3	3		264	234	-5	-1	3	158	172	0	1	3	262	239
-3	3		77	65	-4	-1	3	38	28	1	1	3	422	416
-3	3		28	31	-3	-1	3	68	75	2	1	3	213	226
-3	3		64	71	-2	-1	3	151	154	3	1	3	169	171
-3	3		130	132	-1	-1	3	1528	1548	4	1	3	108	103
-3	3		99	102	0	-1	3	217	210	5	1	3	92	100
-3	3		31	25	1	-1	3	705	688	6	1	3	21	28
-3	3		55	53	2	-1	3	217	213	8	1	3	58	55
-3	3		175	156	3	-1	3	553	517	-9	2	3	44	45
-3	3		75	68	4	-1	3	65	66	-7	2	3	44	37
-3	3		56	30	6	-1	3	61	64	-6	2	3	28	36
-3	3		208	200	7	-1	3	69	81	-5	2	3	30	27
-3	3		53	55	8	-1	3	20	10	-4	2	3	57	37
-3	3		51	55	-9	0	3	29	46	-3	2	3	133	136
-3	3		24	22	-7	0	3	24	25	-2	2	3	139	132
-2	3		34	31	-6	0	3	27	33	0	2	3	358	337
-2	3		29	19	-5	0	3	357	381	1	2	3	145	136
-2	3		25	46	-4	0	3	73	82	2	2	3	285	277
-2	3		37	31	-3	0	3	358	406	3	2	3	208	204

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR V

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K
4	2	3	197	191	8	4	3	39	32	5	7
5	2	3	35	55	9	4	3	43	33	6	7
6	2	3	343	340	-7	5	3	91	89	7	7
7	2	3	69	69	-6	5	3	211	211	-6	8
8	2	3	63	57	-5	5	3	120	125	-5	8
9	2	3	42	37	-4	5	3	76	74	-4	8
-9	3	3	35	37	-3	5	3	95	91	-3	8
-7	3	3	58	54	-2	5	3	254	245	-2	8
-5	3	3	133	139	-1	5	3	25	28	-1	8
-4	3	3	88	96	0	5	3	207	203	0	8
-3	3	3	79	70	1	5	3	83	82	1	8
-2	3	3	225	222	3	5	3	27	23	2	8
-1	3	3	150	137	5	5	3	75	66	3	8
0	3	3	49	54	8	5	3	45	43	5	8
1	3	3	174	161	-6	6	3	26	26	6	8
2	3	3	746	740	-5	6	3	56	53	-5	9
3	3	3	278	279	-4	6	3	55	66	-4	9
4	3	3	131	131	-3	6	3	109	115	-3	9
5	3	3	96	104	-2	6	3	47	54	-2	9
6	3	3	403	388	-1	6	3	74	71	-1	9
7	3	3	87	87	0	6	3	23	27	1	9
-8	4	3	35	38	1	6	3	127	127	3	9
-7	4	3	67	67	2	6	3	172	177	5	9
-6	4	3	309	296	3	6	3	102	104	-2	10
-5	4	3	64	68	5	6	3	27	34	-1	10
-4	4	3	42	36	7	6	3	24	27	0	10
-3	4	3	51	36	-7	7	3	21	17	2	10
-2	4	3	267	260	-6	7	3	27	19	3	10
-1	4	3	162	164	-3	7	3	96	91	-2	-11
0	4	3	93	89	-2	7	3	96	89	-1	-11
2	4	3	220	209	-1	7	3	89	93	0	-11
3	4	3	64	58	0	7	3	82	82	-5	-10
4	4	3	27	28	1	7	3	228	233	-4	-10
5	4	3	79	86	2	7	3	128	129	-3	-10
6	4	3	20	34	3	7	3	73	80	-2	-10
7	4	3	20	26	4	7	3	23	29	-1	-10

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	-10	4	27	33	6	-7	4	55	52	3	50	60		
2	-10	4	26	33	-8	-6	4	38	30	3	35	39		
-6	-9	4	23	25	-7	-6	4	40	28	3	21	16		
-5	-9	4	25	26	-6	-6	4	33	29	3	25	20		
-4	-9	4	72	64	-5	-6	4	42	36	3	71	68		
-3	-9	4	81	78	-4	-6	4	47	51	3	102	103		
-2	-9	4	29	35	-3	-6	4	55	61	3	85	78		
-1	-9	4	30	34	-2	-6	4	70	73	3	86	76		
0	-9	4	154	158	-1	-6	4	44	50	3	48	47		
1	-9	4	108	105	0	-6	4	173	175	3	136	140		
2	-9	4	28	29	1	-6	4	26	25	3	169	175		
3	-9	4	59	63	2	-6	4	59	61	3	143	151		
5	-9	4	51	57	3	-6	4	200	202	3	61	66		
-7	-8	4	24	17	4	-6	4	247	239	3	65	63		
-5	-8	4	42	34	5	-6	4	33	36	3	46	45		
-4	-8	4	99	97	7	-6	4	26	24	3	25	29		
-3	-8	4	26	40	-8	-5	4	47	41	3	61	65		
-2	-8	4	132	135	-7	-5	4	37	28	3	33	32		
-1	-8	4	20	32	-6	-5	4	42	43	3	40	38		
0	-8	4	20	24	-5	-5	4	38	45	3	27	31		
2	-8	4	58	61	-4	-5	4	26	20	3	52	54		
3	-8	4	51	57	-3	-5	4	119	123	3	27	25		
4	-8	4	24	30	-2	-5	4	169	170	3	40	37		
5	-8	4	37	36	-1	-5	4	166	166	3	35	38		
6	-8	4	21	13	0	-5	4	64	38	3	27	24		
-6	-7	4	31	22	1	-5	4	58	65	3	20	12		
-5	-7	4	66	65	2	-5	4	48	50	3	24	20		
-3	-7	4	23	23	3	-5	4	23	21	3	33	37		
-2	-7	4	45	55	4	-5	4	275	269	4	28	26		
-1	-7	4	38	45	6	-5	4	44	53	4	50	51		
0	-7	4	109	113	7	-5	4	28	27	4	39	38		
1	-7	4	95	97	8	-5	4	39	23	4	25	28		
2	-7	4	32	43	-9	-4	4	29	25	4	24	25		
3	-7	4	94	95	-7	-4	4	47	44	4	24	26		
4	-7	4	25	21	-6	-4	4	40	44	4	54	56		
5	-7	4	35	37	-5	-4	4	40	50	4	27	37		

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-4	-4	4	231	228	0	-2	4	307	286	5
-3	-4	4	138	138	1	-2	4	138	139	6
-2	-4	4	46	50	2	-2	4	137	126	7
-1	-4	4	94	92	3	-2	4	233	226	8
0	-4	4	66	53	4	-2	4	21	22	9
1	-4	4	167	163	5	-2	4	36	30	-7
2	-4	4	102	105	7	-2	4	94	101	-6
3	-4	4	35	36	8	-2	4	59	50	-5
4	-4	4	57	60	-7	-1	4	60	50	-4
5	-4	4	35	36	-6	-1	4	111	133	-3
6	-4	4	58	61	-5	-1	4	60	56	-2
7	-4	4	18	19	-4	-1	4	92	89	-1
-8	-3	4	44	32	-3	-1	4	144	141	0
-7	-3	4	51	31	-2	-1	4	388	383	1
-6	-3	4	30	20	-1	-1	4	550	541	2
-5	-3	4	55	58	0	-1	4	36	28	3
-4	-3	4	34	35	1	-1	4	118	114	4
-3	-3	4	109	109	2	-1	4	196	196	5
-2	-3	4	167	177	3	-1	4	534	510	6
-1	-3	4	242	241	4	-1	4	32	34	9
0	-3	4	130	120	5	-1	4	23	24	-9
1	-3	4	82	68	6	-1	4	20	15	-8
2	-3	4	236	239	7	-1	4	108	112	-7
3	-3	4	201	202	9	-1	4	33	31	-6
4	-3	4	71	72	-9	0	4	19	13	-5
6	-3	4	34	31	-8	0	4	24	21	-4
7	-3	4	22	33	-6	0	4	35	40	-3
8	-3	4	19	18	-5	0	4	58	68	-2
-9	-2	4	33	24	-4	0	4	50	54	-1
-8	-2	4	29	38	-2	0	4	153	138	0
-7	-2	4	22	19	-1	0	4	710	691	1
-6	-2	4	29	34	0	0	4	318	301	2
-5	-2	4	22	30	1	0	4	154	146	3
-4	-2	4	75	90	2	0	4	107	100	4
-2	-2	4	155	157	3	0	4	77	79	5
-1	-2	4	302	284	4	0	4	64	70	6

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	4		86	92	7	2	4	31	32	-5	5	4	34	31
0	4		98	96	8	2	4	52	48	-4	5	4	156	156
0	4		40	43	9	2	4	70	57	-3	5	4	44	47
0	4		22	34	-7	3	4	88	76	-2	5	4	110	110
0	4		49	41	-6	3	4	28	29	-1	5	4	45	43
1	4		22	19	-5	3	4	36	35	0	5	4	27	30
1	4		41	55	-4	3	4	34	23	1	5	4	41	50
1	4		68	51	-3	3	4	145	137	2	5	4	25	27
1	4		123	120	-2	3	4	57	48	3	5	4	33	26
1	4		120	124	-1	3	4	175	165	4	5	4	81	81
1	4		44	42	1	3	4	127	106	8	5	4	49	43
1	4		203	201	2	3	4	227	219	-5	6	4	62	57
1	4		58	63	3	3	4	126	127	-4	6	4	21	23
1	4		103	98	4	3	4	27	34	-3	6	4	121	122
1	4		79	80	5	3	4	269	258	-2	6	4	138	133
1	4		82	89	6	3	4	166	162	-1	6	4	34	33
1	4		64	68	7	3	4	18	17	0	6	4	71	72
1	4		100	101	9	3	4	23	16	1	6	4	88	91
1	4		95	82	-7	4	4	248	230	2	6	4	40	44
1	4		35	38	-6	4	4	57	42	3	6	4	22	25
2	4		17	12	-5	4	4	33	35	4	6	4	71	84
2	4		21	30	-4	4	4	105	107	5	6	4	43	43
2	4		28	27	-3	4	4	438	443	6	6	4	34	34
2	4		25	28	-2	4	4	102	106	-6	7	4	83	80
2	4		51	52	-1	4	4	36	35	-5	7	4	36	37
2	4		90	74	0	4	4	26	23	-4	7	4	153	141
2	4		139	147	1	4	4	85	72	-3	7	4	60	60
2	4		250	209	2	4	4	89	84	-2	7	4	19	18
2	4		202	200	3	4	4	64	58	-1	7	4	55	43
2	4		43	27	4	4	4	74	70	0	7	4	409	410
2	4		293	279	5	4	4	92	91	2	7	4	29	28
2	4		202	206	6	4	4	50	53	3	7	4	32	35
2	4		49	45	8	4	4	52	40	4	7	4	203	215
2	4		93	93	-8	5	4	20	24	7	7	4	30	28
2	4		256	255	-7	5	4	131	122	-5	8	4	31	31
2	4		255	245	-6	5	4	74	60	-4	8	4	96	95

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-3	8	4	27	19	0	-9	5	48	49	3
-2	8	4	103	95	1	-9	5	140	131	4
-1	8	4	113	114	4	-9	5	24	29	5
0	8	4	319	321	-7	-8	5	113	100	-9
1	8	4	74	82	-6	-8	5	25	20	-8
2	8	4	29	18	-5	-8	5	26	22	-6
3	8	4	38	35	-4	-8	5	62	69	-5
4	8	4	30	32	-3	-8	5	198	195	-4
6	8	4	18	20	-2	-8	5	50	45	-2
-4	9	4	31	31	-1	-8	5	109	110	-1
-3	9	4	44	48	0	-8	5	64	59	0
-2	9	4	23	26	2	-8	5	108	107	2
-1	9	4	29	28	3	-8	5	61	66	3
0	9	4	19	17	4	-8	5	66	61	4
1	9	4	42	42	-8	-7	5	24	23	5
2	9	4	60	59	-7	-7	5	49	42	6
3	9	4	35	38	-6	-7	5	23	15	7
-2	10	4	48	51	-5	-7	5	58	51	-9
0	10	4	20	21	-4	-7	5	36	31	-8
1	10	4	29	26	-3	-7	5	61	62	-7
3	10	4	56	58	-2	-7	5	21	22	-6
-3	-11	5	26	30	-1	-7	5	102	102	-5
-2	-11	5	20	27	0	-7	5	126	128	-4
-1	-11	5	30	44	1	-7	5	42	37	-3
-5	-10	5	28	28	3	-7	5	81	88	-2
-4	-10	5	31	35	4	-7	5	91	88	-1
-3	-10	5	60	70	-8	-6	5	26	22	1
-2	-10	5	36	30	-7	-6	5	39	41	2
0	-10	5	56	61	-6	-6	5	38	34	3
1	-10	5	52	47	-5	-6	5	39	43	4
2	-10	5	43	52	-3	-6	5	25	29	5
3	-10	5	18	20	-2	-6	5	63	63	6
-4	-9	5	38	32	-1	-6	5	142	150	8
-3	-9	5	330	330	0	-6	5	296	303	-9
-2	-9	5	32	35	1	-6	5	50	52	-8
-1	-9	5	40	40	2	-6	5	120	114	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	5		43	51	-6	-3	5	36	29	2	-1	5	223	219
-6	5		325	319	-5	-3	5	34	32	3	-1	5	161	147
-6	5		116	111	-4	-3	5	84	90	4	-1	5	154	146
-5	5		33	35	-3	-3	5	56	57	6	-1	5	44	46
-5	5		54	49	-2	-3	5	56	54	7	-1	5	32	40
-5	5		46	39	-1	-3	5	275	271	-6	0	5	68	77
-5	5		58	47	0	-3	5	144	138	-5	0	5	105	113
-5	5		73	84	1	-3	5	247	237	-4	0	5	152	150
-5	5		49	62	2	-3	5	55	64	-3	0	5	186	191
-5	5		112	102	3	-3	5	35	35	-2	0	5	123	125
-5	5		398	393	5	-3	5	25	21	-1	0	5	387	378
-5	5		127	127	6	-3	5	82	77	0	0	5	127	116
-5	5		103	114	7	-3	5	87	85	1	0	5	40	42
-5	5		138	143	8	-3	5	59	52	2	0	5	153	154
-5	5		44	47	-9	-2	5	22	19	3	0	5	47	57
-5	5		30	27	-7	-2	5	28	21	4	0	5	39	41
-5	5		36	43	-5	-2	5	43	53	7	0	5	42	44
-4	5		43	40	-4	-2	5	48	53	8	0	5	26	26
-4	5		128	122	-3	-2	5	73	84	-7	1	5	32	32
-4	5		25	20	-2	-2	5	219	210	-5	1	5	33	35
-4	5		68	70	-1	-2	5	82	76	-4	1	5	127	107
-4	5		22	19	0	-2	5	71	75	-2	1	5	110	103
-4	5		152	149	1	-2	5	138	120	-1	1	5	78	72
-4	5		113	116	2	-2	5	109	109	0	1	5	164	153
-4	5		82	86	3	-2	5	54	59	1	1	5	41	31
-4	5		143	137	5	-2	5	63	60	2	1	5	79	84
-4	5		150	134	6	-2	5	47	46	3	1	5	134	138
-4	5		24	25	7	-2	5	82	90	4	1	5	39	40
-4	5		23	21	-8	-1	5	23	37	5	1	5	159	159
-4	5		26	24	-5	-1	5	45	45	6	1	5	47	48
-4	5		39	40	-4	-1	5	43	40	7	1	5	31	34
-4	5		69	67	-3	-1	5	144	144	-9	2	5	24	27
-4	5		39	33	-2	-1	5	74	38	-8	2	5	54	50
-3	5		49	44	-1	-1	5	374	355	-6	2	5	30	33
-3	5		110	101	0	-1	5	233	225	-5	2	5	49	43
-3	5		25	30	1	-1	5	294	280	-4	2	5	135	130

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K
-3	2	5	72	41	3	4	5	140	145	-1	7
-2	2	5	138	135	4	4	5	27	25	0	7
-1	2	5	166	141	5	4	5	84	89	1	7
0	2	5	37	43	8	4	5	35	44	2	7
1	2	5	85	89	-7	5	5	35	40	3	7
2	2	5	44	46	-6	5	5	38	21	4	7
3	2	5	62	61	-5	5	5	87	83	5	7
4	2	5	22	11	-4	5	5	116	115	6	7
5	2	5	169	158	-3	5	5	24	30	-4	8
8	2	5	24	26	-2	5	5	170	171	-3	8
-8	3	5	37	37	-1	5	5	187	178	-2	8
-7	3	5	63	54	0	5	5	49	46	-1	8
-6	3	5	72	69	1	5	5	53	52	0	8
-5	3	5	45	48	2	5	5	25	33	1	8
-4	3	5	94	85	3	5	5	79	80	2	8
-3	3	5	57	65	4	5	5	38	38	3	8
-2	3	5	106	91	7	5	5	46	53	4	8
-1	3	5	183	169	-7	6	5	25	25	-3	9
0	3	5	59	55	-6	6	5	21	20	-2	9
1	3	5	110	91	-5	6	5	63	62	-1	9
2	3	5	52	59	-4	6	5	36	38	0	9
3	3	5	64	65	-3	6	5	95	100	1	9
4	3	5	115	109	-2	6	5	122	124	2	9
5	3	5	139	135	-1	6	5	92	90	3	9
6	3	5	35	40	0	6	5	93	99	1	10
7	3	5	53	54	1	6	5	68	70	-2	-11
8	3	5	21	11	2	6	5	32	30	-1	-11
-7	4	5	33	31	3	6	5	56	65	-4	-10
-6	4	5	29	31	4	6	5	98	101	-3	-10
-5	4	5	26	27	6	6	5	33	40	-2	-10
-4	4	5	21	21	7	6	5	18	16	-1	-10
-3	4	5	125	132	-6	7	5	28	35	0	-10
-2	4	5	79	81	-5	7	5	59	61	1	-10
-1	4	5	27	20	-4	7	5	56	57	2	-10
1	4	5	33	22	-3	7	5	34	26	-5	-9
2	4	5	59	66	-2	7	5	25	19	-4	-9

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	136	142	-3	-9	6	160	153	0	-6	6	118	120		
5	189	198	-2	-9	6	41	47	1	-6	6	64	64		
5	97	110	-1	-9	6	55	61	2	-6	6	87	91		
5	43	58	0	-9	6	89	97	3	-6	6	284	290		
5	35	41	1	-9	6	119	115	4	-6	6	191	198		
5	88	92	2	-9	6	53	59	5	-6	6	29	29		
5	22	23	4	-9	6	34	30	-7	-5	6	26	28		
5	33	30	-7	-8	6	56	48	-6	-5	6	43	58		
5	51	50	-6	-8	6	56	48	-5	-5	6	153	150		
5	64	61	-5	-8	6	30	24	-4	-5	6	23	28		
5	48	56	-4	-8	6	61	68	-3	-5	6	22	18		
5	44	43	-3	-8	6	103	96	-2	-5	6	37	36		
5	140	141	-2	-8	6	36	33	-1	-5	6	173	170		
5	32	39	-1	-8	6	136	142	0	-5	6	146	149		
5	40	32	1	-8	6	97	97	1	-5	6	170	170		
5	24	31	2	-8	6	74	72	2	-5	6	164	167		
5	30	34	3	-8	6	82	86	3	-5	6	117	115		
5	41	43	4	-8	6	46	54	4	-5	6	219	220		
5	42	46	5	-8	6	26	37	5	-5	6	35	37		
5	31	30	-8	-7	6	26	36	6	-5	6	74	72		
5	37	42	-7	-7	6	52	33	-9	-4	6	156	136		
5	29	32	-6	-7	6	47	41	-8	-4	6	61	54		
5	22	17	-2	-7	6	69	67	-7	-4	6	76	66		
5	32	33	-1	-7	6	24	33	-6	-4	6	25	24		
5	20	17	0	-7	6	35	36	-5	-4	6	117	118		
6	21	30	1	-7	6	174	175	-4	-4	6	87	92		
6	54	60	2	-7	6	25	25	-3	-4	6	43	52		
6	69	68	3	-7	6	145	146	-2	-4	6	26	28		
6	32	30	5	-7	6	36	34	-1	-4	6	54	21		
6	35	28	6	-7	6	33	38	0	-4	6	75	57		
6	56	60	-7	-6	6	23	35	1	-4	6	51	25		
6	65	67	-6	-6	6	41	26	2	-4	6	67	67		
6	67	70	-5	-6	6	55	57	4	-4	6	81	82		
6	21	22	-3	-6	6	90	95	5	-4	6	86	81		
6	28	38	-2	-6	6	26	24	6	-4	6	20	17		
6	146	137	-1	-6	6	289	290	7	-4	6	69	67		

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-9	-3	6	83	78	-1	-1	6	115	109	6
-8	-3	6	42	18	0	-1	6	219	214	8
-6	-3	6	30	22	1	-1	6	92	93	-7
-5	-3	6	50	53	2	-1	6	307	286	-6
-4	-3	6	73	81	3	-1	6	80	78	-5
-3	-3	6	113	116	4	-1	6	29	27	-4
-2	-3	6	67	59	5	-1	6	53	56	-3
-1	-3	6	34	43	6	-1	6	20	12	-2
0	-3	6	136	133	7	-1	6	42	44	-1
1	-3	6	134	127	8	-1	6	26	23	0
2	-3	6	50	60	-6	0	6	169	194	1
4	-3	6	20	18	-5	0	6	52	47	2
5	-3	6	47	53	-4	0	6	127	133	3
6	-3	6	106	115	-3	0	6	87	90	4
7	-3	6	35	41	-2	0	6	499	486	5
8	-3	6	19	17	-1	0	6	59	43	6
-7	-2	6	57	56	0	0	6	58	49	7
-5	-2	6	29	30	1	0	6	124	130	8
-4	-2	6	57	60	2	0	6	56	58	-8
-3	-2	6	48	58	3	0	6	128	127	-7
-2	-2	6	452	453	4	0	6	60	57	-6
-1	-2	6	32	42	5	0	6	76	70	-5
0	-2	6	44	45	6	0	6	30	28	-4
1	-2	6	118	100	7	0	6	18	21	-3
2	-2	6	347	323	-8	1	6	27	31	-2
3	-2	6	127	131	-6	1	6	51	60	-1
4	-2	6	23	25	-4	1	6	48	66	0
5	-2	6	90	88	-3	1	6	89	92	1
6	-2	6	129	142	-2	1	6	186	178	2
7	-2	6	45	43	-1	1	6	55	46	3
8	-2	6	27	28	0	1	6	74	73	4
-7	-1	6	31	27	1	1	6	75	72	5
-6	-1	6	152	160	2	1	6	40	33	6
-5	-1	6	42	48	3	1	6	21	12	8
-3	-1	6	126	130	4	1	6	171	176	-8
-2	-1	6	918	884	5	1	6	204	201	-7

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	6		48	49	-6	4	6	79	73	-1	7	6	37	26
1	6		34	47	-5	4	6	29	37	0	7	6	255	270
2	6		25	19	-4	4	6	27	27	1	7	6	126	128
2	6		54	53	-3	4	6	181	192	2	7	6	64	63
2	6		40	33	-2	4	6	129	134	4	7	6	37	39
2	6		49	44	-1	4	6	64	64	5	7	6	48	50
2	6		87	89	0	4	6	98	97	-4	8	6	74	71
2	6		22	38	2	4	6	43	36	-3	8	6	53	54
2	6		129	123	3	4	6	46	46	-2	8	6	57	52
2	6		151	161	4	4	6	31	30	-1	8	6	88	88
2	6		347	333	5	4	6	23	19	0	8	6	95	100
2	6		111	111	7	4	6	80	83	1	8	6	82	88
2	6		77	85	-7	5	6	119	107	2	8	6	24	19
2	6		187	185	-6	5	6	19	29	4	8	6	24	26
2	6		393	384	-5	5	6	38	45	-3	9	6	47	45
2	6		74	75	-4	5	6	21	20	-2	9	6	33	33
2	6		52	57	-3	5	6	57	60	0	9	6	26	33
2	6		18	16	-2	5	6	43	35	1	9	6	47	47
3	6		44	48	-1	5	6	45	43	2	9	6	29	27
3	6		86	90	1	5	6	31	27	3	9	6	28	27
3	6		41	33	2	5	6	113	113	-4	-10	7	27	21
3	6		67	73	3	5	6	33	33	-3	-10	7	23	20
3	6		103	106	4	5	6	70	65	-2	-10	7	46	47
3	6		156	163	-5	6	6	41	52	-1	-10	7	33	34
3	6		29	35	-4	6	6	18	13	0	-10	7	73	77
3	6		62	63	-3	6	6	125	121	1	-10	7	57	55
3	6		164	165	-2	6	6	116	117	-5	-9	7	59	57
3	6		458	441	-1	6	6	50	41	-4	-9	7	65	61
3	6		49	56	0	6	6	64	58	-3	-9	7	39	33
3	6		52	55	1	6	6	24	27	0	-9	7	60	63
3	6		104	108	2	6	6	70	70	1	-9	7	36	33
3	6		129	128	3	6	6	27	39	2	-9	7	41	47
3	6		72	76	4	6	6	49	50	-6	-8	7	29	16
3	6		34	29	-4	7	6	30	31	-4	-8	7	50	56
4	6		55	50	-3	7	6	77	74	-3	-8	7	58	47
4	6		185	178	-2	7	6	34	29	-2	-8	7	52	56

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
0	-8	7	57	54	2	-5	7	23	26	-4
1	-8	7	95	98	3	-5	7	164	167	-3
2	-8	7	24	25	4	-5	7	51	55	-2
-7	-7	7	35	35	5	-5	7	37	38	-1
-4	-7	7	19	13	6	-5	7	18	13	0
-3	-7	7	30	32	-8	-4	7	42	28	1
-2	-7	7	65	64	-5	-4	7	121	122	2
-1	-7	7	77	82	-4	-4	7	42	41	4
0	-7	7	58	60	-3	-4	7	94	89	5
1	-7	7	100	105	-1	-4	7	114	106	6
2	-7	7	20	24	0	-4	7	105	110	7
3	-7	7	56	62	1	-4	7	108	105	-8
4	-7	7	45	46	2	-4	7	45	40	-7
5	-7	7	71	79	4	-4	7	59	57	-6
-8	-6	7	42	33	5	-4	7	47	46	-5
-7	-6	7	18	10	6	-4	7	30	27	-4
-6	-6	7	42	38	7	-4	7	36	41	-3
-5	-6	7	59	62	-9	-3	7	25	9	-2
-3	-6	7	21	34	-8	-3	7	38	29	-1
-1	-6	7	33	26	-7	-3	7	45	36	1
0	-6	7	48	50	-6	-3	7	49	42	2
1	-6	7	42	39	-5	-3	7	89	88	3
2	-6	7	91	95	-4	-3	7	26	25	4
3	-6	7	342	348	-3	-3	7	70	78	5
4	-6	7	26	21	-2	-3	7	69	78	6
5	-6	7	31	38	-1	-3	7	127	132	7
6	-6	7	31	38	0	-3	7	84	85	8
-8	-5	7	22	15	1	-3	7	119	116	-7
-7	-5	7	39	37	4	-3	7	63	63	-6
-6	-5	7	23	23	5	-3	7	22	25	-5
-5	-5	7	107	105	6	-3	7	132	132	-3
-3	-5	7	22	17	7	-3	7	19	15	-2
-2	-5	7	105	101	-8	-2	7	19	25	-1
-1	-5	7	91	103	-7	-2	7	36	37	0
0	-5	7	73	72	-6	-2	7	41	34	1
1	-5	7	59	58	-5	-2	7	63	66	2

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-2	7		74	68	3	0	7	31	16	6	2	7	73	65
-2	7		92	90	4	0	7	109	115	-8	3	7	122	104
-2	7		85	97	5	0	7	94	91	-6	3	7	25	25
-2	7		86	74	6	0	7	71	70	-5	3	7	19	31
-2	7		231	230	8	0	7	22	21	-4	3	7	108	101
-2	7		181	171	-8	1	7	31	26	-3	3	7	58	67
-2	7		219	202	-7	1	7	30	20	-2	3	7	79	78
-2	7		60	58	-6	1	7	39	23	-1	3	7	57	52
-2	7		35	32	-5	1	7	39	53	0	3	7	70	56
-2	7		98	106	-4	1	7	56	57	1	3	7	164	156
-2	7		20	23	-3	1	7	111	111	2	3	7	60	64
-1	7		31	27	-2	1	7	28	22	4	3	7	46	47
-1	7		68	56	-1	1	7	37	30	5	3	7	128	121
-1	7		31	35	0	1	7	21	26	6	3	7	42	51
-1	7		36	47	1	1	7	70	72	7	3	7	51	41
-1	7		110	113	2	1	7	91	95	-7	4	7	33	25
-1	7		173	162	3	1	7	22	16	-6	4	7	19	14
-1	7		373	361	4	1	7	138	140	-5	4	7	22	19
-1	7		50	30	5	1	7	85	83	-4	4	7	169	174
-1	7		126	120	6	1	7	28	33	-2	4	7	52	51
-1	7		311	305	7	1	7	68	67	-1	4	7	42	42
-1	7		62	60	8	1	7	64	65	0	4	7	100	99
-1	7		128	128	-8	2	7	36	32	1	4	7	37	33
-1	7		22	26	-7	2	7	23	31	2	4	7	58	59
-1	7		68	69	-6	2	7	44	45	3	4	7	43	42
-1	7		27	47	-5	2	7	44	22	4	4	7	21	20
-1	7		43	36	-4	2	7	53	57	5	4	7	41	42
0	7		20	14	-3	2	7	34	40	-7	5	7	45	31
0	7		32	32	-2	2	7	30	46	-6	5	7	24	24
0	7		51	43	-1	2	7	21	33	-5	5	7	42	37
0	7		54	57	0	2	7	316	307	-4	5	7	32	28
0	7		215	209	1	2	7	93	98	-3	5	7	33	33
0	7		98	105	2	2	7	77	84	-2	5	7	26	22
0	7		126	127	3	2	7	42	47	-1	5	7	42	45
0	7		43	28	4	2	7	217	214	0	5	7	36	40
0	7		45	43	5	2	7	249	248	1	5	7	39	30

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
2	5	7	49	44	-3	-9	8	41	34	5
3	5	7	26	27	-1	-9	8	21	22	-8
4	5	7	32	26	0	-9	8	80	84	-7
5	5	7	34	33	1	-9	8	43	42	-6
-5	6	7	19	12	-6	-8	8	49	35	-5
-3	6	7	21	13	-5	-8	8	25	31	-4
-2	6	7	45	44	-4	-8	8	83	86	-3
-1	6	7	181	182	-3	-8	8	74	64	-2
0	6	7	33	40	-1	-8	8	42	43	-1
2	6	7	35	42	1	-8	8	20	16	0
3	6	7	128	130	2	-8	8	54	61	1
5	6	7	34	35	3	-8	8	22	26	2
6	6	7	18	11	4	-8	8	21	35	3
-5	7	7	81	76	-7	-7	8	24	8	4
-4	7	7	27	31	-6	-7	8	25	19	6
-3	7	7	34	35	-5	-7	8	29	35	-8
-2	7	7	71	73	-4	-7	8	33	32	-6
-1	7	7	373	369	-3	-7	8	65	63	-5
0	7	7	39	39	-2	-7	8	52	47	-4
1	7	7	43	43	-1	-7	8	95	94	-2
2	7	7	30	26	0	-7	8	56	53	-1
3	7	7	108	112	1	-7	8	34	35	0
-3	8	7	40	42	2	-7	8	66	60	1
-2	8	7	28	16	3	-7	8	69	70	2
-1	8	7	50	54	4	-7	8	37	41	3
0	8	7	23	37	-7	-6	8	22	18	4
2	8	7	68	77	-6	-6	8	49	41	5
3	8	7	20	24	-4	-6	8	68	68	-8
-1	9	7	32	37	-3	-6	8	41	41	-7
0	9	7	28	24	-2	-6	8	20	18	-6
1	9	7	41	51	-1	-6	8	133	141	-5
-2	-10	8	22	26	0	-6	8	77	78	-3
-1	-10	8	48	47	1	-6	8	51	58	-2
0	-10	8	35	42	2	-6	8	24	34	-1
-5	-9	8	31	31	3	-6	8	218	214	0
-4	-9	8	148	150	4	-6	8	34	46	1

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	8		25	21	5	-3	8	25	35	-1	0	8	52	57
-5	8		35	26	6	-3	8	116	123	1	0	8	69	66
-5	8		47	37	7	-3	8	50	51	2	0	8	35	28
-5	8		63	55	-7	-2	8	40	43	3	0	8	35	39
-5	8		63	65	-6	-2	8	57	63	4	0	8	44	42
-5	8		28	43	-5	-2	8	49	51	5	0	8	23	24
-5	8		108	107	-4	-2	8	117	115	6	0	8	52	52
-5	8		41	30	-3	-2	8	156	150	7	0	8	32	28
-5	8		118	124	-2	-2	8	179	187	-8	1	8	23	14
-5	8		25	15	-1	-2	8	137	130	-7	1	8	40	40
-5	8		40	52	0	-2	8	83	78	-6	1	8	39	35
-5	8		79	81	1	-2	8	168	166	-4	1	8	71	73
-5	8		70	62	2	-2	8	46	40	-2	1	8	42	31
-5	8		27	32	3	-2	8	27	19	-1	1	8	24	25
-5	8		29	32	4	-2	8	31	29	0	1	8	126	132
-4	8		26	15	5	-2	8	19	27	1	1	8	46	46
-4	8		24	21	6	-2	8	22	22	2	1	8	43	45
-4	8		84	81	-6	-1	8	78	82	3	1	8	45	43
-4	8		40	43	-5	-1	8	20	23	4	1	8	155	152
-4	8		34	33	-3	-1	8	75	83	5	1	8	74	70
-4	8		75	69	-2	-1	8	51	53	6	1	8	28	33
-4	8		67	56	-1	-1	8	67	69	7	1	8	27	25
-4	8		25	33	0	-1	8	47	40	-7	2	8	33	32
-4	8		31	35	1	-1	8	26	34	-5	2	8	46	48
-4	8		54	56	2	-1	8	114	121	-4	2	8	28	33
-4	8		19	25	3	-1	8	108	110	-3	2	8	22	18
-4	8		89	90	4	-1	8	28	33	-2	2	8	27	39
-3	8		27	27	5	-1	8	30	29	-1	2	8	56	50
-3	8		22	22	6	-1	8	34	37	0	2	8	46	39
-3	8		74	66	7	-1	8	21	25	1	2	8	38	48
-3	8		26	23	-7	0	8	47	51	2	2	8	31	33
-3	8		51	45	-6	0	8	35	35	3	2	8	56	60
-3	8		32	36	-5	0	8	50	26	4	2	8	174	170
-3	8		74	66	-4	0	8	24	28	5	2	8	24	24
-3	8		66	65	-3	0	8	26	30	6	2	8	65	65
-3	8		72	67	-2	0	8	168	168	-6	3	8	29	29

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-4	3	8	124	130	-2	7	8	46	52	4
-3	3	8	42	44	-1	7	8	102	102	-7
-2	3	8	39	44	0	7	8	27	18	-6
-1	3	8	52	47	2	7	8	52	53	-4
0	3	8	20	25	3	7	8	68	74	-3
1	3	8	49	49	-2	8	8	29	22	-2
2	3	8	54	53	-1	8	8	88	89	-1
4	3	8	129	126	0	8	8	36	37	0
5	3	8	32	27	1	8	8	81	85	1
6	3	8	34	31	-4	-9	9	126	124	3
7	3	8	26	25	-3	-9	9	37	40	5
-5	4	8	21	27	0	-9	9	51	52	-8
-4	4	8	27	29	1	-9	9	28	33	-7
-2	4	8	27	35	-5	-8	9	30	33	-5
-1	4	8	42	35	-4	-8	9	55	40	-4
0	4	8	108	108	-3	-8	9	25	26	-2
1	4	8	50	52	-2	-8	9	20	11	-1
2	4	8	39	46	-1	-8	9	21	17	0
3	4	8	29	33	2	-8	9	33	35	2
6	4	8	35	38	3	-8	9	35	36	3
-6	5	8	24	31	-4	-7	9	50	61	4
-4	5	8	36	39	-3	-7	9	21	10	5
-3	5	8	25	23	-2	-7	9	51	50	6
-2	5	8	56	50	-1	-7	9	32	33	-8
-1	5	8	42	41	0	-7	9	21	24	-5
1	5	8	20	14	1	-7	9	36	40	-3
2	5	8	50	35	2	-7	9	148	146	-2
-4	6	8	28	19	3	-7	9	115	118	-1
-3	6	8	18	11	4	-7	9	44	49	0
-2	6	8	117	116	-6	-6	9	45	48	1
-1	6	8	68	74	-2	-6	9	67	69	4
1	6	8	68	72	-1	-6	9	118	128	5
2	6	8	37	43	0	-6	9	65	68	6
3	6	8	103	112	1	-6	9	85	90	-8
4	6	8	19	12	2	-6	9	213	211	-7
-3	7	8	28	27	3	-6	9	179	192	-5

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	9		32	31	-4	-2	9	93	87	-4	1	9	28	34
-5	9		30	37	-3	-2	9	281	284	-3	1	9	26	26
-5	9		122	114	-2	-2	9	72	68	-2	1	9	98	102
-5	9		48	41	0	-2	9	77	64	-1	1	9	87	96
-5	9		39	47	1	-2	9	210	202	0	1	9	73	68
-5	9		133	140	2	-2	9	22	19	1	1	9	26	25
-5	9		99	94	4	-2	9	50	49	2	1	9	40	48
-5	9		30	41	5	-2	9	33	35	3	1	9	63	59
-5	9		40	39	6	-2	9	23	19	4	1	9	220	222
-5	9		53	60	-8	-1	9	21	17	5	1	9	84	86
-5	9		69	70	-7	-1	9	65	63	6	1	9	18	25
-4	9		21	22	-6	-1	9	20	21	-7	2	9	22	28
-4	9		33	38	-5	-1	9	101	94	-5	2	9	60	56
-4	9		101	84	-4	-1	9	110	125	-4	2	9	94	110
-4	9		60	62	-3	-1	9	307	315	-3	2	9	72	67
-4	9		34	38	-2	-1	9	160	160	-2	2	9	20	19
-4	9		71	69	-1	-1	9	94	95	-1	2	9	80	77
-4	9		52	32	0	-1	9	135	134	0	2	9	197	189
-4	9		23	23	1	-1	9	39	42	1	2	9	42	43
-4	9		36	33	2	-1	9	97	103	2	2	9	96	92
-4	9		65	61	3	-1	9	39	31	3	2	9	104	103
-4	9		36	33	6	-1	9	28	20	4	2	9	145	146
-4	9		27	29	-7	0	9	65	60	5	2	9	42	49
-3	9		19	15	-6	0	9	27	35	6	2	9	29	27
-3	9		35	37	-5	0	9	40	28	-7	3	9	48	47
-3	9		92	92	-4	0	9	98	100	-5	3	9	25	36
-3	9		97	106	-3	0	9	114	109	-4	3	9	92	113
-3	9		49	53	-1	0	9	69	62	-3	3	9	24	45
-3	9		48	51	0	0	9	45	48	-2	3	9	69	64
-3	9		97	88	1	0	9	24	29	-1	3	9	59	51
-3	9		20	16	2	0	9	30	17	0	3	9	109	99
-3	9		111	113	3	0	9	76	79	1	3	9	85	87
-3	9		38	43	4	0	9	19	24	2	3	9	46	50
-2	9		22	9	-7	1	9	39	44	3	3	9	47	53
-2	9		86	71	-6	1	9	36	33	4	3	9	67	67
-2	9		40	39	-5	1	9	68	65	6	3	9	44	41

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-5	4	9	46	53	0	-7	10	31	35	1
-4	4	9	49	52	2	-7	10	187	196	2
-3	4	9	42	42	-6	-6	10	30	21	3
-2	4	9	38	40	-5	-6	10	29	22	4
-1	4	9	19	28	-4	-6	10	33	30	5
0	4	9	44	34	-3	-6	10	19	23	-4
1	4	9	41	30	-2	-6	10	119	125	-3
2	4	9	55	58	-1	-6	10	21	17	-2
3	4	9	60	57	1	-6	10	48	44	-1
5	4	9	27	26	2	-6	10	163	174	0
-2	5	9	48	59	3	-6	10	51	50	1
-1	5	9	98	96	-6	-5	10	48	45	2
0	5	9	23	24	-5	-5	10	48	39	3
1	5	9	21	36	-2	-5	10	34	35	5
2	5	9	50	52	-1	-5	10	57	60	-7
3	5	9	39	34	0	-5	10	89	95	-5
4	5	9	21	26	2	-5	10	56	54	-4
-3	6	9	28	25	3	-5	10	69	74	-3
-2	6	9	42	34	4	-5	10	43	44	-2
-1	6	9	121	124	-6	-4	10	23	18	-1
0	6	9	40	37	-5	-4	10	45	33	0
1	6	9	37	38	-4	-4	10	111	95	1
2	6	9	19	20	-3	-4	10	20	16	3
3	6	9	26	27	-2	-4	10	32	13	4
-2	7	9	30	33	-1	-4	10	35	43	5
-1	7	9	81	75	0	-4	10	36	24	6
0	7	9	86	98	2	-4	10	47	54	-7
1	7	9	24	16	4	-4	10	33	29	-6
-4	-8	10	25	27	5	-4	10	57	61	-5
-3	-8	10	22	24	-6	-3	10	38	34	-4
-2	-8	10	18	14	-5	-3	10	57	49	-3
-1	-8	10	33	37	-4	-3	10	65	64	-2
2	-8	10	59	60	-3	-3	10	39	50	0
-4	-7	10	41	40	-2	-3	10	122	126	1
-2	-7	10	55	60	-1	-3	10	58	57	2
-1	-7	10	24	30	0	-3	10	72	75	5

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-3	10		61	59	6	0	10	37	35	-2	4	10	36	32
-3	10		35	39	-6	1	10	21	21	1	4	10	42	25
-3	10		45	52	-5	1	10	19	21	3	4	10	25	26
-3	10		19	15	-3	1	10	20	28	-4	5	10	21	17
-3	10		109	110	-2	1	10	41	35	-3	5	10	24	30
-2	10		126	120	-1	1	10	63	57	-2	5	10	80	81
-2	10		213	221	0	1	10	52	50	-1	5	10	60	61
-2	10		39	35	1	1	10	39	31	0	5	10	50	47
-2	10		30	24	2	1	10	22	31	1	5	10	47	55
-2	10		74	70	3	1	10	124	132	2	5	10	71	70
-2	10		171	172	4	1	10	151	156	-3	6	10	47	42
-2	10		64	63	5	1	10	51	48	-2	6	10	156	164
-2	10		34	32	-6	2	10	32	27	0	6	10	63	56
-2	10		85	86	-5	2	10	100	102	1	6	10	43	35
-1	10		57	59	-4	2	10	22	23	2	6	10	76	84
-1	10		35	41	-3	2	10	41	50	-4	-7	11	22	17
-1	10		60	65	-2	2	10	95	95	-3	-7	11	50	47
-1	10		209	225	-1	2	10	64	57	-2	-7	11	53	58
-1	10		115	101	0	2	10	127	134	-1	-7	11	25	22
-1	10		36	37	1	2	10	48	44	0	-7	11	38	38
-1	10		35	47	2	2	10	24	24	1	-7	11	50	36
-1	10		104	96	3	2	10	116	120	2	-7	11	74	84
-1	10		109	115	4	2	10	194	201	-3	-6	11	22	14
-1	10		19	29	5	2	10	35	40	-2	-6	11	106	118
-1	10		53	54	-5	3	10	78	86	-1	-6	11	22	21
-1	10		26	20	-4	3	10	50	61	2	-6	11	55	52
0	10		47	38	-3	3	10	49	61	3	-6	11	23	12
0	10		28	22	-2	3	10	48	44	-6	-5	11	36	38
0	10		45	45	-1	3	10	55	57	-5	-5	11	18	13
0	10		39	38	0	3	10	66	63	-3	-5	11	48	49
0	10		48	36	1	3	10	61	72	-2	-5	11	26	36
0	10		94	93	2	3	10	30	32	-1	-5	11	36	35
0	10		116	111	3	3	10	38	35	0	-5	11	44	56
0	10		81	83	4	3	10	49	48	2	-5	11	23	20
0	10		60	58	5	3	10	34	38	3	-5	11	23	31
0	10		48	45	-4	4	10	27	32	-6	-4	11	35	26

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H
-5	-4	11	23	18	2	0	11	73	78	2
-4	-4	11	24	24	3	0	11	62	60	3
-2	-4	11	54	46	4	0	11	38	39	-2
-1	-4	11	31	37	5	0	11	30	35	-1
0	-4	11	46	44	-6	1	11	27	23	0
1	-4	11	33	41	-5	1	11	22	16	2
2	-4	11	26	32	-4	1	11	21	12	-3
4	-4	11	62	63	-2	1	11	51	43	-2
-6	-3	11	22	15	-1	1	11	146	149	-1
-4	-3	11	57	51	0	1	11	82	86	1
-2	-3	11	38	45	1	1	11	43	35	-4
-1	-3	11	47	41	2	1	11	44	42	-3
4	-3	11	20	19	3	1	11	178	183	-2
-4	-2	11	111	92	4	1	11	57	53	0
-3	-2	11	134	139	-3	2	11	47	39	1
-2	-2	11	54	70	-1	2	11	48	50	2
-1	-2	11	108	109	0	2	11	40	39	-4
0	-2	11	135	136	2	2	11	30	30	-2
1	-2	11	45	32	3	2	11	115	115	-1
2	-2	11	54	56	4	2	11	36	26	0
3	-2	11	24	24	-5	3	11	46	51	2
5	-2	11	29	25	-4	3	11	56	57	3
-6	-1	11	25	19	-3	3	11	60	61	-5
-5	-1	11	47	54	-1	3	11	27	23	-4
-3	-1	11	97	87	0	3	11	57	59	-2
-2	-1	11	49	57	1	3	11	35	34	-1
-1	-1	11	53	54	2	3	11	23	17	0
2	-1	11	29	27	4	3	11	33	33	1
5	-1	11	32	22	-4	4	11	23	13	-5
-6	0	11	35	19	-3	4	11	45	41	-4
-3	0	11	64	56	-2	4	11	39	36	-3
-2	0	11	29	32	-1	4	11	52	46	-2
0	0	11	68	78	0	4	11	27	36	-1
1	0	11	37	32	1	4	11	31	14	0

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	11		31	31	1	-2	12	32	30	2	3	12	42	41
4	11		32	32	2	-2	12	20	9	-1	4	12	32	28
5	11		31	29	-4	-1	12	85	88	0	4	12	38	47
5	11		28	33	-3	-1	12	35	31	-2	-5	13	28	47
5	11		37	31	-2	-1	12	43	45	-2	-4	13	26	24
5	11		55	53	-1	-1	12	69	62	-1	-4	13	31	34
-6	12		79	83	0	-1	12	34	43	-4	-3	13	54	54
-6	12		49	51	1	-1	12	91	99	-3	-3	13	28	40
-6	12		54	52	2	-1	12	29	24	-2	-3	13	30	29
-6	12		57	54	-5	0	12	42	30	-1	-3	13	44	41
-5	12		21	26	-4	0	12	22	11	0	-3	13	84	97
-5	12		26	29	-3	0	12	48	50	1	-3	13	18	16
-5	12		23	20	-2	0	12	46	52	-4	-2	13	96	110
-5	12		47	46	0	0	12	23	17	-3	-2	13	41	27
-5	12		41	40	1	0	12	39	40	-1	-2	13	65	65
-5	12		49	52	2	0	12	40	44	0	-2	13	71	76
-4	12		34	26	3	0	12	71	77	2	-2	13	23	23
-4	12		34	35	-5	1	12	46	52	-4	-1	13	79	89
-4	12		36	47	-3	1	12	43	40	-3	-1	13	44	44
-4	12		43	42	-1	1	12	114	116	-2	-1	13	57	51
-4	12		56	54	1	1	12	25	35	0	-1	13	24	21
-4	12		19	13	2	1	12	49	45	2	-1	13	45	50
-3	12		28	28	3	1	12	113	114	-2	0	13	28	24
-3	12		92	92	-4	2	12	34	32	-1	0	13	27	28
-3	12		22	22	-2	2	12	43	44	0	0	13	54	51
-3	12		40	37	-1	2	12	69	71	1	0	13	21	28
-3	12		44	41	0	2	12	40	43	2	0	13	58	54
-3	12		27	27	1	2	12	58	60	-3	1	13	41	26
-2	12		44	46	2	2	12	73	70	-2	1	13	61	57
-2	12		150	161	3	2	12	45	48	0	1	13	33	31
-2	12		36	27	-3	3	12	26	22	1	1	13	28	29
-2	12		28	31	-2	3	12	37	36	-1	2	13	58	65
-2	12		38	38	0	3	12	24	19	0	2	13	20	17
-2	12		95	96	1	3	12	19	23					

APPENDIX B

Postgraduate Courses

Postgraduate Courses Attended

1. Radiation Hazards Course at Aberdeen University.
2. A 3rd Year undergraduate course of nine lectures at Aberdeen University which outlined the various types of X-ray analysis.
3. British Crystallographic Association Spring Meeting, London, 1983.
4. A ten session Fortran Course given by the Director of Studies.
5. Multinuclear Magnetic Resonance Spectroscopy (SERC) Summer School, Sheffield, 1983.
6. Phytochemical Society of Europe Spring Meeting, London, 1984.
7. British Crystallographic Association Spring Meeting, Bristol, 1985.

APPENDIX C

Publications and Communications

Publications and Communications

1. 'A Photo Artefact from Linderazulene' B. Alpertunga, S. Imre, H.J. Cowe, P.J. Cox and R.H. Thomson, Tetrahedron Letters 41 (1983) 4461.

2. 'Bromoallenes from the Alga Laurencia microcladia' D.J. Kennedy, I.A. Selby, H.J. Cowe, P.J. Cox, R.H. Thomson, J. Chem. Soc. Chem. Commun., (1984) 153.

3. 'Isofuranonaphthoquinones from Ventilago maderaspatana', Crystal Structure of Ventilone C' T. Hanumaiah, G.S.R. Rao, C.P. Rao, K.V.J. Rao, H.J. Cowe, P.J. Cox, R.A. Howie, D.S. Marshall, R.H. Thomson, Tetrahedron, 41 (1985) 635.

4. 'Traditional Medicinal Plants of Thailand, V. Ancistrotectorine, A new Naphthalene-Isoquinoline Alkaloid From Ancistrocladus Tectorius', N. Ruangrungsi, V. Wongpanich, P.Tantivatana, H.J. Cowe, P.J. Cox, S. Funayama and G.A. Cordell. J.Nat. Prod. (1985) 48 529.

5. Nirurine - A new Securinega Alkaloid from Phyllanthus niruri (Euphorbiaceae). (Oral presentation by Prof. G.A. Cordell), N. Bunyapraphatsara, G.A. Cordell, H.J. Cowe and P.J. Cox, The American Society of Pharmacognosy - 24th Annual Meeting, University of Mississippi, July 1983.

6. 'Molecular Studies of Natural Products' Poster presentation H.J. Cowe, SERC Summer School on nmr spectroscopy, University of Sheffield, September 1983.

7. 'Ancistrotectorine a new Naphthalene-Isoquinoline Alkaloid from Ancistrocladus tectorius' Poster presentation N. Ruangrungsi, V. Wongpanich, P. Tantivartana, H.J. Cowe, P.J. Cox, S. Funayama, G.A. Cordell, The Phytochemical Society of Europe, Isoquinoline Meeting, University of London, April, 1984.

8. 'The Leucoacetate of Ventilatone A - a Disordered Structure' Poster presentation H.J. Cowe, P.J. Cox, R.H. Thomson, British Crystallographic Association, Spring Meeting, University of Bristol, March 1985.

Bromoallenes from the Alga *Laurencia microcladia*

Derek J. Kennedy,[a]* Ian A. Selby,[a] Heather J. Cowe,[b] Philip J. Cox,[b] and Ronald H. Thomson[c]

[a] *Merck, Sharp and Dohme, Hertford Road, Hoddesdon, Herts EN11 9BU, U.K.*
[b] *School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, U.K.*
[c] *Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, U.K.*

The structures of the microcladallenes A, B, and C have been established from spectroscopic data and *X*-ray crystallographic analysis of microcladallene B.

Reprinted from the Journal of The Chemical Society

Chemical Communications 1984

Bromoallenes from the Alga *Laurencia microcladia*

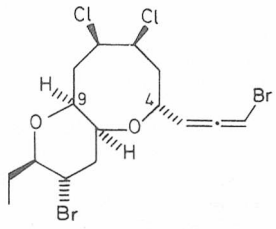
Derek J. Kennedy,[a]* Ian A. Selby,[a] Heather J. Cowe,[b] Philip J. Cox,[b] and Ronald H. Thomson[c]

[a] *Merck, Sharp and Dohme, Hertford Road, Hoddesdon, Herts EN11 9BU, U.K.*
[b] *School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, U.K.*
[c] *Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, U.K.*

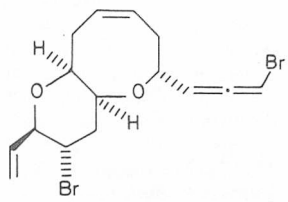
The structures of the microcladallenes A, B, and C have been established from spectroscopic data and *X*-ray crystallographic analysis of microcladallene B.

The red algal genus *Laurencia* elaborates a large number of metabolites[1] which include a C_{15} family of halogenated cyclic ether enynes.[2] A second, closely related, C_{15} family of allenes is now emerging,[3] and we report here on the structures of three new bromoallenes isolated from *L. microcladia* (collected on the French coast at Cap Ferrat) by extraction with methanol–toluene (3:1) followed by extensive chromatography.

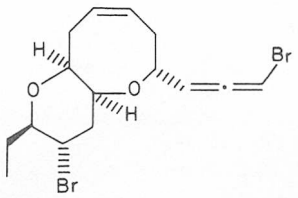
Microcladallene C is an optically active, viscous oil, $[\alpha]_D^{20}$ + 116.7° (*c* 0.5, Me_2CO). The molecular formula, $C_{15}H_{20}Br_2Cl_2O_2$, was derived from accurate mass measurement on the $M^+ - Cl$ (found: *m/z* 424.9516; calc. 424.9517) and



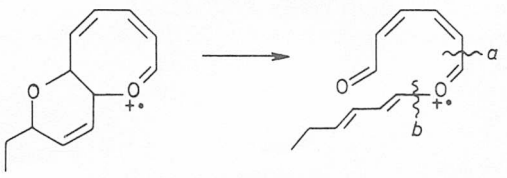
(1)



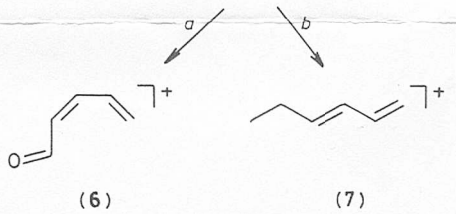
(2)



(3)



(4) (5)



(6) (7)

$M^+-CH=C=CHBr$ (found: *m/z* 342.9865; calc. 342.9866) ions. The ^1H and ^{13}C n.m.r. spectra† show the presence of a bromoallene side chain, an ethyl and three methylene groups, and seven >CHHal and >CHO methine protons, and to fit the molecular formula the compound must be bicyclic. Extensive decoupling of the 360 MHz ^1H n.m.r. spectrum† revealed both the gross structure and the relative stereochemistry (**1**) except at C-4. The gross structure (**1**) is consistent with the mass spectrum. In particular, a peak at *m/z* 191‡ ($C_{12}H_{13}O_2$) can be attributed to the backbone ion (**4**), derived from (**1**) by successive losses of the allenic side chain, HBr, and 2 × HCl, which, after retro-Diels–Alder conversion into (**5**), fragments at *a* and at *b* to give the two *m/z* 81 ions (**6**)

† ^1H δ($CDCl_3$) (*J* in Hz) 0.98 (t, 7, 3H-15), 1.50 (ddq, 14,8,7, H-14), 1.78 (ddd, 14,2.2,2, H-8), 2.00 (ddd, 14,10,10, H-8), 2.04 (ddq, 14,2.2,7, H-14), 2.38 (ddd, 14,5,?, H-5), 2.40 (ddd, 14,12,4, H-11), 2.49 (ddd, 14,6.5,1.5, H-5), 2.73 (ddd, 14,4,3, H-11), 3.40 (ddd, 14,10,3, H-13), 3.88 (ddd, 10,2,1.8, H-9), 4.04 (ddd, 4,3,2, H-10), 4.06 (ddd, 12,10,4, H-12), 4.25 (dt, 10,2,2, H-7), 4.46 (m, H-6), 4.89 (m, H-4), 5.43 (t, 6,6, H-3), and 6.08 (dd, 6,2, H-1); ^{13}C δ ($CDCl_3$) 9.41 (C-15), 26.11 (C-14), 35.94 and 42.79 (each CH_2), 43.83 (C-11), 46.42 (C-12), 61.66 and 62.55 (C-6 and C-7), 74.14 (C-1), 73.54, 75.94 and 77.80 (each –CHO–), 83.63 (C-4), 101.24 (C-3), and 200.8 p.p.m. (C-2).

‡ All ions mentioned gave satisfactory accurate mass values.

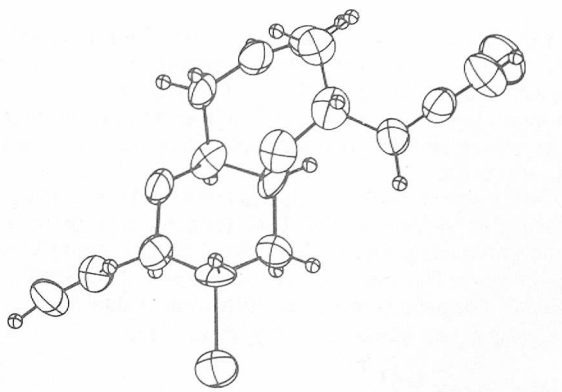
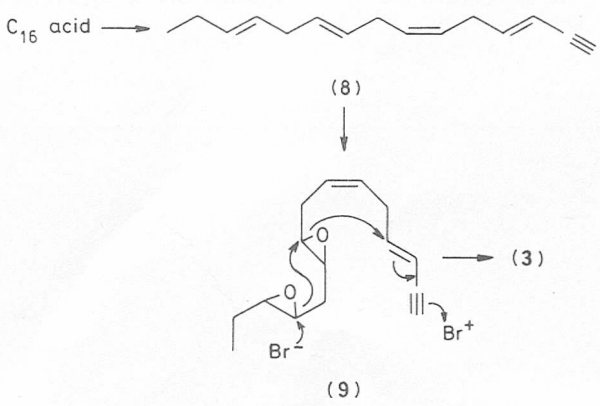


Figure 1. The atomic arrangement in microcladallene B (**2**).



(8) (3) (9)

(C_5H_5O, calc. 81.0340) and (**7**) (C_6H_9, calc. 81.0704), both of which were observed (81.0339 and 81.0701, respectively).

The structures of microcladallenes A {$C_{15}H_{20}Br_2O_2$, crystals, m.p. 90.5 °C, $[\alpha]_D^{20}$ + 114.0°, (*c* 0.5, Me_2CO)} (**3**) and B {$C_{15}H_{18}Br_2O_2$, plates, m.p. 83 °C, $[\alpha]_D^{20}$ + 96.0° (*c* 0.5, Me_2CO)} (**2**) were deduced in the same way from spectral data. Decoupling of the ^1H n.m.r. spectra revealed the presence of the $\Delta^{6,7}$ double bond in both, and the vinyl group at C-13 in B. Final confirmation of structure (**2**) and the absolute configuration was obtained by *X*-ray crystallographic analysis of B which is crystalline. The similarity of the H-4 n.m.r. signal from A, B, and C suggests that all three have the same configuration at C-4.

Crystal data: $C_{15}H_{18}Br_2O_2$, *M* = 389.2, orthorhombic, space group $P2_12_12_1$, *a* = 5.174(5), *b* = 14.234(8), *c* = 21.479(14) Å, *U* = 1581.9 Å^3, *Z* = 4, D_c = 1.634 g cm^{-3}, μ(Mo-K_α) = 50.0 cm^{-1}. The 832 unique observed reflexions [$I>3\sigma(I)$] with $2\theta<50°$ were measured on a Nicolet P3 automated diffractometer and corrected for crystal degradation. The structure was solved by direct methods[4] and refined by full-matrix least-squares[5] (Br, O, and C anisotropic, H calculated). The weighting scheme used was $w=1.48/(\sigma^2F_o)$ and convergence was reached at R=7.2% (for the mirror image R=8.0%.§ As considerable degradation of the crystal in the *X*-ray beam was observed, higher than usual e.s.d.'s are associated with geometrical parameters and the absolute configuration shown in Figure 1 may require future revision.

The biogenesis of these enyne and allenic systems is not known but the cyclisation of diepoxides, originally proposed

§ The atomic co-ordinates for this work are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW. Any request should be accompanied by the full literature citation for this communication.

by Bu'Lock,[6] remains highly attractive. Thus microcladallene A could arise by brominative cyclisation of (**9**) formed by selective epoxidation of (**8**), itself derived from the related C_{16} non-conjugated polyene acid. While (**8**) has not been found the all-*cis* isomer, laurencenyne, has been isolated[7] from *L. okamurai*.

We thank the Edinburgh University WH-360 NMR Service for n.m.r. spectra, the S.E.R.C. for a studentship (to H. J. C.), and particularly Dr. A. Meinesz, Laboratoire de Biologie et D'Ecologie Marines, 28 Av. de Valrose, 06034 Nice Cedex, France, for collection and identification of algal material.

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A PHOTO ARTEFACT FROM LINDERAZULENE

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Abstract: Exposure of an ethanolic solution of linderazulene to direct sunlight yields the sesquiterpene keto-lactone 2.

During the isolation[1] of the violet pigment linderazulene 1 from the gorgonian *Paramuricea chamaeleon* we detected a yellow pigment in minute amount. We subsequently found this to be a photo-oxidation product, and it can be obtained by exposing an ethanol solution of linderazulene to direct sunlight for six days (5 mg from 100 mg linderazulene, after chromatographic purification).

The photo-product, $C_{15}H_{12}O_3$,[2] m.p. 107-110°C (from MeOH) shows $\lambda_{max.}$ (MeOH) 272, 285sh, 415sh, 430 nm; $\nu_{max.}$ (KBr) 1758, 1684, 1620, 1540 cm^{-1}; and a very simple ^1H NMR spectrum ($CDCl_3$) consisting of three 1H singlets at δ 6.82, 6.52 and 6.22, and three 3H singlets at δ 2.69, 2.32, and 2.12. These data are consistent with the keto-lactone structure 2 and the compound is generally similar to the isomer, malaphyllidin 3, isolated[3] from *Ferula malacophylla*. However, if the formation of the photo-product involves skeletal rearrangement as well as oxidation then isomeric structures must be considered, such as 4, which is also consistent with the spectroscopic data. To exclude this possibility the compound was subjected to X-ray crystallography which confirmed structure 2.

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TRADITIONAL MEDICINAL PLANTS OF THAILAND, V. ANCISTROTECTORINE, A NEW NAPHTHALENE-ISOQUINOLINE ALKALOID FROM *ANCISTROCLADUS TECTORIUS*[1]

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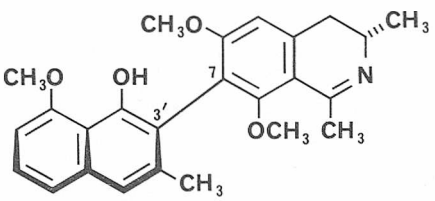
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ABSTRACT.—A new member of the naphthalene-isoquinoline series of alkaloids, ancistrotectorine (**2**), has been isolated from the leaves of the Thai medicinal plant *Ancistrocladus tectorius* (Ancistrocladaceae). The structure was deduced through single crystal X-ray crystallography and complete ^{1}H-nmr spectroscopic assignments were made. Ancistrotectorine (**2**) is the second member of the 7,3′-linked naphthalene-isoquinoline alkaloid group to be isolated.

Ancistrocladus is the only genus of the plant family Ancistrocladaceae and is composed of nearly 20 species distributed in tropical Asia, Malayasia, and West Africa (2). The genus is a source, together with two genera in the Dionchophyllaceae, of the naphthalene-isoquinoline group of alkaloids (3, 4). Eighteen alkaloids in this series have been isolated to date, and the compounds may be grouped according to the location of the σ-bond joining the two units. Most of the alkaloids (ten) are linked 5-1′ (5-12), whilst seven others are linked 7-1′ (9, 13-15). The remaining alkaloid, ancistrocladidine (**1**) from *Ancistrocladus heyneanus* Wall. (16), is linked 7-3′, and the crystallographic analysis of this alkaloid was recently presented (17). We report here the structure determination of a second alkaloid having a 7-3′ linkage, ancistrotectorine (**2**) from the leaves of *Ancistrocladus tectorius* (Lour.) Merr.



1

Previous work on the stem and twigs of *A. tectorius* has yielded ancistrocladeine (10), ancistrocladine (12), hamatine (12), and ancistrocline (12), subsequent to a demonstration that the species contained alkaloids (18). The roots of *A. tectorius* have been used to treat dysentery and malaria.

After partition of the EtOH extract, the $CHCl_3$ fraction was chromatographed on alumina to afford ancistrotectorine (0.016% yield). From its uv spectrum, which displayed λmax 234, 286, 306, 323, and 337 nm, ancistrotectorine was a member of the

[1]For the previous paper in this series see Mukhopadhyay *et al.* (1).

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naphthalene-isoquinoline series of alkaloids containing a phenolic hydroxy group (νmax) 3375 cm^{-1}), even though no positive $FeCl_3$ test or shift in base (2N NaOH solution) in the uv spectrum was observed. No molecular ion was observed in the electron impact (ei) mass spectrum, but a clear ($M^{\cdot}+H^{+}$) was found at *m/z* 422 in the chemical ionization mass spectrum using methane as the reagent gas. Important fragments in the ei mass spectrum were observed at *m/z* 406 and 203.

The low-field ^{1}H-nmr spectrum of ancistrotectorine firmly established the naphthalene-isoquinoline nature of the compound and indicated the presence of two doublet methyl groups, an aromatic methyl group, an *N*-methyl group, and three aromatic methoxy groups. The aromatic region was complex, although two singlets were apparent.

In order to evaluate the structure more explicitly, a detailed examination of the high-field ^{1}H-nmr spectrum of ancistrotectorine was undertaken. Two doublet methyl groups were observed at δ 1.268 and δ 1.466 from the 3-CH_3 and the 1-CH_3, respectively, and their methine protons were observed at δ 2.544 and δ 3.731. Irradiation at δ 1.27 collapsed the signal at δ 2.544 to a doublet of doublets (J=3.0, 10.3 Hz), clearly establishing the 3-CH_3 to be equatorial. The aromatic region was now clearly resolved with singlets observed at δ 6.518 and 7.232, doublets at δ 6.689 and 7.343, and a triplet at δ 7.266. The latter three signals were assigned to adjacent protons on the naphthalene nucleus, and on biogenetic grounds a methyl group (δ 2.169) was placed at C-2′ and oxygenation at C-4′ and C-5′. Irradiation of the methoxyl group singlet at δ 3.691 caused an 8.1% nOe effect in the singlet at δ 6.518, and the reverse irradiation produced an nOe (4%) only in the δ 3.691 singlet. A similar spatial relationship was established between the aromatic methoxy group at δ 3.985 and the doublet at δ 6.689, which could therefore be assigned to a 5′-OCH_3 and its adjacent 6′-H. There were a number of alternatives to explain the former nOe experiments: (a) a 7-1′ linkage, with the OH at C-4′ and the third OCH_3 at C-8, (b) a 7-3′ linkage with the OH/OCH_3 at C-8/C-4′ or reverse, and (c) a 5-3′ linkage with OH at C-8 and OCH_3 at C-4′. Potentially, these structures could be distinguished by the base-induced shift of a proton *ortho* or *para* to the hydroxy group and whether or not the proton shifted is *ortho* to a methoxy group. However, the failure of ancistrotectorine to undergo a bathochromic shift in the uv spectrum on the addition of 2N NaOH did not augur well for the success of this experiment, and consequently, the single crystal X-ray crystallographic analysis was carried out.

Ancistrotectorine crystallized in the monoclinic $P2_1$ space group with unit cell dimensions of a=11.858(14), b=7.043(7), c=14.693(28) Å and β=114.43(12). The crystal structure was elucidated, using the program MULTAN (19), and refined, using the program SHELX (20). The hydrogen bond lengths were restricted to 1.0 Å during refinement, and the largest peak on a final electron density difference map was 0.12 eÅ.$^{-3}$ The final R=8.0% over 1087 independent reflexions using a unit weighting scheme.

Final positional parameters, bond lengths, and valency angles are listed in Tables 1-3, respectively. The crystallographic structure deduced for ancistrotectorine (Figure 1)

TABLE 1. Fractional Atomic Co-ordinates (x10^4) with e.s.d's (in parenthesis)

Atom	x	y	z
O(1)	1240(8)	9594(20)	0433(6)
O(2)	4670(7)	6549(22)	2800(6)
O(3)	3753(9)	11387(23)	2826(7)
O(4)	3645(10)	13613(23)	4207(6)

TABLE 1. *Continued*

Atom	x	y	z
C(1)	1943(12)	8794(27)	−1116(9)
N(2)	2331(11)	7931(24)	−1864(8)
C(3)	2868(13)	6042(27)	−1609(10)
C(4)	3963(11)	6199(29)	−0718(10)
C(4A)	3682(12)	7021(28)	0111(9)
C(5)	4374(11)	6427(28)	1072(10)
C(6)	4056(11)	7043(25)	1837(9)
C(7)	3025(12)	8244(29)	1655(9)
C(8)	2347(12)	8766(27)	0679(9)
C(8A)	2689(11)	8158(25)	−0056(9)
C(1′)	1312(11)	7786(30)	3363(11)
C(2′)	1769(11)	7380(27)	2658(10)
C(3′)	2589(10)	8651(25)	2481(9)
C(4′)	2962(11)	10278(23)	3052(9)
C(4A′)	2490(13)	10752(27)	3776(9)
C(5′)	2789(12)	12383(27)	4364(10)
C(6′)	2339(18)	12833(30)	5069(13)
C(7′)	1546(16)	11543(32)	5221(11)
C(8′)	1207(11)	9955(35)	4686(10)
C(8A′)	1656(13)	9490(26)	3945(9)
C(9)	2074(14)	10965(24)	−1185(11)
C(10)	1288(16)	7917(32)	−2780(12)
C(11)	3135(14)	5103(3)	−2472(10)
C(12)	5591(12)	5143(29)	2970(9)
C(13)	1163(16)	11377(31)	0861(11)
C(14)	1417(14)	5579(28)	2077(12)
C(15)	4087(14)	15272(29)	4814(11)
HO(3)	3799(102)	12787(37)	2937(80)
H(1)	1102(41)	8521(175)	−1144(71)
H(3)	2306(81)	5109(127)	−1480(79)
H(4A)	4436(81)	5016(86)	−0463(68)
H(4B)	4559(77)	7121(122)	−0795(77)
H(5)	5014(69)	5531(121)	1053(74)
H(1′)	0662(71)	6984(134)	3440(73)
H(6′)	2342(100)	14212(56)	5234(79)
H(7′)	1116(75)	11783(176)	5685(55)
H(8′)	0583(72)	9069(124)	4760(74)
H(9A)	1546(77)	11586(160)	−0873(64)
H(9B)	2922(39)	10879(185)	−0680(52)
H(9C)	2105(94)	11794(152)	−1731(60)
H(10A)	0896(106)	9195(78)	−2902(88)
H(10B)	0712(97)	6960(116)	−2689(90)
H(10C)	1516(101)	7534(144)	−3343(62)
H(11A)	3642(84)	3971(104)	−2491(95)
H(11B)	2300(38)	4926(158)	−2963(65)
H(11C)	3510(89)	6260(106)	−2646(91)
H(12A)	5926(88)	4500(150)	3619(40)
H(12B)	5199(96)	4183(126)	2448(56)
H(12C)	6272(70)	5770(154)	2829(70)
H(13A)	0396(52)	12051(147)	0480(69)
H(13B)	1102(88)	10847(161)	1476(52)
H(13C)	1854(67)	12306(135)	1050(79)
H(14A)	2230(49)	5140(165)	2110(79)
H(14B)	0997(82)	4533(126)	2288(81)
H(14C)	0919(74)	5983(177)	1399(43)
H(15A)	4807(59)	15680(158)	4660(75)
H(15B)	3619(94)	16424(107)	4869(83)
H(15C)	4383(87)	14600(147)	5464(49)

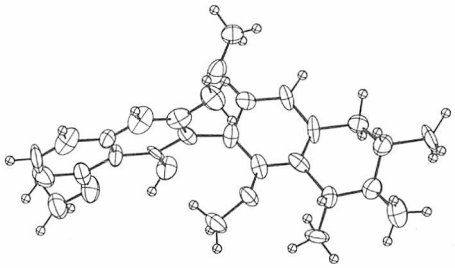
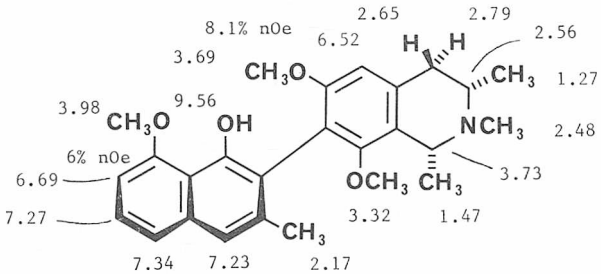


FIGURE 1. ORTEP Display of the Crystallographic Structure of Ancistrotectorine (**2**).

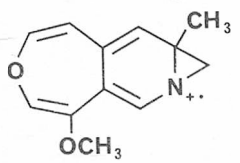
indicates that it is indeed a 7-3′ linked naphthalene-isoquinoline alkaloid having the molecular array shown in **2** in which the C-1 and C-3 methyl groups are *cis* and the N-containing ring of the isoquinoline moiety adopts a sofa conformation with C-3 the out-of-plane atom. Steric hindrance about the diaryl linkage results in a dihedral angle of 56.4° between the two aromatic rings.

It now became possible to assign the ^{1}H spectrum completely. From the nOe experiments the singlets at δ 3.691 and δ 3.985 could be assigned to the 6- and 5′-OCH_3, respectively, leaving the singlet at δ 3.323 to be a somewhat shielded methoxy group at C-8. Similarly, the aromatic singlet at δ 6.518 could be assigned to H-5 and at δ 7.232 to H-1′. Examination of the coupling constants permitted the doublets of doublets at δ 2.655 and δ 2.787 to be assigned to H-4β and H-4α respectively. The complete assignments are shown on structure **2**. Ancistrotectorine (**2**) is therefore a member of the rare 7-3′-linked naphthalene-isoquinoline alkaloids of which ancistrocladidine (**1**) (16, 17), is the only other example.



2

The mass spectrum of ancistrotectorine also merits some additional comment. A facile loss of 15 amu was apparent to give the stable species at *m/z* 406. The most interesting ion though is *m/z* 203 since this does not correspond to either half of the molecule as might be expected, although it clearly is derived from the isoquinoline unit. In our estimation this ion may have the structure **3** in which one of the aliphatic methyl groups has been lost together with one of the methyls from an aromatic methoxy group.



3 *m/z* 203

TABLE 2. Bond Lengths (Å) with eds's (in parentheses)

O(1)—C(8)	1.359(18)	C(5)—C(6)	1.380(22)
O(1)—C(13)	1.422(25)	C(6)—C(7)	1.429(22)
O(2)—C(6)	1.379(14)	C(7)—C(8)	1.413(17)
O(2)—C(12)	1.427(21)	C(7)—C(3′)	1.510(21)
O(3)—C(4′)	1.352(18)	C(8)—C(8A)	1.354(22)
O(4)—C(5′)	1.416(22)	C(1′)—C(2′)	1.363(24)
O(4)—C(15)	1.447(23)	C(1′)—C(8A′)	1.443(25)
C(1)—N(2)	1.469(22)	C(2′)—C(3′)	1.414(23)
C(1)—C(8A)	1.550(17)	C(2′)—C(14)	1.501(25)
C(1)—C(9)	1.544(25)	C(3′)—C(4′)	1.393(21)
N(2)—C(3)	1.463(24)	C(4′)—C(4A′)	1.411(22)
N(2)—C(10)	1.458(17)	C(4A′)—C(5′)	1.403(25)
C(3)—C(4)	1.473(16)	C(4A′)—C(8A′)	1.417(25)
C(3)—C(11)	1.562(25)	C(5′)—C(6′)	1.365(28)
C(4)—C(4A)	1.492(23)	C(6′)—C(7′)	1.383(31)
C(4A)—C(5)	1.413(18)	C(7′)—C(8′)	1.341(30)
C(4A)—C(8A)	1.370(21)	C(8′)—C(8A′)	1.414(23)

Ancistrocladidine (**1**)[3] shows a loss of 15 amu from the molecular ion at *m/z* 405, but no stable species are observed below this mass.

The absolute configuration of naphthalene-isoquinoline alkaloids has been studied previously (8, 21). The cd spectrum of ancistrotectorine in MeOH showed a negative first Cotton effect ($[\theta]_{234}$ −34,179) and a positive second Cotton effect ($[\theta]_{218}$ +70,167) which clearly demonstrates the absolute configuration of ancistrotectorine to be that shown in **2**.

TABLE 3. Valency Angles (Å) with eds's (in parentheses)

C(13)—O(1)—C(8)	118.2(13)	C(7)—C(8)—O(1)	121.2(12)
C(8A)—C(8)—O(1)	117.7(12)	C(12)—O(2)—C(6)	116.0(11)
C(5)—C(6)—O(2)	123.9(14)	C(7)—C(6)—O(2)	115.9(11)
C(3′)—C(4′)—O(3)	115.0(11)	C(4A′)—C(4′)—O(3)	123.4(14)
C(15)—O(4)—C(5′)	120.9(12)	C(4A′)—C(5′)—O(4)	115.8(13)
C(6′)—C(5′)—O(4)	118.9(16)	C(8A)—C(1)—N(2)	114.4(13)
C(9)—C(1)—N(2)	107.1(13)	C(3)—N(2)—C(1)	114.2(12)
C(10)—N(2)—C(1)	107.5(13)	C(9)—C(1)—C(8A)	108.5(12)
C(4A)—C(8A)—C(1)	119.1(12)	C(8)—C(8A)—C(1)	118.9(13)
C(10)—N(2)—C(3)	111.9(14)	C(4)—C(3)—N(2)	108.6(15)
C(11)—C(3)—N(2)	112.1(12)	C(11)—C(3)—C(4)	112.3(14)
C(4A)—C(4)—C(3)	111.6(13)	C(5)—C(4A)—C(4)	118.7(15)
C(8A)—C(4A)—C(4)	120.9(12)	C(8A)—C(4A)—C(5)	120.0(13)
C(6)—C(5)—C(4A)	119.3(14)	C(8)—C(8A)—C(4A)	122.0(13)
C(7)—C(6)—C(5)	120.2(12)	C(8)—C(7)—C(6)	118.3(13)
C(3′)—C(7)—C(6)	118.9(12)	C(3′)—C(7)—C(8)	122.1(14)
C(8A)—C(8)—C(7)	120.1(15)	C(2′)—C(3′)—C(7)	119.2(14)
C(4′)—C(3′)—C(7)	120.9(14)	C(8A′)—C(1′)—C(2′)	121.3(16)
C(3′)—C(2′)—C(1′)	119.9(16)	C(14)—C(2′)—C(1′)	120.3(16)
C(4A′)—C(8A′)—C(1′)	118.8(13)	C(8′)—C(8A′)C(1′)	123.0(16)
C(14)—C(2′)—C(3′)	119.8(13)	C(4′)—C(3′)—C(2′)	119.9(12)
C(4A′)—C(4′)—C(3′)	121.5(14)	C(5′)—C(4A′)—C(4′)	125.3(15)
C(8A′)—C(4A′)—C(4′)	118.6(15)	C(8A′)—C(4A′)—C(5′)	116.2(13)
C(6′)—C(5′)—C(4A′)	125.2(16)	C(8′)—C(8A′)—C(4A′)	118.2(16)
C(7′)—C(6′)—C(5′)	116.6(18)	C(8′)—C(7′)—C(6′)	121.9(16)
C(8A′)—C(8′)—C(7′)	121.9(17)		

[3]Kindly supplied by Dr. P.C. Parthasarathy, Hindustan CIBA-Geigy Ltd., Bombay, India.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Melting points were determined using a Kofler hot-stage microscope and are uncorrected. Uv spectra were recorded on a Beckman model DB-G spectrophotometer and ir spectra on a Nicolet model MX-1 FT-IR interferometer. Mass spectra were obtained with Finnegan model 4500 quadrupole mass spectrometer. ^{1}H-nmr spectra were recorded on a Nicolet NT-360 instrument operating at 360 MHz at the NSF Regional NMR Facility at the University of Illinois at Urbana, Urbana-Champaign. X-Ray intensity data were measured with Mo-K_α radiation on a Nicolet P3 automated diffractometer. Alumina used for column chromatography was obtained from E. Merck, Darmstadt, W. Germany.

PLANT MATERIAL.—The leaf material of *A. tectorius* was collected at Sakae-Raj Environmental Research Station, Rachasima Province, Thailand, in October 1982. The plant material was identified by the Botany Section, Technical Division, Department of Agriculture, Ministry of Agriculture and Cooperatives, Thailand. A herbarium specimen is deposited in the herbarium of the Department of Pharmaceutical Botany, Faculty of Pharmaceutical Sciences, Chulalongkorn University, Bangkok, Thailand.

ISOLATION OF ANCISTROTECTORINE (**2**).—The dried powdered leaves of *A. tectorius* (800 g) were macerated twice for 3-day periods with 95% EtOH (8 liters and 5 liters) and the combined EtOH extracts pooled and evaporated under reduced pressure. The residue (125 g) was suspended in distilled H_2O (5 liters) and the filtrate extracted with $CHCl_3$ (2 ×5 liters). The combined $CHCl_3$ extracts were dried (Na_2SO_4), filtered, and evaporated under reduced pressure to afford a light brown powder (13 g).

The powder was divided into six portions, and each was separately chromatographed as follows. Each portion was dissolved in $CHCl_3$ (10 ml), absorbed onto neutral alumina (5 g), dried, and placed on top of a dry alumina column (6 × 20 cm) and eluted with Et_2O. Twenty fractions were collected, examined by tlc, and fractions of similar composition were combined. Pooled fractions 9-20 were homogeneous by tlc and yielded ancistrotectorine (**2**, 125 mg, 0.016% yield).

PHYSICAL PROPERTIES OF ANCISTROTECTORINE (**2**).—Ancistrotectorine (**2**) crystallized from Me_2CO as pale yellow needles, mp 134-140°; ir νmax (KBr) 3375, 2960, 2925, 2840, 2770, 1640, 1600, 1555, 1458, 1400, 1362, 1320, 1115, 1095, 1085, 825, and 755 cm^{-1}; uv λmax (EtOH) 234 (log ε 4.73), 286 (3.64), 306 (3.61), 323 (3.66), and 337 nm (3.69); ms *m/z* (ei, 70eV, 180°) no M^+ observed, 420 (1%), 407 (28), 406 (100), 360 (17), 203 (72), 202 (5), 195 (6), 189 (4), 188 (9), 187 (5), 181 (8), 180 (36), 172 (28), 167 (9), 165 (28), 158 (31), 151 (33), 145 (8), 144 (8), 143 (8), 137 (7), 136 (8), 131 (7), 129 (10), 128 (8), 115 (12), and 107 (7); ms, *m/z* (ci, CH_4 gas, 70eV) 422 ($M+H^+$, 100%) and 406 (9); ^{1}H-nmr (360 MHz, $CDCl_3$) δ 1.268 (d, 3H, J=6.1 Hz, 3-CH_3), 1.466 (d, 3H, J=6.4 Hz, 1-CH_3), 2.169 (s, 3H, 2′-CH_3), 2.484 (s, 3H, N-CH_3), 2.544 (ddq, 1H, J=3.0, 6.1, 10.3 Hz, 3-H), 2.655 (dd, 1H, J=3.0, 15.3 Hz, 4-Hβ), 2.787 (dd, 1H, J=10.3 15.4 Hz, 4-Hα), 3.323 (s, 3H, 8-OCH_3), 3.691 (s, 3H, 6-OCH_3), 3.731 (q, 1H, J=6.4 Hz, 1-H), 3.985 (s, 3H, 5′-OCH_3), 6.518 (s, 1H, 5-H), 6.689 (d, 1H, J=7.5 Hz, 6′-H), 7.232 (s, 1H, 1′-H), 7.266 (t, 1H, J=7.8 Hz, 7′-H), 7.343 (d, 1H, J=8.0 Hz, 8′-H), and 9.565 (s, 1H, 4′-OH); $[\alpha]_D^{26}$ 0° ($CHCl_3$); cd (MeOH) $[\theta]_{218}$ +70,167, $[\theta]_{234}$ −34,179, $[\theta]_{283}$ −10,291, $[\theta]_{308}$ −5,146, $[\theta]_{321}$ −6,549 and $[\theta]_{336}$ −8,420.

CRYSTALLOGRAPHIC ANALYSIS[4].—Crystal data, $C_{26}H_{31}NO_4$, M=421.5, monoclinic, a=11.858(14), b=7.043(7), c=14.693(28) Å, β=111.43(12)°, U=1142.6 Å^3, Z=2 D_c=1.22 g. cm^{-3}, space group $P2_1$, μ (MO-K_α)=0.46 cm^{-1}.

The crystal structure was elucidated using MULTAN (19) and refined using SHELX (20). In the final cycles of least-squares refinement, the positional parameters for all atoms, anisotropic thermal parameters for the C and O atoms and common isotropic thermal parameters for the methyl and non-methyl hydrogens were varied. Structure amplitudes and thermal parameters are listed in material available from the authors.

ACKNOWLEDGMENTS

The high field nmr spectral data were obtained at the Mid-West Regional NMR Facility, University of Illinois at Urbana. We thank Dr. P.C. Parthasarathy, Hindustan CIBA-Geigy Ltd, Bombay, India, for a sample of ancistrocladidine; Professor C.D. Hufford, School of Pharmacy, University of Mississippi for the cd spectrum of **2**; and Ms. P. Van Leeuwen for the mass spectral data of **1** and **2**.

We also thank Dr. R.A. Howie, Aberdeen University, for assistance in collecting the X-ray data and the SERC for a studentship (to HJC).

[4]Atomic coordinates for this structure have been deposited with the Cambridge Crystallographic Data Centre and can be obtained on request from Dr. Olga Kennard, University Chemical Laboratory, Lensfield Rd., Cambridge, CB2 EW, UK.

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ISOFURANONAPHTHOQUINONES FROM *VENTILAGO MADERASPATANA*

CRYSTAL STRUCTURE OF VENTILONE-C

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Abstract—From the root bark of *Ventilago maderaspatana* (Rhamnaceae) five isofuranonaphthoquinones have been isolated. Ventilone-A is 4,9-dihydro-5,8-dihydroxy-6,7-methylenedioxy-1-methylnaphtho[2,3-c]furan-4,9-dione and B is the 5-methyl ether. Ventilone-C has the structure 1,3,4,9-tetrahydro-8-hydroxy-5-methoxy-6,7-methylenedioxy-1-methylnaphtho[2,3-c]furan-4,9-dione, D is the isomeric 8-methyl ether, and E is the 5,8-dimethyl ether. Structures were determined by spectroscopic methods, and X-ray crystallographic analysis of ventilone-C.

In Perkin and Hummel's original investigation[1] on *Ventilago maderaspatana* they encountered a complex mixture of pigments, apparently anthraquinones and anthrones. In fact, as we have already reported,[2] this plant contains numerous anthraquinones and, as we will show, there are even more naphthoquinones. Herein we describe a new group of five isofurano-naphthoquinones, ventilones A–E, isolated from the acetone extract of the root bark.

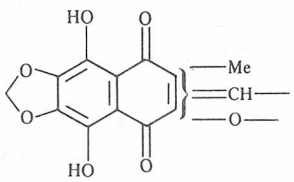
Ventilone-A, $C_{14}H_8O_7$, forms a dimethyl ether and a diacetate, and its ^{1}H-NMR spectrum comprises singlets from a methyl and a methylenedioxy group, an aromatic proton (δ 8.03) and two *peri*-hydroxyls. The latter, and the IR-carbonyl band at 1612 cm^{-1} suggest a naphthazarin structure although the VIS maximum at 433 nm (MeOH), shifting to 488 nm (MeOH—HO$^-$), is inconsistent. However, the dihydro compound, obtained by reduction with zinc and acetic acid, has a VIS spectrum with λ_{max} 527 nm shifting to 590 nm in alkaline solution which is more naphthazarin-like. The ^{1}H-NMR spectrum of the dihydro derivative reveals the presence of a secondary methyl group, and also a methylene group which replaces the aromatic proton in the parent compound.

The PMR evidence indicates partial structure **1** for ventilone-A in which the remaining oxygen must be in an ether function. Two structures, **2** and **4**, are possible, and the latter is most likely as the aromatic signal from **2** would not be as far downfield as δ 8.03 (*cf* **3**[3] and **6**[4]); structure **4** is also preferred on biogenetic grounds, and was confirmed by correlation with ventilone-C (below). It follows that dihydroventilone-A has the tautomeric structure **7**.

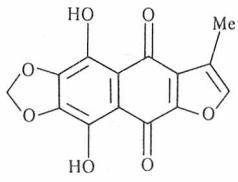
Ventilone-B, $C_{15}H_{10}O_7$, has a ^{1}H-NMR spectrum very similar to that of A but one *peri*-hydroxy signal is replaced by a methoxy. Hence B is a monomethyl ether of A, and on methylation it forms a dimethoxy compound identical with ventilone-A dimethyl ether. As reduction of B with zinc and acetic acid gives racemic ventilone-C of established structure **13** (see below) ventilone-B must be the monomethyl ether **8**. Unlike juglones, ventilone-B shows no peak in the visible region and the long-wave maximum at 392 nm is close to that of 1-hydroxyanthraquinone (402 nm).

After we had identified ventilone-B Barbier *et al.*[5] reported nectriafurone **9**, the first natural isofurano-naphthoquinone, which they isolated from cultures of the fungus *Nectria haematococca*. In principle ventilone-A and nectriafurone have tautomeric structures but we have little evidence. Ventilone-A **4** forms only one diacetate and one dimethyl ether in which H-3 resonates at δ 7.88 and 7.87, respectively. As these values are close to that of H-3 in ventilone-B (δ 7.92) presumably the diacetate and dimethyl ether of A are derivatives of **4** which has a benzenoid ring, rather than **5**. In ventilone-A **4** and nectriafurone **9** H-3 resonates further downfield at 8.03 and 8.10, respectively, suggesting the existence of a naphthazarin-type equilibrium in solution.

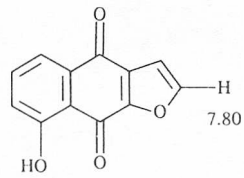
The ^{1}H-NMR spectrum of ventilone-C, $C_{15}H_{12}O_7$, comprises singlets for methoxy, methylenedioxy and *peri*-hydroxy groups, and coupled signals for $CH_3CH<$ and $-CH_2-$ in which the methine (δ 5.40) and the methylene (δ 5.04) protons form an ABX system. The spectrum is very similar to that of dihydroventilone-A **7** but one *peri*-hydroxyl signal is replaced by methoxy. The data, including the ^{13}C-NMR spectrum, are consistent with structures **10** and **11**. Alternative dihydrofuran structures can be excluded by comparison with the ^{1}H-NMR spectrum of haemoventosin **12**.[6] Structure **10** is preferred to **11** on the basis of the long-range coupling constants observed in the dihydroisofuran ring. In the ABX system J_{AB} = 16, J_{AX} = 5, and J_{BX} = 4 Hz the smallest coupling being *cis*.[7] The long-range couplings are larger than those observed in phthalans[8] implying that the dihydrofuran ring is fused to a quinone and not to a benzene ring.



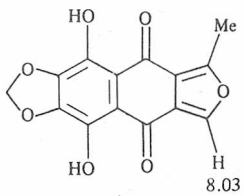
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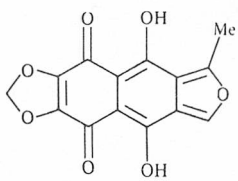
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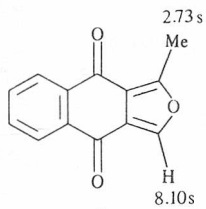
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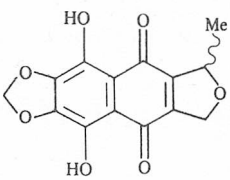
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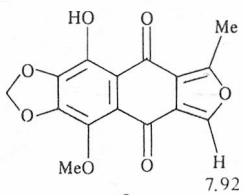
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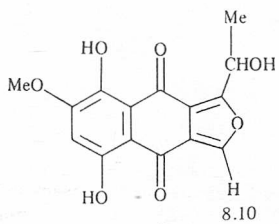
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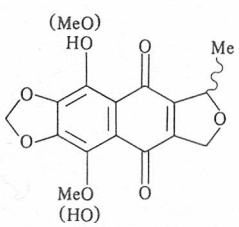
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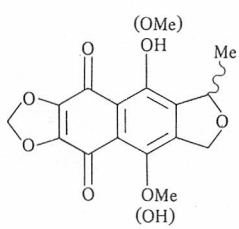
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Further, ventilone-C forms a methyl ether which was reduced to a quinol 15 (R = H); in the ^{1}H-NMR spectra of **15** (R = H) and **15** (R = Ac) the methylene proton signals comprise an AB quartet (J = 12.5 Hz) and long-range coupling to the methine proton was too small to be observed showing that the dihydrofuran ring is now fused to a benzene ring. Thus ventilone-C is one of the structures **10** and the stereochemistry and the position of the methoxy group were finally ascertained by X-ray analysis which established structure **13** for ventilone-C.

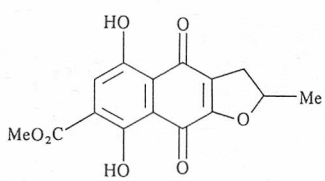
Ventilone-D is isomeric with C and has structure **14**



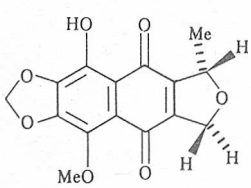
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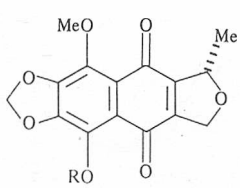
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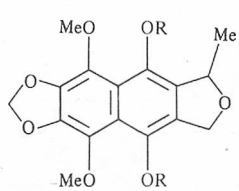
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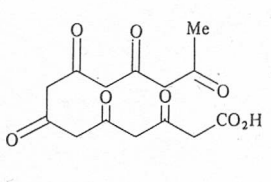
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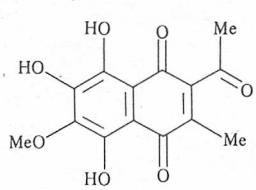
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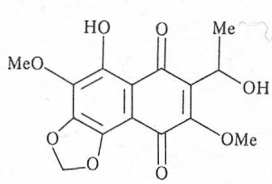
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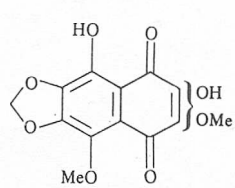
16



17



18



19

(R = H). Both C and D give the same dimethoxy compound on methylation which proved to be identical with ventilone-E; E has therefore structure **15** (R = Me).

Biogenetically these quinones are probably derived from a heptaketide **16** from which the terminal carboxyl group is lost, and it is significant that cordeauxione **17**[9] has also been isolated from *V. maderaspatana*.[10] Formation of a dihydrofuran ring from the side chains of **17**, or an equivalent, is clearly possible in several ways.

It is of interest that all the ventilones possess a methylenedioxy group ($\nu \sim 930$ cm^{-1}, ^{1}H-$\delta \sim 6.2$, ^{13}C-$\delta \sim 104$) which is a rare substituent in natural quinones. Only two methylenedioxynaphthoquinones have been reported previously, lomastilone **18**[11] and nepenthone-A **19**.[12]

EXPERIMENTAL

Plant material was obtained from Srisailam Forest, Andhra Pradesh, India.

Extraction and purification. The air-dried root bark (2.5 kg) was powdered and extracted with acetone. The dark brown extract was fractionated on a column of silica gel, eluting with solvents of increasing polarity, i.e. petroleum ether, C_6H_6 and EtOAc. Individual fractions were examined by TLC and those showing the same spot pattern were combined and subjected to column chromatography again. Ventilone-A was purified on a silica gel column using C_6H_6 as eluting solvent. It crystallised from a large volume of C_6H_6 as orange-red micro crystals, m.p. 275° (dec) (65 mg). Column chromatography of the fractions containing ventilone-B, -C, -D and -E with C_6H_6 : EtOAc (9 : 1) gave ventilone-B which crystallised from C_6H_6 as bright yellow needles, m.p. 236° (dec) (18 mg), and ventilone-C and -D as a mixture. These two compounds were separated by PLC in C_6H_6 : EtOAc (4 : 1) and crystallised from C_6H_6-pet. ether (b.p. 60–80°); ventilone-C red needles, m.p. 196° (32 mg), ventilone-D, dark red needles, m.p. 177–178° (20 mg). Further elution of the column with C_6H_6–EtOAc (4 : 1) gave ventilone-E which on crystallisation from MeOH separated as yellow micro crystals, m.p. 180–181° (48 mg).

Ventilone-A **4**. Found: C, 58.26; H, 2.74%; M$^+$, 288.0270. $C_{14}H_8O_7$ requires C, 58.34; H, 2.80%; M$^+$, 288.0270; UV λ_{max}^{MeOH} 283, 413, 433 nm (log ε 4.09, 3.78, 3.82); $\lambda_{max}^{MeOH/HO^-}$ 488 nm; ν_{max}^{KBr} 930, 1612 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$) δ 2.78 (3H, s, Me), 6.27 (2H, s, —OCH_2O—), 8.03 (1H, s, H-3), 12.98, 13.16 (each 1H, s, exchangeable with D_2O, 2 *peri*-OH); MS *m/z* 288 (100%), 242 (21), 232 (7), 231 (8), 214 (13). The *dimethyl ether* ($Me_2SO_4/K_2CO_3/Me_2CO$) was obtained as pale yellow crystalline globulets, m.p. 189° (C_6H_6-pet. ether); Found: C, 60.72; H, 3.90. $C_{16}H_{12}O_7$ requires: C, 60.76; H, 3.82%; IR ν_{max}^{Nujol} 924, 1658 cm^{-1}; ^{1}H-NMR (90 MHz, $CDCl_3$) δ 2.66 (3H, s, Me), 3.98 (3H, s, OMe), 4.00 (3H, s, OMe), 6.15 (2H, s, —OCH_2O—), 7.87 (1H, s, H-3); MS *m/z* 316 (45%), 315 (100), 301 (17), 299 (10), 282 (15), 273 (29), 271 (13), 270 (13), 269 (36), 260 (17), 259 (13), 258 (35). The *diacetate* (Ac_2O/pyridine) formed light yellow needles, m.p. 230° (C_6H_6-pet. ether); Found: C, 58.24; H, 3.34. $C_{18}H_{12}O_9$ requires: C, 58.09; H, 3.25%; ^{1}H-NMR (90 MHz, $CDCl_3$) δ 2.41, 2.43 (each 3H, s, OAc), 2.65 (3H, s, Me), 6.20 (2H, s, —OCH_2O—), 7.88 (1H, s, H-3). Ventilone-A (25 mg), Zn powder (500 mg) in AcOH (8 ml) was heated on a steam bath for 60 min and worked up in the usual way. The product **7** was obtained as dark red needles, m.p. 174° (C_6H_6-pet. ether); Found: C, 57.86; H, 3.52. $C_{14}H_{10}O_7$ requires: C, 57.94; H, 3.47%; UV λ_{max}^{MeOH} 235, 308, 465, 495, 527 nm; $\lambda_{max}^{MeOH/HO^-}$ 235, 312, 560, 590 nm; IR $\nu_{max}^{CHCl_3}$ 925, 1615 cm^{-1}; ^{1}H-NMR (90 MHz, $CDCl_3$) δ 1.58 (3H, d, J = 7 Hz, Me), 5.10 (2H, m, CH_2-3), 5.50 (1H, m, H-1), 6.20 (2H, s, —OCH_2O—), 11.82, 12.50 (each 1H, exchanged with D_2O, *peri*-OH).

Ventilone-B **8**. Found: C, 59.70; H, 3.36%; M$^+$, 302.0427. $C_{15}H_{10}O_7$ requires C, 59.61; H, 3.33%; M$^+$, 302.0426; UV λ_{max}^{EtOH} 290, 392 nm (log ε 4.18, 3.82); $\lambda_{max}^{EtOH/HO^-}$ 467 nm; IR ν_{max}^{KBr} 928, 1610, 1650 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$) δ 2.70 (3H, s, Me), 3.97 (3H, s, OMe), 6.20 (2H, s, —OCH_2O—), 7.92 (1H, s, H-3), 13.35 (1H, s, exchangeable with D_2O, *peri*-OH); MS *m/z* 302 (100%), 285 (6), 284 (14), 273 (19), 256 (40), 255 (6), 231 (13), 229 (26), 228 (16), 215 (9). The methyl ether of ventilone-B ($Me_2SO_4/K_2CO_3/Me_2CO$) was identical (TLC, m.m.p. and IR) with the dimethyl ether of ventilone-A. Ventilone-B was reduced with zinc and acetic acid, and the product showed the same R_f value (co-TLC) as ventilone-C.

Ventilone-C **13**. Found: C, 59.28; H, 4.00%; M$^+$, 304.0580. $C_{15}H_{12}O_7$ requires C, 59.21; H, 3.98%; M$^+$, 304.0582; $[\alpha]_D^{22}$ + 144° ($CHCl_3$ c 0.16); UV λ_{max}^{EtOH} 230, 270, 323, 454 nm (log ε 4.28, 4.21, 3.14, 3.46); $\lambda_{max}^{EtOH/HO^-}$ 550 nm; IR ν_{max}^{KBr} 930, 1625, 1655 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$), δ 1.54 (3H, d, J = 7 Hz, Me), 4.0 (3H, s, OMe), 5.04 (2H, ddd, J = 16, 5, 4 Hz, 2 × H-3), 5.40 (1H, m, H-1), 6.22 (2H, s, —OCH_2O—), 12.52 (1H, s, exchangeable with D_2O, *peri*-OH); ^{13}C-NMR (90.56 MHz, $CDCl_3$), 20.43 (q, C-1a), 60.76 (q, OMe), 71.97 (t, C-3), 81.14 (d, C-1), 103.75 (t, —O$\underline{C}H_2$O—), 113.39[b] (s, C-8a), 119.09[b] (s, C-

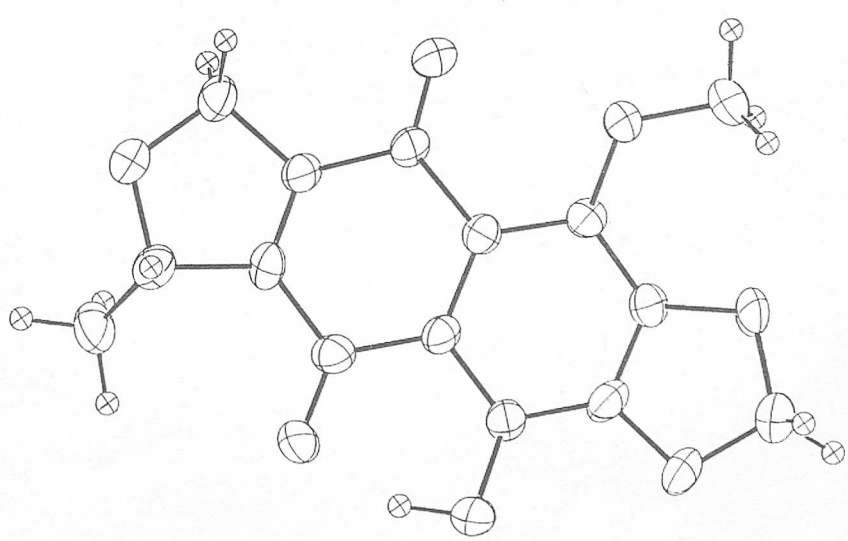


Fig. 1.

Table 1. Fractional atomic co-ordinates ($\times 10^4$) with esds

C(1)	2787(15)	−0611(4)	10 612(8)
O(2)	2772(19)	−1349(3)	10 602(6)
C(3)	2858(17)	−1629(4)	9212(9)
C(3A)	2671(15)	−0982(3)	8336(7)
C(4)	2605(18)	−0950(3)	6832(7)
C(4A)	2465(18)	−0267(3)	6208(6)
C(5)	2443(20)	−0236(3)	4737(7)
C(6)	2465(20)	0409(3)	4155(7)
C(7)	2534(22)	1008(3)	4905(8)
C(8)	2488(18)	1005(3)	6370(7)
C(8A)	2471(16)	0354(3)	7013(7)
C(9)	2476(16)	0292(3)	8552(7)
C(9A)	2568(16)	−0422(3)	9113(7)
O(10)	2444(19)	1564(2)	4024(5)
C(11)	2413(27)	1281(4)	2659(9)
O(12)	2404(18)	0551(3)	2781(6)
C(13)	2739(21)	2245(4)	6554(8)
C(14)	1252(17)	−2162(5)	9027(10)
O(15)	2509(16)	1581(2)	7165(6)
O(16)	2538(14)	−0795(2)	3912(5)
O(17)	2396(12)	0775(2)	9364(5)
O(18)	2551(15)	−1487(2)	6120(5)
H(1A)	1517(76)	−0502(43)	11 109(85)
H(1B)	3814(96)	−0365(38)	11 182(80)
H(3)	4350(37)	−1681(41)	9209(89)
H(11A)	3820(62)	1383(43)	2349(90)
H(11B)	1760(117)	1484(38)	1820(55)
H(13A)	3104(113)	2534(39)	7347(55)
H(13B)	1375(66)	2361(50)	6201(80)
H(13C)	3713(96)	2279(52)	5781(58)
H(14A)	1167(136)	−2381(40)	8078(40)
H(14B)	−0110(69)	−1984(40)	9269(85)
H(14C)	1591(129)	−2545(33)	9702(64)
HO(16)	2297(228)	−1163(38)	4611(82)

Table 2. Bond lengths (Å) with esds

C(1)—O(2)	1.429(9)	C(5)—O(16)	1.346(8)
C(1)—C(9A)	1.501(10)	C(6)—C(7)	1.368(10)
O(2)—C(3)	1.449(10)	C(6)—O(12)	1.356(9)
C(3)—C(3A)	1.517(10)	C(7)—C(8)	1.415(10)
C(3)—C(14)	1.503(14)	C(7)—O(10)	1.374(8)
C(3A)—C(4)	1.455(10)	C(8)—C(8A)	1.405(9)
C(3A)—C(9A)	1.321(9)	C(8)—O(15)	1.354(8)
C(4)—C(4A)	1.456(9)	C(8A)—C(9)	1.491(9)
C(4)—O(18)	1.247(8)	C(9)—C(9A)	1.486(9)
C(4A)—C(5)	1.422(9)	C(9)—O(17)	1.222(8)
C(4A)—C(8A)	1.432(8)	O(10)—C(11)	1.428(10)
C(5)—C(6)	1.370(9)	C(11)—O(12)	1.419(10)
C(13)—O(15)	1.423(9)		

4a), 140.87[c] (s, C-6), 141.02[c] (s, C-7), 144.53[a] (s, C-3a), 146.06[a] (s, C-9a), 146.42 (s, C-5), 148.86 (s, C-8), 179.51 (s, C-4), 186.37 (s, C-9) (values with the same superscript may be interchanged); MS *m/z* 304 (89%), 289 (100), 275 (7), 271 (6), 261 (20); CD λ_{max}^{MeOH} 280, 320, 368, 408 nm ($\Delta\varepsilon$ −0.17, +0.09, −0.28, −0.15). The methyl ether of ventilone-C ($Me_2SO_4/K_2CO_3/Me_2CO$) was identical (TLC, m.m.p.) with ventilone-E.

Ventilone-D **14** (R = H). Found: C, 59.28; H, 4.02%; M^+, 304.0585. $C_{15}H_{12}O_7$ requires C, 59.21; H, 3.98%; M^+, 304.0583; $[\alpha]_D$ +240° ($CHCl_3$, c 0.10); UV λ_{max}^{MeOH} 226, 270, 320, 440 nm (log ε 4.37, 4.19, 3.53, 3.64); IR ν_{max}^{KBr} 930, 1630, 1660 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$) δ 1.56 (3H, d, J = 7 Hz, Me), 4.03 (3H, s, OMe), 5.04 (2H, ddd, J = 15.5, 5.5, 4 Hz, 2 × H-3), 5.39 (1H, m, H-1), 6.20 (2H, s, —OCH_2O—), 12.45 (1H, s, exchangeable with D_2O, *peri*-OH); MS *m/z* 304 (100%), 289.0341 ($C_{14}H_9O_7$ requires 289.0345, 61), 275 (11), 274 (9), 262 (51), 261.0383 ($C_{13}H_9O_6$ requires 261.0389, 47), 247 (18), 246 (9), 233 (15), 219 (19). The *acetate* (Ac_2O/pyridine) formed bright yellow needles, m.p. 198–199° (pet. ether) Found: C, 59.88; H, 4.06. $C_{17}H_{14}O_8$ requires: C, 58.96; H, 4.07%; ^{1}H-NMR (100 MHz, $CDCl_3$) δ 1.56 (3H, d, J = 7 Hz, Me), 2.44 (3H, s, OAc), 4.06 (3H, s, OMe), 5.00 (2H, m, CH_2-3), 5.36 (1H, m, H-1), 6.20 (2H, s, —OCH_2O—). The methyl ether of ventilone-D ($Me_2SO_4/K_2CO_3/Me_2CO$) was found to be identical (TLC, IR, m.m.p.) with ventilone-E.

Ventilone-E **14** (R = Me). Found: C, 60.52; H, 4.46%; M^+, 318.0748. $C_{16}H_{14}O_7$ requires: C, 60.38; H, 4.43%; M^+, 318.0739. $[\alpha]_D^{22}$ +174° ($CHCl_3$, c 0.13); UV λ_{max}^{EtOH} 272, 308, 400 nm (log ε 4.23, 3.55, 3.49); IR ν_{max}^{KBr} 925, 1660 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$) δ 1.56 (3H, d, J = 7 Hz, Me), 4.02 and 4.03 (6H, s, 2 × OMe), 5.00 (2H, ddd, J = 15, 5, 4 Hz, 2 × H-3), 5.36 (1H, m, H-1), 6.16 (2H, s, —OCH_2O—); MS *m/z* 318 (60%), 303.0505 ($C_{15}H_{11}O_7$ requires 303.0504, 100), 290 (6), 289 (8), 288 (9), 285 (10), 275 (17), 273 (20), 261 (6), 260 (8). Ventilone-E (32 mg) in ether (40 ml) was reduced by shaking with a saturated solution of sodium dithionite (4 × 25 ml). The ether layer was evaporated and the residue was immediately sublimed at 177°/0.001 mm Hg to give the quinol (16 mg). Found: M^+, 320.0895. $C_{16}H_{16}O_7$ requires: 320.0896. ^{1}H-NMR (220 MHz, $CDCl_3$), δ 1.60 (3H, d, J = 7 Hz, Me), 4.13 (6H, s, 2 × OMe), 5.09, 5.23 (2H, ABq, J = 12.5 Hz, CH_2-3), 5.50 (1H, q, J = 7 Hz, H-1), 6.03 (2H, s, —OCH_2O—), 8.85, 8.90 (each 1H, s, exchanged with D_2O, —OH). With Ac_2O, Zn dust, and NaOAc, ventilone-E formed a leuco-diacetate as needles (from MeOH) (Found: C, 59.70; H, 4.94%. $C_{20}H_{20}O_9$

Table 3. Bond angles (°) with esds

C(9A)—C(1)—O(2)	103.7(6)	C(3)—O(2)—C(1)	112.3(6)
C(3A)—C(9A)—C(1)	110.0(6)	C(9)—C(9A)—C(1)	125.6(6)
C(3A)—C(3)—O(2)	101.8(6)	C(14)—C(3)—O(2)	109.8(8)
C(14)—C(3)—C(3A)	116.2(8)	C(4)—C(3A)—C(3)	126.5(6)
C(9A)—C(3A)—C(3)	111.4(6)	C(9A)—C(3A)—C(4)	122.1(6)
C(4A)—C(4)—C(3A)	117.0(6)	O(18)—C(4)—C(3A)	121.1(6)
C(9)—C(9A)—C(3A)	124.0(6)	O(18)—C(4)—C(4A)	121.8(6)
C(5)—C(4A)—C(4)	116.9(5)	C(8A)—C(4A)—C(4)	122.5(6)
C(8A)—C(4A)—C(5)	120.5(5)	C(6)—C(5)—C(4A)	116.6(6)
O(16)—C(5)—C(4A)	123.9(5)	C(8)—C(8A)—C(4A)	120.9(6)
C(9)—C(8A)—C(4A)	118.3(5)	O(16)—C(5)—C(6)	119.3(6)
C(7)—C(6)—C(5)	123.8(6)	O(12)—C(6)—C(5)	125.9(6)
O(12)—C(6)—C(7)	110.3(6)	C(8)—C(7)—C(6)	121.7(6)
O(10)—C(7)—C(6)	109.6(6)	C(11)—O(12)—C(6)	106.5(6)
O(10)—C(7)—C(8)	128.4(6)	C(8A)—C(8)—C(7)	116.5(6)
O(15)—C(8)—C(7)	124.3(6)	C(11)—O(10)—C(7)	105.8(5)
O(15)—C(8)—C(8A)	119.2(6)	C(9)—C(8A)—C(8)	120.8(5)
C(13)—O(15)—C(8)	120.7(6)	C(9A)—C(9)—C(8A)	116.0(5)
O(17)—C(9)—C(8A)	125.3(6)	O(17)—C(9)—C(9A)	118.7(6)
O(12)—C(11)—O(10)	107.8(6)		

Table 4. Torsion angles (°) with esds

C(9A)—C(1)—O(2)—C(3)	8.5(9)	O(2)—C(1)—C(9A)—C(3A)	−7.1(9)
O(2)—C(1)—C(9A)—C(9)	179.3(8)	C(1)—O(2)—C(3)—C(3A)	−6.7(9)
C(1)—O(2)—C(3)—C(14)	−130.4(8)	O(2)—C(3)—C(3A)—C(4)	−178.0(8)
O(2)—C(3)—C(3A)—C(9A)	2.0(10)	C(14)—C(3)—C(3A)—C(4)	−58.8(12)
C(14)—C(3)—C(3A)—C(9A)	121.2(9)	C(3)—C(3A)—C(4)—C(4A)	−178.6(8)
C(3)—C(3A)—C(4)—O(18)	5.5(14)	C(9A)—C(3A)—C(4)—C(4A)	1.4(12)
C(9A)—C(3A)—C(4)—O(18)	−174.5(8)	C(3)—C(3A)—C(9A)—C(1)	3.2(10)
C(3)—C(3A)—C(9A)—C(9)	176.9(8)	C(4)—C(3A)—C(9A)—C(1)	−176.8(7)
C(4)—C(3A)—C(9A)—C(9)	−3.1(13)	C(3A)—C(4)—C(4A)—C(5)	178.7(8)
C(3A)—C(4)—C(4A)—C(8A)	2.5(12)	O(18)—C(4)—C(4A)—C(5)	−5.5(13)
O(18)—C(4)—C(4A)—C(8A)	178.3(8)	C(4)—C(4A)—C(5)—C(6)	−175.2(8)
C(4)—C(4A)—C(5)—O(16)	0.2(13)	C(8A)—C(4A)—C(5)—C(6)	1.2(13)
C(8A)—C(4A)—C(5)—O(16)	176.5(8)	C(4)—C(4A)—C(8A)—C(8)	175.1(8)
C(4)—C(4A)—C(8A)—C(9)	−4.4(12)	C(5)—C(4A)—C(8A)—C(8)	−1.0(12)
C(5)—C(4A)—C(8A)—C(9)	179.5(8)	C(4A)—C(5)—C(6)—C(7)	0.9(14)
C(4A)—C(5)—C(6)—O(12)	−178.4(9)	O(16)—C(5)—C(6)—C(7)	−174.6(9)
O(16)—C(5)—C(6)—O(12)	6.1(15)	C(5)—C(6)—C(7)—C(8)	−3.2(16)
C(5)—C(6)—C(7)—O(10)	−177.2(9)	O(12)—C(6)—C(7)—C(8)	176.2(9)
O(12)—C(6)—C(7)—O(10)	2.2(12)	C(5)—C(6)—O(12)—C(11)	178.7(10)
C(7)—C(6)—O(12)—C(11)	−0.7(11)	C(6)—C(7)—C(8)—C(8A)	3.2(14)
C(6)—C(7)—C(8)—O(15)	−178.5(9)	O(10)—C(7)—C(8)—C(8A)	175.9(9)
O(10)—C(7)—C(8)—O(15)	−5.8(16)	C(6)—C(7)—O(10)—C(11)	−2.8(11)
C(8)—C(7)—O(10)—C(11)	−176.2(10)	C(7)—C(8)—C(8A)—C(4A)	−1.1(12)
C(7)—C(8)—C(8A)—C(9)	178.4(8)	O(15)—C(8)—C(8A)—C(4A)	−179.5(8)
O(15)—C(8)—C(8A)—C(9)	0.0(12)	C(7)—C(8)—O(15)—C(13)	−5.0(13)
C(8A)—C(8)—O(15)—C(13)	173.3(8)	C(4A)—C(8A)—C(9)—C(9A)	2.7(11)
C(4A)—C(8A)—C(9)—O(17)	−176.7(8)	C(8)—C(8A)—C(9)—C(9A)	−176.8(7)
C(8)—C(8A)—C(9)—O(17)	3.8(13)	C(8A)—C(9)—C(9A)—C(1)	173.8(7)
C(8A)—C(9)—C(9A)—C(3A)	1.0(12)	O(17)—C(9)—C(9A)—C(1)	−6.8(12)
O(17)—C(9)—C(9A)—C(3A)	−179.6(8)	C(7)—O(10)—C(11)—O(12)	2.4(11)
O(10)—C(11)—O(12)—C(6)	−1.1(11)		

requires: C, 59.41; H, 4.99%); IR ν_{max}^{KBr} 925, 1745 cm^{-1}; ^{1}H-NMR (220 MHz, $CDCl_3$) 1.55 (3H, d, J = 7 Hz, Me), 2.37, 2.36 (6H, s, 2 × OAc), 3.94 (6H, s, 2 × OMe), 5.04, 5.17 (2H, ABq, J = 12.5 Hz, CH_2-3), 5.46 (1H, q, J = 7 Hz, H-1), 6.06 (2H, s, —OCH_2O—).

Crystal data. $C_{15}H_{12}O_7$, M = 304.258. Orthorhombic, a = 6.715 (5), b = 19.365 (11), c = 9.658 (8) Å, U = 1255 (2) $Å^3$, Z = 4, D_c = 1.58 cm^{-3}. Space group $P2_12_12$. Mo-Kα radiation, μ = 0.82 cm^{-1}, 820 independent reflections.

The reflection were measured on a Nicolet P3 automated diffractometer ($2\theta \leqslant 50°$). The structure was solved by direct methods[13] and refined[14] by full matrix least squares. The carbons and oxygens were given anisotropic temperature factors and the hydrogen isotropic temp factors. The final weighting scheme used was $\omega = 1.00/[\sigma^2(F) + 0.00987F^2]$. Convergence was achieved at R = 5.33%.

The molecular structure is shown in Fig. 1. Atomic co-ordinates and other data are listed in Tables 1–5.

Table 5. Observed and calculated structure factors

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	0	0	3376	3734	3	7	0	136	27	2	0	1	520	541	3	6	1	160	153	6	13	1	45	48
4	0	0	1396	1410	0	8	0	130	123	3	0	1	81	87	4	6	1	56	56	0	14	1	142	138
6	0	0	558	580	1	8	0	77	78	4	0	1	297	298	5	6	1	61	65	1	14	1	151	145
8	0	0	239	249	2	8	0	79	82	5	0	1	90	100	1	7	1	149	165	2	14	1	87	87
3	1	0	90	89	4	8	0	59	61	6	0	1	119	117	2	7	1	126	117	3	14	1	126	115
4	1	0	137	125	1	9	0	73	93	7	0	1	57	49	3	7	1	114	126	4	14	1	58	47
5	1	0	75	68	2	9	0	53	57	0	1	1	525	523	4	7	1	92	93	5	14	1	81	77
6	1	0	100	100	3	9	0	53	63	1	1	1	164	174	5	7	1	42	44	6	14	1	41	47
7	1	0	45	52	0	10	0	667	661	2	1	1	421	406	7	7	1	49	36	0	15	1	460	453
0	2	0	1177	1194	2	10	0	481	474	3	1	1	98	104	0	8	1	242	239	1	15	1	101	101
2	2	0	1076	1098	4	10	0	169	164	4	1	1	279	293	1	8	1	93	100	2	15	1	384	382
3	2	0	81	85	2	11	0	96	104	5	1	1	64	72	2	8	1	147	150	3	15	1	94	101
4	2	0	576	598	4	11	0	63	62	6	1	1	173	185	3	8	1	64	78	4	15	1	242	247
6	2	0	276	278	1	12	0	84	80	0	2	1	236	235	4	8	1	94	101	5	15	1	58	51
2	3	0	382	403	2	12	0	64	61	1	2	1	678	677	6	8	1	56	60	6	15	1	141	144
3	3	0	135	115	3	12	0	49	57	3	2	1	197	181	7	8	1	43	47	0	16	1	254	271
4	3	0	200	221	5	12	0	41	30	5	2	1	48	66	0	9	1	283	264	1	16	1	331	335
5	3	0	111	91	2	14	0	56	51	6	2	1	46	47	1	9	1	140	149	2	16	1	236	252
6	3	0	89	111	4	14	0	145	140	0	3	1	246	237	2	9	1	146	140	3	16	1	269	269
7	3	0	42	45	6	14	0	123	123	1	3	1	271	291	3	9	1	107	101	4	16	1	170	169
0	4	0	828	823	0	16	0	555	564	2	3	1	213	222	0	10	1	64	56	5	16	1	172	172
2	4	0	666	683	1	16	0	243	234	3	3	1	178	172	1	10	1	163	155	0	17	1	240	239
3	4	0	74	77	2	16	0	458	455	4	3	1	83	94	2	10	1	117	122	2	17	1	219	206
4	4	0	274	290	3	16	0	163	157	5	3	1	70	62	4	10	1	61	71	4	17	1	157	144

Table 5—*continued.*

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	5	0	101	128	4	16	0	302	294	0	4	1	97	102	6	10	1	39	30	1	18	1	192	189
2	5	0	190	186	5	16	0	84	95	1	4	1	365	350	0	11	1	163	151	3	18	1	134	127
3	5	0	58	71	1	18	0	68	73	2	4	1	73	70	2	11	1	139	133	4	18	1	39	22
4	5	0	59	66	2	18	0	73	74	3	4	1	98	99	4	11	1	73	71	0	19	1	69	67
0	6	0	219	206	3	18	0	71	74	0	5	1	353	348	0	12	1	155	159	1	19	1	52	70
1	6	0	144	150	4	18	0	91	86	1	5	1	273	291	1	12	1	190	186	3	19	1	67	62
2	6	0	153	153	5	18	0	41	29	2	5	1	153	134	2	12	1	97	94	0	20	1	69	77
3	6	0	191	195	1	20	0	51	56	3	5	1	121	128	3	12	1	78	74	1	20	1	58	72
4	6	0	68	67	2	20	0	43	35	5	5	1	63	61	4	12	1	51	58	2	20	1	66	58
6	6	0	53	51	2	21	0	39	29	0	6	1	149	143	6	12	1	47	38	1	21	1	81	79
1	7	0	155	95	1	22	0	41	22	1	6	1	83	77	0	13	1	73	74	3	21	1	60	57
2	7	0	82	83	0	0	1	413	411	2	6	1	160	169	1	13	1	107	101	0	22	1	46	48
1	22	1	74	68	4	7	2	88	95	4	13	2	41	55	0	2	3	100	103	0	9	3	161	153
0	0	2	250	250	5	7	2	105	101	6	13	2	48	48	1	2	3	93	99	1	9	3	497	466
1	0	2	200	192	6	7	2	43	50	0	14	2	89	93	2	2	3	69	75	2	9	3	69	66
2	0	2	100	89	7	7	2	61	61	1	14	2	137	136	4	2	3	53	36	3	9	3	389	372
3	0	2	149	130	1	8	2	773	728	2	14	2	66	73	5	2	3	44	43	5	9	3	197	190
0	1	2	411	405	2	8	2	63	43	3	14	2	108	104	1	3	3	637	623	7	9	3	91	86
2	1	2	292	276	3	8	2	472	448	5	14	2	44	43	2	3	3	159	152	0	10	3	343	316
3	1	2	145	154	5	8	2	234	225	6	14	2	52	57	3	3	3	229	234	1	10	3	78	75
4	1	2	132	136	7	8	2	106	108	3	15	2	82	81	4	3	3	77	92	2	10	3	276	277
5	1	2	45	55	0	9	2	301	293	5	15	2	69	60	5	3	3	64	59	4	10	3	128	130
0	2	2	152	155	1	9	2	451	434	4	16	2	46	29	0	4	3	307	314	0	11	3	83	83
1	2	2	616	620	2	9	2	277	266	0	17	2	44	38	1	4	3	67	88	1	11	3	76	85
2	2	2	173	182	3	9	2	246	238	1	17	2	157	155	2	4	3	233	220	2	11	3	60	54
3	2	2	211	216	4	9	2	164	162	3	17	2	90	88	4	4	3	95	97	3	11	3	84	83
4	2	2	100	101	5	9	2	116	117	5	17	2	56	55	5	4	3	45	32	5	11	3	44	37
5	2	2	47	53	6	9	2	57	53	0	18	2	82	83	6	4	3	59	58	0	12	3	98	96
0	3	2	257	261	7	9	2	59	63	1	18	2	46	54	0	5	3	165	150	1	12	3	77	89
1	3	2	513	522	0	10	2	89	79	2	18	2	79	73	1	5	3	523	521	2	12	3	64	77
2	3	2	329	332	1	10	2	312	291	3	18	2	59	55	3	5	3	289	282	3	12	3	58	46
3	3	2	98	98	2	10	2	92	83	4	18	2	43	53	5	5	3	89	87	0	13	3	69	66
4	3	2	185	174	3	10	2	175	174	1	19	2	78	76	0	6	3	73	72	1	13	3	177	182
6	3	2	61	62	5	10	2	89	101	4	19	2	55	59	1	6	3	240	238	2	13	3	69	70
1	4	2	101	124	7	10	2	57	61	0	20	2	47	49	2	6	3	69	65	3	13	3	116	122
2	4	2	72	59	0	11	2	190	192	2	20	2	47	50	3	6	3	123	122	4	13	3	57	54
1	5	2	251	233	1	11	2	136	135	3	20	2	38	28	4	6	3	48	47	5	13	3	42	31
2	5	2	121	113	2	11	2	144	149	0	21	2	85	92	5	6	3	61	60	0	14	3	224	219
3	5	2	74	74	3	11	2	64	64	2	21	2	68	55	0	7	3	231	240	1	14	3	56	61
4	5	2	58	63	4	11	2	83	75	3	21	2	36	36	1	7	3	695	649	2	14	3	189	179
1	6	2	292	273	0	12	2	72	70	1	22	2	51	55	2	7	3	74	80	3	14	3	59	54
3	6	2	202	187	1	12	2	290	282	0	0	3	844	838	3	7	3	423	397	4	14	3	100	101
5	6	2	111	121	2	12	2	77	78	1	0	3	119	116	4	7	3	45	30	1	15	3	101	99
7	6	2	72	75	3	12	2	161	157	2	0	3	634	621	5	7	3	201	207	0	16	3	45	36
0	7	2	467	440	5	12	2	51	53	3	0	3	81	84	7	7	3	90	94	1	16	3	108	112
1	7	2	429	407	0	13	2	44	37	4	0	3	278	278	0	8	3	134	130	2	16	3	60	70
2	7	2	271	257	1	13	2	154	151	6	0	3	83	83	2	8	3	145	145	3	16	3	74	84
3	7	2	231	213	3	13	2	125	123	1	1	3	125	114	4	8	3	82	77	4	16	3	44	49
5	16	3	66	79	4	4	4	47	40	1	12	4	137	137	5	0	5	57	56	5	6	5	55	68
1	17	3	347	357	5	4	4	70	60	2	12	4	90	85	6	0	5	68	64	6	6	5	36	38
2	17	3	44	34	6	4	4	37	39	3	12	4	87	78	0	1	5	618	579	0	7	5	87	88
3	17	3	212	212	0	5	4	211	199	4	12	4	43	46	2	1	5	471	461	1	7	5	95	89
1	19	3	119	118	1	5	4	195	198	0	13	4	149	150	3	1	5	51	53	3	7	5	57	59
3	19	3	117	117	2	5	4	108	103	1	13	4	71	74	4	1	5	256	253	4	7	5	45	34
1	20	3	59	52	3	5	4	67	64	2	13	4	134	133	5	1	5	51	36	6	7	5	36	26
1	21	3	91	88	4	5	4	51	50	3	13	4	66	65	6	1	5	140	135	0	8	5	107	111
0	0	4	667	672	1	6	4	73	70	0	14	4	58	56	0	2	5	154	147	1	8	5	60	62
1	0	4	75	81	3	6	4	102	115	1	14	4	53	52	1	2	5	199	203	2	8	5	49	50
2	0	4	422	426	5	6	4	65	70	2	14	4	42	32	2	2	5	166	162	3	8	5	66	63
3	0	4	69	66	0	7	4	163	167	3	14	4	44	41	3	2	5	188	191	0	9	5	214	224
4	0	4	196	200	2	7	4	98	95	0	15	4	302	326	4	2	5	107	117	1	9	5	54	61
6	0	4	122	116	4	7	4	45	47	1	15	4	84	86	5	2	5	93	90	2	9	5	180	192
7	0	4	42	14	6	7	4	44	37	2	15	4	269	277	6	2	5	71	65	3	9	5	58	64
0	1	4	466	460	0	8	4	67	73	3	15	4	72	74	7	2	5	39	30	4	9	5	116	123
1	1	4	380	386	1	8	4	54	54	4	15	4	160	159	1	3	5	181	176	6	9	5	61	66
2	1	4	349	337	2	8	4	57	54	0	16	4	253	271	2	3	5	84	76	1	10	5	99	101
3	1	4	253	265	3	8	4	48	57	2	16	4	198	204	3	3	5	103	104	4	10	5	39	34
4	1	4	214	207	5	8	4	46	32	4	16	4	106	107	4	3	5	68	67	0	11	5	227	229
5	1	4	102	95	0	9	4	144	136	5	16	4	58	41	5	3	5	55	51	1	11	5	116	115
6	1	4	122	118	1	9	4	125	122	0	17	4	160	167	0	4	5	126	111	2	11	5	160	171

Table 5—*continued.*

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
7	1	4	39	34	2	9	4	137	124	2	17	4	131	127	1	4	5	228	218	3	11	5	64	70
1	2	4	575	561	4	9	4	89	83	4	17	4	67	86	2	4	5	75	71	4	11	5	60	66
2	2	4	105	88	1	10	4	130	125	0	18	4	54	65	3	4	5	75	82	5	11	5	51	44
3	2	4	283	281	2	10	4	88	82	2	18	4	72	73	4	4	5	45	54	0	12	5	149	162
4	2	4	103	99	3	10	4	102	111	3	18	4	45	34	6	4	5	41	43	1	12	5	46	41
5	2	4	119	135	4	10	4	54	51	4	18	4	66	71	0	5	5	114	102	2	12	5	100	95
6	2	4	58	65	5	10	4	45	54	0	19	4	57	56	1	5	5	166	160	3	12	5	42	50
7	2	4	51	59	0	11	4	164	160	2	19	4	56	40	2	5	5	50	53	5	12	5	43	34
0	3	4	103	115	1	11	4	178	175	0	21	4	79	73	3	5	5	80	71	0	13	5	162	170
1	3	4	279	260	2	11	4	126	126	0	0	5	379	382	0	6	5	77	77	2	13	5	155	152
2	3	4	55	64	3	11	4	92	89	1	0	5	58	57	1	6	5	75	72	4	13	5	111	102
3	3	4	92	97	4	11	4	51	53	2	0	5	223	218	2	6	5	74	76	0	14	5	87	87
1	4	4	201	197	5	11	4	47	45	3	0	5	82	77	3	6	5	44	53	1	14	5	66	59
3	4	4	137	135	0	12	4	83	81	4	0	5	102	97	4	6	5	54	60	2	14	5	111	111
3	14	5	71	74	0	6	6	200	204	0	13	6	61	62	4	4	7	73	70	0	11	7	108	108
4	14	5	98	99	1	6	6	72	78	1	13	6	182	178	5	4	7	46	41	2	11	7	98	97
5	14	5	66	60	2	6	6	152	158	2	13	6	49	37	6	4	7	54	49	3	11	7	51	42
0	15	5	80	88	4	6	6	88	95	3	13	6	103	106	0	5	7	218	218	4	11	7	58	61
1	15	5	121	138	5	6	6	40	41	5	13	6	44	48	2	5	7	175	165	5	11	7	56	46
2	15	5	97	105	0	7	6	60	59	0	14	6	196	190	4	5	7	102	96	1	12	7	141	144
3	15	5	105	101	1	7	6	405	429	2	14	6	147	138	5	5	7	54	55	2	12	7	63	64
4	15	5	94	98	2	7	6	45	49	4	14	6	75	82	6	5	7	60	67	3	12	7	85	84
1	16	5	104	99	3	7	6	316	317	1	15	6	99	104	0	6	7	165	170	4	12	7	62	67
3	16	5	69	81	4	7	6	54	46	3	15	6	85	88	1	6	7	231	229	1	13	7	142	141
1	17	5	144	137	5	7	6	196	198	0	16	6	62	74	2	6	7	150	156	2	13	7	47	39
3	17	5	99	92	6	7	6	49	43	2	16	6	86	94	3	6	7	177	182	3	13	7	67	72
4	17	5	47	45	0	8	6	209	214	0	17	6	42	46	4	6	7	95	95	0	14	7	94	94
0	18	5	97	95	2	8	6	159	167	1	17	6	84	81	5	6	7	146	152	1	14	7	79	81
1	18	5	49	35	3	8	6	42	50	2	17	6	41	31	6	6	7	64	53	2	14	7	57	66
2	18	5	70	59	4	8	6	100	114	3	17	6	43	69	0	7	7	179	178	3	14	7	60	59
0	0	6	535	531	6	8	6	65	67	1	19	6	67	61	1	7	7	359	352	0	15	7	39	27
2	0	6	307	297	0	9	6	105	104	0	0	7	122	133	2	7	7	176	177	1	16	7	36	32
3	0	6	48	49	1	9	6	445	442	2	0	7	60	68	3	7	7	272	269	2	16	7	47	38
4	0	6	82	77	2	9	6	105	111	5	0	7	40	27	4	7	7	124	131	0	1	8	104	101
1	1	6	63	68	3	9	6	328	328	0	1	7	77	84	5	7	7	181	174	1	1	8	42	45
4	1	6	50	53	4	9	6	90	93	1	1	7	197	198	0	8	7	57	56	2	1	8	72	64
6	1	6	54	40	5	9	6	221	208	2	1	7	71	69	1	8	7	584	605	3	1	8	78	79
2	2	6	69	76	6	9	6	60	52	3	1	7	87	95	2	8	7	61	61	4	1	8	45	36
4	2	6	45	59	0	10	6	432	442	4	1	7	60	58	3	8	7	452	463	5	1	8	43	46
0	3	6	140	142	1	10	6	111	119	6	1	7	53	31	5	8	7	275	267	1	2	8	118	127
1	3	6	266	256	2	10	6	383	386	0	2	7	65	68	1	9	7	400	399	3	2	8	84	82
2	3	6	83	89	3	10	6	64	57	1	2	7	260	273	3	9	7	316	308	5	2	8	56	53
3	3	6	175	172	4	10	6	242	238	3	2	7	150	143	4	9	7	60	45	0	3	8	70	66
5	3	6	71	75	5	10	6	38	29	5	2	7	46	51	5	9	7	189	183	1	3	8	56	49
0	4	6	178	173	1	11	6	71	81	2	3	7	59	57	0	10	7	158	156	2	3	8	44	42
1	4	6	81	82	2	11	6	45	62	3	3	7	43	39	1	10	7	139	137	4	3	8	46	38
2	4	6	131	145	3	11	6	84	86	4	3	7	78	67	2	10	7	134	123	0	4	8	42	52
1	5	6	235	237	4	11	6	45	45	0	4	7	110	110	3	10	7	127	129	2	4	8	48	45
3	5	6	132	125	5	11	6	79	73	2	4	7	96	96	4	10	7	78	78	2	5	8	58	63
5	5	6	53	46	0	12	6	47	45	3	4	7	63	62	5	10	7	103	103	4	5	8	49	67
0	6	8	58	62	0	10	8	52	60	2	0	9	427	452	1	9	9	47	48	0	6	10	81	75
1	6	8	199	202	1	10	8	52	52	4	0	9	274	287	3	9	9	53	50	1	6	10	44	54
2	6	8	68	76	2	10	8	66	68	0	1	9	52	41	0	10	9	160	162	2	6	10	78	67
3	6	8	148	143	4	10	8	38	41	1	1	9	72	88	2	10	9	130	128	0	7	10	54	57
4	6	8	58	52	0	11	8	92	101	2	1	9	69	66	3	10	9	43	37	1	7	10	50	45
5	6	8	90	83	2	11	8	71	71	4	1	9	62	54	0	12	9	63	63	2	7	10	65	58
0	7	8	194	197	3	11	8	60	59	0	2	9	173	186	2	12	9	49	47	0	8	10	62	59
2	7	8	141	141	1	12	8	52	53	2	2	9	145	154	1	13	9	78	71	1	8	10	107	101
3	7	8	51	47	3	12	8	44	48	4	2	9	97	93	2	13	9	42	37	2	8	10	47	46
4	7	8	78	75	0	13	8	53	52	1	3	9	38	33	0	14	9	44	63	0	9	10	69	73
0	8	8	97	109	1	13	8	46	47	3	3	9	48	58	0	0	10	94	102	1	9	10	83	74
1	8	8	102	92	2	13	8	48	37	0	4	9	244	250	2	0	10	66	71	2	9	10	63	67
2	8	8	119	116	3	13	8	42	45	2	4	9	189	191	0	3	10	68	67	0	10	10	68	76
3	8	8	108	97	0	14	8	56	39	4	4	9	94	88	1	3	10	45	48	0	11	10	37	37
4	8	8	91	86	1	14	8	68	65	3	5	9	52	45	2	3	10	44	48	1	1	11	58	46
5	8	8	90	94	0	15	8	54	57	1	6	9	47	45	3	3	10	41	37	2	1	11	42	21
0	9	8	53	67	2	15	8	45	49	3	6	9	37	19	1	4	10	40	42	2	2	11	37	17
1	9	8	62	60	0	16	8	61	60	1	7	9	93	93	3	4	10	41	52	1	4	11	34	16
2	9	8	78	73	1	16	8	109	98	3	7	9	68	62	0	5	10	60	58	0	5	11	55	56
4	9	8	53	58	0	0	9	499	531	0	8	9	48	39	2	5	10	52	38	1	6	11	122	116

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