STRUCTURAL STUDIES ON PEPTIDES, ALKALOIDS AND THEIR COMPLEXES

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A Thesis presented for the degree of Doctor of Philosophy.

University of Edinburgh, 1984

DECLARATION

This thesis is the original composition of the author's work, unless otherwise stated, and it has not been submitted for any other degree.

LECTURES AND COURSES ATTENDED

In the course of this work the following lectures and courses were attended.

Natural Products	5 lectures	Prof. A. I. Scott
Biosynthesis	5 lectures	Dr. T. J. Simpson
Current Topics In Organic Chemistry	15 lectures	Members of the Organic Staff
Bio-organic Chemistry	5 lectures	Invited Speakers
Medicinal Chemistry	5 lectures	Dr. R. M. Paton
Synthesis and mode of action of β-lactam anti-biotics	5 lectures	Speakers from Glaxo Research
Chemistry of the Photographic process	5 lectures	Speakers from Kodak
Pulse sequences and Applications in NMR	5 lectures	Dr. G. A. Morris
X-ray crystallography	10 lectures	Dr. R. O. Gould Dr. M. D. Walkinshaw Dr. A. J. Welch
One week course in FORTRAN77		Staff of E.R.C.C.

In addition, the following group seminars have been attended:

Bio-organic group weekly meetings	One year
Crystallography " "	Two years
Organic Chemistry evening seminars	Two years

& departmental colloquia over a period of three years.

ACKNOWLEDGEMENTS

I would like to thank Dr. R. L. Baxter, Dr. R. O. Gould and Professor A. I. Scott for their help, encouragement and patient instruction while I was under their supervision. I would also like to thank Dr. M. D. Walkinshaw, who has been a supervisor in all but name, for all the guidance he has given me both in practical technique and the many discussions of the results presented here.

I would like to thank Miss Rosemary Kelly for the crystals of N-acetyl methionine and strychnine nitrate and Dr. E. M. Gordon, who prepared both the two disulphide compounds whilst working for Professor Scott in 1973.

The N.M.R spectra discussed in the disulphide chapters were measured by Dr. A. Boyd and Dr. D. Reid, to both of whom I would like to express my thanks.

During the period in which this work was carried out, I was supported by a post-graduate studentship from the Department of Education for Northern Ireland, for which I am very grateful.

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Finally, I must thank my wife Jennifer for every kind of immeasurable assistance while I was doing this work. In a very real sense this thesis is as much the result of her work as it is of mine.

TO JENNIFER
AND TO ANTARA DAWN

ABSTRACT

This dissertation reports eight crystal structure determinations carried out on amino acids, peptides, alkaloids and their complexes. Two of the compounds (N-phenylacetyl cysteinyl penicillamine cyclic disulphide methyl ester and bis(N-phenylacetyl) cystinyl bis(valine methyl ester)) were examined as part of a project on model compounds for penicillin biosynthesis, while the others (N-acetyl methionine, DL-glutamic acid monohydrate, strychnine, strychnine nitrate, brucine ethanolate dihydrate and strychnine: N-acetyl tyrosine tetrahydrate) were examined during a project on molecular recognition.

The first chapter contains a brief description of the monocyclic intermediate hypothesis of penicillin biosynthesis and the attempts to verify it, along with a discussion of molecular recognition phenomena ranging from substrate: receptor interactions of biological molecules to the resolution of racemic mixtures of amino acids by complexation with chiral bases.

The second chapter is a description of the methods used in determining X-ray structures, from the initial photographs and data collection, through the solution and refinement processes to the production of the results.

The structure of N-phenylacetyl cysteinyl penicillamine cyclic disulphide methyl ester is described in chapter 3. This compound was refined to R = 0.0511 on the basis of 1259 data. The conformation of the ring is compared with that of two other cyclic cysteine derivatives and the conformation of the disulphide linkage is compared with those of a selection of cystine derivatives from the Cambridge Crystallographic Database. The chapter also includes a discussion of an nmr solution conformation study carried out on the cyclic disulphide.

Chapter 4 is a description of the structure of bis(N-phenylacetyl) cystinyl bis(valine methyl ester). The asymmetric unit of this compound contains 4 independent half molecules, one of which is disordered. The

structure was refined to R = 0.0912 based on 2201 data. The conformation is compared with that of the antibiotic tetra N-desmethyl triostin A (TANDEM), which also contains an intramolecularly hydrogen bonded cystinyl valinyl fragment. This chapter also contains a description of a solution study on the disulphide.

The next chapter describes the structure of N-acetyl methionine, which was refined to R = 0.0465 on the basis of 947 data. The conformation of the methionine side chain is compared with that of 20 other methionine residues from the Cambridge database.

Chapter 6 describes the structure of DL-glutamic acid monohydrate, which was refined to R = 0.0376 with 1154 independent reflections. Once again the Cambridge database was used to provide a comparison of side chain conformation, with 24 independent glutamate residues found.

The next two chapters describe the structures of strychnine and strychnine nitrate. The free base refined to R = 0.0370 based on 1195 data, while the salt refined to R = 0.0555 based on 1076 reflections. The structures are compared with each other, and in the case of the nitrate salt, with a range of other strychnine salts. The chapter on the nitrate salt also includes a description of the calculation of an "idealised" strychnine cation from all the coordinates measured to date, which could be used for the rotation searching of Patterson vector maps of other strychnine structures. Listings of both the programs used are included in this chapter.

Chapter 9 describes the structure of brucine ethanolate dihydrate, which may be considered as an alcohol inclusion complex. This structure refined to R = 0.0387 with 1684 data. The structure is compared with the strychnine free base.

The last chapter discusses the molecular complex of strychnine with N-acetyl tyrosine. In this complex the tyrosine moiety is disordered over two sites of equal occupancy and the water molecules are also disordered. However, it was possible to refine the structure to R = 0.104 based on 1162 data by using rigid groups and distance constraints for the disordered molecule. The structure is compared with that of

a strychnine: N-benzoyl alanine complex.

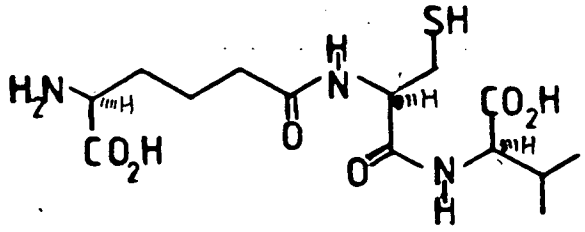
CONTENTS

CHAPTER ONE
INTRODUCTION

INTRODUCTION

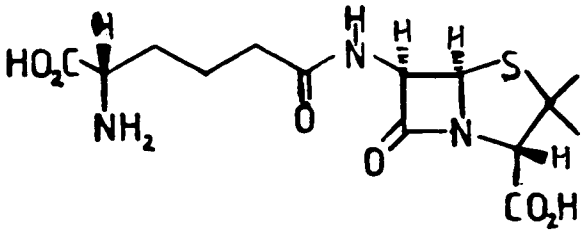
Cystine Peptides

Although it has been known for nearly twenty five years that the penicillins are derived from the tripeptide L-α-aminoadipyl L-cysteinyl D-valine (I) (Arnstein *et al*, 1960), the exact mechanism of the cyclisation of the tripeptide to the bicyclic isopenicillin N ring system (II) is unknown. Several proposals have been made for the existence of a cyclic peptide or thiazepine derivative, either of which could possibly be oxidised to the bicyclic system.



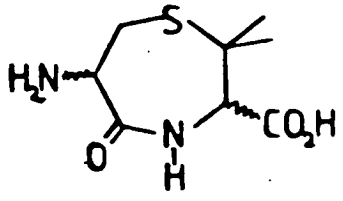
(I)

(II)



The first attempt to verify this hypothesis was in 1958 when it was reported by Arnstein that a cyclic peptide (III), possibly formed *in vivo* by an attack by the cysteine sulphur on the hydroxyl function of β-hydroxy valine, did not lower the rate at which a colony of *Penicillium chrysogenum* utilised a substrate for β-lactam manufacture. This failure to validate the hypothesis did not however immediately disprove it as the peptide used was a statistical mixture of all possible stereoisomers, and there was no evidence that it was even taken into

the mycelia. In 1965, van Tamelin found that the N-phenylacetyl derivative (IV) of the correct stereoisomer was not incorporated into penicillin. Again, it was considered possible that the material was not taken into the mycelia. It was also considered possible that the L-α-aminoadipyl side chain was necessary for recognition by the enzyme. It has since been shown that this is the case. A compound bearing the L-α-aminoadipyl side chain (V) has been synthesised (Wolfe, 1981) but it has since been shown to take no part in the biosynthetic pathway (Baldwin *et al*, 1981).



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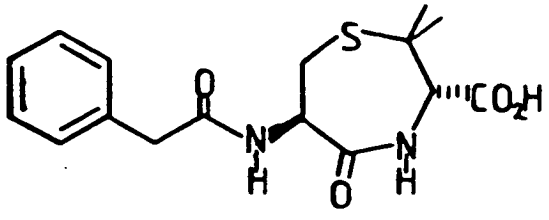

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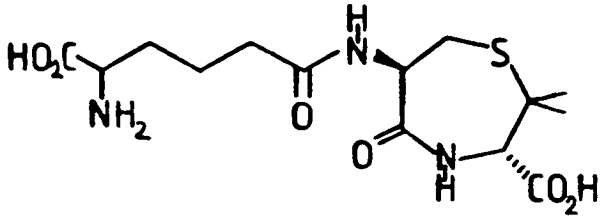
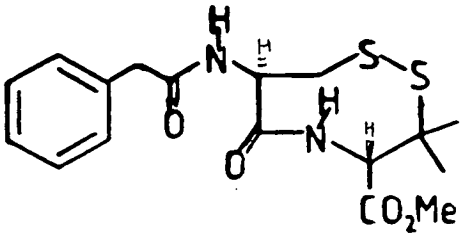


(V)

The cyclic disulphide compound (VI) was prepared as a model for the oxidation of the cyclic peptide to a bicyclic system (VII). No β-lactam product was obtained under any of the oxidation conditions used, however, which suggests that there is some form of steric hindrance to the reaction pathway.



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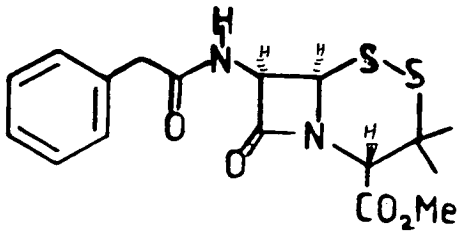

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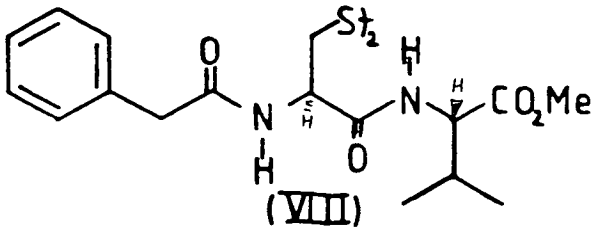
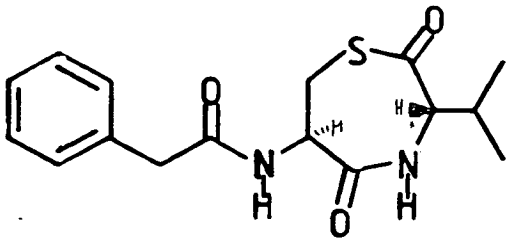


(VIII)

The difficulty of closing the ring by oxidation depends greatly on the conformation of the peptide bond, as can be seen by examination of space-filling models of both isomers. It was therefore of importance to determine the configuration of the amide linkage, so that the probability of a successful bicyclisation could be assessed accurately.

The dimeric disulphide (VIII) was prepared during the same investigation of β-lactam biosynthesis as a synthetic intermediate in the preparation of the thiolactone (IX), which was also at one point considered to be a valid model for the cyclisation of the Arnstein tripeptide to the β-lactam system. As the disulphide (VIII) is structurally related to the cyclic compound (VI), its crystal structure is of interest from the point of view of a comparison of molecular conformation.



(IX)

Molecular Recognition

Molecular recognition is a fundamental part of a wide range of processes including catalysis, diastereomeric salt resolutions, drug receptor interactions and enzyme substrate reactions. (The latter two processes may involve similar effects, and are examples of biological recognition.)

Biological recognition at the molecular level involves the binding of two mutually recognising species. In most cases, these are a macromolecule with an "active site" or *receptor* (usually a protein) and a much smaller, possibly flexible molecule or *drug* such as a substrate, inhibitor, neurotransmitter, hormone or other pharmacologically active substance. (Richards *et al*, 1975).

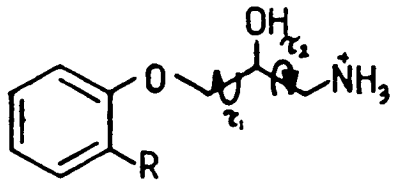
The ideal method for the study of biological recognition is to extract and purify the macromolecule and solve its crystal structure both in the native state, and with a variety of smaller molecules bound to the active site. An example of this approach is Lipscomb's work on zinc carboxy-peptidase (1970, 1971 & 1972), using both enzyme inhibitors and model substrates, but it has not yet been possible to extend this treatment to drug receptor complexes. In any case, this method is extremely time-consuming and not really rewarding in terms of detailed measurement of the recognition interactions as the resolution of crystal structures of macromolecules may not be sufficiently high.

In general then, the detailed structure of the receptor is unknown, as is its environment. It is possible that the substrate molecule may be required to adopt a different conformation from that adopted in the solid state or in solution. This becomes more likely when it is noted that some transmitters have more than one biological role and react with more than one specific receptor. Examples of this include acetylcholine, histamine and noradrenaline, each of which have synthetic analogues that stimulate or inhibit their different receptors to greater or lesser degrees.

As direct crystallographic examination of receptor substrate complexes is virtually impossible, the use of some form of model system is mandatory. The types of model system used can be divided into physical models, where the interactions between molecules are measured from crystal structures, thermodynamic data and the like, and theoretical methods, where molecular interactions are measured or inferred from energy calculations.

A simple example of the theoretical approach is that of Richards *et al* (1975). Working from the premise that as molecules bound into active sites are often distorted from their equilibrium conformation, standard structural methods (such as X-ray crystallography or nmr of solutions) cannot provide a full picture, they have correlated the degree of biological activity with the theoretically calculated potential energy surfaces for the phenoxy propanolamine β-adrenoceptor agonists (X) and the histamine H1 activity of a range of methyl histidines (XI).

(X)



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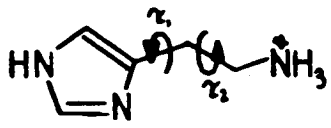


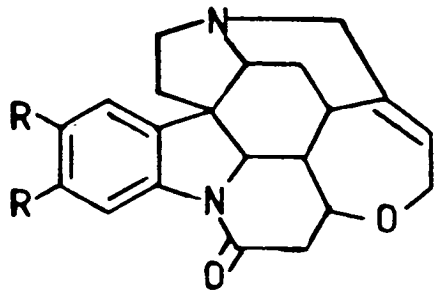
They show that the efficacy of a drug has a linear correlation with the population of the relevant conformational form. The method is, however, incapable of indicating whether the conformation found is that of the molecule when at the active site, or if it is a transition state between the free and the bound conformations that the molecule must pass through to reach the active site.

A more rigorous approach to the problem is taken by Pincus and Scheraga (1981), who examine molecular recognition and catalysis from

theoretical calculations on the enzyme substrate complex. This entails two stages of calculations. The first stage is the calculation of energy over all conformational space for the enzyme, substrate and bound complex so that the global minimum energy conformation can be found. The second stage of the calculation is the use of quantum mechanical calculations on the enzyme-substrate complexes produced in the first step to identify "good" or "bad" substrates. Although this method is very demanding of computer resources, it has been successfully applied to the prediction of the structures of a complex of α-chymotrypsin with N-acetyl-L-phenyl alaninamide and of lysozyme with N-acetyl glucosamine hexamer and the results compare favourably with crystal structures involving similar substrates.

At its simplest level, the physical approach to molecular recognition involves the examination of a crystal structure of a molecule (or series of related molecules) with reference to its preferred environment and configuration. The underlying assumption is that this environment will be similar to that of the molecule when bound to an active site, as almost all the interactions possible between protein residues may be found in crystal structures of amino acids and peptides, if enough structural determinations are compared.

The series of strychnine salts examined in Edinburgh show that the strychnine cation (XIIa, R = H) has a strong preference for a particular environment. Other reasons for using strychnine as a model for recognition studies are that it has a fairly rigid structure (causing it to take the same conformation in solution or on a receptor as it has in the solid state) and that it has several possible "interactive sites". These include an aromatic ring which could interact with a hydrophobic pocket in the receptor, a protonated nitrogen which could form a hydrogen bond to glutamate or aspartate residues and a carbonyl group which could act as a hydrogen bond acceptor. The alkaloid brucine (XIIb, R = OCH_3) has similar interactive capabilities, but possibly due to its larger hydrophobic region it does not have quite the same properties as strychnine either *in vivo* as a central stimulant nor *in vitro* as a resolving agent.



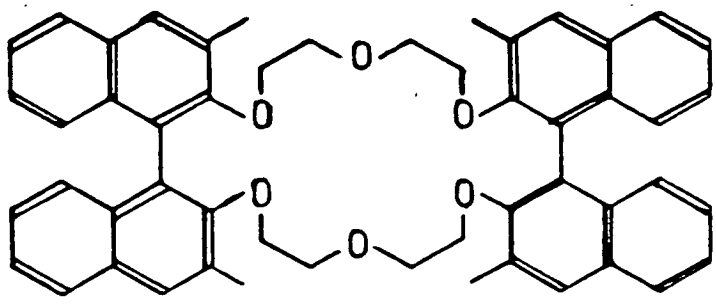
(XIIa) R=H

(XIIb) R=OCH_3

It is of interest that the relative positions of these interactive sites correspond closely to the positions of similar residues in the major loop of α-cobratoxin (Walkinshaw & Saenger, 1984), which has similar physiological effects to strychnine.

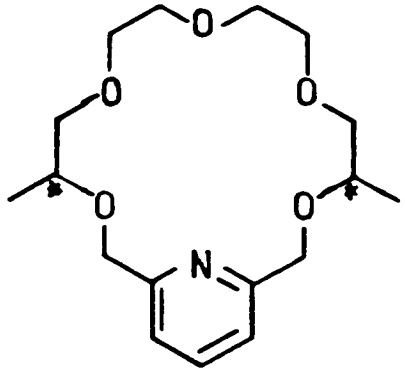
There have been several studies on host:guest complexation both in the solid state and in solution. Solution studies have been carried out on chiral recognition by crown ether derivatives or cryptands using a number of techniques to measure enantioselectivity.

In a study of chiral recognition of six amino acids and their methyl esters (alanine, methionine, valine, phenylalanine tryptophan and phenylglycine, all as the perchlorate salt) by crown ether analogues (XIII) containing substituted dinaphthyl residues (Lingenfelter *et al*, 1981), aqueous solutions of the racemic amino acids or esters were extracted enantioselectively with organic solvent containing the crown ether (this method is the basis of an amino ester resolving machine (Newcomb *et al*, 1974)).



(XIII)

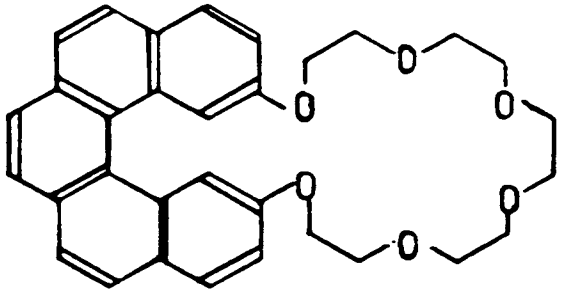
A study of chiral recognition of chiral alkylammonium salts by dimethyldioxopyridino-18-crown-6 (XIV) (Bradshaw *et al*, 1982) used the three independent methods of temperature dependent nmr, titration calorimetry and selective crystallisation to demonstrate the efficiency of their host. This report also contains the first measurement of the enthalpy and entropy of a chiral recognition reaction. The authors also note that the dimethyl substituted compound is more enantioselective than the diphenyl analogue.



(XIV)

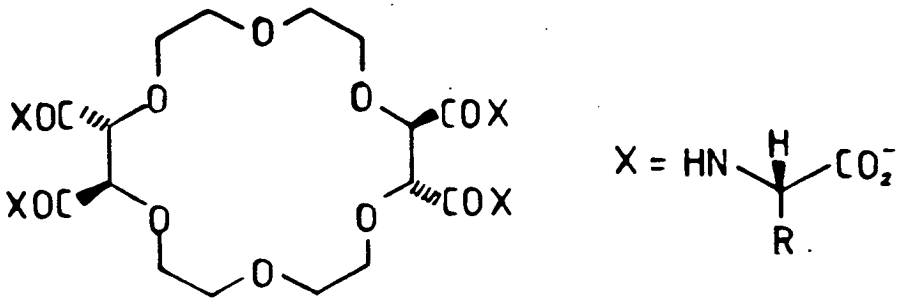
It has recently been shown (Nakazaki *et al*, 1983) that pentahelicene and hexahelicene containing crown ethers were able to resolve methyl phenylglycinate and phenylethylamine hydrochlorides to about 25% optical

purity, except that the pentahelicene compound (XV) showed a 75% purity when used with the methyl phenylglycinate hydrochloride. No rationalisation of this is given, other than to say it is consistent with studies made using Corey Pauling Koltun space-filling models.



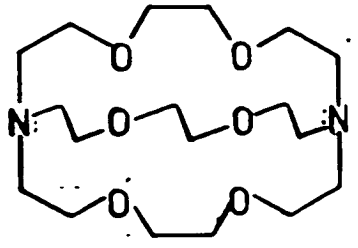
(XV)

Crown ethers bearing amino acid side chains (XVI) have been used as model receptors in a study of structural effects and substrate recognition of organic and biogenic ammonium ions (Behr *et al*, 1982), in which the interactions examined are divided into "central" and "lateral" discrimination. The central discrimination is derived from the size of the Cα substituent and the number of protons on the quaternary nitrogen, and the lateral discrimination is derived from electrostatic and hydrophobic interactions between the guest molecule and the macrocycle side chains. However, the host molecule still binds potassium or ammonium cations more tightly than any of the organic ammonium ions.



(XVI)

Recently, a comparison of the receptor properties of 18-crown-6, the (2-2-2) cryptand (XVII) and α-chymotrypsin using both experimental (stability constant) and theoretical (molecular mechanics calculation) methods has been published (Wipff *et al*, 1983). The authors suggest that the active site of a receptor tends to mimic the solvation shell of the substrate.

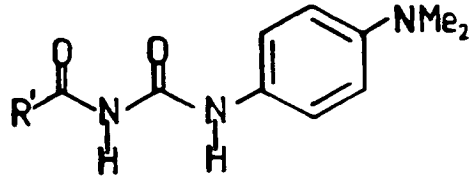


(XVII)

The design of a molecular receptor is briefly discussed in terms of the benefits of rigidity ("lock and key") and flexibility ("induced fit", allostery or cooperativity). It is also stated that the chiral discrimination by α-chymotrypsin takes place in the transition state rather than on initial binding.

Host:guest complexes in the solid state can be divided into clathrates, where one molecular species is imprisoned in closed cages formed by another species, the channel type (e.g. brucine ethanol dihydrate) where continuous channels running through a crystal accommodate the guest molecule, and the layer type (e.g the strychnine salts) where layers of one species alternate with layers of the other. Although several clathrate compounds have been analysed by X-ray crystallography, they do not seem to have any specifically biomimetic interactions (MacNicol *et al*, 1978).

Endo and co-workers (1982, 1983) have shown shape specific weak interactions between the alkyl groups of aromatic acylurea alkyl compounds (XVIII, XIX), both by reaction in solution to form disulphides and by crystallisation of 1:1 molecular complexes.



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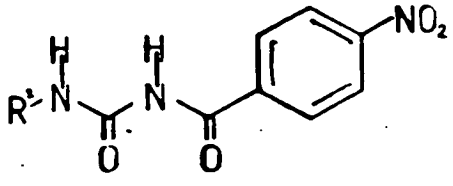


(XIX)

It might appear that the molecular association would be caused by hydrogen bonding at the acylurea sites, with bulky alkyl groups causing a decrease in association, but it is here suggested that complex formation is enhanced by the similarity of the alkyl groups rather than a simple lack of steric bulk. No mechanism for this interaction is proposed, although it is suggested that the mechanism would be useful for distinguishing between hydrophobic amino acids.

The series of complexes whose structures were determined by Ishida, Inoue and their co-workers over the last seven years under the general title of "Structural studies of the interactions between indole

derivatives and biologically important aromatic compounds" provides interesting examples of the type of results that may be obtained by using molecular complexes to model larger biological molecules, although it also shows some of the possible pitfalls inherent in the procedure. The main problem with preparing molecular complexes to model biological interactions is that the model compounds may have another mode of interaction available that is more favourable in the solid state than the one under examination. This may at first seem to imply that the model system is invalid, but it provides information on the relative strengths of possible interactions in the complex.

An example of this occurs in the structure of the indole-3-acetic acid: nicotinamide (1:1) complex (Ishida, *et al*, 1978i), which was prepared in order to investigate whether protonation of the pyridine ring to give the pyridinium ion is a necessary pre-condition for formation of a charge-transfer complex. When the structure was solved, it was seen that there were no charge-transfer or "ring stacking" interactions, but that the whole structure was held together by hydrogen bonds to form infinite layers perpendicular to the c axis. This result was taken as implying the necessity for a quaternary nitrogen in the nicotinamide moiety as a prerequisite for charge transfer, but it could also be possible that the hydrogen bonding interaction is energetically more favourable.

Examples of other biological interactions investigated in this manner are:

1) Between indole and benzene rings (Inoue *et al*, 1978ii; Ishida & Inoue , 1981ii) using tryptamine: phenylacetic acid (1:1) and tyramine: indoleacetic acid (1:1) complex. No charge transfer interactions.

2) Between flavins and indoles or pyrimidines (Ishida & Inoue, 1979; Inoue *et al*, 1980; Inoue *et al*, 1981; Ishida *et al*, 1981; Inoue *et al*, 1983)
These complexes all exhibit an interaction between the aromatic systems

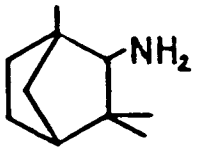
3) between nucleotides and indoles (Ishida *et al*, 1979i; Ishida *et al*, 1979ii; Ishida *et al*, 1980ii; Ishida *et al*, 1980iii; Ishida *et al*, 1981i; Ishida *et al*, 1982ii) These complexes model the interaction between proteins and nucleic acids, and again hydrogen bonding is prominent though there is some ring stacking.

There are also a number of other complexes in the series that do not quite fit this classification scheme. These are: thiamine indole-3-propionate (Inoue *et al*, 1982), which is a model for coenzyme: tryptophan interactions; a uracil: phenylethylamine complex (Ishida *et al*, 1983), which is a model for protein: nucleic acid interactions and two adenine compounds that are not strictly complexes, but discrete molecules. These are 1-[2(-(Adenin-9-yl) ethyl]-3-carbamoyl pyridinium chloride monohydrate, which is a pyridinium coenzyme model without any form of ring stacking(Ishida *et al*, 1982i), and a model nucleotide of 5'-deoxy -5'-adenosine acetic acid (Ishida *et al*, 1980i).

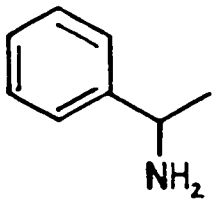
Other complexes involving nucleosides and aromatic compounds have been prepared in order to clarify the mode of action of intercalating antibiotics. The antitrypanosomal drug ethidium has been used to form a complex with 5'-iodouridylyl-(3'-5')-adenosine (Tsai *et al*, 1975) and the antimalarial drug proflavine has been co-crystallised with cytidylyl-(3'-5')-guanosine sulphate (Berman *et al*, 1979).

The diastereomeric salts of amino acids and alkaloids have been used in optical resolutions since the turn of the century (Fischer, 1899), and such complexes would be good models for drug receptor interaction as well as being suitable for a study of chiral recognition. Strychnine and brucine are the most commonly used resolving agents for N-protected amino acids (Dunn & Rockland, 1947; Greenstein, 1954 & 1961), but almost any chiral compound that can form a diastereomeric salt with the racemic amino acid may be used. Some examples of the use of resolving agents other than the indole alkaloids occur in the work of Ingersoll's group in the early 1950's and more recently, in the series of complexes prepared by Toda and his co-workers. Both groups made use of a "chain of resolutions", where each resolved compound may be used

to resolve another racemic mixture. Ingersoll used the N-acetyl derivative of the naturally occurring L-leucine to resolve crude dl-α-fenchylamine (XX) (Ingersoll & DeWitt, 1951) and then used the resulting optically pure amines for the resolution of DL-phenylalanine and DL-valine (via the N-acetyl or formyl derivatives). In the same paper (Overby & Ingersoll, 1951) the use of α-phenylethylamine (XXI) in resolution is discussed and it is mentioned that the resolved acetyl amino acids were capable of resolving dl-fenchylamine.

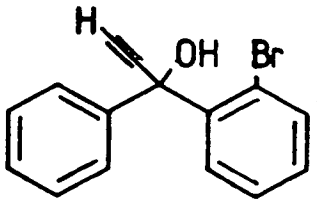


(XX)

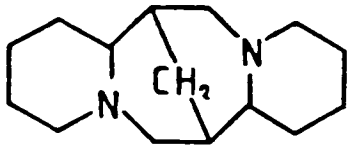


(XXI)

Toda used brucine to resolve the enantiomers of 1-(o-bromophenyl) 1-phenyl-2-propynol (XXII) (Toda *et al*, 1981) and determined the crystal structure of the resulting complex. It is suggested that the linearity of the acetylenyl group may be important in formation of the complex and further suggested that other racemic compounds with linear side chains (cyanohydrins, allenic or allyl alcohols) may also be resolved with brucine. The chiral alcohol was then used to resolve dl-sparteine (XXIII) (Toda *et al*, 1983i).



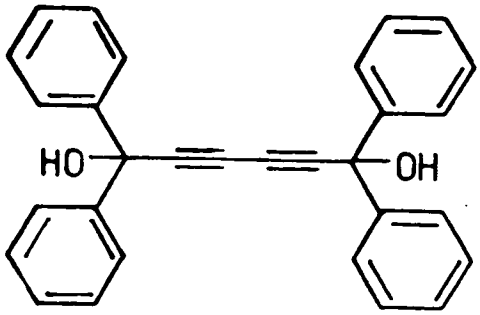
(XXII)



(XXIII)

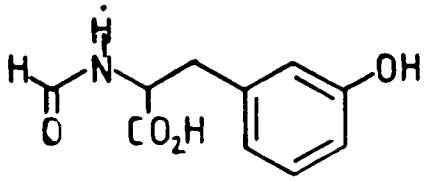
It is reported that resolution by complexation with sparteine is more efficient than with brucine and furthermore, that sparteine is very much less toxic. Moreover, sparteine has an advantage over "natural" bases in that both enantiomers are available for complex formation from the same synthetic material, but d-brucine would be at best a prohibitively expensive curiosity. Toda and his colleagues carried the concept of diastereomeric salt formation a step further than simple optical resolution, when they used brucine to resolve compounds with two chiral centres (Toda *et al*, 1983ii). However, in the case of the compounds examined here (α- and β-haloacetylenic alcohols), several complexations were required to give optically pure alcohol, and the resolution did not work at all for a β-alkyl acetylenic alcohol.

They have also used a dimeric analogue of the acetylenic alcohol resolved with sparteine to resolve 3-methylcycloalkanones and 5-methyl-γ-butyrolactone (Toda *et al*, 1983iii). It is noted that an attempt at the resolution of 2-methylcyclohexanone was not successful and it is suggested that the distance between the chiral centre and the carbonyl group is crucial to the success of the resolution. Replacing bromine with chlorine does not greatly alter the resolving power of the complex, but the o-fluorophenyl derivative is much less efficient. Although no crystal structure of this complex has been published as yet, the structure of the analogous 2:1 acetone tetraphenyl-2,4-hexadiyne-1,6-diol complex is available (Toda *et al*, 1981) and it can be seen that the "wheel and axle" geometry of the diol (XXIV) that allows complexation to occur by holding the structure open. The infinite channels containing nothing other than acetone are similar in appearance to the channels containing 2-propanol in the cyclotricatechylene di-2-propanolate complex whose structure was determined by Hyatt's group (1980). The environment of the solvent in these cases is quite similar to that of the ethanol and water of the brucine complex whose structural determination is discussed below.



(XXIV)

The procedure used for the resolution is essentially unchanged since the Fischer resolution of N-benzoylalanine, and consists of dissolving equimolar amounts of racemate and resolving agent in a suitable solvent (usually water or aqueous alcohol) by warming the suspension, filtering the resulting solution, and allowing it to slowly cool to room temperature as the less soluble diastereomeric salt is precipitated. The amino acid may be recovered from the diastereomeric salt by metathesis with a stronger base than that used in the precipitation or by acidification and extraction with a non-polar solvent. It should be noted though that the solvent chosen for the resolution may influence the relative solubilities of the diastereomeric salts obtained. Shabica and Tishler (1949) have shown that the brucine salt of N-acetyl D-tryptophan separated cleanly from dry ethanol while the residual L-isomer crystallised from methanol/ether only after concentration and charcoal filtration. Recently, the N-acetyl D-tryptophan complex with brucine was prepared by crystallisation from aqueous alcohol solution (Gould *et al*, 1984). This complex contains two molecules of brucine per tryptophan residue, of which only one is charged. This can be compared with the observations of Sealock and co-workers (1951), that brucine forms an ethanol insoluble complex with N-formyl D-meta tyrosine (XXV) while the same preparation carried out in aqueous solution yields a precipitate containing the L-tyrosine derivative. Presumably an attempt at resolving this compound using aqueous alcohol would not succeed as both salts could crystallise together.



(XXV)

Information on molecular recognition phenomena may also be obtained from crystal structures of amino acids and their derivatives by comparing the common features in environment and conformation over a range of independent crystal structure determinations containing the same amino acid residues. This can lead to an understanding of the relative magnitudes of the interactions between an amino acid and its environment. This may be considered as a form of autorecognition as the preferred environment of each part of the molecule should be of the same type in the simple amino acid as it is when the amino acid is part of the active site of a receptor protein.

CHAPTER TWO
METHODS USED IN X-RAY STRUCTURAL DETERMINATION

METHODS USED IN X-RAY STRUCTURAL DETERMINATION

The first stage in the determination of a crystal structure is the selection and mounting of a suitable crystal. The crystal should be large enough to diffract X-rays well, but small enough to remain within the X-ray beam at all times. To minimise absorption effects, the crystal should be of uniform size in all dimensions (though this is less important for crystals of compounds containing only the lighter elements, as they absorb the X-rays to a lesser extent), so the crystal may be cut to shape. A cuboidal or spherical crystal of about 0.3 mm in diameter is an ideal size. The crystal should also have plane faces without surface cracks and extinguish a beam of polarised light uniformly. A crystal fulfilling these requirements is then mounted, either by cementing it to a glass fibre, or in the case of unstable crystals, by sealing it into a glass tube with some mother liquor. The fibre or tube is then attached to a goniometer head in order to take preliminary photographs on the Weissenberg camera. These initial photographs are used to set the crystal accurately on the the goniometer head, and the crystal is then photographed again for unit cell and space group determination. An oscillation photograph and a zero-level Weissenberg photograph are used for cell measurement. For a correctly set crystal, the oscillation photograph will give the length of the unit cell axis parallel to the oscillation axis in the form of an interlayer spacing. When the camera is set up to take a Weissenberg photograph, screens are positioned so that the film is exposed to only one of these layers of reflections, and the film case is set in lateral motion by means of a gear system connected to the oscillating goniometer head in order to spread out the zone of reflections into an interpretable two dimensional net. For a first level Weissenberg photograph the screens are shifted to capture the first layer only. A precession photograph, showing an undistorted zone of the reciprocal lattice, may also be examined. The local program FILMFIT was used to refine the cell dimensions using accurately measured positions of reflections from Weissenberg or precession photographs.

With the exception of the cyclic disulphide, N-acetyl methionine and

the strychnine:N-acetyl tyrosine molecular complex, it was possible to determine the space groups uniquely from the systematic absences, for all the compounds examined during this work. It was, however, possible to deduce the space groups of these three structures given that they contained chiral molecules and that cell dimensions and density could be used to calculate the number of asymmetric units likely per cell.

Data collection was carried out using an Enraf-Nonius CAD-4 four circle diffractometer controlled by a PDP-11 computer for all but one of the structures examined. The strychnine nitrate data set was collected on the STADI-2 two circle diffractometer under the control of a PDP-8 computer. The output file of either type of data set contains intensity information for each reflection and its background along with the angle at which the reflection was measured. One or more reflections are measured several times at regular intervals to provide a means of measuring drift. This provides an indication of whether the crystal is decomposing or moving partially out of the X-ray beam.

The program used for initial data processing depends on whether the data was collected on the CAD-4 or STADI-2 diffractometers, although both CADABS and STOEABS perform similar operations on the input file. First the input file is read in order to calculate the drift curve. Values for the observed intensities are calculated by subtracting the background on either side of a reflection from the peak value and scaling according to the relative time taken for each measurement. The standard deviation may be derived from the square root of the scaled peak and background measurements.

The amplitudes of the structure factors are calculated from the intensities by first applying empirically calculated corrections for absorption and drift (derived from the curves plotted earlier), and then corrections for the effects of polarisation of the diffracted beam and the Lorentz effect (these are effects of data collection geometry). The structure factor amplitude e.s.d's are calculated from the intensity e.s.d's in a similar manner. As well as listing out the reflections, structure factors and e.s.d's, the data reduction program

provides statistical data which can be of use in space group determination.

The next stage of the procedure depends on the compound under examination. If the compound contains a few atoms of markedly higher atomic number than the rest of the structure (e.g. sulphur in an organic compound) then the structure may be solved by the Patterson or heavy-atom method. On the other hand, when the structure consists of atoms of approximately equal atomic number (neglecting hydrogen) then solution by direct methods is more applicable.

The process of structure determination is based on the fact that the data set collected is a Fourier transform of the unit cell. To determine the positions of the unit cell contents it is necessary to have two pieces of information for each structure factor. These are the amplitude and the relative phase of the diffracted beam producing the reflection. The amplitude information may be obtained from the intensity, but the phase information is impossible to record. It is this lack of phase information that prevents X-ray structural determination from being totally automatic.

The Patterson method avoids the need for phase information by using the squares of the structure factors and phase angles of zero in a Fourier synthesis. The resultant map does not contain information on the position of atoms relative to an origin, but only the inter-atomic vectors. The heights of the peaks on the map are proportional to the product of the atomic numbers of the atoms they connect, so the highest non-origin peaks should be those connecting the heavy atoms. As the map also contains vectors connecting symmetry related atoms, it may be possible to deduce a set of coordinates for the heavy atoms relative to an origin. A difference map can then be calculated using the phases calculated from the coordinates of the heavy atoms.

The structure of the cysteinyl penicillamine cyclic disulphide and of N-acetyl methionine were solved in this manner using the program SHELX to calculate the map, but the Patterson map calculated for the

dimeric disulphide contained so many vectors that the sulphur-sulphur vectors were hidden.

The program SHELX was also used to solve the structure of DL Glutamic acid by a direct method. It can be shown that for a centrosymmetric structure, all the phase angles are either zero or π radians. It can further be shown that for any three strong reflections **H1**, **H2** and **H3** with indices such that **H1** + **H2** = **H3**, the products of the cosines of their phases is usually positive. In principle, phases can therefore be determined for all the strong reflections if a suitable starting set of phases is chosen. In practice, several starting sets are chosen and the most consistent sets of phases calculated are used to calculate the electron density map.

For the non-centrosymmetric case the application of direct methods is not quite as straightforward as phase angles are no longer restricted to zero or π radians. The MULTAN system of programs was used to solve the structures in this category. The MULTAN system contains five programs which perform all the necessary calculations from normalisation of the structure factors to output of a list of coordinates for further refinement.

The first program in the suite is NORMAL, which normalises the structure factors by means of a calculated expected intensity for each reflection. This expected intensity is calculated using either randomly positioned atoms in the cell (using the Wilson plot), or if the information is available, randomly positioned spherically averaged groups of atoms (with a Debye curve). The more stereochemical information that is input in this way, the easier the solution of the structure should be. If no stereochemical information is read in, then the points on the Debye curve are the same as those of the Wilson plot. A line of best fit is calculated through the Wilson or Debye curves, which is used to obtain the scale multiplier and the temperature coefficient. It is also possible to input an arbitrary temperature coefficient to the program (this is how the disordered strychnine:N-acetyl tyrosine structure was solved). The scaling factors calculated are applied to the

reflections in such a way that the mean value of the square of the normalised structure factor is unity for each index group. After producing a comprehensive set of intensity statistics for use in space group determination, a file is prepared containing the strongest and weakest reflections (about the top 200 and bottom 50) for input to MULTAN.

The program MULTAN is itself divided into three sub-programs: SIGMA2, CONVERGE and FASTAN. The SIGMA2 section sets up the phase relationships which will be used by the tangent formula. These are relations between triplets of reflections such that the indices of **H1** minus those of **H2** equal those of **H3** such that the phases may be related similarly:

$$\phi(H1)=\phi(H2)+\phi(H1-H2)$$

The weight given to each of these relationships is proportional to the product of their normalised structure factors. The CONVERGE sub-program uses the space group information to determine which reflections are structure seminvariants and which phase restrictions are imposed by symmetry for use in the application of the sigma 1 formula and phase determinations. This section also choses the reflections that define the origin and fix an enantiomorph along with a few other starting reflections. The sigma 1 relation is similar to the phase relationship defined in SIGMA2, but it applies when two of the three reflections are the same. The calculated phases are once again stated with a probability of being correct, and the most probably correct phases are used as part of the tangent formula starting set with a weight of one less than twice their probability. The phases of the starting set reflections are assigned using a "magic integer" system unless the phase is restricted, when it is allowed to take two possible values only. The magic integer principle is a means of increasing the size of the starting set of reflections without making the total number of phase permutations prohibitively large, by expressing approximate values of several phases in terms of one parameter and writing

$$\phi = mx \pmod 1$$

where ϕ is the phase in cycles and m is one of a set of integers. This means that with a set of five magic integers, starting phases of fifteen reflections may be defined with only three parameters. The relationship between phases in magic integer notation can be expressed as

$$Hx + Ky + Lz + b = 0$$

where H, K, L are sums of magic integers. It is not usually possible to find values of the parameters x, y, z to satisfy all the possible relationships, but the best solutions can be selected for by relating phase probabilities to intensities. These sets of phases provide the multiple starting points for the tangent formula, which are evenly distributed over the possible range of phase permutations to ensure that at least one is close to the correct set.

The final sub-program FASTAN develops these starting sets of phases by using them with the relationships derived by SIGMA2. The phases are then refined using the weighted tangent formula (Germain *et al*, 1971):

$$\mathrm{Tan}\phi(H) = \frac{\Sigma\left(W(K)\ W(H-K)\,|E(K)\ E(H-K)|\,\mathrm{Sin}(\phi(K)+\phi(H-K))\right)}{\Sigma\left(W(K)\ W(H-K)\,|E(K)\ E(H-K)|\,\mathrm{Cos}(\phi(K)+\phi(H-K))\right)} = \frac{T(H)}{B(H)}$$

where W(H) is the weight given to the phase of the reflections. This weighting leads to an efficient propagation of phase information as poorly determined phases have little effect on the values of well determined (and hence higher weighted) phases.

After the phases for each set have been determined by refinement with the tangent formula, three parameters - the figures of merit - are calculated as an indication of which phase sets lead to valid solutions. The absolute figure of merit is a measure of internal consistency among the SIGMA2 relationships and is zero if the phases

are randomly determined and greater than unity if the relationships are wholly consistent. The second figure of merit, $\Psi(0)$ is calculated using the weakest E-value reflections and should have as low a value as possible. This figure is sensitive to molecular positions and totally independent of the tangent formula. The last figure of merit is the residual calculated for the equation

$$E(H) = S * \langle E(K) * E(H-K) \rangle$$

where 'S' is a scale factor calculated to minimise the value of the residual and <X> means 'the average value of X'. All three figures of merit are combined into one, with the option of altering the weight given to each figure. This combined figure of merit is used to rank the phase sets for the generation of the electron density maps.

The last three programs, EXPAND, FFT and SEARCH are used together to produce an electron density map and analyse it for possible molecular fragments. A complete hemisphere of reflections is generated from the input reflections by the program EXPAND, which passes the result to FFT which synthesises the Fourier map corresponding to the highest unused figure of merit. (In the MULTAN 80 system, used for the solution of Strychnine:N-acetyl tyrosine complex, these two programs are combined into EFFT 80). Unless specifically requested, the map is not explicitly printed out, but passed to the program SEARCH. This program locates the highest peaks in the output map and lists their coordinates in order of peak height. The list of peaks is analysed to detect molecular fragments and a table of inter-peak distances and angles is calculated. A fragment interpretation routine applies minimum and maximum bond lengths and angles to eliminate spurious peaks and prints out an interpretation for each significantly different subset of the fragment. Up to three molecular plots are output depending on molecular shape. If the molecular fragment found is roughly planar, a plot of the peaks on the least-squares plane is sufficient to yield a clearly recognisable representation, but if the fragment is more spherical, then the plot of peaks on the least-squares plane can be somewhat confusing, so plots of peaks on the most-squares plane and

the plane orthogonal to these two are also output. In the intermediate case, of a "cylindrical" molecule, the plot of peaks on the most-squares plane is omitted. Finally, a list of coordinates is output for possible phase recycling. At this stage of the determination, the program SHELX can be used to calculate a difference map for the purpose of locating the rest of the non-hydrogen atoms in the structure, or to start the refinement procedure.

It is, however, possible that the known part of the structure is insufficient to phase all the reflections well enough to locate the unknown part on a difference map. This problem may be solved by the use of the DIRDIF system. This program takes as input the coordinates of the part structure and the F(obs) values for all reflections, which are used for the calculation of difference structure factors and then phase extension and refinement with the weighted tangent formula. The improved phases are then output to the MULTAN system programs EFFT and SEARCH for Fourier map generation and interpretation. The resulting fragment may be used for least squares refinement, or recycled through DIRDIF to produce a larger fragment. The DIRDIF system contains four sub-programs for use when both the position and orientation of the input molecular fragment are known, and two others for use when only orientation is known (the TRADIR system). The first subprogram, ENTER, is used to prepare the data file for use later in the system and to check how well the origin and enantiomorph are fixed. Structure factors and temperature factors are then calculated by part 1 of SFANDB. After calculation of the structure factors, this subprogram calculates a scale factor based on default thermal parameters and then an overall temperature factor from a Wilson-Parthasarathy plot. This is a form of Wilson plot which has been modified to allow for the contributions from the known part-structure. It is followed by a two dimensional refinement of the temperature factors of the known and unknown part-structure. In the second part of SFANDB, the temperature and scale factors are used to calculate maximum and minimum values for the E-values of the unknown part-structure, depending on whether F(obs) is in or out of phase with F(cal). A probability of the two being in phase rather than out of

phase is calculated. At this stage, several diagnostic parameters are calculated using the new temperature and scale factors.

The subprogram DIFTAN applies the weighted tangent formula for origin and enantiomorph fixation (if necessary) and for refinement and expansion of the phase information. For any phase that changes during refinement, the most probable E-value for the unknown part-structure is recalculated. After each cycle, the average shifts of the special and general reflections are output, with a consistency factor. If there are entire parity groups of reflections that are unphased by the known atoms, or if less than 60% of the reflections have phases assigned, then symbols are assigned to the phases of some reflections. A similar procedure is used for enantiomorph fixation in the case where the known atoms in an acentric structure have a pseudo centre of symmetry. Numerical values are then assigned to the symbols by a method using weighted symbol relations.

The last part of the DIRDIF system is the subprogram EXIT, which prepares a file containing the new phase information for output to EFFT and SEARCH of the MULTAN system (or optionally to the XRAY system) in the form of Fourier coefficients for a difference map, a weighted difference map, or a full electron density map.

The next subprogram is EXPAND, which is used only as part of TRADIR, to prepare a file for the program EXTRA. The file contains the reduced symmetry, as the next subprograms (SFANDB and DIFTAN) must be run with space group P1, the expanded data set and the original space group information (used by EXTRA only).

The program EXTRA (only used as part of TRADIR) calculates the structure factors of a symmetry related fragment by applying the relevant symmetry operators to the structure factors of the input fragment. These structure factors are combined with those of the unknown part-structure and a translation function is produced by the Fourier transform of the result. This translation function can be interpreted to obtain a shift vector which may be applied to the

input fragment in a run of DIRDIF.

The program SHELX is used for the last stages of structural determination. The molecular fragment found by DIRDIF (or by MULTAN, if that fragment was large enough) is used to phase a difference map based on all observed data (it is possible to vary the criteria for deciding if a reflection is observed). This difference map should indicate if any atoms are missing, or wrongly positioned. In some cases, the hydrogen atoms will be visible at this stage and these may be input to the next run of SHELX either as independent atoms or constrained to be in idealised positions relative to their environment. The thermal parameters of all non-hydrogen atoms may be either isotropically or anisotropically calculated, depending on the available data, but they are usually isotropic in the initial stages of refinement. Unless they are allowed to vary independently the hydrogen thermal parameters are usually either fixed to the same value (which may or may not refine) or they may be derived from the thermal parameter of the atom they are attached to. There are a number of mechanisms for constraining parameters during least squares refinement which may be of great use if the data does not permit all the parameters to vary independently, or if the structure is disordered. At its simplest, the constraint mechanism involves fixing a parameter so that it does not vary (e.g. for origin fixation in P1 or $P2_1$, or to hold all occupancy factors at unity), or it may be used to constrain a group of atoms to maintain a specific geometry (either as a rigid group or by explicitly listing interatomic distances and permissable e.s.d's). Another use of the constraint mechanism is when an atom is disordered over two sites and the total occupancy must be equal to that of a non-disordered atom (it is necessary in this situation to constrain the thermal parameters), as in the strychnine: N-acetyl tyrosine structure.

Another useful feature of the SHELX program is the facility for printing out the entire electron density map and not just the plot of peaks on the least squares plane. This map may be used as input to a transformation and interpolation program, FMAPO, which produces an output file suitable for contouring and detailed examination for the

location of atomic positions in disordered regions. This is essential, as it is possible for the two possible sites of a disordered atom to be so close to each other that the overlap region appears higher in electron density than the atomic centres, which is a virtually impossible situation for the peak search routines of SHELX to cope with.

Although it is possible to "refine" a structure by repetitively generating Fourier maps and manually altering the file of atomic parameters to improve the fit between the model and the structure, the use of least squares refinement is more efficient as it directly minimises the difference between F(obs) and F(cal). If each of the data points involved in the minimisation is assigned a weight varying with the precision of measurement, and there are many more observations than parameters, the quantity to be minimised may be expressed as:

$$Q = \Sigma w_H (|F_{oH}| - |F_{cH}|)^2$$

which is summed over all observed data. To minimise this figure, it is necessary to find the point at which the derivative of Q with respect to each parameter is zero, leading to the same number of independent simultaneous equations as these are parameters. This would imply that a solution to these equations is easily determinable as there are usually 5-10 observations (reflections) for each parameter. The method of least squares, however, applies only to linear equations, and the above differential equations are anything but linear. The solution to this problem is to use the shift in the parameters and not the parameters themselves as variables. This method requires a trial structure to be reasonably close to the final structure, so that a Taylor series expanded from the initial parameters will lead to a better set. A further assumption is that the trial structure is sufficiently close to the final structure to permit the use of the first derivative term only, leading to the relation

$$|F_o| - |F_c| = \Sigma \frac{\delta |F_c| . \Delta p_i}{\delta p_i}$$

for all parameters P_i. As this method involves several approximations, several iterations are needed before final convergence is achieved. At each end of each cycle, the R factors are output, along with a list of parameters (with e.s.d's and shift/e.s.d.). The difference map is then calculated and the peaks plotted. The final part of the output shows the number and variance of the reflections in each angular zone and by parity group. These may be used to determine if a weighting scheme is necessary.

After the final convergence is reached and the structure is fully determined SHELX may be used to print out a list of reflections with F(obs) and F(cal) values and the program PAPER used to format the SHELX output into suitable tables of coordinates and thermal parameters with e.s.d's for eventual publication. The molecular geometry may be fully explored using the program CALC to produce lists of bond-lengths, angles, torsion angles and contact distances as well as planes through the molecule (and their dihedral angles) or to fit one molecule or fragment to another. Finally, the effects of thermal vibration or of molecular packing may be shown graphically by means of the programs ORTEP and PLUTO.

<u>List of Programs</u>

DIRDIF

Direct Methods for Difference Structures. An Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.

P. T. Beurskens, W. P. Bosman, H. M. Doesburg, R. O. Gould, Th. E. M. van den Hark, P. A. J. Prick, J. H. Noordik, G. Beurskens and V. Parthasarathy.

Technical report 1981/2, Crystallography lab. Toerooiveld, 6525 ED, Nijmegen,Netherlands

MULTAN-77

A System of Computer Programs for the Automatic Solution of Crystal Structures from the X-ray Diffraction Data

P. Main, L. Lessinger, M. M. Woolfson, G. Germain and J-P. Declerq, 1977.

SHELX-76

A Program for Crystal Structure Solution.

G. Sheldrick, University of Cambridge, 1976.

OR TEP-II

A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations.

C. K. Johnson, Oak Ridge National Laboratory, Tennessee, 1976.

PLUTO

Molecular Plotting Program

W. D. S. Motherwell, University of Cambridge, 1976.

In addition, the following 'local' programs were used:

FILMFIT: a program for refining cell constants from film data

CADABS & STOEABS: programs for the reduction of diffractometer collected data

TABLES: a program for formatting tables of bonds, angles and torsion angles with their e.s.d's

PAPER: a program for formatting tables of atomic coordinates and thermal parameters with their e.s.d's

All the above written by Dr. R. O. Gould.

FMAPO: a program for the interpolation and scaling of SHELX electron density output for line-printer listing and contouring

By Dr. M. M. Harding

CALC: a program for molecular geometry

By R. O. Gould and P. Taylor

A NOTE ON STEREOCHEMISTRY

Of the eight crystal structures presented here, seven are chiral compounds crystallising in acentric space groups. As it is not possible to determine the absolute configuration of a compound crystallographically without the use of anomalous dispersion measurements, it is necessary to decide the configuration of the compounds on the basis of chemical information and by reference to the structures of known compounds.

For the cyclic disulphide, the stereochemistry of the penicillamine used in synthesis is known from optical rotation measurements, indicating that the compound contains l-cysteine and d-penicillamine. This indirectly allows determination of the stereochemistry of the dimeric disulphide, as it was prepared from the same batch of l-cystine.

The N-acetyl methionine was supplied as the l-isomer, and was assumed to be so as it formed an insoluble complex with brucine, but not with strychnine (Greenstein, 1954).

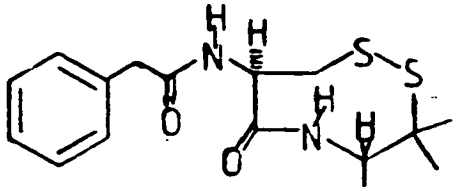
The remaining structures all contain the naturally produced strychnine molecular skeleton, and their configurations are in agreement with that found by anomalous dispersion methods on strychnine hydrobromide (Peerdeman, 1956). Even without this study, it would have been possible to determine the absolute configuration of strychnine from the strychnine tyrosine complex if the absolute configuration of the tyrosine used was known.

CHAPTER THREE
N-PHENYLACETYL CYSTEINYL PENICILLAMINE METHYL ESTER CYCLIC DISULPHIDE

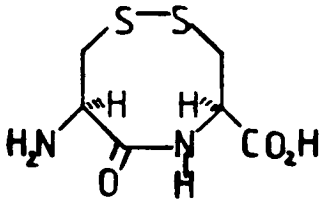
3,3-DIMETHYL-4-D-CARBOMETHOXY-6-OXO-7-L-PHENYLACETYLAMINO PERHYDRO-1,2,5-DITHIAZOCINE

Introduction

Although this cyclic disulphide (VI) was first prepared as a biosynthetic model for β-lactam formation (as described above), the structure is of interest in its own right as the smallest ring system containing both a disulphide bridge and a *trans* amide linkage. There is a similar system, *cyclo* L-cysteinyl cysteine disulphide (XXVII), but this compound contains a *cis* amide linkage.



(VI)



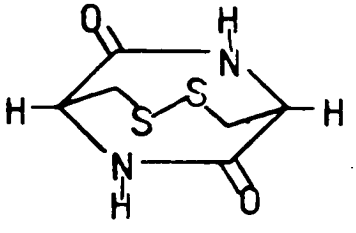
(XXVII)

Energy calculations carried out on the *cis* compound (Chandresekharan & Balasubramanian, 1969) have indicated the possibility of two distinct conformations of this compound, differing only in the sense of the torsion angle χ^3 (C-S-S-C). The conformation is referred to as P-helical when the angle χ^3 is positive (or *circa* Plus 90°) and as M-helical when the angle is negative (Minus 90°). In neither of these conformations is the amide linkage planar and some of the intra-annular angles are distorted, as the potential function used for the energy minimisation included the effects of changes in non-bonded interactions, electrostatic interactions, dihedral angle distortion and variations in bond angles, while keeping bond lengths constant at standard values. Both the predicted conformations have amide linkages distorted by over

ten degrees, but in opposite directions to the sign of the disulphide torsion.

Conformation I	$\Delta\omega = -12°$	$E = -3.55$ kcal mol^{-1}	P-helical
Conformation II	$\Delta\omega = 14°$	$E = -2.92$ kcal mol^{-1}	M-helical

Conformation I with its smaller distortion in amide planarity is therefore the expected conformation, but the difference is not extreme and it may be possible for either conformation to exist, depending on the size of the barrier between the states. Later work by Chandresekharan and Mitra (1977) on the conformation of the disulphide bridge in *cyclo* L-cystine (cystine diketopiperazine or cys-DKP) (XXVI) again predicts two possible conformations of the disulphide bridge with two-fold rotational symmetry in each case.



(XXVI)

The M-helical conformation is the more stable (-9.2 kcal mol^{-1} as against -8.9 kcal mol^{-1} for the P-helical structure) and there are no other differences in gross conformation. At first glance, this would appear to contradict the work of Ottnad and co-workers (1975) who studied the conformation of cys-DKP in DMSO solution and found it to contain a P-helical disulphide bridge, but the crystal structure of the acetic acid solvate of cys-DKP (Mez, 1974) shows the bridge to be M-helical.

More recently, the crystal structures of two derivatives (XXVI,XXVII) of *cyclo* L-cysteinyl L-cysteine have been determined (Capasso *et al*, 1977; Ashida *et al*, 1977), showing both to be P-helical but to have the

opposite distortion at the amide linkage. These compounds will be discussed in more detail below. The seeming contradiction in the work of Ottnad and Mez may be resolved if it is assumed that the helicity of the disulphide bridge may change depending on the molecular environment, even in a rigid molecule such as cys-DKP. However, a solution conformation study on the *cyclo* L-cysteinyl L-cysteine (Capasso *et al*, 1979) presented no real evidence for either configuration of the bridge to the exclusion of the other. This being the case, it was decided that an investigation of the solution conformation of the cysteinyl penicillamine cyclic disulphide would indicate whether or not its disulphide bridge was able to alter its configuration in solution. The results of this nmr study are also presented below.

Preparation and Crystallisation

The compound was prepared and crystallised by Dr. E. M. Gordon, according to the procedure outlined in the experimental section below. The crystals were grown by dissolving the compound in a slight excess of chloroform, adding 40/60 petrol ether until crystallisation began and then leaving the solution at room temperature overnight.

Crystal Data

Formula $C_{17}H_{22}N_2O_4S_2$ M = 382.47 F(000) = 404
Space group P1 Int. Tab. No. 1
Cell dimensions a = 9.240(4) b = 9.642(4) c = 11.317(4) A
α = 94.97(5) β = 98.85(5) γ = 110.37(5)°
Volume = 923.2 cubic Angstroms Z = 2
Dc = 1.376 gcm^{-3} Dm = 1.348 gcm^{-3}
Radiation Mo Kα λ = 0.71069 Angstrom μ = 2.99 cm^{-1}
Final R = 0.0511 based on 1259 independent reflections
h range 0 to 10 k range -11 to 10 l range -13 to 13
θ max = 25° Sin(θ max)/λ = 0.5947
3282 reflections measured
3246 after merging
1987 considered unobserved ($F<6\sigma(F)$)
Parameters refined 290
Max shift/σ of last cycle 0.186
Final difference Fourier max. 0.3444 eA^{-3} min. -0.2407 eA^{-3}
Weight = $1.3833/(\sigma^2(F)+0.000363F^2)$

Data Collection and Reduction

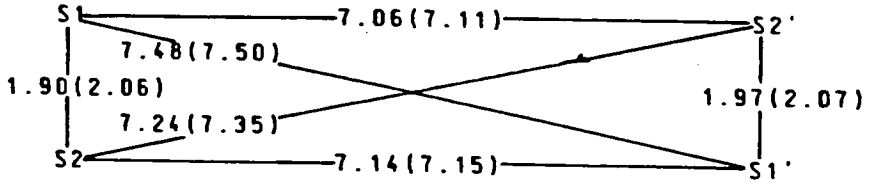
The data set was collected on the CAD4, and corrected for Lorentz and polarisation effects using the program CADABS.

Solution and Refinement

The structure was solved from the Patterson map produced by the Program SHELX. The top four non-origin peaks in the map were interpreted as being consistent with a centrosymmetric arrangement of four sulphur atoms in a parallelogram, such that two of the vectors were double weighted. These four peaks, and the next two highest peaks are tabulated below.

No.	Height	x/A	y/B	z/C	length
1	231	0.6804	0.4704	0.2990	7.105
2	191	0.1066	0.1603	0.9042	2.023
3	111	0.2224	0.3822	0.7866	7.287
4	105	0.5626	0.2994	0.3950	7.490
5	92	0.7922	0.4243	0.1865	5.486
6	88	0.8693	0.4777	0.1040	5.237

The diagram below shows the four sulphur-sulphur vectors and their final lengths.



The coordinates derived from these vectors were used as input for the program SHELX, but the resulting difference map showed only a part of the two molecules, and recycling this fragment through SHELX did not lead to any further improvement in the difference map. This situation, where the only part of the structure known is the position of a few heavy atoms around a pseudo-inversion centre, is an almost ideal case for the use of the DIRDIF procedure.

The first run of DIRDIF produced a map containing a fragment of one molecule, which was used as input to a new run. The new map was then examined for a new input fragment for a further run of the procedure. The diagnostics produced by the program are tabulated below.

Input Fragment Test	S_4	S_2C_5N	$S_2C_9N_2O_3$
R	0.63	0.66	0.60
BH	4.50	3.04	3.41
Bl	3.70	4.96	5.39
σC(EH)	2.4*	5.4	5.31
(EL)	0.6*	2.3	2.18
σA(EH)	6.4	1.9*	0.68
(EL)	2.5	1.0*	1.91
Consistency			
cycle 1	0.30(332)	0.26(400)	0.27(399)
2	0.21(526)	0.17(688)	0.19(828)
3	0.22(562)	0.21(687)	0.26(841)

In this table BH and BL are the overall temperature factors for the known and unknown part-structures respectively.* The next set of figures relate to the distribution function of the expectation values of E calculated from both centric and acentric expressions. The change in these values on going from a centric to an acentric input fragment is marked thus '*'. The last part of the table shows the progress of the weighted tangent phase extension and refinement, with the figures in brackets being the number of reflections in the basis set for each cycle.

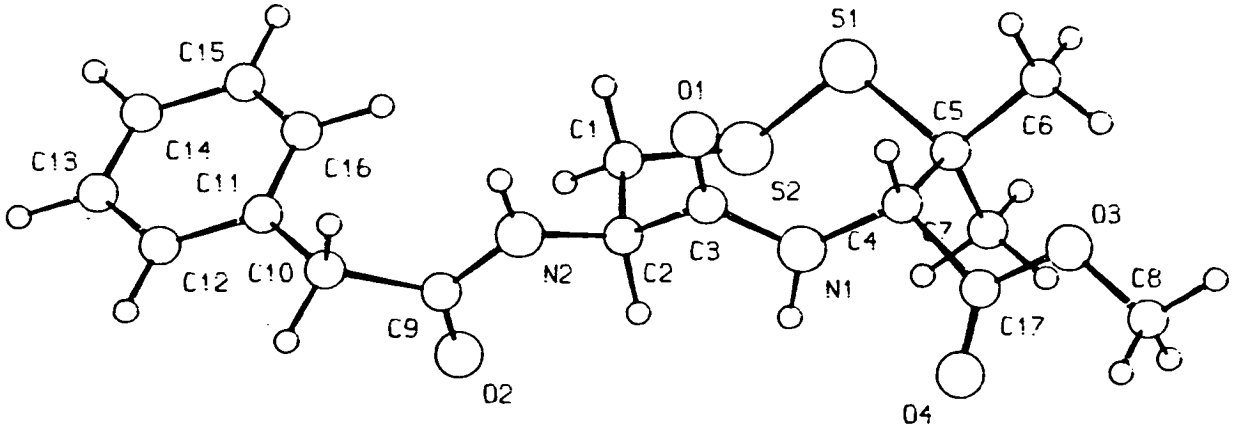
After another couple of cycles through DIRDIF, the structure was complete enough to use the program SHELX for refinement. The progress of refinement is summarised in the table below.

*In this table, the letters H and L indicate the expressions involving the known fragment of the molecule (or Heavy atoms) and the unknown fragment (or the Light atoms).

R	Comment
0.2554	Almost two complete molecules input (2 carbons missing)
0.2435	All of one molecule located, and all of the other but for a phenyl ring
0.1406	All non-H atoms found, isotropic thermal parameters tied to a free variable
0.0947	All thermal parameters freed
0.0943	Some H atoms located in the difference map H atoms input in calculated positions
0.0714	Sulphur atoms with anisotropic thermal parameters and phenyl rings constrained to ideal geometry
0.0644	Amide oxygens anisotropic
0.0592	Nitrogen atoms anisotropic
0.0567	Convergence
0.0569	Thermal parameters of H atoms 'riding' on those of atoms they bond to, aromatic rings no longer constrained to ideal geometry
0.0546	Absolute stereochemistry corrected
0.0511	Final convergence*

Description of Structure

The unit cell contains two independent molecules arranged head to tail (causing the pseudocentric arrangement of the four sulphur atoms) and connected by hydrogen bonding into an infinite ribbon parallel to the y axis, in a manner analogous to the β-pleated sheet found in most polypeptides and proteins. The numbering scheme used in this section is illustrated on the diagram below.



*All hydrogens were constrained to remain at calculated positions, with their thermal parameters 'riding' on those of the atom to which they are bonded

The following tables show all unconstrained bond lengths, angles and torsion angles for both molecules with the addition of some torsion angles involving hydrogen which are referred to in the section on solution conformation below.

S(1) - S(2)	2.058(6)	C(2) - C(3)	1.515(20)
S(1) - C(5)	1.861(15)	C(4) - C(5)	1.565(21)
S(2) - C(1)	1.814(16)	C(4) -C(17)	1.562(22)
O(1) - C(3)	1.252(18)	C(5) - C(6)	1.569(23)
O(2) - C(9)	1.195(19)	C(5) - C(7)	1.523(22)
O(3) - C(8)	1.427(19)	C(9) -C(10)	1.581(23)
O(3) -C(17)	1.365(19)	C(10) -C(11)	1.482(21)
O(4) -C(17)	1.161(20)	C(11) -C(12)	1.418(22)
N(1) - C(3)	1.367(19)	C(11) -C(16)	1.344(21)
N(1) - C(4)	1.459(19)	C(12) -C(13)	1.41(3)
N(2) - C(2)	1.465(19)	C(13) -C(14)	1.30(3)
N(2) - C(9)	1.364(20)	C(14) -C(15)	1.41(3)
C(1) - C(2)	1.500(20)	C(15) -C(16)	1.34(3)

S(1') -S(2')	2.066(8)	C(2') -C(3')	1.529(19)
S(1') -C(5')	1.855(16)	C(4') -C(5')	1.478(21)
S(2') -C(1')	1.823(16)	C(4') -C(17')	1.515(23)
O(1') -C(3')	1.207(18)	C(5') -C(6')	1.538(22)
O(2') -C(9')	1.217(20)	C(5') -C(7')	1.549(21)
O(3') -C(8')	1.446(21)	C(9') -C(10')	1.551(22)
O(3') -C(17')	1.332(21)	C(10')-C(11')	1.476(22)
O(4') -C(17')	1.199(21)	C(11')-C(12')	1.438(25)
N(1') -C(3')	1.385(19)	C(11')-C(16')	1.348(24)
N(1') -C(4')	1.455(19)	C(12')-C(13')	1.37(3)
N(2') -C(2')	1.475(18)	C(13')-C(14')	1.27(3)
N(2') -C(9')	1.354(21)	C(14')-C(15')	1.39(3)
C(1') -C(2')	1.479(20)	C(15')-C(16')	1.40(3)

S(2)-S(1)-C(5)	110.0(5)	C(4)-C(5)-C(6)	109.5(12)
S(1)-S(2)-C(1)	108.5(5)	C(4)-C(5)-C(7)	111.4(12)
C(8)-O(3)-C(17)	115.1(12)	C(6)-C(5)-C(7)	111.9(13)
C(3)-N(1)-C(4)	124.0(12)	O(2)-C(9)-N(2)	121.5(15)
C(2)-N(2)-C(9)	120.2(12)	O(2)-C(9)-C(10)	121.8(14)
S(2)-C(1)-C(2)	115.4(10)	N(2)-C(9)-C(10)	116.8(13)
N(2)-C(2)-C(1)	107.2(11)	C(9)-C(10)-C(11)	111.9(13)
N(2)-C(2)-C(3)	113.4(11)	C(10)-C(11)-C(12)	121.0(13)
C(1)-C(2)-C(3)	107.0(11)	C(10)-C(11)-C(16)	120.5(14)
O(1)-C(3)-N(1)	123.0(13)	C(12)-C(11)-C(16)	118.5(14)
O(1)-C(3)-C(2)	122.2(13)	C(11)-C(12)-C(13)	121.5(15)
N(1)-C(3)-C(2)	114.3(12)	C(12)-C(13)-C(14)	116.7(17)
N(1)-C(4)-C(5)	109.2(12)	C(13)-C(14)-C(15)	122.1(17)
N(1)-C(4)-C(17)	10.0(12)	C(14)-C(15)-C(16)	121.7(18)
C(5)-C(4)-C(17)	106.7(12)	C(11)-C(16)-C(15)	119.4(16)
S(1)-C(5)-C(4)	106.5(10)	O(3)-C(17)-O(4)	126.9(15)
S(1)-C(5)-C(6)	104.9(10)	O(3)-C(17)-C(4)	110.8(13)
S(1)-C(5)-C(7)	112.4(11)	O(4)-C(17)-C(4)	122.3(15)

S(2')-S(1')-C(5')	110.1(6)	C(4')-C(5')-C(6')	112.4(13)
S(1')-S(2')-C(1')	107.1(6)	C(4')-C(5')-C(7')	113.7(12)
C(8')-O(3')-C(17')	115.7(13)	C(6')-C(5')-C(7')	109.2(12)
C(3')-N(1')-C(4')	122.4(12)	O(2')-C(9')-N(2')	121.5(15)
C(2')-N(2')-C(9')	120.7(12)	O(2')-C(9')-C(10')	121.5(14)
S(2')-C(1')-C(2')	116.0(11)	N(2')-C(9')-C(10')	117.0(14)
N(2')-C(2')-C(1')	106.6(11)	C(9')-C(10')-C(11')	112.6(13)
N(2')-C(2')-C(3')	114.1(11)	C(10')-C(11')-C(12')	120.4(14)
C(1')-C(2')-C(3')	107.5(11)	C(10')-C(11')-C(16')	119.7(15)
O(1')-C(3')-N(1')	124.1(13)	C(12')-C(11')-C(16')	119.8(15)
O(1')-C(3')-C(2')	121.7(13)	C(11')-C(12')-C(13')	120.9(17)
N(1')-C(3')-C(2')	113.5(12)	C(12')-C(13')-C(14')	118.7(20)
N(1')-C(4')-C(5')	110.8(12)	C(13')-C(14')-C(15')	122.6(21)
N(1')-C(4')-C(17')	110.0(12)	C(14')-C(15')-C(16')	121.6(19)
C(5')-C(4')-C(17')	109.4(12)	C(11')-C(16')-C(15')	116.1(17)
S(1')-C(5')-C(4')	106.9(10)	O(3')-C(17')-O(4')	123.9(16)
S(1')-C(5')-C(6')	104.0(10)	O(3')-C(17')-C(4')	113.1(14)
S(1')-C(5')-C(7')	110.2(10)	O(4')-C(17')-C(4')	122.8(15)

C(5) - S(1) - S(2) - C(1)	107.7(7)
S(2) - S(1) - C(5) - C(4)	-81.8(10)
S(2) - S(1) - C(5) - C(6)	162.2(9)
S(2) - S(1) - C(5) - C(7)	40.4(12)
S(1) - S(2) - C(1) - C(2)	-76.2(11)
C(8) - O(3) -C(17) - O(4)	8.3(23)
C(8) - O(3) -C(17) - C(4)	-173.0(12)
C(4) - N(1) - C(3) - O(1)	-19.0(22)
C(4) - N(1) - C(3) - C(2)	152.9(13)
C(3) - N(1) - C(4) - C(5)	-98.5(15)
C(3) - N(1) - C(4) -C(17)	144.7(13)
C(9) - N(2) - C(2) - C(1)	84.6(16)
C(9) - N(2) - C(2) - C(3)	-157.5(13)
C(2) - N(2) - C(9) - O(2)	10.6(22)
C(2) - N(2) - C(9) -C(10)	-168.2(12)
S(2) - C(1) - C(2) - N(2)	180.0(9)
S(2) - C(1) - C(2) - C(3)	58.0(14)
N(2) - C(2) - C(3) - O(1)	-38.2(19)
N(2) - C(2) - C(3) - N(1)	149.9(12)
C(1) - C(2) - C(3) - O(1)	79.8(17)
C(1) - C(2) - C(3) - N(1)	-92.1(14)
N(1) - C(4) - C(5) - S(1)	51.8(13)
N(1) - C(4) - C(5) - C(6)	164.7(12)
N(1) - C(4) - C(5) - C(7)	-71.1(15)
C(17) - C(4) - C(5) - S(1)	170.7(10)
C(17) - C(4) - C(5) - C(6)	-76.4(15)
C(17) - C(4) - C(5) - C(7)	47.8(16)
N(1) - C(4) -C(17) - O(3)	-172.8(12)
N(1) - C(4) -C(17) - O(4)	6.0(21)
C(5) - C(4) -C(17) - O(3)	68.9(15)
C(5) - C(4) -C(17) - O(4)	-112.3(17)
O(2) - C(9) -C(10) -C(11)	-73.2(19)
N(2) - C(9) -C(10) -C(11)	105.6(16)
C(9) -C(10) -C(11) -C(12)	133.0(15)
C(9) -C(10) -C(11) -C(16)	-48.1(19)
C(10) -C(11) -C(12) -C(13)	-180.0(15)
C(16) -C(11) -C(12) -C(13)	1.0(24)
C(10) -C(11) -C(16) -C(15)	179.0(16)
C(12) -C(11) -C(16) -C(15)	-2.1(24)
C(11) -C(12) -C(13) -C(14)	-1.3(26)
C(12) -C(13) -C(14) -C(15)	2.7(27)
C(13) -C(14) -C(15) -C(16)	-4 (3)
C(14) -C(15) -C(16) -C(11)	4 (3)

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C(5') -S(1') -S(2') -C(1')  105.0( 8)
S(2') -S(1') -C(5') -C(4')  -80.8(10)
S(2') -S(1') -C(5') -C(6')  160.1( 9)
S(2') -S(1') -C(5') -C(7')   43.1(11)
S(1') -S(2') -C(1') -C(2')  -80.2(11)
C(8') -O(3') -C(17')-O(4')   -1.7(24)
C(8') -O(3') -C(17')-C(4') -176.9(13)
C(4') -N(1') -C(3') -O(1')  -22.5(22)
C(4') -N(1') -C(3') -C(2')  148.4(13)
C(3') -N(1') -C(4') -C(5') -104.6(15)
C(3') -N(1') -C(4') -C(17') 134.3(14)
C(9') -N(2') -C(2') -C(1')   82.7(16)
C(9') -N(2') -C(2') -C(3') -158.8(13)
C(2') -N(2') -C(9') -O(2')    7.2(23)
C(2') -N(2') -C(9') -C(10')-171.9(12)
S(2') -C(1') -C(2') -N(2') -175.2(10)
S(2') -C(1') -C(2') -C(3')   62.0(14)
N(2') -C(2') -C(3') -O(1')  -35.1(19)
N(2') -C(2') -C(3') -N(1')  153.8(12)
C(1') -C(2') -C(3') -O(1')   82.9(16)
C(1') -C(2') -C(3') -N(1')  -88.2(14)
N(1') -C(4') -C(5') -S(1')   55.6(13)
N(1') -C(4') -C(5') -C(6')  169.1(12)
N(1') -C(4') -C(5') -C(7')  -66.3(16)
C(17')-C(4') -C(5') -S(1')  177.1(10)
C(17')-C(4') -C(5') -C(6')  -69.4(16)
C(17')-C(4') -C(5') -C(7')   55.2(16)
N(1') -C(4') -C(17')-O(3') -155.0(13)
N(1') -C(4') -C(17')-O(4')   29.7(22)
C(5') -C(4') -C(17')-O(3')   83.0(16)
C(5') -C(4') -C(17')-O(4')  -92.2(19)
O(2') -C(9') -C(10')-C(11') -64.0(20)
N(2') -C(9') -C(10')-C(11') 115.1(16)
C(9') -C(10')-C(11')-C(12') 133.7(16)
C(9') -C(10')-C(11')-C(16') -49.8(20)
C(10')-C(11')-C(12')-C(13')-178.6(17)
C(16')-C(11')-C(12')-C(13')   4.9(27)
C(10')-C(11')-C(16')-C(15') 177.9(16)
C(12')-C(11')-C(16')-C(15')  -5.6(25)
C(11')-C(12')-C(13')-C(14')  -4 ( 3)
C(12')-C(13')-C(14')-C(15')   3 ( 3)
C(13')-C(14')-C(15')-C(16')  -4 ( 3)
C(14')-C(15')-C(16')-C(11')   5 ( 3)
```

```
H(1A) - N(1) - C(4) -H(041) -158 ( 9)
H(2A) - N(2) - C(2) -H(021) 164 (10)
H(011)- C(1) - C(2) -H(021)  59.7(20)
H(012)- C(1) - C(2) -H(021) 178.0(16)
H(1'A)-N(1') -C(4') -H(41') -137
H(2'A)-N(2') -C(2') -H(21') 121 ( 9)
H(11')-C(1') -C(2') -H(21')  64.6(20)
H(12')-C(1') -C(2') -H(21') -177.3(16)
```

The two molecules are very similar in conformation, with no significant differences in bond lengths or angles. The largest difference in bond length occurs in the C4-C5 bond, but it is not significant.

C(4)-C(5) 1.565(21)A C(4')-C(5') 1.478(21) 0.087A difference

There are no significant differences in torsion angle around the dithiazocine ring, but the amide linkage is distorted significantly from planarity.

	mol. 1	mol. 2	Ideal
C(2)-C(3)-N(1)-C(4)	152.9(13)	148.4(13)	180.00

This can be explained on grounds of ring strain, as this is the smallest ring to contain a *trans* amide linkage. The smallest lactam to have a *trans* amide linkage in the solid state is caprylolactam ($C_8H_{15}NO$), with a torsion angle of 148.4° in a nine membered ring (Dunitz and Winkler, 1975).

The only significant conformational difference between the two molecules lies in the carbomethoxy group.

Torsion	mol. 1	mol. 2
C(5)-C(4)-C(17)-O(4)	-112.3(17)	-92.2(19)
C(5)-C(4)-C(17)-O(3)	68.9(15)	83.0(16)
N(1)-C(4)-C(17)-O(3)	-172.8(12)	-155.0(13)
N(1)-C(4)-C(17)-O(4)	6.0(21)	29.7(13)

C(4)-C(17)-O(3)-C(8)	-173.0(12)	-176.9(13)
O(4)-C(17)-O(3)-C(8)	8.3(23)	-1.7(24)

Intermolecular Contacts

The tables below show all significant intermolecular contacts. They are organised into three tables, on the following basis:

table 1) All contacts not involving hydrogen

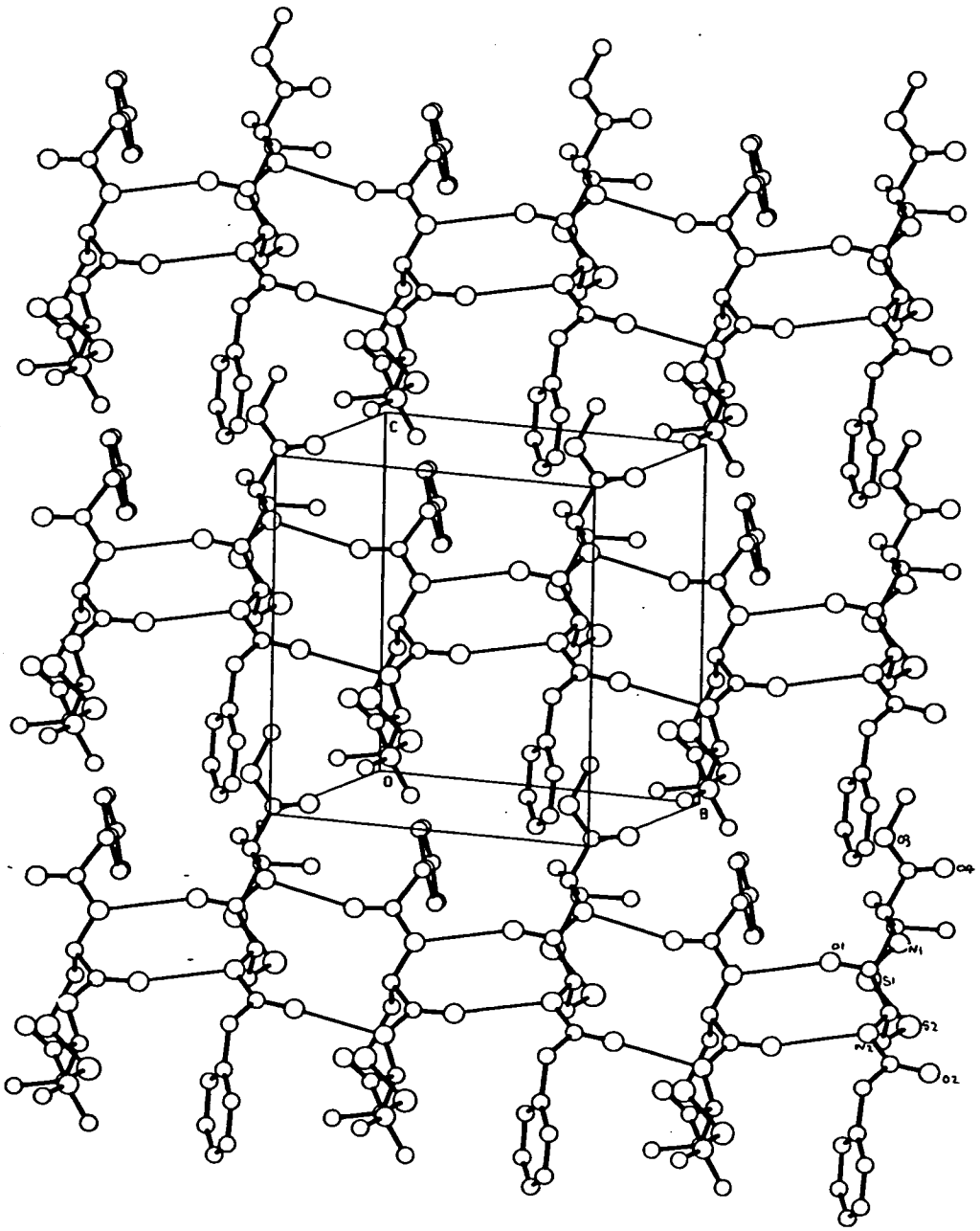
table 2) Contacts involving well-defined hydrogen atoms such as methene, methine or trigonal hydrogens

table 3) Contacts involving methyl group hydrogens, (which are not necessarily in the position calculated for them by SHELX as the methyl groups may rotate)

The most important of these contacts are the oxygen-nitrogen hydrogen bonds listed in the first table, which can be seen in the three packing diagrams below.

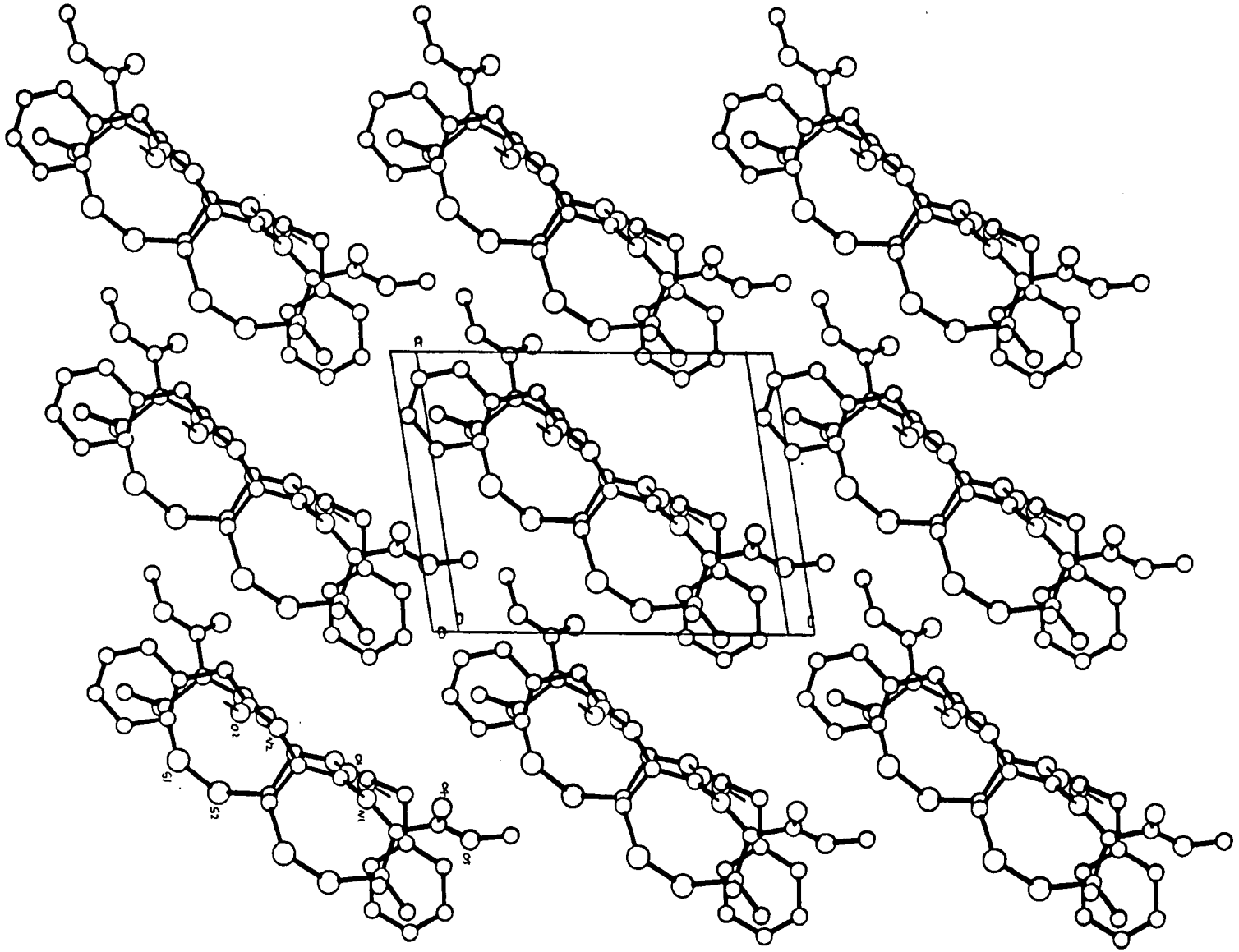
N-PHENYLACETYL CYSTEINYL VALINE METHYL ESTER CYCLIC DISULPHIDE

PROJECTION DOWN THE X-AXIS



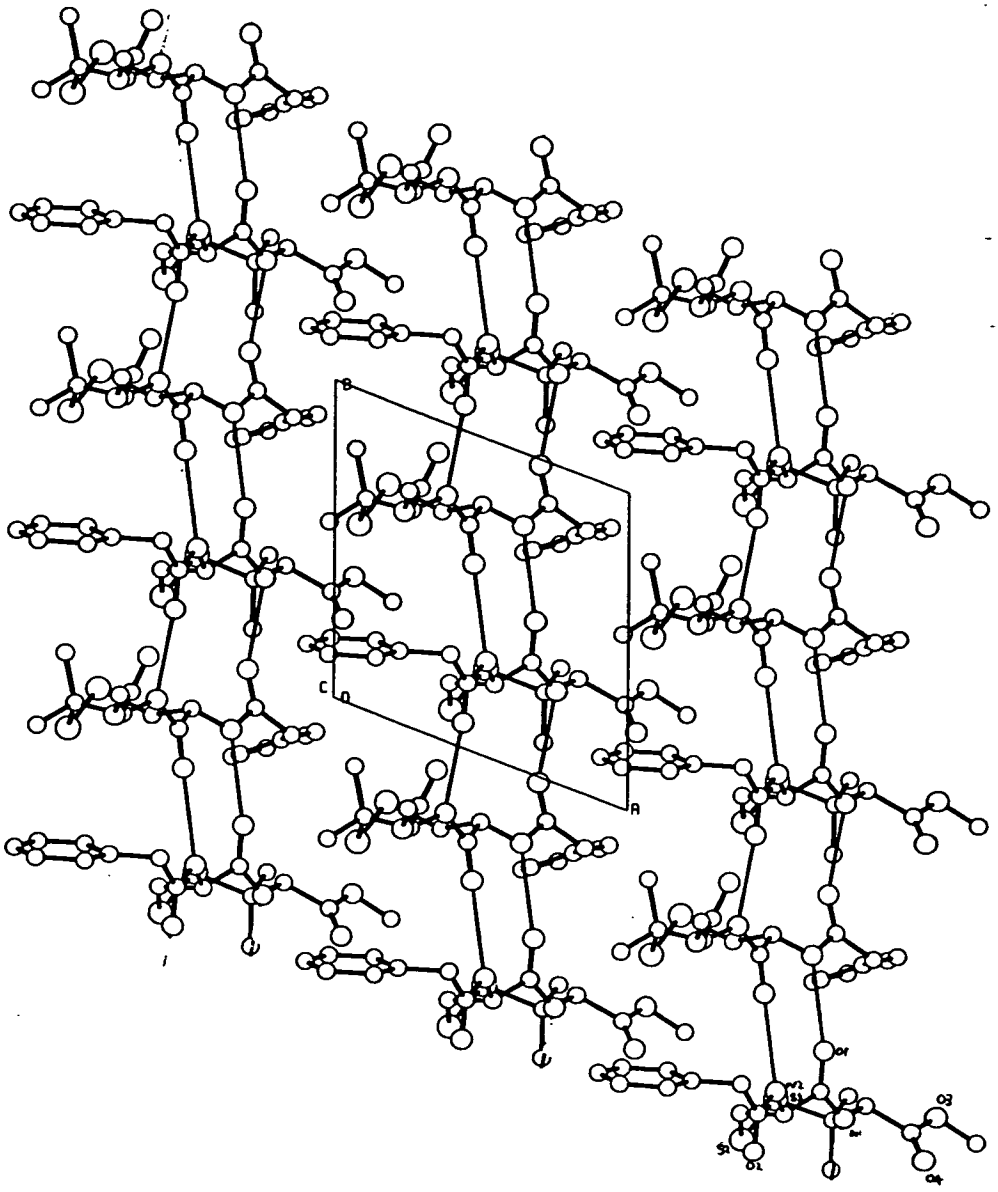
N-PHENYLACETYL CYSTEINYL VALINE METHYL ESTER
CYCLIC DISULPHIDE

PROJECTION DOWN THE Y-AXIS



N-PHENYLACETYL CYSTEINYL VALINE METHYL ESTER CYCLIC DISULPHIDE

PROJECTION DOWN THE Z-AXIS



S2	4.482 (9)	S2'	(X,-1 +Y,Z)
S1	3.938 (12)	O4'	(X,-1 +Y,-1 +Z)
S2	3.649 (12)	O2'	(X,-1 +Y,Z)
S2	3.837 (13)	O4'	(X,-1 +Y,-1 +Z)
S1'	3.479 (12)	O1	(-1 +X,Y,Z)
S1'	3.609 (13)	O4	(-1 +X,Y,Z)
S2'	3.721 (13)	O2	(X,1 +Y,Z)
S2'	3.899 (13)	O3	(-1 +X,Y,Z)
S1'	3.522 (13)	N1	(-1 +X,Y,Z)
S1	3.410 (15)	C8	(-1 +X,Y,Z)
S1	3.857 (18)	C16'	(X,Y,Z)
S2	3.752 (16)	C8	(-1 +X,Y,Z)
S2	3.444 (18)	C8'	(X,-1 +Y,-1 +Z)
S1'	3.620 (16)	C3	(-1 +X,Y,Z)
S1'	3.658 (16)	C4	(-1 +X,Y,Z)
S1'	3.816 (21)	C15	(X,Y,Z)
S1'	3.791 (17)	C16	(X,Y,Z)
S1'	3.611 (17)	C17	(-1 +X,Y,Z)
S2'	3.931 (18)	C17	(-1 +X,Y,Z)
S2'	3.506 (16)	C10'	(-1 +X,Y,Z)
O1	2.949 (16)	N2'	(X,Y,Z)
O2	2.915 (18)	N1'	(X,-1 +Y,Z)
O1'	3.001 (16)	N2	(X,Y,Z)
O2'	2.919 (16)	N1	(X,1 +Y,Z)
O1	3.362 (18)	C10'	(X,Y,Z)
O2	3.337 (17)	C2'	(X,-1 +Y,Z)
O3	3.443 (21)	C8'	(1 +X,Y,-1 +Z)
O1'	3.372 (19)	C10	(X,Y,Z)
O2'	3.343 (17)	C2	(X,1 +Y,Z)
O2'	3.333 (23)	C15	(1 +X,1 +Y,Z)
O4'	3.491 (20)	C7	(X,1 +Y,1 +Z)

table 1

S1	3.207 (16)	H102	(X,Y,-1 +Z)
S1	3.241 (17)	H121	(X,Y,-1 +Z)
S2	3.553 (16)	H11'	(X,-1 +Y,Z)
S2	3.033 (20)	H2'1	(-1 +X,-1 +Y,Z)
S1'	3.304 (16)	H041	(-1 +X,Y,Z)
S1'	3.529 (21)	H151	(X,Y,Z)
S1'	3.524 (17)	H161	(X,Y,Z)
S1'	3.294 (16)	H0'1	(-1 +X,Y,Z)
S2'	3.386 (16)	H011	(X,1 +Y,Z)
S2'	3.405 (16)	H041	(-1 +X,Y,Z)
S2'	2.930 (16)	H0'1	(-1 +X,Y,Z)
S2'	3.180 (16)	H0'2	(-1 +X,Y,Z)
O1'	2.44 (14)	H2'A	(X,Y,Z)
O1	2.478 (18)	H0'1	(X,Y,Z)
O2	2.989 (21)	H141	(1 +X,Y,Z)
O2	2.29 (16)	H1'A	(X,-1 +Y,Z)
O2	2.412 (17)	H21'	(X,-1 +Y,Z)
O4	2.818 (23)	H151	(1 +X,Y,Z)
O4	2.999 (20)	H161	(1 +X,Y,Z)
O1'	2.01 (14)	H2A	(X,Y,Z)
O1'	2.488 (19)	H101	(X,Y,Z)
O2'	1.93 (13)	H1A	(X,1 +Y,Z)
O2'	2.446 (17)	H021	(X,1 +Y,Z)
O2'	2.524 (23)	H151	(1 +X,1 +Y,Z)
O3'	2.890 (25)	H5'1	(X,Y,1 +Z)
N1	2.843 (23)	H151	(1 +X,Y,Z)
C8	2.991 (21)	H012	(1 +X,Y,Z)
C13	2.995 (29)	H3'1	(-1 +X,-1 +Y,1 +Z)
C8'	2.887 (23)	H11'	(X,Y,1 +Z)
C13'	2.904 (29)	H131	(1 +X,1 +Y,-1 +Z)
H1A	2.15 (13)	H151	(1 +X,Y,Z)
H021	2.378 (19)	H21'	(X,-1 +Y,Z)

table 2

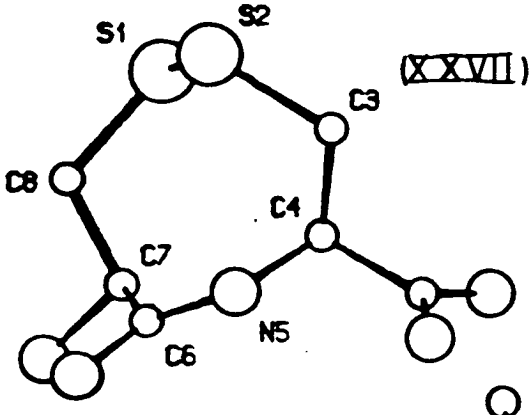
S1	3.335 (15)	H081	(-1 +X,Y,Z)
S1	3.162 (15)	H082	(-1 +X,Y,Z)
S1	3.152 (15)	H083	(-1 +X,Y,Z)
S1	3.477 (18)	H6'1	(X,Y,Z)
S2	3.150 (16)	H082	(-1 +X,Y,Z)
S2	3.414 (16)	H083	(-1 +X,Y,Z)
S2	2.935 (18)	H82'	(X,-1 +Y,-1 +Z)
S2	2.995 (18)	H83'	(X,-1 +Y,-1 +Z)
S2'	3.432 (19)	H81'	(X,Y,-1 +Z)
O2	2.645 (19)	H71'	(X,-1 +Y,Z)
O3	2.434 (21)	H81'	(1 +X,Y,-1 +Z)
O2'	2.605 (19)	H072	(X,1 +Y,Z)
O3'	2.813 (19)	H081	(-1 +X,Y,1 +Z)
O4'	2.551 (20)	H071	(X,1 +Y,1 +Z)
C8	2.732 (23)	H81'	(1 +X,Y,-1 +Z)
C10	2.961 (23)	H062	(X,Y,1 +Z)
C12	2.953 (23)	H72'	(X,-1 +Y,Z)
C14	2.745 (24)	H63'	(X,Y,Z)
C15	2.676 (26)	H63'	(X,Y,Z)
C8'	2.928 (23)	H081	(-1 +X,Y,1 +Z)
C12'	2.957 (25)	H073	(X,1 +Y,Z)
C12'	2.818 (26)	H82'	(1 +X,Y,-1 +Z)
C13'	2.988 (27)	H073	(X,1 +Y,Z)
C13'	2.798 (28)	H82'	(1 +X,Y,-1 +Z)
C14'	2.908 (27)	H061	(X,Y,Z)
C15'	2.659 (28)	H061	(X,Y,Z)
C16'	2.903 (24)	H061	(X,Y,Z)
H012	2.126 (21)	H083	(-1 +X,Y,Z)
H062	2.187 (23)	H102	(X,Y,-1 +Z)
H081	2.333 (23)	H81'	(1 +X,Y,-1 +Z)
H083	2.312 (22)	H12'	(1 +X,Y,Z)
H102	2.187 (23)	H062	(X,Y,1 +Z)

table 3

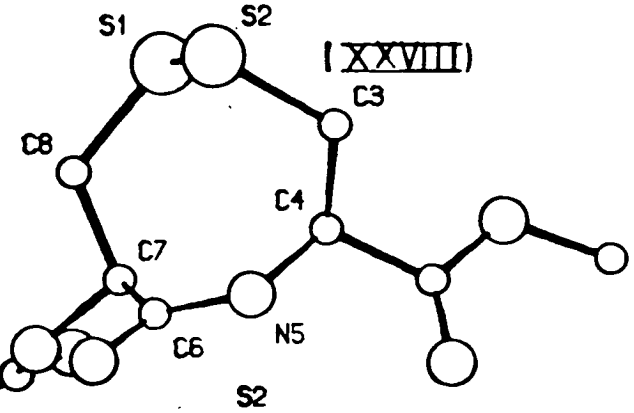
Comparison With Other Structures

There are only two crystal structures in the literature of cyclic disulphides containing an amide linkage. These are L-cysteinylcysteine cyclic disulphide (XXVII) (Capasso *et al*, 1977) and N-tert-Butyloxycarbonyl cysteinylcysteine methyl ester cyclic disulphide (XXVIII) (Ashida *et al*, 1977).

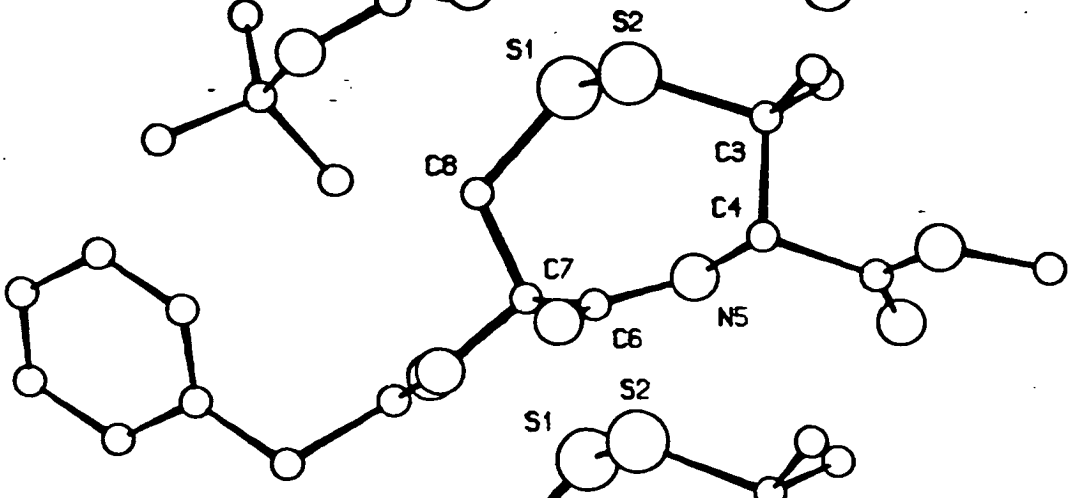
As can be seen from the diagrams, the heterocyclic rings in these compounds have almost exactly the same conformation. They are however significantly different from cyclic disulphide (VI), as they have the 'L' configuration at both chiral centres. This means that a *cis* amide linkage is necessary in order to allow the ring substituents to take up the energetically more favourable equatorial positions.



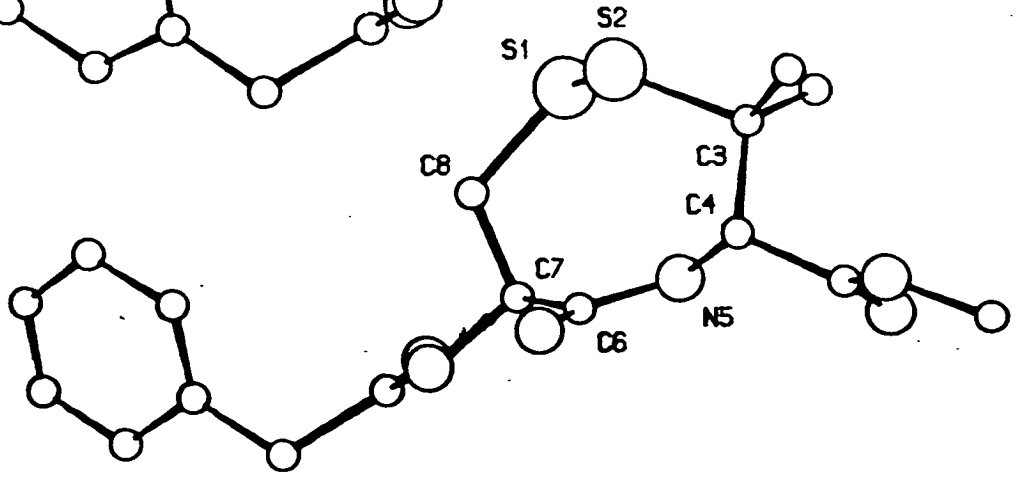
(XXVII)



(XXVIII)



(VIa)



(VIb)

A comparison of the torsion angles around the ring is of interest, as it shows the essential similarity of conformation at the sulphur bridge in both ring systems.

Torsion	VIa	VIb	XXVI	XXVII
S1-S2-C3-C4	-81.8	-80.8	-48.43	-48.63
S2-C3-C4-N5	51.8	55.6	-54.06	-59.43
C3-C4-N5-C6	-98.5	-104.6	112.44	101.85
C4-N5-C6-C7	152.9	148.4	-7.23	10.80
N5-C6-C7-C8	-92.1	-88.2	-84.2	-89.42
C6-C7-C8-S1	58.0	62.0	87.12	80.30
C7-C8-S1-S2	-76.2	-80.2	-80.66	-89.42
C8-S1-S2-C3	107.7	105.2	94.99	95.68

It is also noticeable that the amide linkages in (XXVII) and (XXVIII) are a lot less strained than the *trans* amide in (VI) as the two Cα's do not need to be so far apart.

It is also useful to compare the sulphur bridges with those of other cystine type compounds. In general, disulphide bridges can be divided into two classes on the basis of the sign of their torsion angles. The type I bridge has C-C-S-S and C-S-S-C torsion angles of approximately + 90°, whilst the type II bridge has angles of about - 90°, and hence opposite chirality.

In both these conformations the distance between the Cα's is a maximum, compared with the few cases in which the bridge conformation is not of these types, for example in the bicyclic cyclo-L-cystine diketopiperazine (Varughese *et al*, 1981; Mez, 1973) or the sulphur bridges in proteins, where they are constrained to be closer for other reasons.

The table below shows the parameters of the sulphur bridges from a selection of cystine derivatives.

COMPOUND CODE	S-S	S-CB	S'SCB	SCBCA	CSSC	SSCC
CLCYST10	2.019	1.818	103.86	114.97	-93.53	66.22
			103.73	114.07		61.63
CYLCYS	2.003	1.834	105.17	116.33	-91.44	63.11
		1.787	104.65	115.48		67.22
CYSMEC	2.045	1.808	103.08	113.61	-84.44	-77.37
		1.805	99.99	114.89		-79.19
CYSTBR01	2.024	1.864	103.89	111.97	-81.34	-88.93
CYSTCL02	2.038	1.801	103.83	115.84	-82.55	-88.45
CYSTIN10	2.044	1.820	100.08	112.65	-79.75	-69.99
		1.804	102.33	114.62		-82.35
DGLYCH01	2.037	1.824	103.43	112.75	-84.43	-95.22
LCSTIM	2.051	1.757	102.87	119.09	-81.41	-94.56
LCYSCC	2.040	1.818	99.52	113.32	-79.93	-70.79
		1.832	103.26	114.07		-80.62
LCYSTI10	2.032	1.820	104.51	116.21	73.67	81.64
LCYSTI11	2.042	1.800	104.17	116.79	69.32	66.93
		1.830	105.74	115.72		75.14
PENICS10	2.049	1.865	105.40	109.95	114.71	67.11
		1.866	105.44	106.83		69.61

A key to compound codes giving names of compounds and references will be found at the end of the chapter.

Solution Conformation study

The solid state structure of the cyclic disulphide (VI) shows two independent molecules of similar conformation in the unit cell. To determine whether this similarity was due to packing constraints, or to the rigidity of the molecule itself, a study of the solution

conformation was carried out by nuclear magnetic resonance.

The first step in an nmr study of molecular configuration is to assign each signal to the relevant proton. This can be done by means of irradiation techniques if the complexity of the spectrum warrants it, but in this case it was possible to assign almost all the resonances on the basis of the information contained in one spectrum run in deuterochloroform at ambient temperature. The table below summarises the assignment of the spectrum. (the two methyl resonances were assigned on the basis of their n.O.e's to C(4)-H and N(1)-H)

Shift/ppm	Assignment
1.435	$C(6)H_3$ singlet
1.447	$C(7)H_3$ singlet
2.842	A of ABMX system $C(1)H_2$ $^2J_{AB}$ = 14.04 Hz
3.350	B of ABMX system $^3J_{AM}$ = 11.19 Hz $^3J_{BM}$ = 5.15 Hz
3.570	$C(10)H_2$ singlet
3.741	$C(8)H_3$ singlet
4.555	M of ABMX system C(2)H $^3J_{MX}$ = 7.02 Hz
4.890	$C(4)H_2$ doublet ^3J = 11.10 Hz
6.386	X of ABMX system N(2)
6.642	N(1)H doublet
7.297	Aromatic multiplet (C(12)-C(16))

The coupling constants in the above table were of use in the assignment of the spectrum because it is known that the vicinal coupling constant depends on the dihedral angle between the protons involved. This relationship was first quantified for the H-C-C-H moiety (Karplus, 1959; Karplus, 1963; Barfield and Karplus, 1969) as an equation of the form:

$$^3J_{HH} = A + B\cos\theta + C\cos 2\theta$$

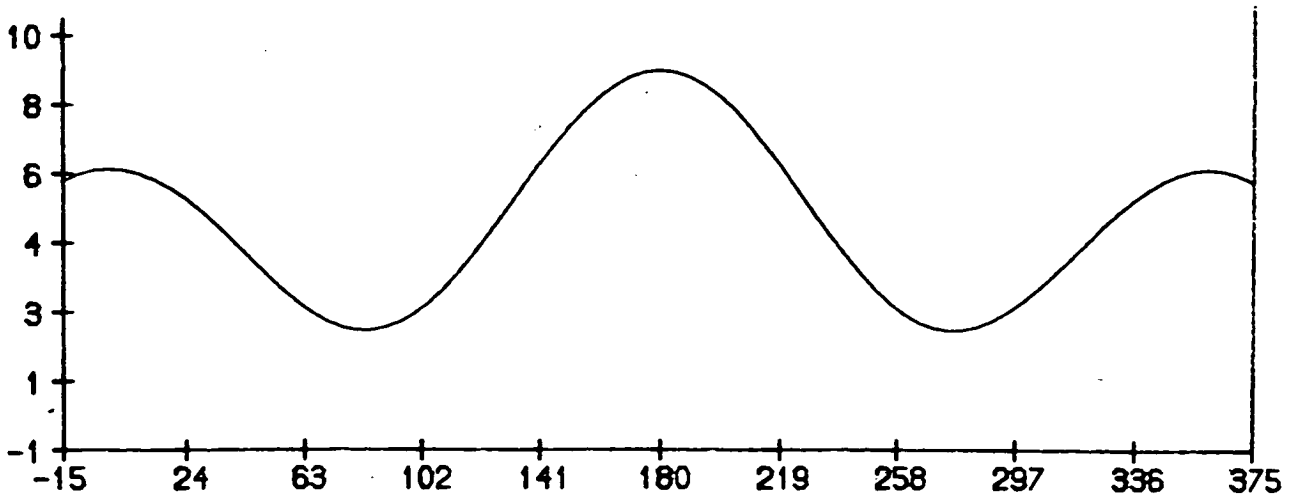
later rewritten as:

$$^3J_{HH} = A\cos^2\theta + B\cos\theta + C$$

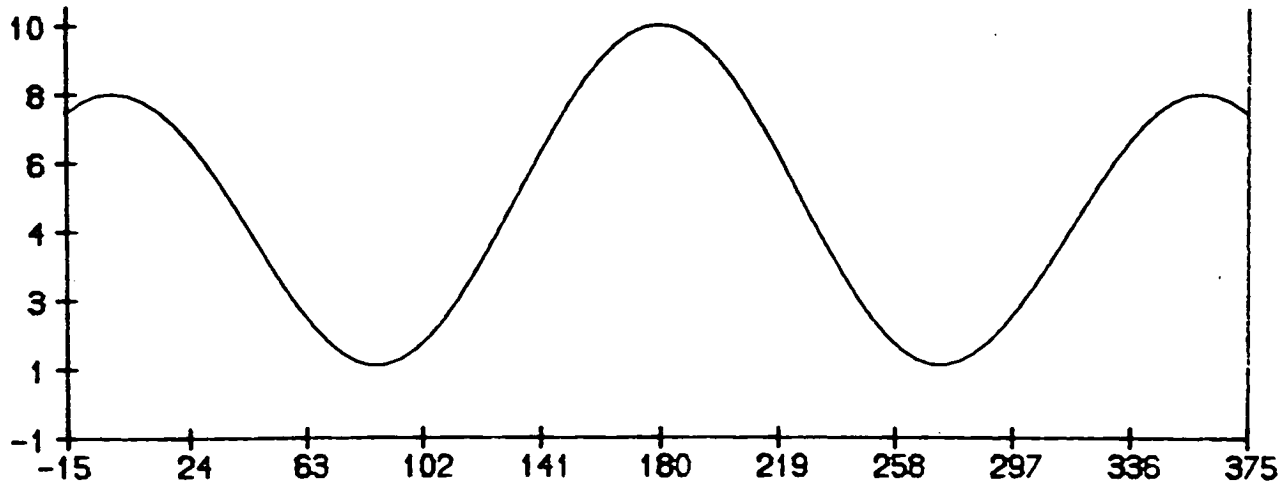
or:

$$^{3}J_{HH} = A\cos^{2}\theta + B\cos\theta + C\sin^{2}\theta$$

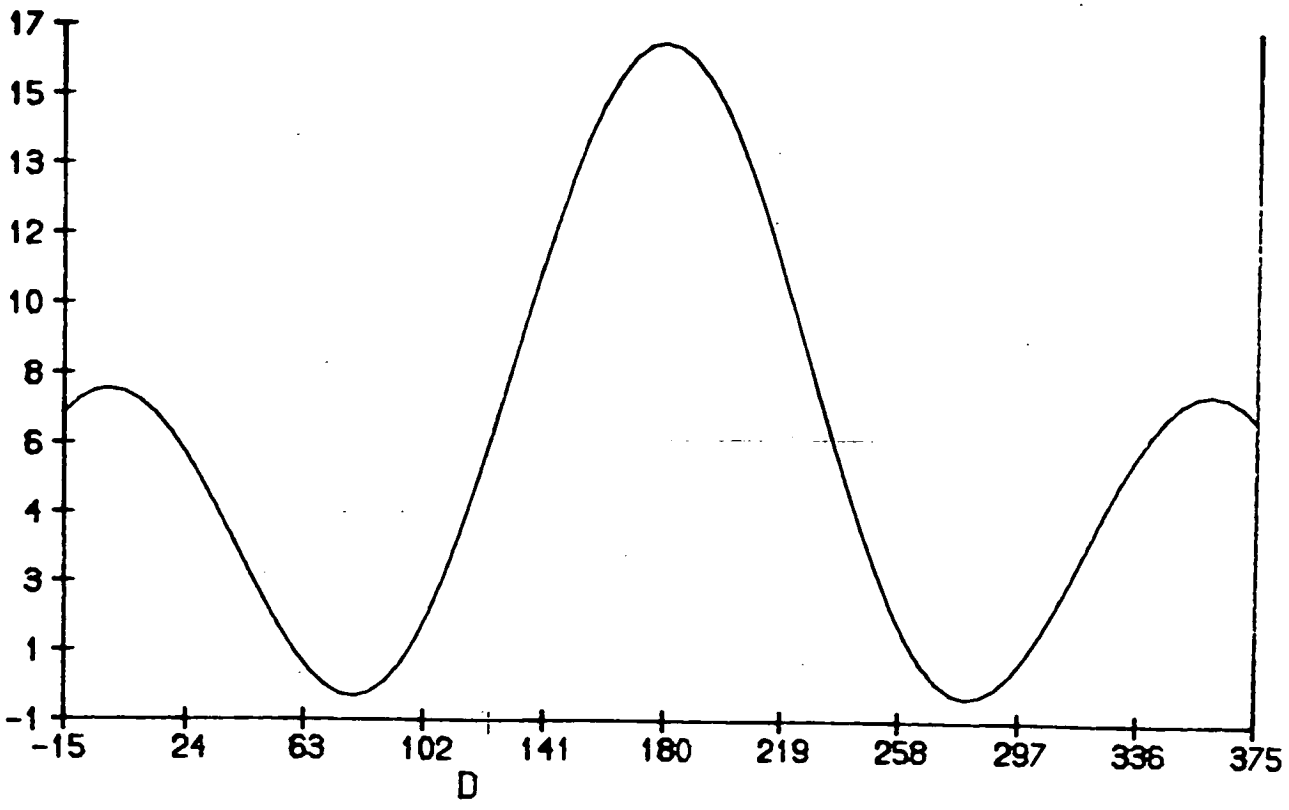
This equation has been used for the H-N-Cα-H system by changing the values used for the Karplus constants (Bystrov *et al*, 1969; Ramachandran *et al*, 1971; Barfield and Gearhart, 1973; and DeMarco *et al*, 1978). The graphs below show how coupling constant varies with dihedral angle for each set of parameters used in the Karplus relationship.



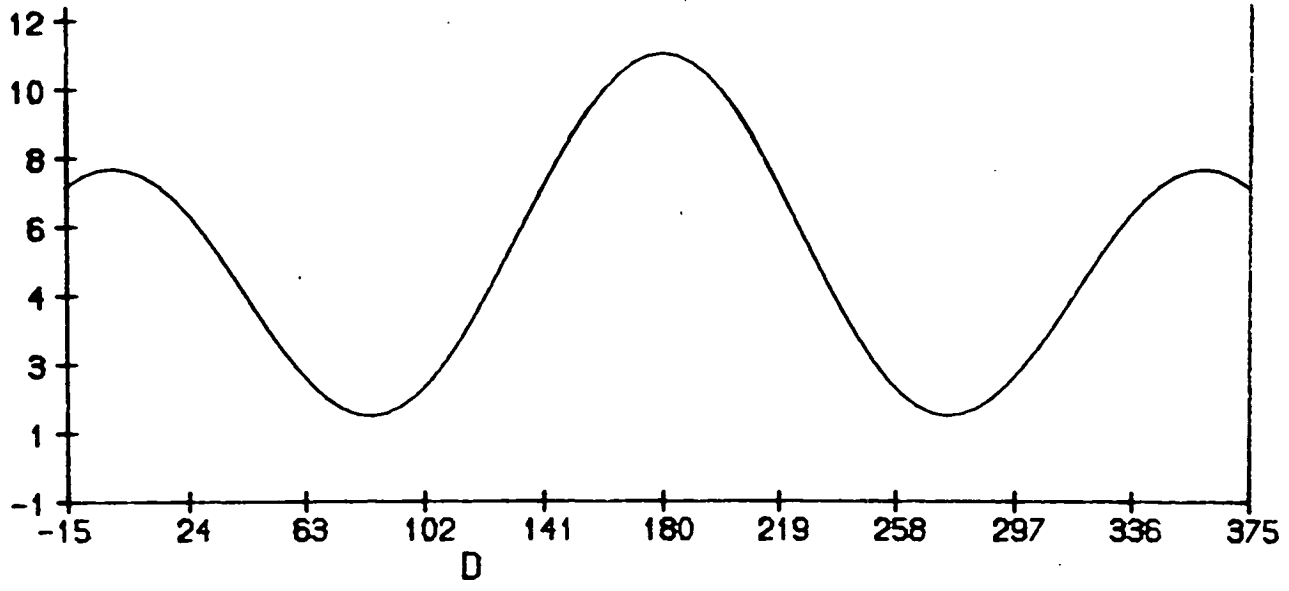
DEMARCO et al



BYSTROV et al



BARFIELD et al



RAMACHANDRAN et al

The method that Bystrov and his co-workers used to calculate values for the constants in the Karplus equation, was to use infra-red spectroscopy to find the conformational states of a range of alanine dipeptides in solution, which could then be correlated with nmr spectra of the same solutions. Each coupling constant measured was taken as an average of the values for the different conformations possible at that site, and the equation below derived.

$$^3J_{HH} = (8.9\pm0.9)\cos^2\theta - (0.9\pm0.9)\cos\theta + (0.9\pm0.9)\sin^2\theta$$

Ramachandran's group used a similar method, correlating nmr spectra with conformational energy calculations for a range of model compounds. A least squares procedure was then used to calculate the values of the constants in the equation below

$$^3J_{HH} = 7.9\cos^2\theta - 1.5_5\cos\theta + 1.3_5\sin^2\theta$$

This equation was then tested by applying it both to simple model peptides (N-acetylamino acid N-methylamides) and to three cyclic oligopeptide systems. Good agreement was found for the model peptides when their calculated conformations were compared with the coupling constants, unless the side chain extended beyond the Cβ. In the case of valine, isoleucine, phenylalanine and tyrosine compounds, the observed coupling constants were larger than those expected from the calculation. As the values derived for leucine, methionine and tryptophan fitted the calculated conformational distribution, it was considered that the disagreements were the result of interactions not taken into account in the conformational calculations. The results obtained by checking solution nmr data for the cyclic oligopeptides against independently determined structural parameters were considered to be more satisfactory. The peptides used were ferrichrome, for which an nmr spectrum (Llinas *et al*, 1970) and an X-ray structure (Zalkin *et al*, 1966) have been published, valinomycin, for which the nmr data (Ivanov *et al*, 1969; Ohnishi and Urry, 1969) were compared with conformational calculations (Stern *et al*, 1968) and gramicidin S-A, for which the only structural information available was derived from the nmr data in that paper.

The curve presented by Barfield and Gearhart was derived for the model compound N-methylacetamide by means of self-consistent perturbation theory in the semi-empirical INDO (intermediate neglect of differential overlap) approximation of self-consistent-field molecular orbital theory. Values were calculated for the coupling constant at 30° intervals for both *cis* and *trans* orientations of the amide bond, leading to the equations below, which correspond to within 0.05 Hz of the calculated values:

$$cis\ {}^{3}J_{HH} = 11.27\cos^{2}\theta - 4.32\cos\theta + 0.01 \text{ Hz}$$

$$trans\ {}^{3}J_{HH} = 12.06\cos^{2}\theta - 4.48\cos\theta + 0.01 \text{ Hz}$$

The method used by DeMarco and Llinas is the most empirical, owing nothing to molecular energy calculations, but being derived directly from a comparison of nmr data with the results of crystallographic analysis (Zalkin *et al*, 1966; Norrestam *et al*, 1975). The use of two independent structures of ferrichrome peptides allows a range of dihedral angles to be associated with each coupling constant, leading to a more accurate set of parameters. The equation they derive (DeMarco *et al*, 1978b) is given by:

$${}^{3}J_{HH} = (5.41\pm0.02)\cos^{2}\theta - (1.27\pm0.15)\cos\theta + (2.17\pm0.54) \text{ Hz}$$

In the same paper, they suggest that seriously nonplanar amide linkages may have higher coupling constants than would ordinarily be calculated by this formula. In a later paper (DeMarco and Llinas, 1980) they present three similar curves derived for different solvents and show that solvent effects on peptides in solution are mainly of conformational origin and are relatively minor. The same group have also produced a set of constants for a Karplus curve to describe the non-amide protons in the ferrichrome series of peptides. (Llinas *et al*, 1978i)

$${}^{3}J_{HH} = (9.5\pm0.3)\cos^{2}\theta - (1.6\pm0.2)\cos\theta + (1.8\pm0.6) \text{ Hz}$$

These equations, all of which are soundly based in theory,give expected values for the H-C(4)-N(1)-H coupling constant in the cyclic disulphide

ranging from 5.7 Hz (Bystrov *et al*,1969) to 12.4 Hz (Barfield and Gearhart, 1973), making it quite clear that the curves only give a rough guide to molecular conformation in most cases. It is however possible to deduce that the ring amide linkage in (VI) is *trans*, while the exocyclic amide is best treated as either rotating freely about the bond C(2)-N(2), or having three possible conformations about this bond (^3J of 7 Hz can be rationalised as an average of two *gauche* and one *trans* rotamer - J = 5 Hz for *gauche* and 11 Hz for *trans*).

In a similar fashion the conformation about the C(1)-C(2) bond can be deduced from the coupling constants, showing that the upfield methene proton must be *trans* to the methine proton.

Useful structural information may also be obtained by comparing spectra of a compound run in two different solvents (Llinas and Klein, 1975; Williams *et al*, 1982). When the spectrum of the cyclic disulphide (VI) is measured in d_6-DMSO (perdeutero dimethyl sulphoxide), the most striking change was the downfield shift of the amide proton resonances by over 2 ppm. This is caused by these protons hydrogen bonding to the solvent, indicating that they are exposed to the medium and are involved in neither inter- nor intramolecular hydrogen bonding. The changes in the spectrum on going from $CDCl_3$ to d_6-DMSO are tabulated below.

Resonance	$CDCl_3$	d_6-DMSO	Δδ/ppm
C_6H_5	7.297	7.246	-0.051
N(2)-H	6.624	8.808	2.165
N(1)-H	6.387	8.396	2.010
C(4)-H	4.890	4.755	-0.135
C(2)-H	4.555	4.677	0.122
$C(8)H_3$	3.741	3.687	-0.054
$C(10)H_2$	3.570	3.310	-0.260
C(1)H(*gauche*)	3.350	3.137	-0.213
C(1)H(*trans*)	2.842	2.901	0.059

The effects of variation in temperature on the amide proton resonances provide confirmatory evidence for the solvent exposure of

these protons. It has been shown (Llinas and Klein, 1975) that the resonance of a hydrogen bonding proton exposed to the solvent shifts upfield on warming as hydrogen bonding to the solvent is thermally disrupted.

peak	$CDCl_3$	d_6-DMSO
N(2)-H	-7.5(8)	-6.21(6)
N(1)-H	-0.1(11)	-3.83(8)

temperature coefficients in ppm/°C * 10^3

The table above shows the temperature coefficients of the amide protons in d_6-DMSO and in $CDCl_3$. These are calculated by measuring the chemical shift of the amide protons at various temperatures and then calculating a best-fit line through the plot of chemical shift versus temperature. In this case the temperature range covered was 20 - 80°C in d_6-DMSO and -40 - 49°C in $CDCl_3$, rising in 30°C increments in both cases. As would be expected, the coefficients for DMSO show hydrogen bonding to the solution, and the coefficient for the penicillamine amide (N(1)-H) in chloroform shows no hydrogen bonding. It is possible that at low temperatures the compound exists in chloroform solution as an hydrogen bonded dimer, as the coefficient for the cysteine amide (N(2)-H) does seem to show a loss of hydrogen bonding on warming.

The final method used to examine the solution conformation of the cyclic disulphide (VI) was nuclear Overhauser effect difference spectroscopy, which can be used to obtain information on the relative proximities of protons (Williams *et al*, 1982). A brief description of the theory of the nuclear Overhauser effect is as follows (Sanders and Mersh, 1982).

In the nmr experiment, excess spin population is induced to move between energy levels by an appropriate frequency of electromagnetic radiation, this is followed by a radiationless return to equilibrium, which requires fluctuating magnetic fields of appropriate frequencies. This is

spin-lattice relaxation. The main sources of this relaxation for diamagnetic organic molecules are the magnetic dipoles of protons in the same molecule (dipole-dipole interactions). The efficiency of this relaxation depends on the magnitude and frequency of the generated fluctuating magnetic fields. This in turn depends on the length and correlation time (or tumbling time) τ_c of the vector connecting the nuclei. Nuclear Overhauser effects, and their growth or decay rates, are essentially measures of the strength of dipole-dipole interactions between the spins involved. This is why they depend on interatomic distance and correlation time, giving them their power as structural probes. It can be demonstrated that the n.O.e. is a kinetic effect, as it can take some time to build up after the start of the irradiation causing it, and it persists for some time when the irradiating frequency is switched off. It can also be shown that for a rigid molecule tumbling isotropically, the rate at which the n.O.e. builds up and decays is inversely proportional to the sixth power of the internuclear distance. The constant of proportionality is related to the correlation time for any given system. There are a few specific occasions when the picture is not quite this simple. These are:

1) Nuclei with close proton neighbours are relaxed quickly and when observed in an n.O.e. experiment will reveal little long-range information; what long-range information is available will take a while to develop. Methyl and methene protons are a good example of this situation.

2) Nuclei without nearby neighbours obtain relaxation and n.O.e's from distant protons. This causes them to relax slowly, and when observed in an n.O.e experiment they show large, slowly growing n.O.e's from distant protons.

As a consequence of these points, a proton equidistant to a methine and a methene proton will in general give a larger n.O.e to the former. Methyl groups have even smaller n.O.e's than those of methene groups. The most reliable measure of distance is to observe n.O.e's at a methine proton whilst irradiating at a methyl or methene proton and not *vice versa*. The single most important application of the n.O.e is

its ability to make through-space connections between nuclei independently of chemical bonding considerations, but there are cases where the n.O.e is too small to be visible with conventional methods. In these circumstances, use of difference spectroscopy allows the measurement of n.O.e's down to a fraction of one percent.

The usual methodology for a difference n.O.e experiment is as follows:

1) A data file is prepared containing the frequencies of all resonances to be irradiated.

2) A subfile is created for each irradiating frequency (and possibly also an extra one for the control spectrum, which is not irradiated).

3) The pulse sequence is started with a slight delay.

4) A weak 'tickling' decoupling power is switched on at one of the irradiating frequencies.

5) One 'scan' or accumulation of data is collected.

6) The above sequence is repeated for the next irradiating frequency.

7) When a scan has been collected into each subfile, the next scan is recorded at each frequency of irradiation.

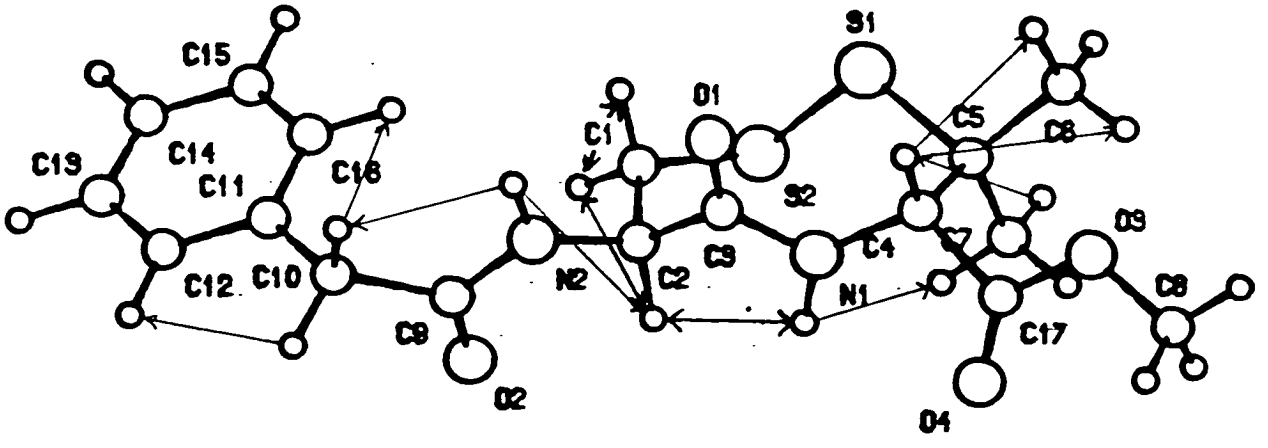
8) The entire sequence is then repeated until each subfile contains *ca* 1000 scans.

The reason for collecting the spectral information in this piecemeal, scan by scan, manner is that it minimises perturbation effects caused by continual or prolonged irradiation at the same frequency. When the control spectrum is subtracted from each irradiated spectrum, the resultant trace shows only the changes caused by irradiation at that frequency.

When this pulse sequence was carried out on the cyclic disulphide (VI), the following n.O.e's were observed.

Irradiated peak	Effect
N(2)-H	Small effect on C(2)-H and $C(10)H_2$
N(1)-H	Larger effect on C(2)-H and $C(7)H_3$ (small)
C(2)-H	N(1)-H and pro-S proton of $C(1)H_2$
C(4)-H	Slight effect on $C(6)H_3$
C(1)-H *trans*	Effect on pro-S proton of $C(1)H_2$
C(1)-H *gauche*	Effect on pro-R proton of $C(1)H_2$ and on C(2)-H
$C(10)H_2$	Slight effect on aromatic protons and $C(1)H_2$
$C(8)H_3$	None
$C(7)H_3$ or $C(6)H_3$	slight effect on C(4)-H

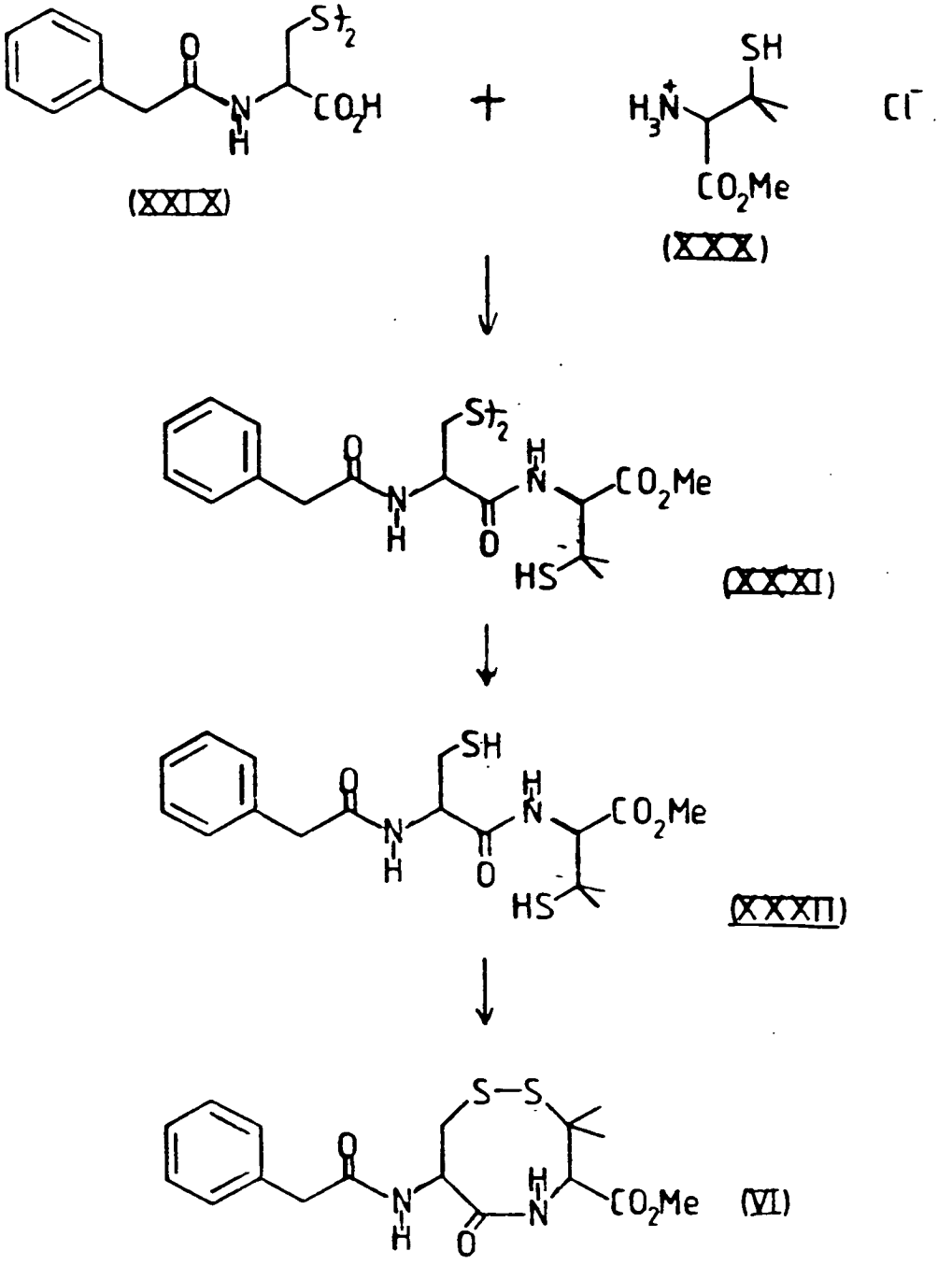
All these results are consistent with the solid state structure, and it may be stated with confidence that the cyclic disulphide (VI) exists in solution as monomers of one conformation only. The diagram below shows the measured n.O.e's graphically.



Experimental

(Synthesis of the cyclic disulphide (VI) as carried by E. M. Gordon, 1973)

The course of this synthesis is illustrated in the following diagram.



Nmr spectra were measured on Perkin Elmer EM 360, Bruker WM 300 or WM 200 spectrometers as deuterochloroform solutions using deuterochloroform as an internal standard for the FT machines, or tetramethylsilane for the CW machine. Optical rotations were recorded on a Mettler 141 polarimeter. Thin layer chromatography (t.l.c.) was carried out on 0.25mm layers of Merck GF_{254} silica. Melting points are uncorrected. Organic solvents were purified and dried by established procedures, and organic extracts typically dried over $MgSO_4$.

D-Penicillamine methyl ester hydrochloride (XXX) was prepared from D-Penicillamine (Aldrich Chemical Co. Inc.) by the standard method (Clarke, Johnson and Robinson, 1949) m.p. 178-180°C (lit. 180-182);$[\alpha]_D$ -10.39° (c 2,H_2O) (lit. $[\alpha]_D$ -7.50°, $[\alpha]$ -10.82°)

N,N'-bisphenylacetyl-L-cystine (XXIX) was prepared by the method of Foldi (1954) and had m.p. 171°C (lit 170-171°C)

N,N'-bisphenylacetyl-L-cystinyl-bis(D-penicillamine methyl ester) (XXXI)

To a solution of the acid (XXIX)(2.38g, 5mM) and D-penicillamine methyl ester hydrochloride (XXX) (1.63g, 10mM) in tetrahydrofuran (140 cm^3), triethylamine (1.5 cm^3) and N-ethyloxycarbonyl-2-ethyloxy-1,2-dihydroquinoline (EEDQ) (2.6g, 10.5mM) in tetrahydrofuran (40 cm^3) were added sequentially and the solution stirred at room temperature for 24hrs. The solution was evaporated and the residue dissolved in ethyl acetate (50 cm^3), washed sequentially with aqueous HCl (1M, 20 cm^3 * 2), sat. aqueous $NaHCO_3$ (20 cm^3 * 2) and water (20 cm^3 * 2) before being dried and evaporated. The residue was chromatographed on 2 * 200 * 200 mm silica plates, eluting with benzene:ethyl acetate (2:1) to afford crude (XXXI) which was crystallised from $CHCl_3$:hexanes (1.91g, 2.45mM) mp 208°C

3,3-dimethyl-4-D-carbomethoxy-6-oxo-7-L-phenylacetylamino perhydro-1,2,5-dithiazocine (VI)

The dipeptide (XXXI) (1.00g, 1.28mM) was dissolved in MeOH:aq. 5M HCl (120 cm^3, 5:1) at 0°C and Zn dust (10g) added to the cooled, stirred liquid over a period of 10 min. After a further 30 min. the solution was filtered, concentrated *in vacuo* to 30 cm^3 and poured into cold aqueous NaOH. The basic solution was washed with $CHCl_3$ (50 cm^3) then taken to pH 1 with conc. HCl, and the acidic solution extracted with $CHCl_3$ (100 cm^3 * 3). Evaporation of the $CHCl_3$ afforded free dithiol dipeptide (XXXII) as a colourless oil (0.97g).

The dithiol (XXXII) was then dissolved in MeOH (70 cm^3), iodine (0.64g) added, the solution heated under reflux for 2hrs, then concentrated *in vacuo* to 20 cm^3 and ethyl acetate (50 cm^3) added. The resultant solution was washed sequentially with sat. aqueous $Na_2S_2O_3$ (50 cm^3 * 2), aqueous HCl (1M, 50 cm^3), aqueous NaOH (1M, 50 cm^3) and water (50 cm^3). Evaporation of the ethyl acetate layer and crystallisation of the residue from $CHCl_3$/petrol afforded the cyclic disulphide (VI) (0.60g, 1.57mM) m.p.210°C transition, remelting at 218-220° C. (found C 53.37; H 5.81; N 7.29; S 16.61. $C_{17}H_{22}N_2O_4S_2$ requires C 53.40; H 5.80; N 7.33; S 16.73)

1Hnmr see text; MS (EI) m/e 382 (M+, exact mass 382.1021; required 382.1022 for $C_{17}H_{22}N_2O_4S_2$

Key to compound codes

CLCYST10 cyclo-L-cystine

K. I. Varughese, C. T. Lu, G. Kartha, Int. J. Pept. Protein Res., **18**, 88, 1981

CYLCYS cyclo-L-cystine acetic acid solvate

H.-C. Mez, Cryst. Struct. Commun., **3**, 657, 1974

CYSMEC L-cystine dimethyl ester dihydrochloride monohydrate

B. K. Vijayalakshmi, R. Srinivasan, Acta Cryst., **B31**, 993, 1975

CYSTBR01 L-cystine dihydrobromide

J. Peterson, L. K. Steinrauf, L. H. Jensen, Acta Cryst., **13**, 104, 1960

CYSTCL02 L-cystine dihydrochloride
(Neutron study)

D. D. Jones, I. Bernal, M. N. Frey, T. F. Koetzle, Acta Cryst., **B30**, 1220, 1974

CYSTIN10 L-cystine dihydrobromide dihydrate

R. E. Rosenfield Jnr., R. Parthasarathy, Acta Cryst., **B31**, 816, 1975

DGLYCH01 N, N'-diglycyl-L-cystine dihydrate
(Re-refinement)

W. C. Stallings Jnr., J. Donohue, Acta Cryst., **B32**, 1916, 1976

LCSTIM L-cystine diamide dihydrochloride

M. O. Chaney, L. K. Steinrauf, Acta Cryst., **B24**, 1564, 1968

LCYSCC L-cystine dihydrochloride dihydrate
(Copper(II) doped)

S. Kominami, P. Riesz, T. Akiyama, J. V. Silverton, J. Phys. Chem., **80**, 203, 1976

LCYSTI10 L-cystine
(Hexagonal form)

B. M. Oughton, P. M. Harrison, Acta Cryst., **12**, 396, 1959

LCYSTI11 L-cystine
(Tetragonal form)

M. O. Chaney, L. K. Steinrauf, Acta Cryst., **B30**, 711, 1974

PENICS10 D-penicillamine disulphide dihydrochloride
(Absolute configuration)

R. E. Rosenfield Jnr., R. Parthasarathy, Acta Cryst., **B31**, 462, 1975

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
S(1)	0.52140(0)	0.27580(0)	0.17070(0)	0.0562
S(2)	0.4055(6)	0.1064(6)	0.2599(5)	0.0564
O(1)	0.6872(12)	0.4844(11)	0.4510(9)	0.0477
O(2)	0.4466(13)	0.0798(11)	0.6796(11)	0.0723
O(3)	1.0667(11)	0.4041(11)	0.2033(10)	0.0584
O(4)	1.0290(12)	0.2567(12)	0.3493(10)	0.0677
N(1)	0.7577(12)	0.2898(12)	0.3825(10)	0.0335
N(2)	0.5234(14)	0.3003(13)	0.6128(11)	0.0379
C(1)	0.4000(16)	0.1884(16)	0.4088(13)	0.0418
C(2)	0.5514(14)	0.2379(14)	0.4996(12)	0.0284
C(3)	0.6752(16)	0.3506(16)	0.4472(13)	0.0357
C(4)	0.8302(16)	0.3548(16)	0.2845(13)	0.0355
C(5)	0.7192(16)	0.2723(16)	0.1605(13)	0.0454
C(6)	0.7715(19)	0.3670(17)	0.0580(14)	0.0622
C(7)	0.7162(18)	0.1138(16)	0.1328(14)	0.0538
C(8)	1.2034(16)	0.3739(16)	0.1866(14)	0.0528
C(9)	0.4601(16)	0.2083(18)	0.6916(13)	0.0315
C(10)	0.4026(17)	0.2792(17)	0.7977(14)	0.0520
C(11)	0.2286(15)	0.2284(15)	0.7770(12)	0.0369
C(12)	0.1462(19)	0.1779(17)	0.8703(16)	0.0603
C(13)	-0.0196(20)	0.1293(18)	0.8517(17)	0.0671
C(14)	-0.0923(20)	0.1360(17)	0.7457(15)	0.0571
C(15)	-0.0116(22)	0.1830(20)	0.6521(18)	0.0733
C(16)	0.1456(18)	0.2316(16)	0.6694(14)	0.0526
C(17)	0.9880(17)	0.3290(17)	0.2849(15)	0.0351
S(1')	0.0853(6)	0.5756(6)	0.5681(5)	0.0649
S(2')	0.1872(7)	0.7368(7)	0.4659(5)	0.0776
O(1')	0.4798(12)	0.5918(10)	0.6672(9)	0.0557
O(2')	0.7000(12)	0.9811(11)	0.4206(10)	0.0537
O(3')	0.2426(13)	0.6723(12)	0.9931(9)	0.0607
O(4')	0.3569(14)	0.8986(11)	0.9428(10)	0.0743
N(1')	0.3862(15)	0.7732(12)	0.7240(13)	0.0412
N(2')	0.6445(14)	0.7709(14)	0.5047(11)	0.0380
C(1')	0.3715(16)	0.7186(16)	0.4439(13)	0.0478
C(2')	0.5069(14)	0.7878(14)	0.5461(11)	0.0298
C(3')	0.4641(15)	0.7091(16)	0.6545(12)	0.0301
C(4')	0.2695(15)	0.6860(15)	0.7884(12)	0.0318
C(5')	0.1086(17)	0.6634(16)	0.7258(14)	0.0454
C(6')	-0.0189(17)	0.5476(17)	0.7765(14)	0.0591
C(7')	0.0743(17)	0.8097(15)	0.7264(13)	0.0489
C(8')	0.2596(19)	0.7421(18)	1.1154(15)	0.0661
C(9')	0.7268(18)	0.8689(18)	0.4383(14)	0.0436
C(10')	0.8550(16)	0.8304(17)	0.3842(13)	0.0420
C(11')	0.8185(17)	0.8017(16)	0.2507(14)	0.0429
C(12')	0.9374(21)	0.8688(19)	0.1828(17)	0.0710
C(13')	0.904(3)	0.8455(22)	0.0589(19)	0.0917
C(14')	0.7673(23)	0.7571(20)	0.0046(19)	0.0830
C(15')	0.6483(25)	0.6887(23)	0.0659(18)	0.0856
C(16')	0.6752(19)	0.7057(18)	0.1923(16)	0.0674
C(17')	0.2987(18)	0.7651(20)	0.9163(16)	0.0483

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(1A)	0.7570	0.1924	0.4130	0.0500
H(2A)	0.5141	0.3921	0.6189	0.0500
H(011)	0.3108	0.1062	0.4439	0.0436
H(012)	0.3677	0.2847	0.3986	0.0436
H(021)	0.5922	0.1485	0.5188	0.0565
H(041)	0.8498	0.4727	0.2992	0.0483
H(061)	0.7721	0.4783	0.0812	0.0537
H(062)	0.6902	0.3142	-0.0265	0.0537
H(063)	0.8883	0.3739	0.0489	0.0537
H(071)	0.6386	0.0602	0.0469	0.0465
H(072)	0.6742	0.0510	0.2031	0.0465
H(073)	0.8336	0.1179	0.1287	0.0465
H(081)	1.2572	0.4383	0.1208	0.0547
H(082)	1.1700	0.2561	0.1547	0.0547
H(083)	1.2864	0.4042	0.2715	0.0547
H(101)	0.4507	0.3996	0.8042	0.0494
H(102)	0.4447	0.2472	0.8815	0.0494
H(121)	0.2114	0.1758	0.9575	0.0501
H(131)	-0.0811	0.0908	0.9234	0.0699
H(141)	-0.2191	0.0996	0.7302	0.0631
H(151)	-0.0769	0.1851	0.5649	0.0700
H(161)	0.2071	0.2701	0.5977	0.0511
H(1'A)	0.4273	0.8476	0.7230	0.0500
H(2'A)	0.6741	0.7130	0.5505	0.0500
H(11')	0.4035	0.7696	0.3658	0.0367
H(12')	0.3510	0.6007	0.4267	0.0367
H(21')	0.5353	0.9057	0.5744	0.0462
H(41')	0.3082	0.6060	0.8081	0.0339
H(61')	-0.1340	0.5350	0.7280	0.0561
H(62')	-0.0082	0.5850	0.8713	0.0561
H(63')	-0.0040	0.4413	0.7659	0.0561
H(71')	0.1635	0.8913	0.6908	0.0521
H(72')	0.0756	0.8531	0.8180	0.0521
H(73')	-0.0399	0.7869	0.6710	0.0521
H(81')	0.2107	0.6571	1.1696	0.0484
H(82')	0.1979	0.8188	1.1144	0.0484
H(83')	0.3829	0.8024	1.1535	0.0484
H(0'1)	0.8644	0.7315	0.4172	0.0487
H(0'2)	0.9661	0.9230	0.4137	0.0487
H(2'1)	1.0534	0.9417	0.2306	0.0492
H(3'1)	0.9938	0.8980	0.0090	0.0723
H(4'1)	0.7412	0.7366	-0.0931	0.0692
H(5'1)	0.5323	0.6158	0.0182	0.0750
H(6'1)	0.5853	0.6532	0.2422	0.0497

Thermal Vibration Parameters with Standard Deviations

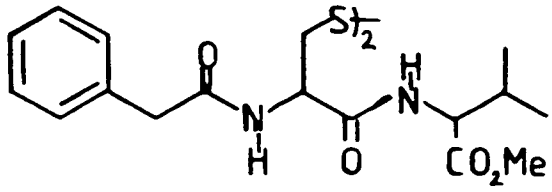
	U11	U22	U33	U23	U13	U12
S(1)	0.0393(26)	0.0747(33)	0.0482(31)	0.0092(26)	0.0056(24)	0.0334(25)
S(2)	0.0419(28)	0.0503(29)	0.0608(34)	-0.0143(24)	0.0063(23)	0.0077(24)
O(1)	0.0588(65)	0.0294(59)	0.0503(66)	0.0017(50)	0.0285(55)	0.0206(49)
O(2)	0.0890(83)	0.0360(64)	0.0913(93)	0.0297(63)	0.0470(74)	0.0371(58)
O(3)	0.0384(60)	0.0602(71)	0.0745(79)	0.0166(62)	0.0259(59)	0.0307(54)
O(4)	0.0569(67)	0.0829(81)	0.0585(73)	0.0163(65)	0.0096(57)	0.0467(63)
N(1)	0.0275(62)	0.0309(70)	0.0431(78)	0.0125(61)	0.0141(59)	0.0236(56)
N(2)	0.0473(73)	0.0203(66)	0.0415(81)	0.0067(64)	0.0182(65)	0.0139(62)
S(1')	0.0439(26)	0.0762(36)	0.0535(33)	-0.0249(27)	0.0039(23)	0.0092(26)
S(2')	0.0537(31)	0.1216(44)	0.0492(35)	0.0124(33)	0.0042(26)	0.0533(31)
O(1')	0.0795(75)	0.0245(56)	0.0651(76)	0.0198(51)	0.0282(61)	0.0415(54)
O(2')	0.0625(65)	0.0314(58)	0.0646(77)	0.0169(54)	0.0255(60)	0.0272(50)
O(3')	0.0900(79)	0.0562(69)	0.0260(63)	0.0098(57)	0.0223(59)	0.0304(60)
O(4')	0.1141(102)	0.0327(69)	0.0483(75)	-0.0047(56)	0.0008(67)	0.0034(64)
N(1')	0.0453(85)	0.0242(74)	0.0469(82)	0.0016(75)	0.0143(71)	0.0119(68)
N(2')	0.0328(68)	0.0429(80)	0.0373(79)	0.0174(64)	0.0061(63)	0.0276(59)

CHAPTER FOUR
BIS-(N-PHENYLACETYL) CYSTINYL BIS(VALINE METHYL ESTER)

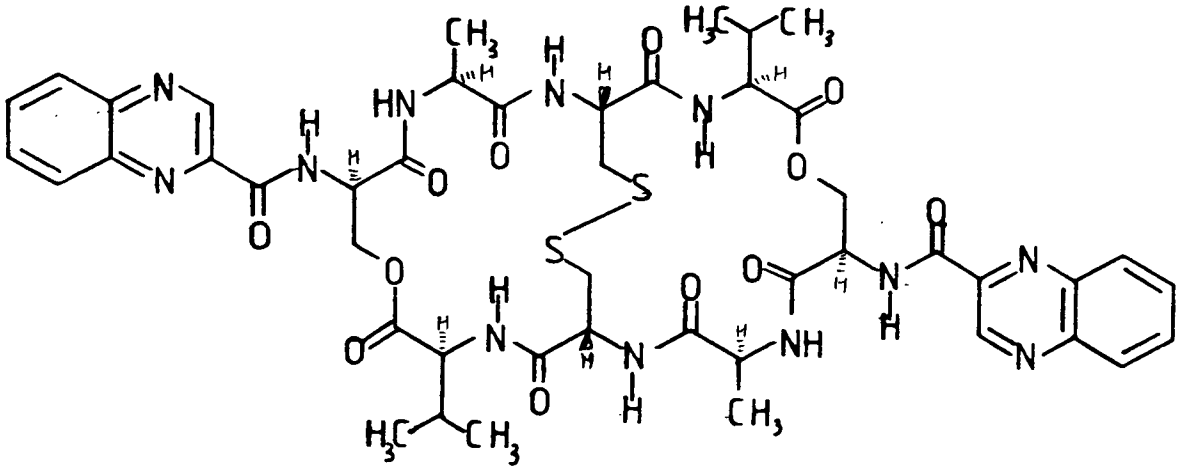
BIS(N-PHENYLACETYL) L-CYSTINYL BIS(L-VALINE METHYL ESTER)

Introduction

This compound (VIII) was a synthetic intermediate prepared during a study on the biosynthesis of β-lactam antibiotics. It is of structural interest because of its similarity to the cysteinyl penicillamine cyclic disulphide described previously. The central portion of this molecule is also similar in conformation to the anti-tumour drug Triostin and its synthetic analogue TANDEM (XXXIV) or tetra N-desmethyl Triostin A (Williams *et al*, 1982), although this compound is not expected to have any such activity, as Triostin functions by intercalating two quinoxaline chromophores into the minor groove of the DNA helix between the adenine and thymine bases (Viswamitra *et al*,1981).



(VIII)



(XXXIV)

Preparation and crystallisation

The compound was prepared by Dr. E. M. Gordon, according to the procedure described in the experimental section below. The crystal used for both preliminary photographs and data collection was grown from a solution of the compound in a slight excess of chloroform, to which enough petrol-ether was added to cause a slight turbidity of the solution. The solution was then left for two days at room temperature.

Crystal Data

Formula $C_{34}H_{46}N_4O_8S_2$ Mol.Wt. = 702.84 F(000) = 5984

Space Group $P4_32_12$ Int. Tab. No. 96
Cell dimensions a = 21.534(5) c = 34.507(12)
Dc = 1.167 gcm^{-3} Dm = 1.166 gcm^{-3}
Z = 16 Vol. = 16001.3 cubic Angstroms
Radiation MoKα λ = 0.71069 Angstroms μ = 1.73 cm^{-1}
Final R = 0.0912 based on 2201 data
h_{max} = 16 k_{max} = 23 l_{max} = 37

θ_{max} = 22.5° $Sin(\theta_{max})$ = 0.3867 $Sin(\theta_{max})/\lambda$ = 0.5385

Reflections measured 5934 (36 systematically absent)
After merging 5877
Unobserved 3676 ($F<4\sigma(F)$)
No. parameters refined = 396
Max. Shift/ σ in last cycle = 0.452
Final difference Fourier Max. = 0.3847 eA^{-3} Min. = -0.2655 eA^{-3}

Space Group Determination

The space group was determined to be one of the enantiomorphous pair $P4_32_12$ or $P4_12_12$ from the systematic absences on the preliminary photographs (conditions for reflection: 00l, l=4n; h00, h=2n). These space groups differ in the sense of rotation of the 4-fold screw axis and it is only possible to distinguish them when the absolute configuration of the compound is known. In this case the choice was made on the basis of the known configuration of the cystinyl Cα carbon.

Data Collection and Reduction

The data set was collected on the CAD4 and the program CADABS was used to correct the data for Lorentz and polarisation effects.

Structure Solution and Refinement

Due to the large number of atoms in the asymmetric unit (96 non-hydrogen atoms in four half-molecules, of which 4 were sulphur atoms), it was not possible to solve the structure by means of the Patterson map. Accordingly, the program NORMAL in the MULTAN suite of programs was used to produce a set of normalised structure factors. An automatic run of the MULTAN system was unsuccessful in solving the structure, but normalisation of the data with the number of atoms in the unit cell set to a small fraction of the actual number, revealed the positions of the four sulphur atoms. This may be because a lower value for the total electron density in the cell leads to an upgrading of all phase relationships and an emphasis on the contributions from heavy atoms. The coordinates of these sulphur atoms were used as a starting point for the application of the DIRDIF procedure. Although the first run of the program led to a recognisable molecular fragment, there was not quite enough information available from this to allow the program SHELX to improve significantly on it. Subsequently, the fragment was recycled thrice more through DIRDIF before a fragment large enough for use in SHELX was obtained. The diagnostics produced for the first and the last two of these runs are tabulated below.

Test	S_4	$S_4O_4N_4C_{13}$	$S_4O_6N_8C_{32}$
R	0.74	0.44	0.38
BH	7.0	4.4	5.6
BL	8.4	4.9	5.7
σC(EH)*	4.0*	4.2	4.9
(EL)	2.7*	2.5	0.8
σA(EH)	8.4	1.6*	2.07
(EL)	4.0	0.8*	0.9

CONSISTENCY

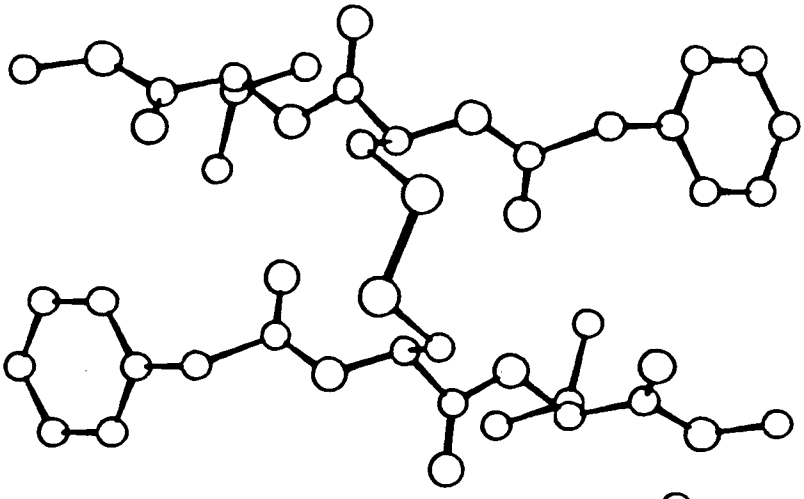
CYCLE 1	0.15(352)	0.38(87)	0.40(91)
2	0.21(738)	0.31(139)	0.32(144)
3	0.13(734)	0.31(138)	0.31(152)

Refinement of the structure progressed slowly, due to the disorder in one molecule (a phenylacetyl group occupying two possible sites) and the high temperature factors of the atoms in each molecule furthest from the region held in position by the sulphur bridge and hydrogen bonding systems. The final model has anisotropic thermal parameters for sulphur, nitrogen and the amide oxygen atoms, all carbon atoms and the ester oxygens having isotropic temperature factors. All hydrogen atoms are in calculated positions with temperature factors constrained to be equal. The phenyl rings are rigid groups, idealised to fit the positions of the rings found in the difference maps. The occupancy ratio for the sites of the disordered ring is 0.54 : 0.46 when the temperature factors are constrained to be equal for both rings. The methyl ester group on one of the molecules (molecule 4) is very poorly defined without having another possible location visible in the difference map.

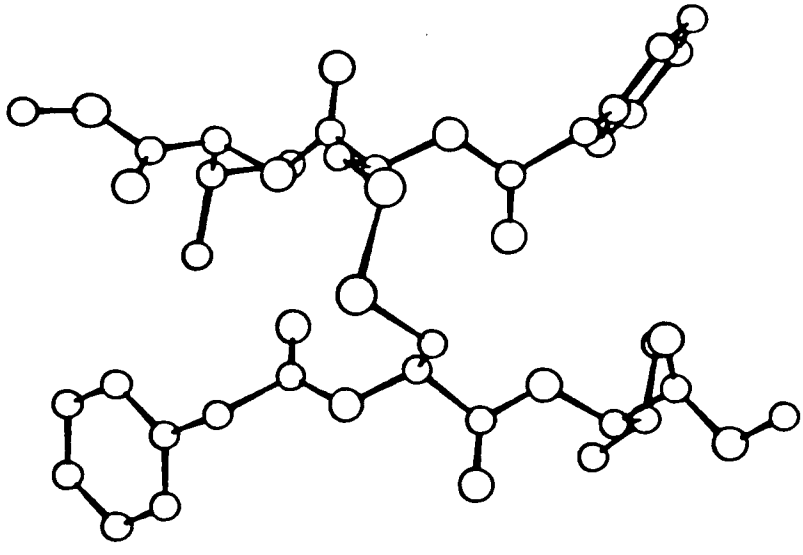
* For an explanation of the notation used in this table, please see p36

Description of Structure

The unit cell contains 16 molecules, distributed over eight asymmetric units; with one full molecule in a general position, and two half molecules lying across crystallographic dyads in each asymmetric unit. These molecules are illustrated below.

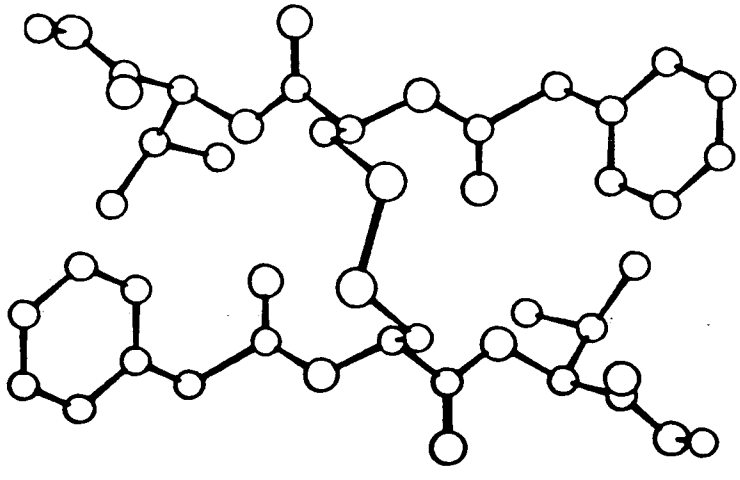


Molecule 1



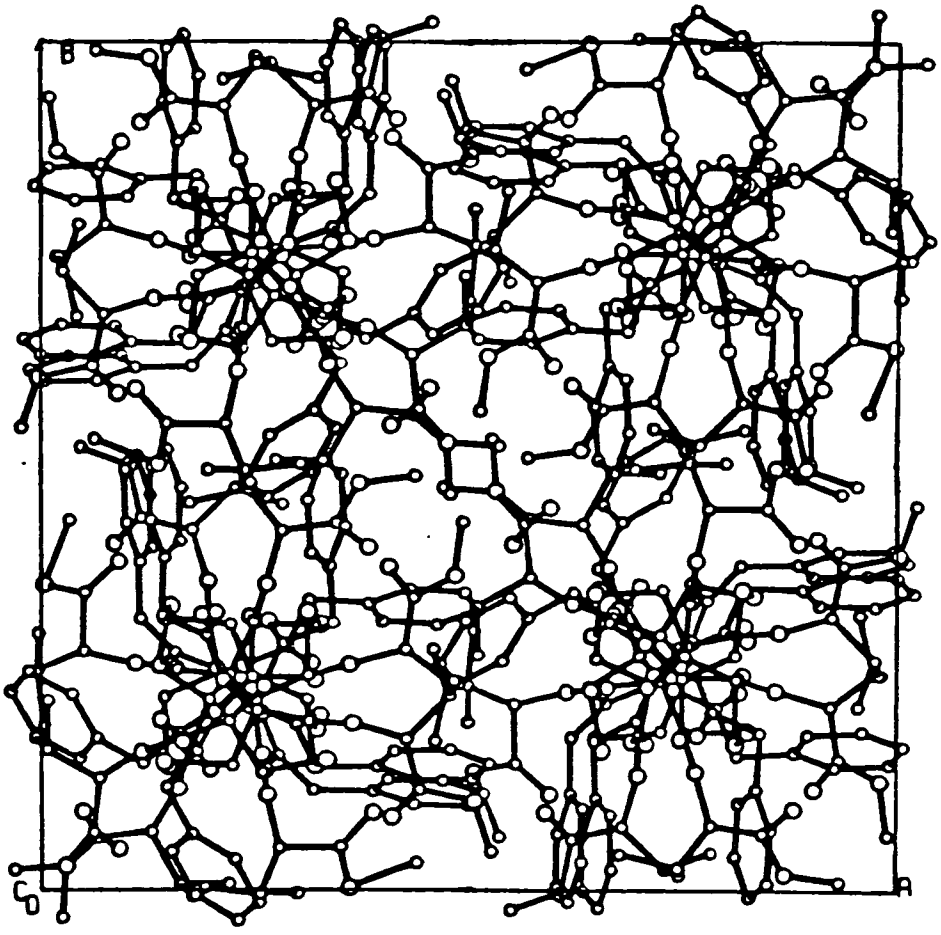
Molecule 2

Molecule 3



Molecule 4

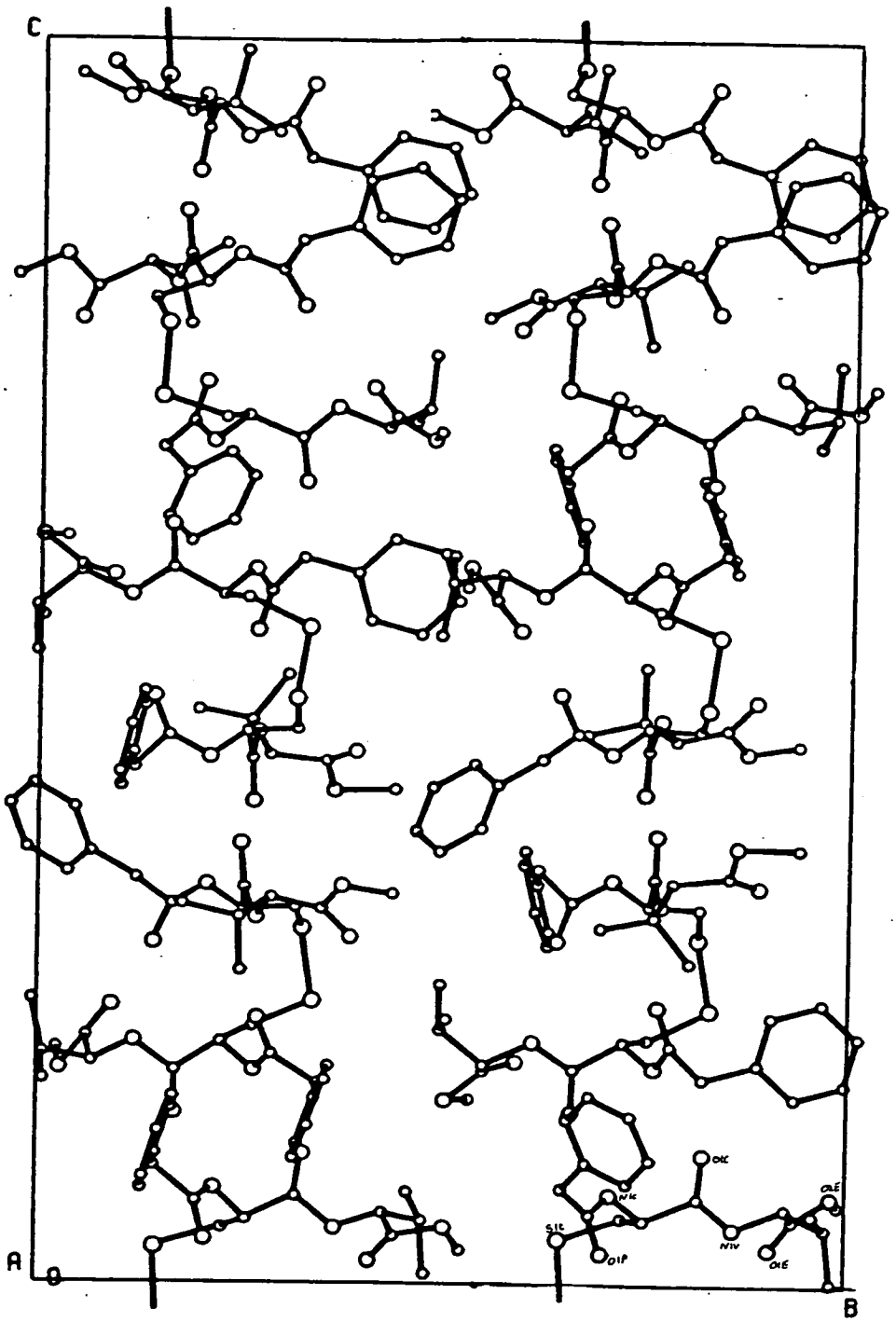
The molecule in the general position is disordered about one of its two phenylacetyl groups. Hydrogen bonding is both inter- and intramolecular, and occurs parallel to the z-axis in a manner analogous to the β-pleated sheet, which is the most common hydrogen bonding pattern for peptides. As a consequence of the twofold rotation axes at z = 0 and z = 1/2, the hydrogen bonding continues as an infinite ribbon as can be seen in the projection down half the x-axis. This helical arrangement occurs four times in the cell due to the 4_3 screw axis. Each column of molecules is well defined at the centre, giving way to a looser arrangement of the molecules at the periphery of each quadrant. This shows up strikingly in the projection down the z-axis, which has a surprising amount of clear space for a projection down 35 angstroms.



PROJECTION DOWN THE Z AXIS

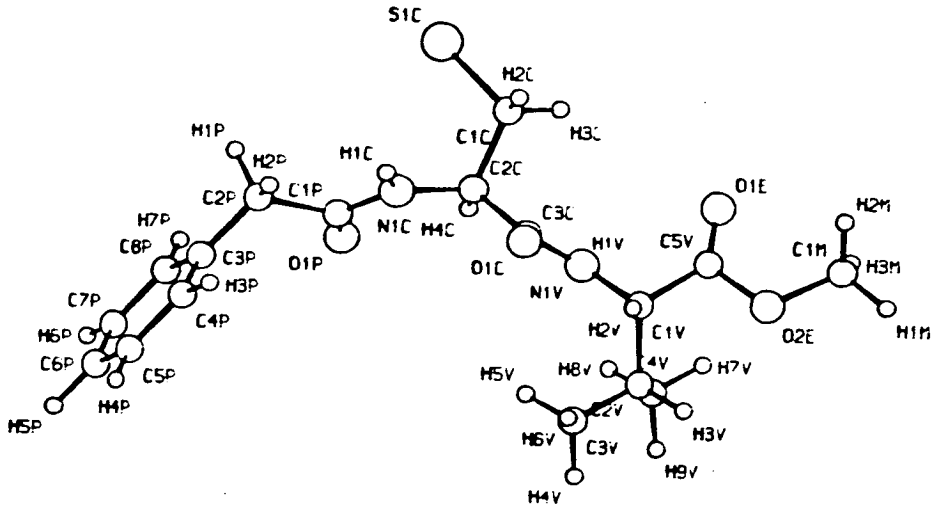
BIS-(N-PHENYLACETYL) CYSTINYL BIS-(VALINE METHYL ESTER)

BIS-(N-PHENYLACETYL) CYSTINYL BIS-(VALINE METHYL ESTER)



HALF PROJECTION DOWN THE X AXIS

Almost all the bond lengths are within their usual ranges, except that the valine Cα to C' distance is significantly different between molecule 2 (1.57(3) Angstrom) and molecule 3 (1.36(3) Angstrom). All bond angles are well within the usual ranges except for the ether oxygen angle in molecule 4, which is 7.6 e.s.d.'s from the mean (85.6(27)° *versus* 106°). The diagram below shows the numbering scheme used in these tables.



The amide groups are all somewhat twisted out of planarity, as can be seen from the deviations from the mean plane and torsion angles below (the extra value in the column for molecule 3 is due to the alternate position of the phenylacetyl group).

Amide bond	M1	M2	M3	M4
Phen Cys	0.06	0.09	0.05	0.03
			0.08	
Cys Val	0.04	0.06	0.08	0.04

RMS deviation from mean plane of C-CO-N-C
(in Angstrom units)

Torsions	M1	M2	M3	M4
C2C-N1C-C1P-C2P	-169.5(14)	-162.8(14)	-177.5(16)	-178.5(16)
			-160.6(19)	
C2C-N1C-C1P-O1P	1.7(27)	14.8(28)	12 (4)	10 (3)
			22 (5)	
C1V-N1V-C3C-C2C	173.7(14)	169.2(15)	164.9(16)	172.9(17)
C1V-N1V-C3C-O1C	-3.6(25)	-6.8(28)	-5 (3)	-2 (3)

This deviation is probably due to the hydrogen bonding pattern, and allows for the rotation between each molecule and its neighbours on the z-axis. There are a number of conformational differences between the phenylacetyl groups on the different molecules. Half molecules 1 and 2 have similar torsions here, but the major occupancy ring on half molecule 3 points away from the main body of the molecule by about 45° compared with the first two, and it is also twisted a similar amount about the methene to carbonyl bond. The minor occupancy ring is closer in orientation to those of half molecules 1 and 2, but it still differs by more than 30° in torsion about the methene to carbonyl bond. In half molecule 4, the conformation of the phenylacetyl group is totally different, with the torsions being of the opposite sense to those in the rest of the structure, as can be seen from examination of the molecular plots. At the other end of the chain, the valine residue adopts a similar conformation in all molecules, except for molecule 4, where it is twisted *circa* 30° about the N-Cα bond compared to the rest of the molecules. The only important difference in conformation of the ester group occurs in molecule 2, where the torsion angle Cα-C'-O-C is *cis* rather than *trans*. The most significant contacts are those involved in hydrogen bonding either within or between molecules, although there are also a number of oxygen to carbon contact distances less than 3.5 Angstroms. There is one nitrogen to nitrogen and one carbon carbon contact less than 3.5 Angstroms, both between molecules 1 and 2. There are 10 contacts involving sulphur atoms less than 4.0 Angstroms and 11 sulphur to hydrogen contacts less than 3.5 Angstroms. There are numerous contacts between hydrogen and carbon, nitrogen or oxygen atoms, of which the most significant are those involved in hydrogen bonding or

the intramolecular close contacts between the cystine Cα protons.

LABEL	M1	M2	M3	M4	MX
S1C-S1C	2.019(7)	2.045(7)		2.019(9)	
S1C-C1C	1.853(17)	1.767(17)	1.795(17)	1.834(21)	
O1C-C3C	1.217(21)	1.210(23)	1.226(24)	1.21(3)	
O1P-C1P	1.214(21)	1.176(22)	1.21(4)	1.22(3)	1.21(4)
O1E-C5V	1.166(25)	1.15(3)	1.26(3)	1.21(4)	
O2E-C5V	1.37(3)	1.42(3)	1.34(3)	1.30(4)	
O2E-C1M	1.47(3)	1.37(4)	1.36(3)	1.42(5)	
N1C-C2C	1.427(20)	1.445(20)	1.489(20)	1.455(23)	
N1C-C1P	1.304(22)	1.301(23)	1.32(4)	1.26(3)	1.32(4)
N1V-C3C	1.368(22)	1.361(23)	1.32(3)	1.32(3)	
N1V-C1V	1.463(22)	1.432(24)	1.53(3)	1.51(3)	
C1C-C2C	1.557(23)	1.569(22)	1.501(23)	1.48(3)	
C2C-C3C	1.486(23)	1.531(25)	1.57(3)	1.50(3)	
C1V-C2V	1.52(3)	1.48(3)	1.47(3)	1.57(4)	
C1V-C5V	1.48(3)	1.57(3)	1.36(3)	1.47(4)	
C2V-C3V	1.46(3)	1.46(3)	1.59(3)	1.47(4)	
C2V-C4V	1.52(3)	1.51(3)	1.51(3)	1.52(4)	
C1P-C2P	1.56(3)	1.54(3)	1.53(4)	1.59(3)	1.53(4)
C2P-C3P	1.492(18)	1.532(18)	1.520(14)	1.476(22)	1.526(15)

The table above shows all the unconstrained bond distances in the structure. The fifth column shows the bond distances in the minor site of the phenylacetyl group of molecule 3.

LABEL	M1	M2	M3	M4	MX
S1C-S1C-C1C	103.8(6)	105.0(6)	105.5(6)	108.5(7)	
C5V-O2E-C1M	113.3(16)	108.5(22)	116.6(21)	85.6(27)	
C2C-N1C-C1P	122.5(14)	123.0(14)	118.6(19)	122.2(16)	117.5(20)
C3C-N1V-C1V	120.2(14)	120.1(15)	118.8(16)	121.2(17)	
S1C-C1C-C2C	111.8(11)	112.6(11)	116.2(12)	113.8(14)	
N1C-C2C-C1C	111.3(13)	111.7(13)	108.5(13)	112.0(15)	
N1C-C2C-C3C	112.4(13)	112.1(13)	108.1(13)	108.5(15)	
C1C-C2C-C3C	104.2(13)	106.6(13)	108.9(14)	109.7(16)	
O1C-C3C-N1V	121.1(16)	124.7(17)	125.5(18)	121.6(19)	
O1C-C3C-C2C	124.9(16)	121.4(16)	120.4(17)	121.5(19)	
N1V-C3C-C2C	113.9(14)	113.9(15)	113.2(16)	116.7(17)	
N1V-C1V-C2V	110.8(15)	113.0(17)	111.3(17)	108.4(19)	
N1V-C1V-C5V	106.8(15)	106.9(17)	105.6(19)	105.1(21)	
C2V-C1V-C5V	116.0(16)	107.9(18)	111.2(21)	113.9(24)	
C1V-C2V-C3V	111.0(18)	109.3(19)	107.7(19)	108.0(24)	
C1V-C2V-C4V	111.9(17)	115.9(20)	115.7(19)	107.4(24)	
C3V-C2V-C4V	111.9(18)	107.5(20)	105.3(19)	120.6(27)	
O1E-C5V-O2E	122.1(19)	124.2(24)	112.3(23)	123 (3)	
O1E-C5V-C1V	130.0(19)	126.5(23)	135.7(25)	129 (3)	
O2E-C5V-C1V	107.8(16)	105.1(19)	112.0(22)	107.6(28)	
O1P-C1P-N1C	125.4(16)	125.9(17)	125 (3)	126.4(21)	125 (3)
O1P-C1P-C2P	121.3(16)	124.3(17)	122.4(27)	115.8(19)	123 (3)
N1C-C1P-C2P	112.7(15)	109.7(15)	112.1(25)	117.3(19)	112.1(28)
C1P-C2P-C3P	117.1(13)	115.3(13)	110.2(16)	112.7(16)	111.9(17)
C2P-C3P-C4P	120.8(7)	118.5(7)	112.6(5)	119.3(9)	124.4(6)
C2P-C3P-C8P	119.2(7)	121.5(7)	127.4(5)	120.6(9)	115.6(6)

This table gives a comparison of all the bond angles in the structure. It can easily be seen that the methyl ester on molecule 4 is very badly distorted. The table of comparative torsion angles is shown overleaf.

C1C-S1C-S1C-C1C	97.4	(8)		99.0	(8)		99.6	(10)			
S1C-S1C-C1C-C2C	-96.5	(11)	-100.0	(11)	-84.9	(12)	-89.2	(14)			
C1M-O2E-C5V-O1E	3	(3)	-22	(4)	4	(3)	1	(4)			
C1M-O2E-C5V-C1V	179.7	(16)	179.8	(21)	-174.4	(22)	-174	(3)			
C1P-N1C-C2C-C1C	123.2	(16)	109.4	(17)	122.4	(21)	117.5	(20)	118.4	(23)	
C1P-N1C-C2C-C3C	-120.3	(17)	-131.1	(17)	-119.7	(22)	-121.2	(20)	-123.6	(23)	
C2C-N1C-C1P-O1P	1.7	(27)	15	(3)	12	(4)	10	(3)	22	(5)	
C2C-N1C-C1P-C2P	-169.5	(14)	-162.8	(14)	-177.5	(16)	-178.5	(16)	-160.6	(19)	
C1V-N1V-C3C-O1C	-3.6	(25)	-7	(3)	-5	(3)	-2	(3)			
C1V-N1V-C3C-C2C	173.7	(14)	169.2	(15)	164.9	(16)	172.9	(17)			
C3C-N1V-C1V-C2V	118.4	(17)	121.8	(19)	123.9	(20)	155.1	(20)			
C3C-N1V-C1V-C5V	-114.4	(17)	-119.7	(19)	-115.2	(21)	-82.7	(25)			
S1C-C1C-C2C-N1C	-49.3	(15)	-48.6	(16)	-57.6	(16)	-59.5	(18)			
S1C-C1C-C2C-C3C	-170.7	(11)	-171.3	(11)	-175.1	(12)	180.0	(13)			
N1C-C2C-C3C-O1C	-36.3	(23)	-29.0	(23)	-35.7	(22)	-43.3	(25)			
N1C-C2C-C3C-N1V	146.5	(14)	154.8	(14)	154.4	(15)	141.9	(17)			
C1C-C2C-C3C-O1C	84.3	(20)	93.4	(19)	82.1	(21)	79.4	(24)			
C1C-C2C-C3C-N1V	-92.8	(16)	-82.7	(17)	-87.9	(18)	-95.4	(21)			
N1V-C1V-C2V-C3V	-63.9	(21)	-66.6	(23)	-59.2	(22)	-59.2	(27)			
N1V-C1V-C2V-C4V	61.9	(21)	54.9	(25)	58.2	(25)	72.3	(26)			
C5V-C1V-C2V-C3V	174.2	(17)	175.4	(19)	-176.8	(20)	-175.8	(26)			
C5V-C1V-C2V-C4V	-60.1	(23)	-63.0	(25)	-59.4	(27)	-44	(3)			
N1V-C1V-C5V-O1E	6	(3)	-2	(3)	-11	(4)	-6	(5)			
N1V-C1V-C5V-O2E	-171.3	(14)	155.9	(17)	167.2	(19)	169.0	(24)			
C2V-C1V-C5V-O1E	129.7	(23)	120	(3)	110	(3)	113	(4)			
C2V-C1V-C5V-O2E	-47.2	(22)	-82.3	(22)	-71.9	(27)	-72	(3)			
O1P-C1P-C2P-C3P	50.0	(22)	48.6	(24)	72	(3)	-80.2	(23)	84	(4)	
N1C-C1P-C2P-C3P	-138.4	(14)	-133.8	(14)	-98.5	(24)	107.2	(21)	-93.7	(27)	
C1P-C2P-C3P-C4P	107.6	(13)	100.0	(13)	154.4	(15)	-153.7	(12)	100.8	(18)	
C1P-C2P-C3P-C8P	-72.5	(14)	-78.4	(14)	-24.6	(18)	29.8	(19)	-78.9	(18)	
C2P-C3P-C4P-C5P	-180.0	(8)	-178.4	(8)	-179.1	(6)	-176.5	(10)	-179.7	(7)	
C2P-C3P-C8P-C7P	180.0	(8)	178.4	(8)	179.0	(7)	176.5	(10)	179.7	(6)	

Intramolecular Contacts

O1P1	2.893	(18)	N1V1	(Y,X,1 -Z)
O1P2	2.961	(20)	N1V3	(X,Y,Z)
O1P3	2.865	(18)	N1V2	(X,Y,Z)
O1P4	2.893	(21)	N1V4	(Y,X,1 -Z)
O1P1	3.405	(20)	C2C1	(Y,X,1 -Z)
O1E1	3.465	(14)	C8P1	(Y,X,1 -Z)
O1P2	3.337	(20)	C2C3	(X,Y,Z)
O1P3	3.256	(20)	C2C2	(X,Y,Z)
O1P4	3.364	(23)	C2C4	(Y,X,1 -Z)
O1P4	3.46	(4)	C4V4	(Y,X,1 -Z)

Intermolecular Contacts

S1C1	3.795	(13)	O1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
S1C1	3.852	(19)	O2E3	(Y,1 +X,1 -Z)
S1C3	3.488	(16)	O2E1	(Y,X,1 -Z)
S1C3	3.781	(15)	O1C4	(1/2-X,1/2+Y,3/4-Z)
S1C1	3.927	(5)	C7P2	(1 +X,Y,Z)
S1C2	3.60	(3)	C4V4	(Y,1 +X,1 -Z)
S1C3	3.919	(23)	C2V1	(Y,X,1 -Z)
S1C3	3.989	(20)	C4V1	(Y,X,1 -Z)
S1C3	3.896	(25)	C1M1	(Y,X,1 -Z)
S1C4	3.542	(27)	C4V2	(Y,X,1 -Z)
O1C1	2.897	(17)	N1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C2	2.817	(17)	N1C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1C3	2.874	(20)	N1C4	(1/2-X,1/2+Y,3/4-Z)
O1C4	2.765	(19)	N1C3	(1/2-X,-1/2+Y,3/4-Z)
O1C1	3.344	(21)	C2P2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C2	3.496	(20)	C1C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1C4	3.455	(22)	C1C3	(1/2-X,-1/2+Y,3/4-Z)
N1C1	3.477	(18)	N1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
C4P1	3.463		C4P2	(1/2+X,1 1/2-Y,1 1/4-Z)

Intramolecular Hydrogen Contacts

O1P1	2.662	(20)	H4C1	(Y,X,1 -Z)
O1P2	2.486	(20)	H4C3	(X,Y,Z)
O1P3	1.812	(18)	H1V2	(X,Y,Z)
O1P3	2.71	(3)	H8V2	(X,Y,Z)
O1P4	1.834	(21)	H1V4	(Y,X,1 -Z)
O1P4	2.43	(4)	H9V4	(Y,X,1 -Z)
O1P1	1.849	(18)	H1V1	(Y,X,1 -Z)
O1P1	2.904	(23)	H8V1	(Y,X,1 -Z)
O1E1	2.399	(14)	H7P1	(Y,X,1 -Z)
O1P2	1.904	(20)	H1V3	(X,Y,Z)
O1P2	2.843	(26)	H8V3	(X,Y,Z)
O1E2	2.999	(23)	H2PX	(X,Y,Z)
O1P3	2.434	(20)	H4C2	(X,Y,Z)
O1E3	2.644	(17)	H7P2	(X,Y,Z)
O1P4	2.514	(23)	H4C4	(Y,X,1 -Z)
C2C1	2.920	(22)	H4C1	(Y,X,1 -Z)
C2C2	2.875	(23)	H4C3	(X,Y,Z)
C7P2	2.993	(23)	H7V3	(X,Y,Z)
C4V2	2.709	(26)	H7PX	(X,Y,Z)
C2C3	2.941	(23)	H4C2	(X,Y,Z)
C1P3	2.87	(4)	H1V2	(X,Y,Z)
C1PX	2.85	(4)	H1V2	(X,Y,Z)
C2C4	2.957	(24)	H4C4	(Y,X,1 -Z)
H4C1	1.913	(22)	H4C1	(Y,X,1 -Z)
H4C2	1.914	(23)	H4C3	(X,Y,Z)
H7V2	2.076	(26)	H7PX	(X,Y,Z)
H8V2	2.447	(26)	H7P3	(X,Y,Z)
H4C4	1.932	(24)	H4C4	(Y,X,1 -Z)

Intermolecular Hydrogen Contacts

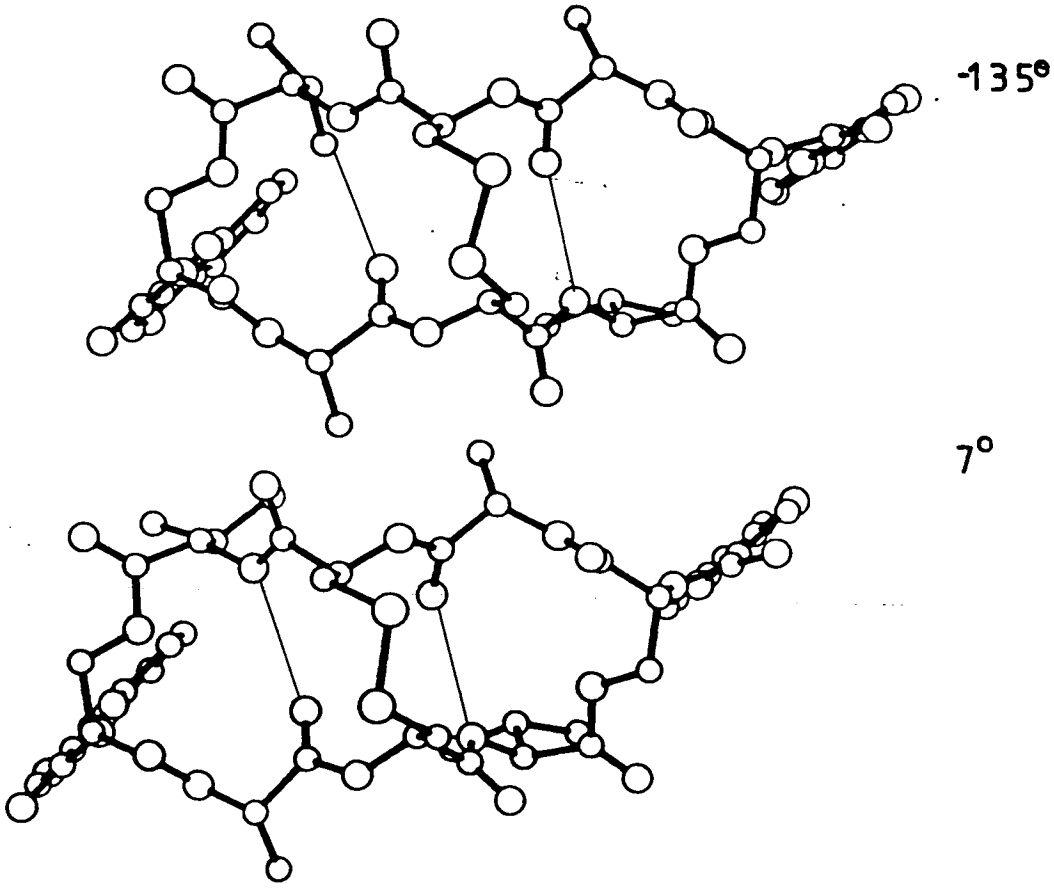
S1C1	2.923	(5)	H6P2	(1 +X,Y,Z)
S1C1	3.482	(23)	H3V3	(Y,1 +X,1 -Z)
S1C2	3.099	(5)	H6P1	(X,Y,Z)
S1C2	3.31	(3)	H3V4	(Y,1 +X,1 -Z)
S1C2	3.16	(3)	H7V4	(Y,1 +X,1 -Z)
S1C2	3.25	(3)	H8V4	(Y,1 +X,1 -Z)
S1C3	2.982	(23)	H3V1	(Y,X,1 -Z)
S1C3	3.418	(25)	H1M1	(Y,X,1 -Z)
S1C	3.393	(3)	H7V4	(Y,1 +X,1 -Z)
S1C4	2.612	(27)	H9V2	(Y,X,1 -Z)
S1C4	3.139	(6)	H6PX	(X,Y,Z)
O1C1	2.700	(11)	H5P1	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C1	1.878	(17)	H1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C1	2.381	(21)	H2P2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1P1	2.967	(12)	H3P2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C2	1.805	(17)	H1C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1C2	2.808	(21)	H2P1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1P2	2.940	(12)	H3P1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1E2	2.71	(4)	H3M2	(Y,X,1 -Z)
O1C3	1.871	(20)	H1C4	(1/2-X,1/2+Y,3/4-Z)
O1E3	2.916	(17)	H4P3	(1/2-X,1/2+Y,3/4-Z)
O1C4	2.344	(19)	H1C3	(1/2-X,-1/2+Y,3/4-Z)
O1C4	2.732	(20)	H2P3	(1/2-X,-1/2+Y,3/4-Z)
O1C2	2.904	(20)	H2C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
O1C2	2.563	(11)	H5P2	(1/2+X,1 1/2-Y,1 1/4-Z)
O1C3	2.824	(26)	H1P4	(1/2-X,1/2+Y,3/4-Z)
O2E3	2.93	(3)	H6V2	(1/2-Y,1/2+X,-1/4+Z)
O1C4	2.786	(22)	H2C3	(1/2-X,-1/2+Y,3/4-Z)
O1C4	2.663	(21)	H1PX	(1/2-X,-1/2+Y,3/4-Z)
O2E4	2.68	(3)	H2V1	(1/2-Y,-1/2+X,-1/4+Z)

Intermolecular Hydrogen Contacts - continued

N1C1	2.686	(18)	H1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
N1C2	2.691	(18)	H1C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
N1C3	2.805	(19)	H1C4	(1/2-X,1/2+Y,3/4-Z)
C3C1	2.731	(22)	H1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
C3C1	2.970	(25)	H2P2	(1/2+X,1 1/2-Y,1 1/4-Z)
C1P1	2.842	(18)	H3P2	(1/2+X,1 1/2-Y,1 1/4-Z)
C4P1	2.845	(0)	H4P4	(1/2+Y,1 1/2-X,1/4+Z)
C3P1	2.788	(0)	H4P4	(1/2+Y,1 1/2-X,1/4+Z)
C3C2	2.693	(23)	H1C1	(-1/2+X,1 1/2-Y,1 1/4-Z)
C3C2	2.988	(19)	H5P2	(1/2+X,1 1/2-Y,1 1/4-Z)
C7P2	2.886	(28)	H1M3	(-1 +Y,1 +X,1 -Z)
C1C3	2.931	(16)	H5P3	(1/2-X,1/2+Y,3/4-Z)
C3C3	2.863	(24)	H1C4	(1/2-X,1/2+Y,3/4-Z)
C4V3	2.906	(23)	H4P1	(-1/2+X,1 1/2-Y,1 1/4-Z)
C6P3	2.952	(22)	H2P4	(1/2-X,1/2+Y,3/4-Z)
C5PX	2.990	(22)	H2P4	(1/2-X,1/2+Y,3/4-Z)
C3C4	2.957	(21)	H3PX	(1/2-X,-1/2+Y,3/4-Z)
C4P4	2.701	(25)	H4V3	(1/2-X,-1/2+Y,3/4-Z)
C5P4	2.856	(24)	H2M1	(Y,-1 +X,1 -Z)
C6P4	2.628	(24)	H2M1	(Y,-1 +X,1 -Z)
C3V4	2.91	(4)	H1P2	(-1 +Y,X,1 -Z)
H1C1	2.060	(18)	H1C2	(1/2+X,1 1/2-Y,1 1/4-Z)
H3C1	2.412	(29)	H9V3	(1 +X,Y,Z)
H4P1	2.429	(23)	H9V	(1/2+X,1 1/2-Y,1 1/4-Z)
H6V1	2.433	(3)	H2V4	(1/2+Y,1/2-X,1/4+Z)
H9V1	2.293	(25)	H3C2	(Y,X,1 -Z)
H1P2	2.14	(4)	H3V4	(Y,1 +X,1 -Z)
H6P2	2.415	(28)	H1M3	(-1 +Y,1 +X,1 -Z)
H6V2	2.37	(3)	H2V3	(-1/2+Y,1/2-X,1/4+Z)
H1C3	2.237	(19)	H1C4	(1/2-X,1/2+Y,3/4-Z)
H2C3	2.210	(16)	H5P3	(1/2-X,1/2+Y,3/4-Z)
H4V3	2.225	(25)	H3P4	(1/2-X,1/2+Y,3/4-Z)
H2M3	2.460	(28)	H4P3	(1/2-X,1/2+Y,3/4-Z)
H6PX	2.469	(20)	H2C4	(X,Y,Z)

Comparison with the cyclic octadepsipeptide TANDEM

A particularly interesting feature of this structure is the sulphur bridge connecting two peptide chains hydrogen bonded in a manner analogous to the β-sheet. Although there are more than a thousand crystal structures of peptides on file with the Cambridge Crystallographic Database, only about twenty of these also contain sulphur bridges. Most of these compounds are simple cystine derivatives with no intramolecular hydrogen bonding, such as the compounds discussed in the previous chapter. After an exhaustive search of the database, in both the interactive and the batch modes, one other compound was found to contain a similar hydrogen bonded sulphur bridge. This compound, known as TANDEM (an abbreviation for des-N-tetramethyl triostin A) (XXXIV), is a cyclic octadepsipeptide antibiotic with a strong binding affinity for the alternating sequence of adenine and thymine. It is suggested (Viswamitra *et al*, 1981) that the selectivity shown in binding of a factor of 7,500 in favour of poly(dA-dT) against poly(dG-dC) is explainable in terms of the backbone conformation, which is controlled by the intramolecular hydrogen bonds. The atomic coordinates from the two independent crystal structures at -135° and 7° C (Hossain *et al*, 1981) were used to produce the diagrams below, in which each molecule is projected onto the best plane of its peptide backbone.



The tables below give a numerical comparison of the central portion of the cystinyl valine dimer with the equivalent portion of the antibiotic. Standard deviations quoted for each figure are based on the unweighted mean of the relevant parameters and do not reflect the e.s.d's involved as the e.s.d's for the antibiotic were unavailable. In the case of the torsion angles, the standard deviation calculated is not quoted if it is greater than 10°, as this is taken to indicate a genuine difference between the structures and not merely a minor difference in conformation.

As with the cyclic disulphide discussed in the previous chapter, the torsion angles at the sulphur bridge belong to neither to class I nor to class II, but to the group of those compounds that are geometrically constrained to have the two Cα's closer to one another than is consistent with a type I or II structure. This would seem to indicate that the gain in conformational stability from intramolecular hydrogen bonding is more than enough to offset the slight steric crowding of the disulphide bridge. With the exception of the sulphur bridge, the side chains have different conformations entirely in the two compounds. However, the conformation at the amide bond is approximately the same in each compound.

bond	M1	M2	M3	M4	T1	T2	T3	T4	mean	e.s.d
B1C-N1C'	2.02	2.05	2.05	2.02	2.04	2.04	2.01	2.01	2.03	0.02
B1C-C1C	1.85	1.79	1.79	1.83	1.82	1.80	1.81	1.82	1.82	0.02
B1C'-C1C'	1.85	1.77	1.77	1.83	1.80	1.82	1.82	1.81	1.81	0.03
O1C-C3C	1.22	1.21	1.23	1.21	1.24	1.23	1.26	1.20	1.22	0.02
O1F-C5V	1.17	1.15	1.26	1.21	1.24	1.19	1.15	1.21	1.20	0.04
O2E-C1M	1.47	1.37	1.36	1.42	1.45	1.44	1.41	1.38	1.41	0.04
O2E-C5V	1.37	1.42	1.34	1.30	1.35	1.37	1.29	1.30	1.34	0.04
O1P-C1P	1.21	1.18	1.21	1.22	1.25	1.20	1.26	1.25	1.22	0.03
N1C-C2C	1.43	1.45	1.49	1.45	1.44	1.46	1.43	1.43	1.45	0.02
N1C-C1P	1.30	1.30	1.32	1.26	1.34	1.36	1.32	1.26	1.31	0.03
N1V-C3C	1.37	1.34	1.32	1.32	1.33	1.36	1.35	1.34	1.34	0.02
N1V-C1V	1.46	1.48	1.53	1.51	1.47	1.44	1.46	1.40	1.46	0.04
C1C-C2C	1.56	1.57	1.50	1.48	1.52	1.50	1.49	1.52	1.52	0.03
C2C-C3C	1.49	1.53	1.57	1.50	1.52	1.52	1.39	1.50	1.50	0.05
C1P-C2P	1.56	1.54	1.53	1.59	1.49	1.56	1.47	1.61	1.54	0.04
C1V-C2V	1.52	1.48	1.47	1.57	1.54	1.55	1.64	1.52	1.54	0.05
C1V-C5V	1.48	1.57	1.36	1.47	1.46	1.51	1.54	1.51	1.49	0.06
C2V-C3V	1.46	1.46	1.59	1.47	1.54	1.56	1.42	1.52	1.50	0.05
C2V-C4V	1.52	1.51	1.51	1.52	1.52	1.57	1.59	1.61	1.54	0.04

angle										
B1C'-B1C-C1C	103.84	104.98	104.98	108.46	105.85	105.90	105.65	106.83	105.81	1.29
B1C-N1C'-C1C'	103.84	105.46	105.46	108.46	105.90	105.85	106.83	105.65	105.93	1.23
C1M-O2E-C5V	113.30	108.55	116.57	85.61	117.41	117.17	116.63	116.20		
C2C-N1C-C1P	122.54	123.04	118.62	122.17	123.84	122.53	123.50	122.07	122.29	1.51
C3C-N1V-C1V	120.23	120.14	118.76	121.21	123.79	119.32	127.39	125.80	122.08	2.99
B1C-C1C-C2C	111.81	112.57	116.23	113.84	116.31	114.92	115.21	115.70	114.57	1.58
N1C-C2C-C1C	111.27	111.65	108.53	112.04	110.81	110.00	112.85	108.74	110.74	1.45
N1C-C2C-C3C	112.44	112.13	108.12	108.48	107.97	107.60	109.05	109.26	109.38	1.75
C1C-C2C-C3C	104.24	106.56	108.91	109.73	109.71	111.55	108.50	109.62	108.60	2.11
O1C-C3C-N1V	121.13	124.66	125.47	121.62	121.23	123.16	111.29	119.10	120.96	4.13
O1C-C3C-C2C	124.91	121.36	120.43	121.46	121.31	121.05	124.31	122.95	122.22	1.94
N1V-C3C-C2C	113.90	113.85	113.24	116.70	117.43	115.65	124.39	117.49	116.98	3.34
O1P-C1P-N1C	125.37	125.90	124.75	126.44	121.63	124.43	122.20	129.08	124.98	2.27
O1P-C1P-C2P	121.30	124.32	122.36	115.80	122.33	123.02	120.89	115.24	120.66	3.12
N1C-C1P-C2P	112.74	109.74	112.11	117.27	116.03	112.39	116.91	115.51	114.09	2.54
N1V-C1V-C2V	110.79	113.00	111.31	108.40	111.66	110.15	109.61	111.27	110.77	1.31
N1V-C1V-C5V	106.83	106.85	105.65	105.08	111.67	110.93	110.79	108.08	108.24	2.40
C2V-C1V-C5V	116.00	107.90	111.24	113.92	116.25	112.48	109.92	115.11	112.85	2.82
C1V-C2V-C3V	111.01	109.29	107.65	107.98	109.40	108.35	125.68	110.72	111.26	5.57
C1V-C2V-C4V	111.87	115.92	115.70	107.35	111.15	112.26	98.97	111.17	110.55	5.07
C3V-C2V-C4V	111.89	107.47	105.27	120.64	112.49	111.10	123.11	114.64	113.33	5.68
O1E-C5V-O2E	122.09	124.24	112.27	123.33	120.99	122.92	124.14	124.41	121.80	3.77
O1E-C5V-C1V	130.04	126.49	135.69	128.86	127.70	127.35	120.97	124.23	127.66	4.03
O2E-C5V-C1V	107.80	105.10	112.01	107.57	110.85	109.47	114.89	111.26	109.87	2.86

torsion	M1	M2	M3	M4	T1	T2	T3	T4	mean	esd
C1C-B1C-B1C'-C1C'	97.37	99.01	99.01	99.65	101.71	101.71	99.69	99.69	99.73	1.35
B1C'-B1C-C1C-C2C	-96.51	-100.02	-84.91	-89.21	-87.85	-84.23	-99.88	-79.05	-90.21	7.24
C1M-O2E-C5V-O1C	2.48	-21.99	4.06	0.87	-10.24	-10.30	-0.67	-5.85	-5.20	8.18
C1M-O2E-C5V-C1V	179.70	179.79	-174.35	-174.05	176.94	175.21	179.91	177.60	180.09	3.63
C1P-N1C-C2C-C1C	123.19	109.40	122.35	117.53	147.36	141.90	134.89	148.64		
C1P-N1C-C2C-C3C	-120.30	-131.08	-119.66	-121.19	-92.47	-96.39	-104.47	-91.74		
C2C-N1C-C1P-O1P	1.70	14.79	12.35	9.83	-4.52	-2.65	-9.92	-6.36	1.90	8.72
C2C-N1C-C1P-C2P	-169.51	-162.78	-177.54	-178.54	174.34	172.95	170.12	168.68	179.71	9.90
C1V-N1V-C3C-O1C	-3.61	-6.80	-4.48	-1.81	2.28	0.00	8.94	5.02	-0.06	4.91
C1V-N1V-C3C-C2C	173.67	169.18	164.89	172.90	-179.52	175.83	-172.22	177.50	175.28	6.54
C3C-N1V-C1V-C2V	118.42	121.80	123.90	159.13	126.17	179.40	106.96	126.49		
C3C-N1V-C1V-C5V	-114.37	-119.68	-115.23	-87.72	-101.78	-55.40	-131.56	-106.20		
B1C-C1C-C2C-N1C	-49.27	-48.58	-57.60	-59.90	-53.85	-60.98	-52.02	-63.96	-55.72	5.28
B1C-C1C-C2C-C3C	-170.69	-171.33	-175.10	179.96	-172.98	179.70	-172.98	176.65	184.16	4.45
N1C-C2C-C3C-O1C	-36.31	-29.04	-39.66	-43.33	-66.71	-94.32	-47.10	-53.83		
N1C-C2C-C3C-N1V	146.52	154.82	154.38	141.94	115.10	89.86	134.21	133.99		
C1C-C2C-C3C-O1C	84.34	93.40	82.09	79.37	54.15	26.41	76.18	65.24		
C1C-C2C-C3C-N1V	-92.83	-82.73	-87.87	-95.37	-124.05	-149.40	-102.91	-106.94		
N1V-C1V-C2V-C3V	-63.86	-66.65	-59.22	-59.18	-65.83	-67.07	50.78	-64.21		
N1V-C1V-C2V-C4V	61.91	54.94	58.17	72.33	169.34	169.86	-166.65	167.15		
C5V-C1V-C2V-C3V	174.16	175.45	-176.75	-175.77	164.47	168.62	-71.21	172.41		
C5V-C1V-C2V-C4V	-60.08	-62.96	-59.37	-44.25	39.64	45.55	71.36	43.77		
N1V-C1V-C5V-O1E	5.68	-1.64	-10.70	-5.94	141.40	141.40	137.82	127.21		
N1V-C1V-C5V-O2E	-171.25	155.93	167.20	164.01	-46.38	-44.41	-42.76	-56.23		
C2V-C1V-C5V-O1E	129.74	120.15	110.22	112.96	-88.90	-94.71	-100.88	-107.73		
C2V-C1V-C5V-O2E	-47.19	-82.27	-71.88	-72.49	83.31	79.47	78.54	68.83		

Solution Study on the Dimeric Disulphide

In the crystalline state the central portion of the molecule is held in a rigid conformation by the disulphide bridge and the intramolecular hydrogen bonds while the periphery of the molecule is not so constrained and thus has a far higher degree of thermal motion. In order to determine whether this was also the case in solution, it was decided to examine the conformation of the molecule by the method of nuclear magnetic resonance. The results of such a study could be compared with those obtained in the study on TANDEM (Williams *et al*, 1982) and in the study of the cyclic disulphide discussed in the previous chapter. It was expected that the study would show the molecule to be symmetrical on the nmr timescale with a well resolved ABMX system (the cystine methylene, methine and amide protons) and a similarly well resolved A_3B_3MRX system (the valine methyls, methines and amide protons). It was further expected that the coupling constants for these systems would indicate a rigid central portion of the molecule held together by hydrogen bonding and free rotation about the bonds in the valine and phenylacetyl group regions of the spectrum. Accordingly, the spectrum of the compound was measured at 360 MHz in deuterochloroform solution (It was felt that using perdeutero dimethyl sulphoxide would have severely lessened the probability of intramolecular hydrogen bonding) and the results are tabulated below.

Shift/ppm(m)		Assignment		
0.823d		Cγ2 of valine	$^3J_{MA}$ = 6.9 Hz	(A_3B_3MR system)
0.849d		Cγ1 of valine	$^3J_{MB}$ = 6.9 Hz	" "
2.094dqq		Cβ of valine	$^3J_{RM}$ = 6.8 Hz $^3J_{MA}$ = 6.8 Hz $^3J_{MB}$ = 6.8 Hz	" "
2.725)	m	Cystine Cβ poorly resolved AB		
2.881)		of ABMX system		
3.585s		Phenylacetyl methylene		
3.677s		Methyl ester		
4.379dd		Cα of valine	$^3J_{RX}$ = 8.3 Hz $^3J_{MR}$ = 5.1 Hz	(A_3B_3MR system)
4.755ddd		Cα of cystine	$^3J_{MX}$ = 7.7 Hz $^3J_{MA}$ = 5.2 Hz $^3J_{MB}$ = 6.4 Hz	(ABMX system)
6.862d		cystine amide	$^3J_{MX}$ = 7.9 Hz	" "
7.181d		valine amide	$^3J_{RX}$ = 8.3 Hz	(A_3B_3MR system)
7.255m		aromatic ring		

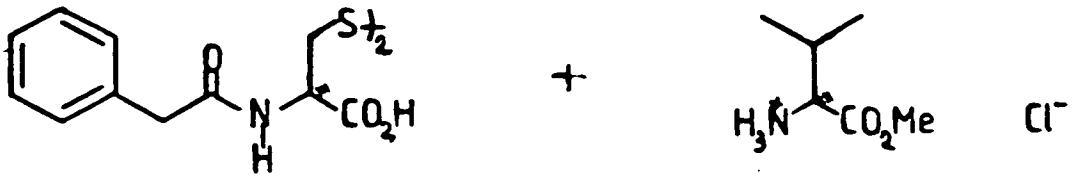
Where
s = singlet
d = doublet
t = triplet
q = quartet
and m = unresolved multiplet
with dd meaning "doublet of doublets" etc.

As the cystine methylene protons are so poorly resolved, it is unlikely that the molecule takes up one conformation about the disulphide bridge and the values of all measurable coupling constants (^3J =7±2 Hz) lend credence to the suggestion that the molecule is freely rotating about all possible bonds and hence does not exist in its intramolecular hydrogen bonded state. More evidence for this is obtained from the results of a nuclear Overhauser effect study of the molecule which was almost totally negative, showing only a slight interaction between the *ortho*- protons of the phenyl ring and the adjacent methylene group. It was impossible to assign the valine methyl resonances on the basis of n.O.e as was done for the cyclic disulphide. A reason for the difference in conformation between the crystalline compound and the compound in solution might lie in the close contact between the

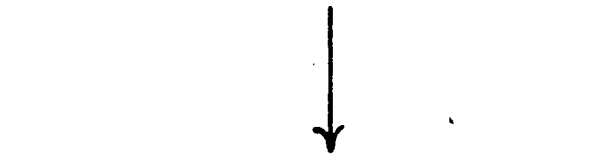
cystine C(a) protons (see above) and the unusual conformation of the cystine bridge. Presumably the combination of hydrogen bonding interactions with the crystal packing forces is strong enough to cause this close contact and to prevent the disulphide linkage from taking up either a type I or a type II conformation, but in solution the hydrogen bonds alone are not strong enough to hold this region of the molecule together. If the molecule is undergoing free rotation, it would be expected that the molecule would appear to have two-fold symmetry on the nmr timescale with the exception of the cystine methylene protons which may only have a symmetrical arrangement in the non-helical type of bridge conformation (as in the crystalline state), leading to the superposition in the spectrum of at least two possible AB subsystems of the ABMX spin system and hence to the poor resolution and concomitant lack of measurable coupling constant that is observed in this spectrum.

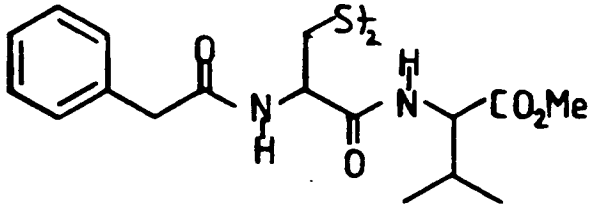
Experimental

(Synthesis of the dimeric disulphide (VIII) as carried by E. M. Gordon, 1973) The scheme below shows the compounds involved in this synthesis



XXIX + XXXIII





VIII

Organic solvents were purified and dried by established procedures, and organic extracts typically dried over Na_2SO_4. Chemicals were purchased from the Aldrich Chemical Co. Inc.

N,N'-bisphenylacetyl-L-cystine (XXIX) was prepared by the method of Foldi (Foldi, 1954) and had m.p. 171°C (lit 170-171°C)

Preparation of L-valine methyl ester hydrochloride (XXXIII)

To a slurry of L-valine (100g, 0.854mol) in absolute methanol (600ml) held at a temperature between -5°C and 0°C, thionyl chloride (130g, 0.96mol, 77ml) was added dropwise with vigorous stirring. After addition, the mixture was allowed to rise slowly to room temperature for two hours. The mixture was again cooled and another equal portion of thionyl chloride added dropwise. The mixture was then left stirring overnight, rising to room temperature.

The mixture was then filtered, and the methanol evaporated off at reduced pressure. The residue was dissolved in chloroform (400ml), filtered and again evaporated to dryness under reduced pressure, yielding an off-white crystalline solid (141g, 0.84mol).

Preparation of bis(N-phenylacetyl) L-cystinyl bis(L-valine methyl ester) (VIII)

Finely powdered L-valine methyl ester hydrochloride (XXXIII) (16.8g, 0.1mol) was mixed with bis(N-phenylacetyl) L-cystine (XXIX) (23.8g, 0.05mol) and EEDQ (24.7g, 0.1mol) in dry tetrahydrofuran (600ml). The mixture was stirred vigorously and N-methyl morpholine (10.8ml, 0.1mol) was injected in one portion. The mixture was then stirred overnight. The THF was removed by evaporation at reduced pressure and the residue was dissolved in ethyl acetate (50ml). The solution was washed twice with 1N HCl, once with 1N NaOH and once with saturated brine, before being dried over Na_2SO_4 and filtered. The solvent was removed under reduced pressure, and the residue was crystallised from hot chloroform and hexane (17.3g, 24.6mmol, fine white crystals, melting at 166°-168°C. Analysis (expected for $C_{34}H_{46}N_4O_8S_2$, found): C (58.103, 58.06%); N (7.972, 7.92%); S (9.123, 9.12%); H (6.597, 6.37%). For ^1H nmr see text.

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
S(1C1)	0.85003(25)	0.85438(22)	0.52920(15)	0.0812
N(1C1)	0.7526(6)	0.7837(5)	0.5765(4)	0.0511
O(1C1)	0.8112(5)	0.6747(5)	0.6047(3)	0.0721
O(1P1)	0.6725(5)	0.7939(6)	0.5357(4)	0.0787
N(1V1)	0.8043(7)	0.6348(6)	0.5445(4)	0.0701
C(1C1)	0.8543(8)	0.7721(7)	0.5446(5)	0.0604
C(2C1)	0.7888(7)	0.7442(7)	0.5521(5)	0.0486
C(3C1)	0.8015(7)	0.6829(8)	0.5703(5)	0.0483
C(1P1)	0.6986(9)	0.8054(8)	0.5661(5)	0.0449
C(2P1)	0.6739(8)	0.8567(8)	0.5941(5)	0.0797
C(4P1)	0.58751(0)	0.83613(0)	0.64120(0)	0.1163
C(5P1)	0.52441(0)	0.83439(0)	0.65027(0)	0.1404
C(6P1)	0.48043(0)	0.85108(0)	0.62247(0)	0.1535
C(7P1)	0.49956(0)	0.86951(0)	0.58560(0)	0.1406
C(8P1)	0.56267(0)	0.87125(0)	0.57653(0)	0.0939
C(3P1)	0.60664(0)	0.85456(0)	0.60432(0)	0.0664
C(1V1)	0.8227(8)	0.5728(8)	0.5575(5)	0.0607
C(2V1)	0.7705(10)	0.5262(10)	0.5509(6)	0.1017
C(3V1)	0.7163(10)	0.5417(9)	0.5743(6)	0.1055
C(4V1)	0.7544(9)	0.5199(9)	0.5083(6)	0.1003
C(5V1)	0.8825(9)	0.5585(10)	0.5383(6)	0.0776
O(1E1)	0.9140(6)	0.5897(7)	0.5188(4)	0.1041
O(2E1)	0.8962(7)	0.4973(7)	0.5454(4)	0.1196
C(1M1)	0.9548(11)	0.4769(11)	0.5277(7)	0.1483
S(1C4)	0.3283(3)	0.3131(3)	0.47153(20)	0.1214
O(1C4)	0.1597(7)	0.2614(8)	0.3882(4)	0.1230
O(1P4)	0.2649(8)	0.1402(6)	0.4731(4)	0.1242
N(1C4)	0.2604(7)	0.2073(7)	0.4244(4)	0.0779
N(1V4)	0.1118(6)	0.2668(7)	0.4450(4)	0.0792
C(1C4)	0.2533(10)	0.3143(9)	0.4463(6)	0.0969
C(2C4)	0.2225(8)	0.2532(8)	0.4444(5)	0.0616
C(3C4)	0.1620(10)	0.2592(9)	0.4234(7)	0.0721
C(1P4)	0.2802(9)	0.1590(11)	0.4411(7)	0.0758
C(2P4)	0.3225(10)	0.1134(11)	0.4164(6)	0.1162
C(4P4)	0.43409(0)	0.10031(0)	0.40089(0)	0.1441
C(5P4)	0.49640(0)	0.09900(0)	0.41193(0)	0.1477
C(6P4)	0.51305(0)	0.11307(0)	0.45000(0)	0.1744
C(7P4)	0.46739(0)	0.12844(0)	0.47703(0)	0.1804
C(8P4)	0.40509(0)	0.12974(0)	0.46599(0)	0.1492
C(3P4)	0.38844(0)	0.11568(0)	0.42792(0)	0.1283
C(1V4)	0.0494(11)	0.2807(12)	0.4269(7)	0.1120
C(2V4)	-0.0029(15)	0.2583(15)	0.4556(9)	0.1699
C(3V4)	0.0054(15)	0.1914(15)	0.4621(8)	0.2088
C(4V4)	-0.0038(15)	0.3034(15)	0.4895(9)	0.2225
C(5V4)	0.0495(18)	0.3480(17)	0.4200(10)	0.1645
O(1E4)	0.0881(12)	0.3862(13)	0.4292(7)	0.2150
O(2E4)	0.0019(12)	0.3608(12)	0.3984(7)	0.2166

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
S(1C2)	0.34669(23)	0.83575(23)	0.52261(15)	0.0746
N(1C2)	0.2602(6)	0.7646(6)	0.5775(4)	0.0545
O(1C2)	0.3259(6)	0.6686(5)	0.6128(3)	0.0761
O(1P2)	0.1821(5)	0.7678(6)	0.5362(4)	0.0764
N(1V2)	0.3385(7)	0.6191(6)	0.5549(4)	0.0604
C(1C2)	0.3622(7)	0.7609(7)	0.5433(5)	0.0553
C(2C2)	0.3014(7)	0.7255(7)	0.5552(5)	0.0439
C(3C2)	0.3220(8)	0.6679(9)	0.5778(6)	0.0599
C(1P2)	0.2079(9)	0.7857(8)	0.5641(5)	0.0437
C(2P2)	0.1856(8)	0.8403(8)	0.5892(5)	0.0797
C(4P2)	0.10151(0)	0.81722(0)	0.63774(0)	0.0911
C(5P2)	0.03937(0)	0.81331(0)	0.64889(0)	0.1145
C(6P2)	-0.00738(0)	0.83047(0)	0.62303(0)	0.1118
C(7P2)	0.00801(0)	0.85153(0)	0.58603(0)	0.1210
C(8P2)	0.07016(0)	0.85544(0)	0.57488(0)	0.0887
C(3P2)	0.11691(0)	0.83828(0)	0.60073(0)	0.0713
C(1V2)	0.3687(9)	0.5662(10)	0.5715(6)	0.0813
C(2V2)	0.3344(11)	0.5076(11)	0.5650(7)	0.1041
C(3V2)	0.2760(11)	0.5096(12)	0.5862(7)	0.1396
C(4V2)	0.3194(12)	0.4924(13)	0.5233(7)	0.1687
O(1E2)	0.4525(7)	0.5917(8)	0.5268(5)	0.1368
O(2E2)	0.4713(9)	0.5271(8)	0.5777(6)	0.1591
C(5V2)	0.4335(12)	0.5603(12)	0.5511(8)	0.1119
C(1M2)	0.5292(15)	0.5199(16)	0.5617(9)	0.2552
S(1C3)	0.35363(23)	0.82302(25)	0.46403(16)	0.0836
N(1C3)	0.2895(6)	0.7076(6)	0.4272(4)	0.0579
O(1C3)	0.1779(5)	0.7470(7)	0.3952(4)	0.1056
O(1P3)	0.2982(6)	0.6409(6)	0.4770(3)	0.1049
N(1V3)	0.1383(7)	0.7663(8)	0.4549(4)	0.0983
C(1C3)	0.2765(7)	0.8148(7)	0.4474(5)	0.0563
C(2C3)	0.2492(7)	0.7507(7)	0.4497(5)	0.0558
C(3C3)	0.1833(9)	0.7513(8)	0.4304(6)	0.0598
C(1V3)	0.0749(10)	0.7845(10)	0.4385(6)	0.0925
C(2V3)	0.0252(11)	0.7452(10)	0.4545(7)	0.1035
C(3V3)	0.0408(12)	0.6751(11)	0.4436(7)	0.1591
C(4V3)	0.0189(11)	0.7459(11)	0.4980(7)	0.1303
O(1E3)	0.0937(8)	0.8831(8)	0.4703(5)	0.1438
O(2E3)	0.0191(8)	0.8709(9)	0.4292(5)	0.1407
C(5V3)	0.0667(12)	0.8450(13)	0.4484(7)	0.1206
C(1M3)	0.0104(12)	0.9327(13)	0.4345(8)	0.1739
C(1P3)	0.3137(20)	0.6590(16)	0.4452(8)	0.0956
C(2P3)	0.3561(6)	0.6209(6)	0.4185(5)	0.0956
C(4P3)	0.34512(0)	0.54460(0)	0.36728(0)	0.0956
C(5P3)	0.31785(0)	0.49429(0)	0.34834(0)	0.0956
C(6P3)	0.26657(0)	0.46459(0)	0.36468(0)	0.0956
C(7P3)	0.24256(0)	0.48521(0)	0.39995(0)	0.0956
C(8P3)	0.26984(0)	0.53553(0)	0.41889(0)	0.0956
C(3P3)	0.32112(0)	0.56522(0)	0.40256(0)	0.0956
C(1PX)	0.3161(21)	0.6615(20)	0.4463(8)	0.0956
C(2PX)	0.3718(7)	0.6347(7)	0.4243(5)	0.0956
C(4PX)	0.34372(0)	0.58174(0)	0.35943(0)	0.0956
C(5PX)	0.32688(0)	0.52824(0)	0.33920(0)	0.0956
C(6PX)	0.31988(0)	0.47210(0)	0.35889(0)	0.0956
C(7PX)	0.32973(0)	0.46946(0)	0.39881(0)	0.0956
C(8PX)	0.34657(0)	0.52296(0)	0.41904(0)	0.0956
C(3PX)	0.35356(0)	0.57910(0)	0.39935(0)	0.0956

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(1C1)	0.7706	0.7961	0.6047	0.1984
H(1V1)	0.7924	0.6425	0.5145	0.1984
H(2C1)	0.8812	0.7692	0.5710	0.1984
H(3C1)	0.8770	0.7455	0.5221	0.1984
H(4C1)	0.7618	0.7394	0.5259	0.1984
H(1P1)	0.6828	0.9012	0.5807	0.1984
H(2P1)	0.7001	0.8533	0.6207	0.1984
H(3P1)	0.6216	0.8232	0.6627	0.1984
H(4P1)	0.5096	0.8201	0.6788	0.1984
H(5P1)	0.4316	0.8497	0.6295	0.1984
H(6P1)	0.4655	0.8824	0.5641	0.1984
H(7P1)	0.5775	0.8855	0.5480	0.1984
H(2V1)	0.8306	0.5706	0.5883	0.1984
H(3V1)	0.7870	0.4814	0.5605	0.1984
H(4V1)	0.6802	0.5080	0.5693	0.1984
H(5V1)	0.6997	0.5872	0.5662	0.1984
H(6V1)	0.7287	0.5416	0.6046	0.1984
H(7V1)	0.7955	0.5083	0.4921	0.1984
H(8V1)	0.7358	0.5633	0.4978	0.1984
H(9V1)	0.7202	0.4836	0.5046	0.1984
H(1M1)	0.9629	0.4287	0.5342	0.1984
H(2M1)	0.9927	0.5044	0.5389	0.1984
H(3M1)	0.9517	0.4830	0.4967	0.1984
H(1C4)	0.2720	0.2147	0.3943	0.1984
H(1V4)	0.1153	0.2629	0.4761	0.1984
H(2C4)	0.2610	0.3305	0.4171	0.1984
H(3C4)	0.2229	0.3462	0.4613	0.1984
H(4C4)	0.2154	0.2377	0.4738	0.1984
H(1P4)	0.3187	0.1259	0.3862	0.1984
H(2P4)	0.3057	0.0666	0.4204	0.1984
H(3P4)	0.4212	0.0894	0.3714	0.1984
H(4P4)	0.5317	0.0871	0.3910	0.1984
H(5P4)	0.5613	0.1121	0.4585	0.1984
H(6P4)	0.4803	0.1393	0.5065	0.1984
H(7P4)	0.3697	0.1416	0.4869	0.1984
H(2V4)	0.0402	0.2572	0.3998	0.1984
H(3V4)	-0.0493	0.2603	0.4438	0.1984
H(4V4)	-0.0305	0.1759	0.4815	0.1984
H(5V4)	0.0503	0.1835	0.4751	0.1984
H(6V4)	0.0023	0.1660	0.4352	0.1984
H(7V4)	-0.0394	0.2894	0.5097	0.1984
H(8V4)	-0.0135	0.3497	0.4793	0.1984
H(9V4)	0.0407	0.3028	0.5039	0.1984

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(1C2)	0.2736	0.7767	0.6067	0.1984
H(1V2)	0.3285	0.6205	0.5243	0.1984
H(2C2)	0.3906	0.7670	0.5688	0.1984
H(3C2)	0.3871	0.7332	0.5223	0.1984
H(4C2)	0.2751	0.7122	0.5298	0.1984
H(1P2)	0.1935	0.8827	0.5732	0.1984
H(2P2)	0.2128	0.8408	0.6154	0.1984
H(3P2)	0.1377	0.8039	0.6578	0.1984
H(4P2)	0.0274	0.7970	0.6775	0.1984
H(5P2)	-0.0555	0.8274	0.6317	0.1984
H(6P2)	-0.0282	0.8648	0.5660	0.1984
H(7P2)	0.0821	0.8717	0.5462	0.1984
H(2V2)	0.3724	0.5730	0.6024	0.1984
H(3V2)	0.3650	0.4715	0.5753	0.1984
H(4V2)	0.2508	0.4670	0.5816	0.1984
H(5V2)	0.2485	0.5482	0.5760	0.1984
H(6V2)	0.2855	0.5151	0.6167	0.1984
H(7V2)	0.3619	0.4906	0.5069	0.1984
H(8V2)	0.2895	0.5278	0.5113	0.1984
H(9V2)	0.2964	0.4480	0.5218	0.1984
H(1M2)	0.5591	0.4949	0.5813	0.1984
H(2M2)	0.5482	0.5655	0.5565	0.1984
H(3M2)	0.5259	0.4948	0.5347	0.1984
H(1C3)	0.2687	0.7462	0.4120	0.1984
H(1V3)	0.1464	0.7654	0.4857	0.1984
H(2C3)	0.2753	0.8291	0.4173	0.1984
H(3C3)	0.2474	0.8454	0.4643	0.1984
H(4C3)	0.2462	0.7363	0.4796	0.1984
H(2V3)	0.0733	0.7786	0.4074	0.1984
H(3V3)	-0.0172	0.7632	0.4421	0.1984
H(4V3)	0.0460	0.6708	0.4126	0.1984
H(5V3)	0.0034	0.6456	0.4533	0.1984
H(6V3)	0.0834	0.6613	0.4576	0.1984
H(7V3)	0.0084	0.7924	0.5075	0.1984
H(8V3)	0.0620	0.7308	0.5110	0.1984
H(9V3)	-0.0181	0.7151	0.5066	0.1984
H(1M3)	-0.0288	0.9479	0.4176	0.1984
H(2M3)	0.0514	0.9576	0.4253	0.1984
H(3M3)	0.0017	0.9418	0.4647	0.1984
H(1P3)	0.3313	0.6050	0.4438	0.1500
H(2P3)	0.3276	0.6537	0.4028	0.1500
H(3P3)	0.3848	0.5676	0.3546	0.1500
H(4P3)	0.3364	0.4783	0.3210	0.1500
H(5P3)	0.2455	0.4256	0.3500	0.1500
H(6P3)	0.2029	0.4622	0.4126	0.1500
H(7P3)	0.2513	0.5515	0.4462	0.1500
H(1PX)	0.3841	0.6798	0.4129	0.1500
H(2PX)	0.3877	0.6310	0.4539	0.1500
H(3PX)	0.3491	0.6252	0.3442	0.1500
H(4PX)	0.3193	0.5303	0.3083	0.1500
H(5PX)	0.3068	0.4307	0.3432	0.1500
H(6PX)	0.3243	0.4260	0.4141	0.1500
H(7PX)	0.3542	0.5209	0.4499	0.1500

Thermal Vibration Parameters with Standard Deviations

	U or U11	U22	U33	U23	U13	U12
S(1C1)	0.1111(46)	0.0644(37)	0.0681(34)	0.0004(30)	-0.0176(34)	-0.0242(31)
N(1C1)	0.0558(103)	0.0482(91)	0.0494(98)	-0.0237(76)	-0.0086(79)	0.0111(76)
O(1C1)	0.1175(108)	0.0675(86)	0.0313(74)	-0.0036(66)	-0.0233(70)	0.0152(74)
O(1P1)	0.0631(88)	0.1092(106)	0.0640(94)	-0.0003(80)	-0.0036(77)	0.0183(73)
N(1V1)	0.1337(144)	0.0301(91)	0.0465(98)	0.0049(80)	-0.0080(95)	0.0135(85)
S(1C2)	0.0727(39)	0.0694(38)	0.0818(39)	0.0045(32)	-0.0055(32)	0.0010(28)
N(1C2)	0.0721(115)	0.0563(102)	0.0352(89)	-0.0045(76)	0.0116(84)	0.0123(85)
O(1C2)	0.1209(113)	0.0965(104)	0.0109(70)	0.0049(66)	-0.0056(69)	0.0333(80)
O(1P2)	0.0530(87)	0.1079(105)	0.0684(97)	-0.0202(83)	-0.0253(77)	0.0138(72)
N(1V2)	0.0981(125)	0.0542(102)	0.0289(88)	0.0091(82)	0.0118(88)	0.0147(90)
S(1C3)	0.0672(38)	0.0971(44)	0.0864(42)	0.0106(34)	0.0132(33)	-0.0065(31)
N(1C3)	0.0608(102)	0.0687(108)	0.0444(93)	-0.0235(82)	-0.0026(80)	0.0101(76)
O(1C3)	0.0633(92)	0.1975(156)	0.0560(101)	-0.0061(97)	0.0016(78)	0.0119(93)
O(1P3)	0.1696(138)	0.0877(103)	0.0575(98)	-0.0059(81)	-0.0224(96)	0.0343(94)
N(1V3)	0.0496(110)	0.1847(185)	0.0607(117)	-0.0398(117)	-0.0076(96)	0.0103(113)
S(1C4)	0.0747(44)	0.1350(58)	0.1543(59)	0.0280(51)	-0.0070(44)	-0.0228(37)
O(1C4)	0.1226(130)	0.2113(171)	0.0350(86)	0.0051(97)	-0.0074(90)	0.0600(115)
O(1P4)	0.2275(175)	0.0675(99)	0.0775(108)	0.0380(84)	0.0601(114)	0.0536(104)
N(1C4)	0.1028(133)	0.0911(128)	0.0399(98)	0.0216(95)	0.0225(89)	0.0523(106)
N(1V4)	0.0358(99)	0.1556(162)	0.0460(104)	-0.0164(101)	-0.0168(86)	0.0116(98)

CHAPTER FIVE
N-ACETYL METHIONINE

N-ACETYL METHIONINE

Introduction

N-Acetyl amino acids are useful model compounds for the peptide linkage in the shorter peptides. They have an advantage over simpler compounds like acetamide or N-methyl formamide in that they can participate more fully in the network of hydrogen bonds characteristic of amino acid and peptide structures. Methionine is itself an essential amino acid for human growth. Its main function (apart from as a 'structural' amino acid in proteins) is as a source of methyl groups as in S-adenosyl methionine. Although methionine is found in one of the enkephalin peptides, it is frequently replaced in this role by leucine - this would imply that the sole function of methionine here is to fill a hydrophobic pocket in the receptor. The crystals used in this determination were grown in the course of an attempted preparation of a molecular complex of acetylcholine and the amino acid. (These crystals were prepared by Miss R. Kelly as part of an Honours project on amino-acid:alkaloid complexes.)

Crystallisation

The crystals were grown from an equimolar solution of N-acetyl methionine and acetylcholine chloride in water, to which had been added some acetone as an aid to crystallisation.

Crystal Data

Formula $C_7H_{13}O_3NS$ Mol. Wt. 191.23 F(000) = 204

Space group $P2_1$ Int. Tab. No. 4
Cell dimensions a = 6.002(3) b = 9.2499(13) c = 9.1737(9) A
β = 91.234(20)°

V = 508.71 A^3 Z = 2 Dc = 1.24 gcm^{-3}
Radiation MoKα λ = 0.71069 A μ = 2.77 cm-1
Final R = 0.0465 based on 840 data
h_{max} = 7 k_{max} = 11 l_{max} = 10

θ_{max} = 25° $Sin\theta_{max}$ = 0.4226 $Sin\theta/\lambda$ = 0.5947

Reflections measured 1011 (5 systematic absences)
After merging 947 (R = 0.0326)
Unobserved reflections 107 ($F<4\sigma(F)$)
Intensity standards 1 5 3 2 3 5
0.993 < drift curve < 1.007
115 parameters refined
Max shift/σ for final cycle 0.005
Final difference Fourier max. 0.1967 min. -0.3283 e A^{-3}
Weighting scheme :weight = $10.3571/(\sigma^2(F)+0.00059F^2)$

Data collection and reduction

The data set was collected on the CAD4 and corrected for Lorentz and polarisation effects using the program CADABS. The space group was determined from the systematic absences (reflection conditions, 0k0, k=2n) and the chirality of the compound to be $P2_1$.

Solution and refinement

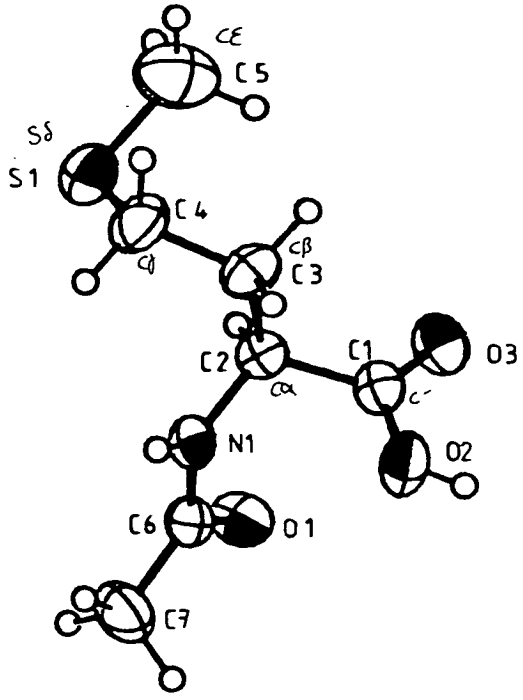
The structure was solved by location of the sulphur sulphur vector in the sharpened Patterson map calculated by the program SHELX-76. The progress of the refinement is shown below.

R	Model
0.4574	One Sulphur atom only
0.1271	All non-Hydrogen atoms included with isotropic thermal parameters
0.1203	Hydrogens input in calculated positions with constrained geometry and thermal parameters
0.0775	All non-Hydrogen atoms with anisotropic thermal parameters
0.0707	O-H and N-H hydrogens input (not constrained)
0.0539	Three reflections with deviation greater than 6σ omitted and weighting scheme applied
0.0536	Converged Absolute stereochemistry corrected
0.0465	Final Convergence

All hydrogens on carbon atoms were constrained to calculated positions, with all their thermal parameters constrained to be equal

Structure Description

The numbering scheme used for this molecule is shown in the diagram below.



As can be seen from the tables of molecular geometry below, all the bond distances, angles and dihedral angles are within the usual ranges for their types.

C(1) - C(2)	1.525(6)
C(1) - O(2)	1.311(6)
C(1) - O(3)	1.202(6)
C(2) - C(3)	1.519(6)
C(2) - N(1)	1.458(5)
C(3) - C(4)	1.532(8)
S(1) - C(4)	1.807(6)
S(1) - C(5)	1.766(8)
C(6) - C(7)	1.510(8)
C(6) - O(1)	1.239(6)
C(6) - N(1)	1.324(6)

C(4)	-	S(1)	-	C(5)	102.5(	3)
C(2)	-	C(1)	-	O(3)	122.2(	4)
O(2)	-	C(1)	-	O(3)	124.4(	4)
C(2)	-	C(1)	-	O(2)	113.2(	4)
C(1)	-	C(2)	-	C(3)	108.8(	3)
C(1)	-	C(2)	-	N(1)	111.5(	3)
C(3)	-	C(2)	-	N(1)	110.9(	3)
C(2)	-	C(3)	-	C(4)	115.1(	4)
S(1)	-	C(4)	-	C(3)	115.3(	4)
O(1)	-	C(6)	-	N(1)	120.0(	4)
C(7)	-	C(6)	-	O(1)	121.6(	4)
C(2)	-	N(1)	-	C(6)	121.0(	4)
C(7)	-	C(6)	-	N(1)	118.4(	4)

C(5)	-	S(1)	-	C(4)	-	C(3)	-63.5(	5)
O(2)	-	C(1)	-	C(2)	-	C(3)	88.7(	4)
O(2)	-	C(1)	-	C(2)	-	N(1)	-33.9(	5)
O(3)	-	C(1)	-	C(2)	-	C(3)	-87.0(	5)
O(3)	-	C(1)	-	C(2)	-	N(1)	150.4(	4)
C(1)	-	C(2)	-	C(3)	-	C(4)	174.9(	4)
N(1)	-	C(2)	-	C(3)	-	C(4)	-62.1(	5)
C(1)	-	C(2)	-	N(1)	-	C(6)	-59.0(	5)
C(3)	-	C(2)	-	N(1)	-	C(6)	179.6(	4)
C(2)	-	C(3)	-	C(4)	-	S(1)	-54.7(	5)
C(7)	-	C(6)	-	N(1)	-	C(2)	175.8(	4)
O(1)	-	C(6)	-	N(1)	-	C(2)	-3.5(	6)

The bond distances and angles involving the positionally constrained hydrogen atoms are not listed, but the table below gives the figures for the unconstrained hydrogens of the amide and carboxylic acid functional groups. Both of the bond distances are shorter than usual, but not significantly so, and the angles are well within the usual ranges.

O(2)	-H(OO2)	0.80(	8)
N(1)	-H(ON1)	0.87(	7)

C(1)	-	O(2)	-H(OO2)	120(	6)
C(2)	-	N(1)	-H(ON1)	123(	5)
C(6)	-	N(1)	-H(ON1)	116(	5)

C(2)	- C(1) - O(2)	-H(OO2)	-179(7)
O(3)	- C(1) - O(2)	-H(OO2)	-4(7)
C(1)	- C(2) - N(1)	-H(ON1)	113(6)
H(O21)	- C(2) - N(1)	-H(ON1)	-128(6)
C(3)	- C(2) - N(1)	-H(ON1)	-8(6)
C(7)	- C(6) - N(1)	-H(ON1)	3(5)
O(1)	- C(6) - N(1)	-H(ON1)	-176(5)

Molecular Packing

The hydrogen bonding scheme for N-acetyl methionine consists of parallel sheets of molecules perpendicular to the c axis. The hydrogen bonded contacts are listed below.

O2	2.560 (5)	O1	(1 -X,1/2+Y,-Z)
O3	2.909 (5)	N1	(1 +X,Y,Z)
HOO2	1.78 (8)	O1	(1 -X,1/2+Y,-Z)
O3	2.04 (7)	HON1	(1 +X,Y,Z)

These hydrogen bonding contacts may be seen clearly on all three unit cell axis projections (shown on the next three pages after this section). On the projection down the a axis they can be seen to form a staggered sheet connecting the carboxyl hydrogens to the amide carbonyls and on the projection down the b axis the amide hydrogens can be seen hydrogen bonding to the carboxyl carbonyls. Between these sheets of hydrophilic atoms lie the hydrophobic regions containing the side chains arranged head to tail about the screw axis. The close contacts to sulphur in this region can be seen in the table below.

C1	3.960 (4)	S1	(1 -X,1/2+Y,-1 -Z)
C3	3.856 (5)	S1	(-X,1/2+Y,-1 -Z)
C4	3.937 (6)	S1	(-X,1/2+Y,-1 -Z)
O3	3.451 (4)	S1	(1 -X,1/2+Y,-1 -Z)
HO31	2.946 (5)	S1	(-X,1/2+Y,-1 -Z)
HO32	3.427 (5)	S1	(1 -X,1/2+Y,-1 -Z)
HO72	3.062 (6)	S1	(X,Y,1 +Z)

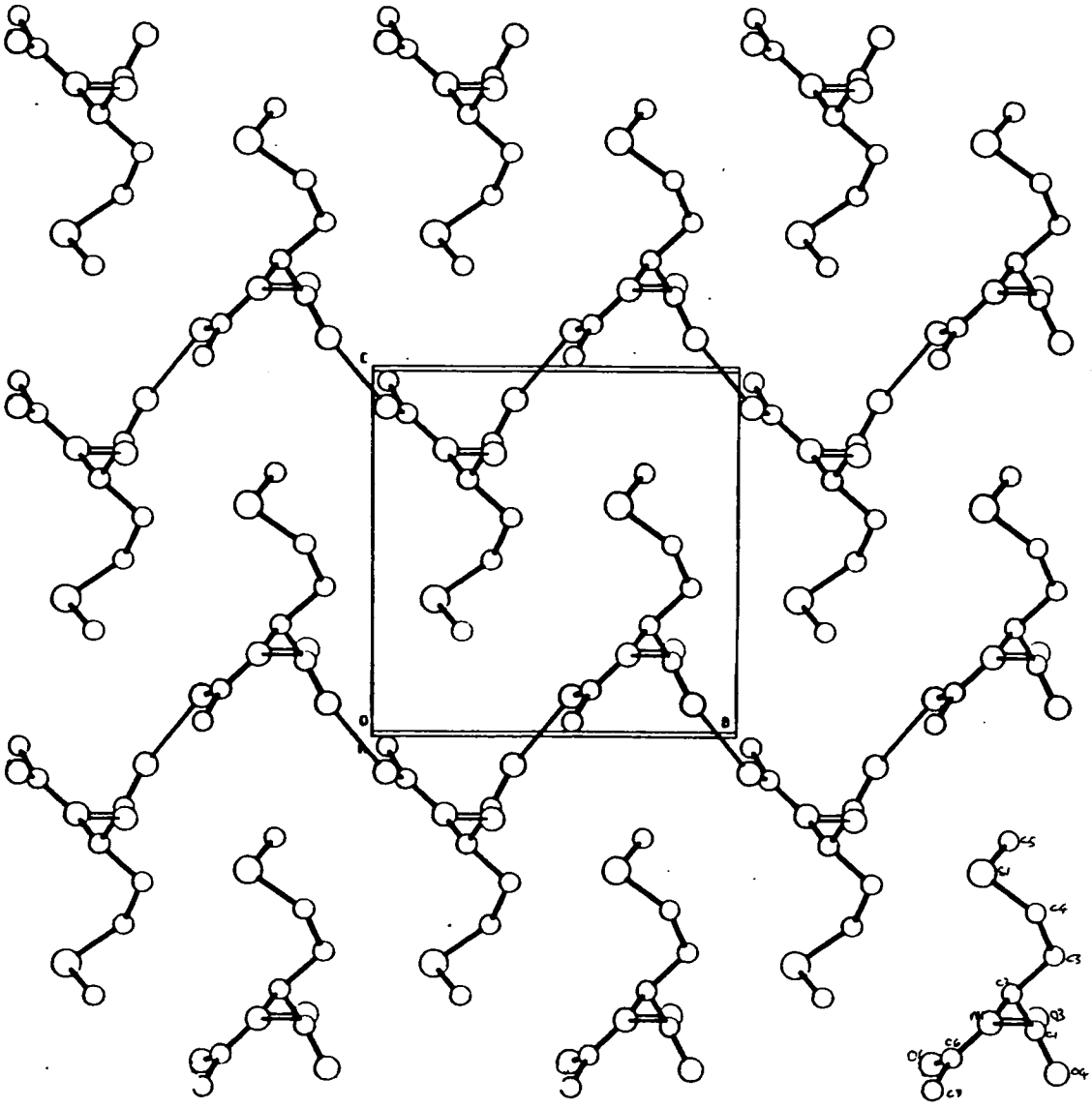
There are eight significant oxygen hydrogen contacts, but these involve

mostly methyl groups with hydrogens in calculated positions so they may not be genuine contacts.

H053	2.639	(9)	O1	(1 -X,1/2+Y,-1 -Z)
H071	2.869	(7)	O1	(-1 +X,Y,Z)
O2	2.846	(9)	H051	(1 -X,1/2+Y,-1 -Z)
O2	2.839	(9)	H053	(X,Y,1 +Z)
O2	2.978	(7)	H071	(-X,1/2+Y,-Z)
O2	2.714	(7)	H073	(-X,1/2+Y,-Z)
O3	2.745	(7)	H041	(1 +X,Y,Z)
O3	2.600	(7)	H071	(1 +X,Y,Z)

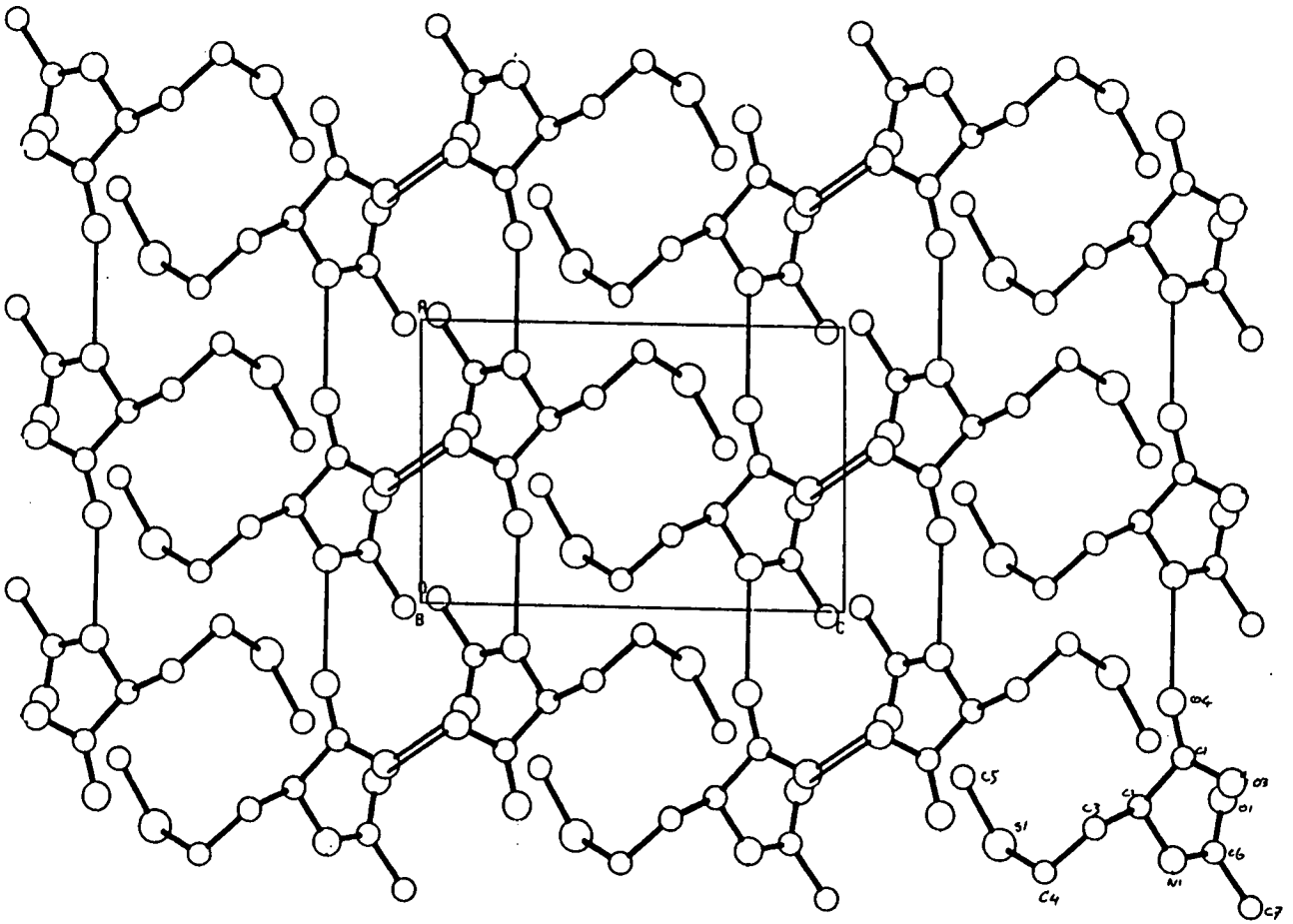
The remaining contact distances involve carbon atoms.

C5	3.416	(9)	O1	(1 -X,1/2+Y,-1 -Z)
O2	3.114	(7)	C7	(-X,1/2+Y,-Z)
N1	3.14	(7)	C1	(-1 +X,Y,Z)
H002	2.80	(8)	C6	(1 -X,1/2+Y,-Z)



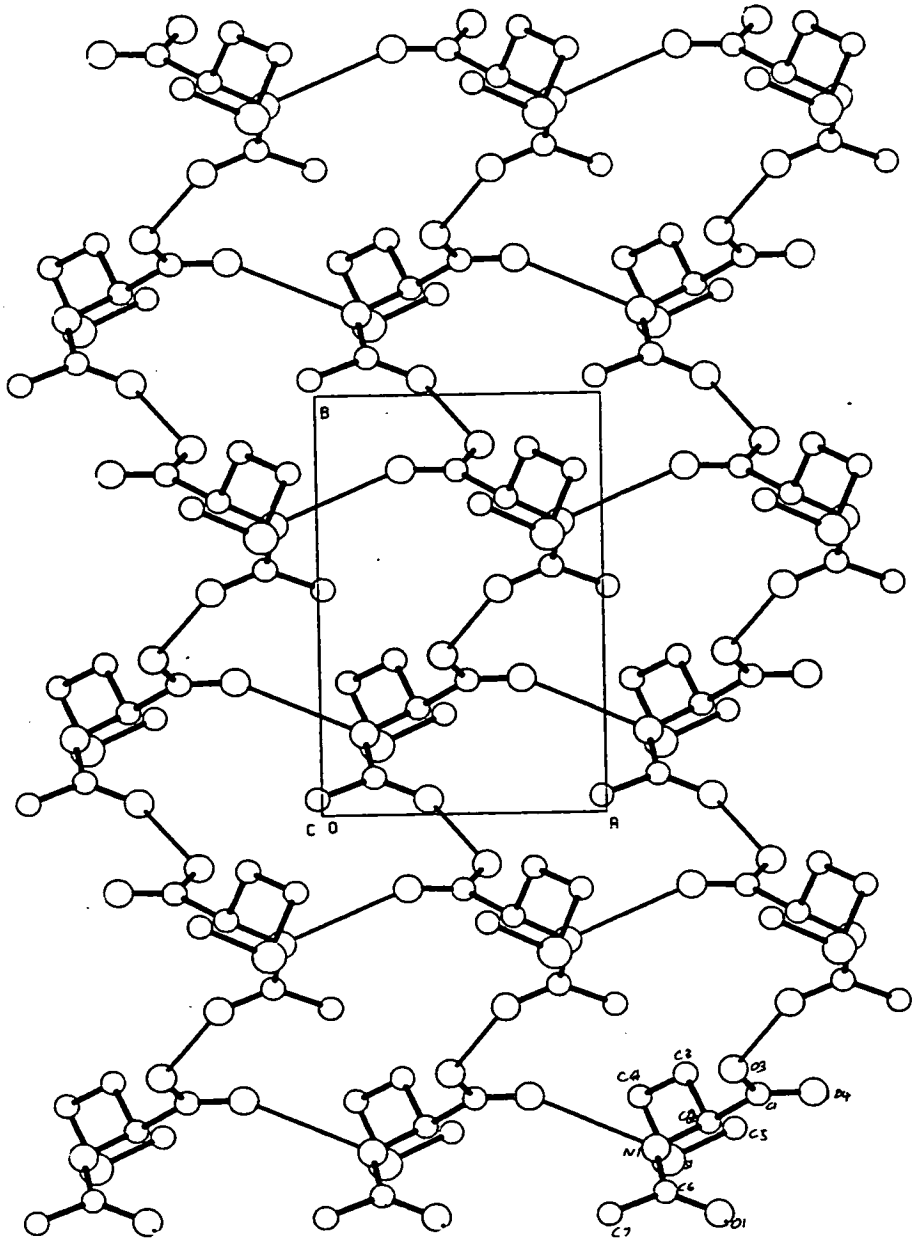
N-ACETYL-L-METHIONINE

PROJECTION DOWN THE XAXIS



PROJECTION DOWN THE YAXIS

N-ACETYL-L-METHIONINE



PROJECTION DOWN THE Z AXIS

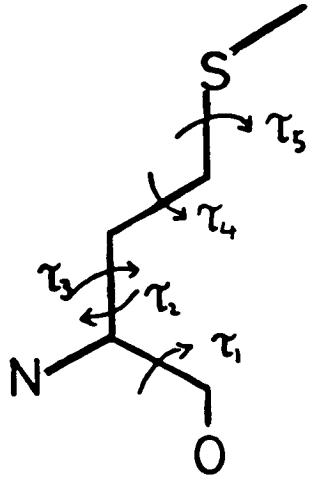
N-ACETYL-L-METHIONINE

Comparison with other structures

The table overleaf lists the torsion angles of twenty independent methionine residues from sixteen structure determinations. Bond lengths and angles are not included in this table as they are all within normal variation of the standard values. There would appear to be three types of side chain found. These may be described as follows:

a) Fully extended ($\tau_4 = \tau_5 \approx 180°$)
b) Fully folded ($\tau_4 = \tau_5 \approx 60°$)
c) An intermediate conformation with the side chain half extended ($\tau_4 \approx 180°, \tau_5 \approx \pm 60°$).

The torsion angles tabulated are marked on the diagram below.



τ_1 is χ(O-C'-Cα-Cβ)
τ_2 is χ(C'-Cα-Cβ-Cγ)
τ_3 is χ(N-Cα-Cβ-Cγ)
τ_4 is χ(Cα-Cβ-Cγ-Sδ)
τ_5 is χ(Cβ-Cγ-Sδ-Cε)

N-acetyl-L-methionine is an example of the second type, while N-acetyl methionine methyl ester is of the first type, but otherwise similar in conformation along the peptide backbone. Both the α and β forms of L-methionine differ in side chain conformation from the N-acetyl methionine, but they are of similar conformation about the C'-Cα and Cα-Cβ bonds to N-acetyl methionine. The side chain of cyclo-L-methionyl glycine trihydrate has the same conformation as that

of N-acetyl methionine.

	τ_1	τ_2	τ_3	τ_4	τ_5
ACMETM	94.20	175.36	-63.63	177.46	-178.61
	-83.41				
ACMTDE	70.16	62.13	-179.53	176.03	-74.52
ALAMET10	119.06	-56.24	67.26	175.00	-173.77
	-59.96				
AMETMA	100.66	169.51	-72.30	173.36	-176.72
BCMEGL	51.94	165.58	-73.51	175.29	174.87
BOMETM	-47.01	-58.02	177.62	-60.33	-66.45
	-19.61	-177.67	-53.38	-177.35	70.67
	163.25				
DLMETA02	-86.35	-178.62	-59.87	177.36	68.95
	89.46				
DLMETA03	-87.14	-174.36	-54.35	-179.32	-174.43
	89.95				
FMLPLA	95.56	-58.61	61.74	172.90	-79.29
FOLMET10	108.56	-67.88	54.37	176.10	173.64
	-70.57				
LMETON10	105.97	71.49	-166.06	174.17	179.71
	-72.97				
	84.25	74.29	-165.62	73.57	73.56
	-90.70				
METHCL	124.77	-60.85	61.30	-178.19	68.74
	-58.56				
METMET	70.70	74.63	-167.24	-168.29	178.39
	114.94	-57.65	68.08	-170.50	-57.65
	-61.74				
METTSO10	-74.95	-46.56	77.00	-164.11	-65.90
	102.22				

	τ_1	τ_2	τ_3	τ_4	τ_5
SMMETC10	-37.07	82.50	-157.15	169.60	-177.31
	141.74				-72.25
GASTRN10	101.89	174.97	-67.39	-164.48	-165.63
	92.13	179.21	-63.84	178.09	81.06
BIVMUO	-56.36	-69.19	58.18	71.40	52.26
MGHPHE20	61.36	71.72	-170.20	176.11	120.15

Key to compounds in the above table

ACMETM N-acetyl-methionine methyl ester

A. J. Geddes, S. Hamodrakas, B. Sheldrick, Cryst. Struct. Commun., **3**, 97, 1974

ACMTDE N-acetyl-D,L-methionine-diethylamide

A. Aubry, J. Protas, M. T. Cung, M. Marraud, Acta Cryst., **B35**, 2634, 1979

ALAMET10 D,L-alanyl-L,D-methionine

R. E. Stenkamp, L. H. Jensen, Acta Cryst., **B30**, 1541, 1974

AMETMA N-acetyl-methionyl-dimethylamide

A. Aubry, M. Marraud, J. Protas, J. Neel, C. R. Acad. Sci., Ser. C, **273**, 959, 1971

BCMEGL N-(T-butoxy-carbonyl)-L-methionyl-glycine-benzyl ester

T. Yamane, T. Umemura, T. Kojima, Y. Yamada, T. Ashida, Bull. Chem. Soc. Jpn., **53**, 908, 1980

BOMETM T-butoxycarbonyl-D-methionyl-L-methionine methyl ester

A. Immirzi, P. Avena, M. R. Ciajolo, J. M. Becker, F. Naider, Acta Cryst., **B34**, 179, 1978

DLMETA02 D,L-methionine (α-form, at 333° K)

T. Taniguchi, Y. Takaki, K. Sakurai, Bull. Chem. Soc. Jpn., **53**, 803, 1980

DLMETA03 D,L-methionine (β-form, at 293° K)

T. Taniguchi, Y. Takaki, K. Sakurai, Bull. Chem. Soc. Jpn., **53**, 803, 1980

FMLPLA N-formyl-methionyl-leucyl-phenylalanine

A. J. Morffew, I. Tickle, Cryst. Struct. Commun., **10**, 781, 1981

FOLMET10 N-formyl-L-methionine (Absolute configuration)

C. Chen, R. Parthasarathy, Acta Cryst., **B33**, 3332, 1977

LMETON10 L-methionine

K. Torii, Y. Iitaka, Acta Cryst., **B29**, 2799, 1973

METHCL L-methionine hydrochloride

B. Di Blasio, V. Pavone, C. Pedone, Cryst. Struct. Commun., **6**, 845, 1977

METMET L-methionyl-L-methionine

R. E. Stenkamp, L. H. Jensen, Acta Cryst., **B31**, 857, 1975

METTSO10 (2S,SR)-methionine sulphoximine (Absolute configuration)

S. Neidle, D. Rogers, J. Chem. Soc. B., 694, 1970

SMMETC10 S-methyl-L-methionine chloride hydrochloride (Vitamin U hydrochloride)

G. Del Re, E. Gavuzzo, E. Giglio, F. Lelj, F. Mazza, V. Zappia, Acta Cryst., **B33**, 3289, 1977

GASTRN10 L-tryptophanyl-L-methionyl-L-aspartyl -L-phenylalanylamide hydrochloride methanol diethyl ether solvate

W. B. T. Cruse, E. Egert, M. A. Viswamitra, O. Kennard, Acta Cryst., **B38**, 1758, 1982

BIVMUO cyclo-L-methionyl-glycine trihydrate

M. Bressan, R. Ettore, F. Marchiori, G. Valle, Int. J. Pept. Protein Res., **19**, 402, 1982

MGHPHE20 L-methionyl-L-α-glutamyl-L-histidyl -L-phenylalanine monohydrate

G. Admiraal, A. Vos, Acta Cryst., **C39**, 82, 1983

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
S(1)	0.1933(3)	-0.83453(0)	-0.63280(14)	0.0780
C(1)	0.5128(6)	-0.6803(5)	-0.1997(4)	0.0418
C(2)	0.3375(6)	-0.7462(5)	-0.3021(4)	0.0379
C(3)	0.2590(8)	-0.6296(6)	-0.4080(5)	0.0490
C(4)	0.0967(8)	-0.6824(8)	-0.5269(5)	0.0644
C(5)	0.4286(14)	-0.7610(10)	-0.7182(7)	0.0970
C(6)	0.1813(6)	-0.9104(5)	-0.1259(4)	0.0451
C(7)	-0.0165(8)	-0.9605(9)	-0.0407(6)	0.0737
O(1)	0.3694(5)	-0.9632(4)	-0.1054(4)	0.0572
O(2)	0.4276(5)	-0.6204(4)	-0.0846(4)	0.0582
O(3)	0.7074(4)	-0.6770(5)	-0.2282(4)	0.0589
N(1)	0.1504(5)	-0.8062(5)	-0.2234(4)	0.0412
H(021)	0.4111	-0.8355	-0.3602	0.0932
H(031)	0.1772	-0.5450	-0.3476	0.0932
H(032)	0.4039	-0.5848	-0.4593	0.0932
H(041)	-0.0564	-0.7146	-0.4764	0.0932
H(042)	0.0639	-0.5924	-0.5996	0.0932
H(051)	0.5023	-0.8431	-0.7858	0.0932
H(052)	0.5493	-0.7259	-0.6371	0.0932
H(053)	0.3781	-0.6688	-0.7839	0.0932
H(071)	-0.1631	-0.9007	-0.0755	0.0932
H(072)	0.0155	-0.9411	0.0730	0.0932
H(073)	-0.0433	-1.0756	-0.0586	0.0932
H(002)	0.5081	-0.5823	-0.0260	0.0932
H(0N1)	0.0161	-0.7692	-0.2291	0.0932

Thermal Vibration Parameters with Standard Deviations

	U11	U22	U33	U23	U13	U12
S(1)	0.1011(12)	0.0770(10)	0.0557(8)	-0.0102(7)	-0.0059(7)	-0.0254(8)
C(1)	0.0354(19)	0.0392(20)	0.0507(24)	0.0040(20)	-0.0052(17)	0.0049(18)
C(2)	0.0336(18)	0.0401(21)	0.0399(21)	-0.0006(17)	0.0008(16)	0.0074(16)
C(3)	0.0552(23)	0.0468(24)	0.0447(22)	-0.0009(20)	-0.0063(19)	0.0099(20)
C(4)	0.0597(26)	0.0824(35)	0.0507(27)	0.0022(29)	-0.0168(22)	0.0033(29)
C(5)	0.1199(53)	0.0906(51)	0.0811(44)	0.0013(40)	0.0290(40)	0.0043(46)
C(6)	0.0395(22)	0.0509(26)	0.0448(25)	0.0021(20)	-0.0019(18)	-0.0021(18)
C(7)	0.0467(24)	0.0957(46)	0.0785(39)	0.0254(35)	-0.0021(25)	-0.0140(30)
O(1)	0.0464(17)	0.0627(22)	0.0624(21)	0.0185(18)	0.0016(15)	0.0103(15)
O(2)	0.0385(15)	0.0751(24)	0.0611(20)	-0.0279(19)	0.0003(14)	-0.0033(16)
O(3)	0.0271(13)	0.0782(23)	0.0713(21)	-0.0001(19)	0.0045(12)	0.0046(15)
N(1)	0.0294(15)	0.0453(21)	0.0488(18)	0.0013(16)	-0.0036(14)	0.0042(15)

CHAPTER SIX
DL-GLUTAMIC ACID MONOHYDRATE

DL GLUTAMIC ACID MONOHYDRATE

Introduction

Crystals of this compound were grown from a mixture of glutamic acid and brucine in aqueous ethanol as part of an attempted preparation of brucine glutamate crystals. Although the crystals formed were obviously not those of an amino-acid:alkaloid complex, it was decided that the X-ray structure would be of value as it could provide more information on the preferred environment and conformation of the glutamate residue as it might appear in biologically important molecules. Glutamic acid plays an important role in the biosynthesis of amino acids, by acting as a source of nitrogen for transamination. It is also likely that it acts as a transmitter in the central nervous system, so a structural study on glutamic acid could possibly provide more information on which to base the rational design of inhibitors or stimulants for this system.

It should also be noted here that the crystal structure of this compound was determined previously by Ciunik and Glowiak and published by them after the refinement process here was completed (Ciunik & Glowiak, 1983).

Crystallisation

Equimolar quantities of DL-glutamic acid and brucine tetrahydrate were dissolved in the minimum of hot aqueous ethanol (50% v/v), and allowed to cool slowly to room temperature. The crystals were cuboidal in shape, rather like flattened needles.

Crystal Data

Formula $C_5H_9NO_4.H_2O$ Mol. Wt. 165.2 F(000) = 704
Space Group Pbca Int. Tab. No. 61
Cell Dimensions a = 9.1469(15) b = 15.5228(13) c = 10.6407(10)
V = 1510.8 A^3 Z = 8 Dc = 1.4524 gcm^{-3}
Radiation MoKα λ = 0.71069 A μ = 1.22cm^{-1}
Final R = 0.0376 based on 1154 data
h_{max} = 11 k_{max} = 19 l_{max} = 13

θ_{max} = 25° $Sin\theta_{max}$ = 0.4226 $Sin\theta_{max}/\lambda$ = 0.5947

1979 reflections measured (283 systematic absences)
1641 data remaining after merging (R = 0.0166)
487 data considered unobserved ($F<4\sigma(F)$)
Crystal dimensions 0.8 * 0.08 * 0.2 mm^3
Intensity standards * 6 1 3 and 1 4 7
0.976 < drift curve < 1.019
135 parameters refined
Max. shift/σ for final cycle = 0.015
Final difference Fourier map max. 0.346 eA^{-3} min. -0.248 eA^{-3}

Data collection and reduction

The data was collected on the CAD4 and the program CADABS used to correct for Lorentz and polarisation effects.

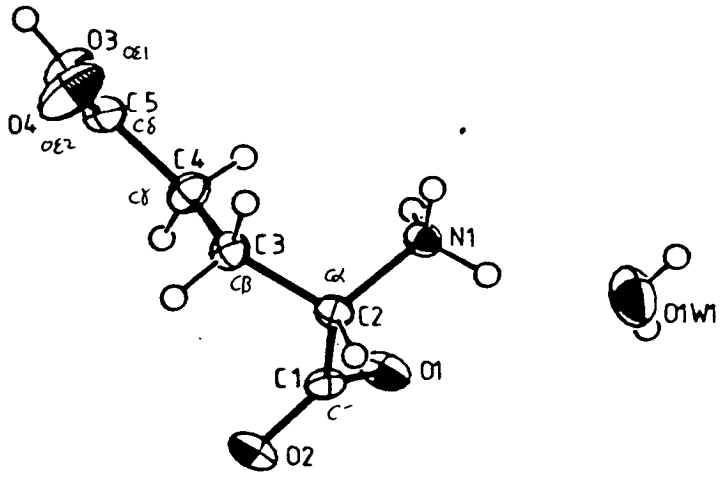
Solution and refinement

The structure was solved automatically, using the centrosymmetric structure solution routine of SHELX-76. The first difference map output showed all the non-hydrogen atoms, and the subsequent progress of refinement is described in the table below.

R	Model
0.4384	All non Hydrogen atoms input with isotropic thermal parameter of 0.05
0.2475	One cycle of least squares refinement.
0.1184	Hydrogens on carbon and nitrogen input in calculated positions with thermal parameter fixed at 0.05
0.1031	Thermal parameters for hydrogens on carbon and nitrogen tied to a free variable
0.0559	All non hydrogen atoms now with anisotropic thermal parameters
0.0474	Hydrogen atoms on oxygens found and given temperature factors fixed at 0.05
0.0495	Oxygen hydrogens thermal parameters tied to a free variable, and reflections with uneven backgrounds omitted
0.0376	Final Convergence

No positional constraints were applied to the hydrogen atoms, but the thermal parameters of the hydrogens bonded to carbon and nitrogen were constrained to be equal

Description of structure



The diagram above shows a perspective plot of the molecule, together with its numbering scheme as used in the tables below. All bond distances and angles are within the usual ranges, although one bond (C(4)-C(5)) is significantly shorter than the average carbon carbon bond length in this structure (1.496(3) angstroms vs. 1.5185 angstroms, a difference of 7.5σ). There is a significant difference between the lengths of the two carbon oxygen bonds in the carboxylate group (5.6σ), which may be due to one oxygen taking part in two hydrogen bonds, and the other only in one.

C(1)-O(1)	1.2421(25)	O(1)--N(1)	2.763(3)
C(1)-O(2)	1.2694(24)	O(2)--N(1)	2.827(3)
		O(2)--O(3)	2.572(3)

With the exception of one bond in the ammonium group, and the two on the water molecule, all bonds to hydrogen are within the usual range. The overall geometry of the molecule can be described in terms of two planes which intersect at Cα (C(2)) with an angle of almost 90° (89.4°). The first of these planes is defined by the carboxylate group and the nitrogen atom, with a root mean square deviation from planarity of 0.014 angstroms, and the second, less well defined plane is that of the carboxyl group and the rest of the carbon skeleton, with a deviation of 0.087 angstroms from planarity.

C(1) - C(2)	1.526(3)
C(1) - O(1)	1.2422(24)
C(1) - O(2)	1.2679(24)
C(2) - C(3)	1.527(3)
C(2) - N(1)	1.4968(25)
C(3) - C(4)	1.526(3)
C(4) - C(5)	1.498(3)
C(5) - O(3)	1.327(3)
C(5) - O(4)	1.209(3)

C(2) - C(1) - O(1)	118.49(17)
C(2) - C(1) - O(2)	115.65(17)
O(1) - C(1) - O(2)	125.86(18)
C(1) - C(2) - C(3)	113.52(16)
C(1) - C(2) - N(1)	108.59(15)
C(3) - C(2) - N(1)	110.69(15)
C(2) - C(3) - C(4)	113.78(17)
C(3) - C(4) - C(5)	113.28(17)
C(4) - C(5) - O(3)	112.77(18)
C(4) - C(5) - O(4)	125.24(20)
O(3) - C(5) - O(4)	121.99(20)

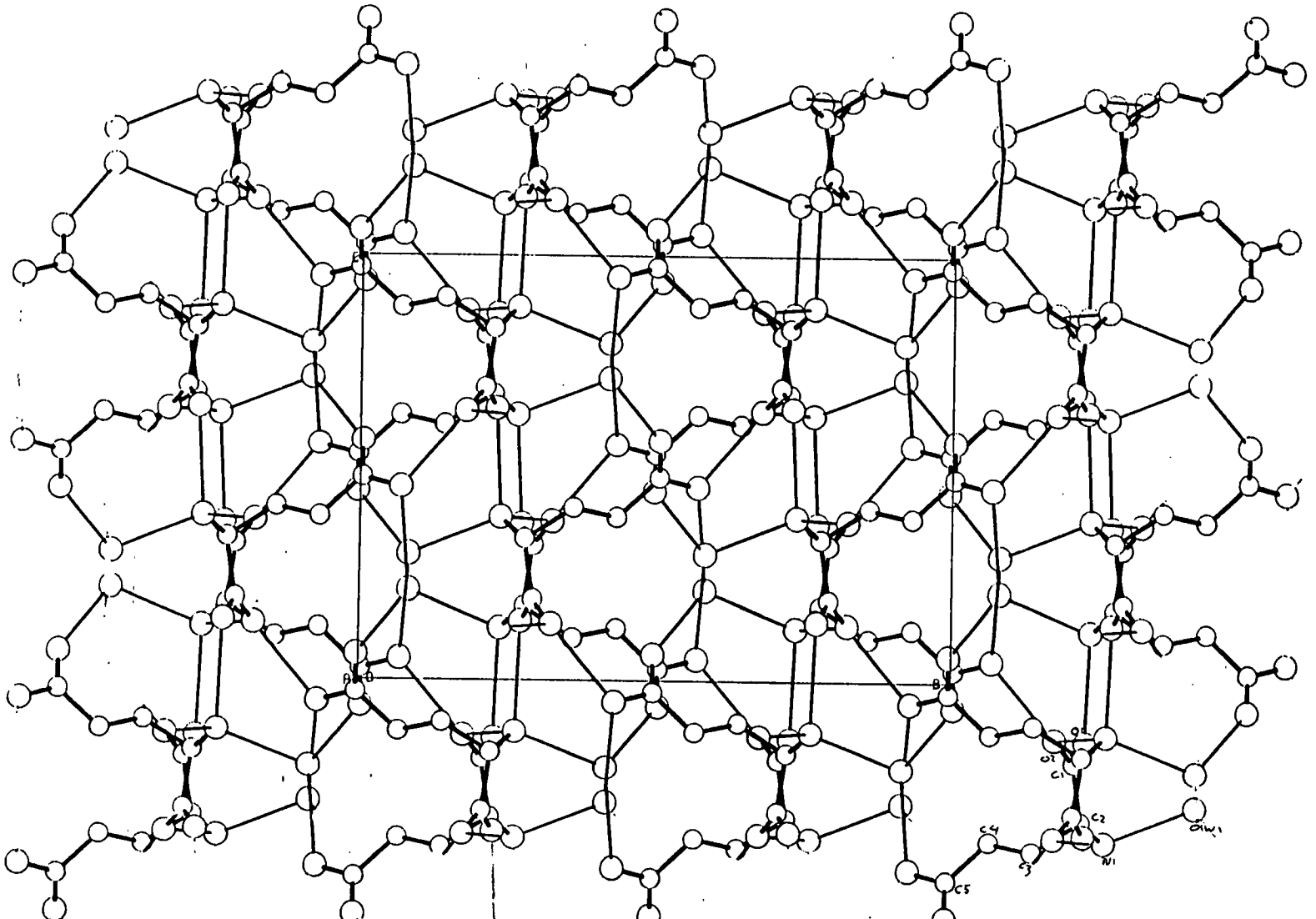
O(1) - C(1) - C(2) - C(3)	121.30(19)
O(1) - C(1) - C(2) - N(1)	-2.26(24)
O(2) - C(1) - C(2) - C(3)	-58.69(23)
O(2) - C(1) - C(2) - N(1)	177.74(16)
C(1) - C(2) - C(3) - C(4)	-53.97(22)
N(1) - C(2) - C(3) - C(4)	68.44(21)
C(2) - C(3) - C(4) - C(5)	-165.59(17)
C(3) - C(4) - C(5) - O(3)	-177.43(17)
C(3) - C(4) - C(5) - O(4)	2.6(3)

C(2) -H(21)	0.979(23)
C(3) -H(31)	0.949(24)
C(3) -H(32)	0.967(24)
C(4) -H(41)	0.938(24)
C(4) -H(42)	0.949(24)
O(3) -H(103)	0.879(24)
N(1) -H(1N1)	0.840(25)
N(1) -H(2N1)	0.944(24)
N(1) -H(3N1)	0.956(24)
O(1W1)-H(10W)	0.83(4)
O(1W1)-H(20W)	0.78(4)

C(1) - C(2) -H(21)	106.2(14)
C(3) - C(2) -H(21)	111.3(14)
N(1) - C(2) -H(21)	106.2(14)
C(2) - C(3) -H(31)	109.3(14)
C(2) - C(3) -H(32)	106.3(14)
C(4) - C(3) -H(31)	106.9(14)
C(4) - C(3) -H(32)	111.2(14)
H(31) - C(3) -H(32)	109.3(20)
C(3) - C(4) -H(41)	110.6(14)
C(3) - C(4) -H(42)	112.5(15)
C(5) - C(4) -H(41)	109.1(14)
C(5) - C(4) -H(42)	106.0(15)
H(41) - C(4) -H(42)	104.9(21)
C(5) - O(3) -H(103)	111.6(16)
C(2) - N(1) -H(1N1)	108.0(17)
C(2) - N(1) -H(2N1)	112.1(15)
C(2) - N(1) -H(3N1)	112.7(14)
H(1N1)- N(1) -H(2N1)	106.3(22)
H(1N1)- N(1) -H(3N1)	110.3(22)
H(2N1)- N(1) -H(3N1)	107.3(21)
H(10W)-O(1W1)-H(20W)	113 (4)

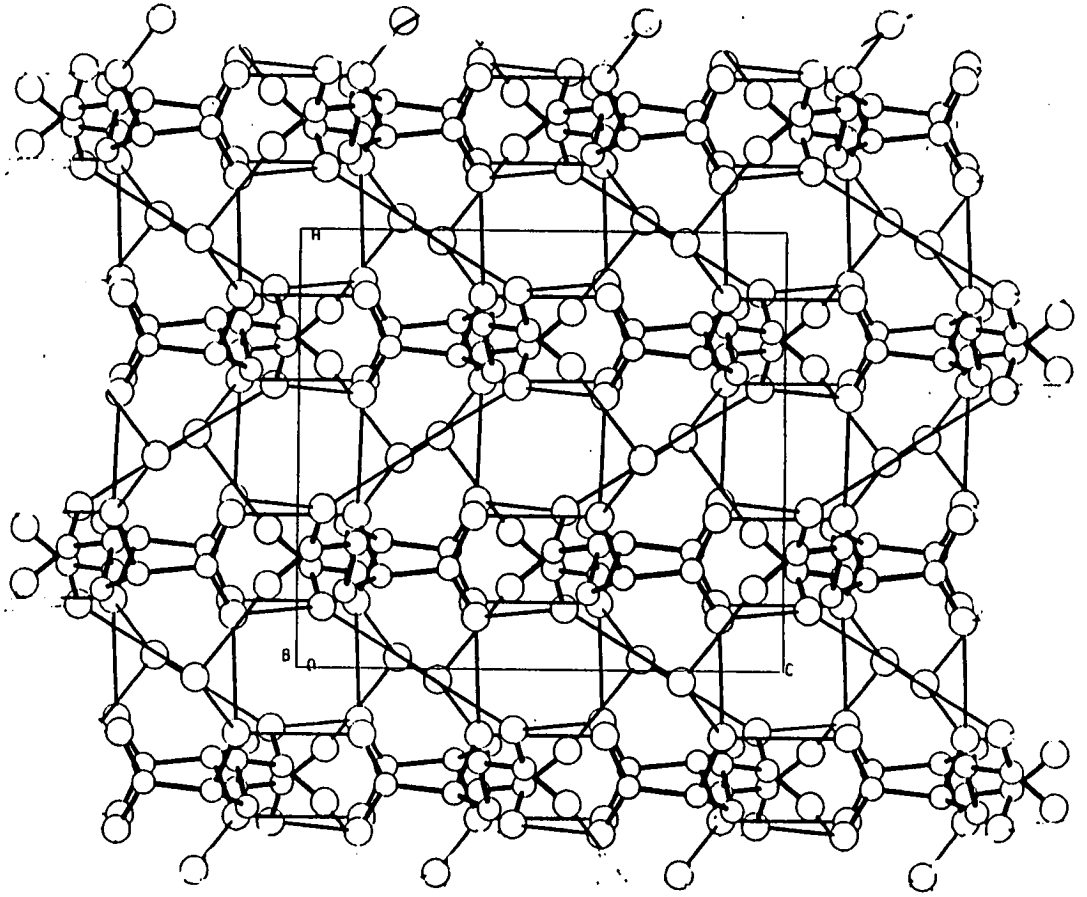
```
O(1) - C(1) - C(2) -H(21) -116.1(14)
O(2) - C(1) - C(2) -H(21)   63.9(14)
C(1) - C(2) - C(3) -H(31)   65.5(15)
C(1) - C(2) - C(3) -H(32) -176.7(15)
N(1) - C(2) - C(3) -H(31) -172.1(15)
N(1) - C(2) - C(3) -H(32)  -54.3(15)
H(21) - C(2) - C(3) - C(4) -173.7(15)
H(21) - C(2) - C(3) -H(31)  -54.3(21)
H(21) - C(2) - C(3) -H(32)   63.6(21)
C(1) - C(2) - N(1) -H(1N1)  56.1(18)
C(1) - C(2) - N(1) -H(2N1) -60.6(16)
C(1) - C(2) - N(1) -H(3N1) 178.2(16)
C(3) - C(2) - N(1) -H(1N1) -69.1(18)
C(3) - C(2) - N(1) -H(2N1) 174.2(16)
C(3) - C(2) - N(1) -H(3N1)  53.0(16)
H(21) - C(2) - N(1) -H(1N1) 169.9(23)
H(21) - C(2) - N(1) -H(2N1)  53.2(21)
H(21) - C(2) - N(1) -H(3N1) -68.0(21)
C(2) - C(3) - C(4) -H(41)   71.6(16)
C(2) - C(3) - C(4) -H(42)  -45.4(16)
H(31) - C(3) - C(4) - C(5)   73.7(15)
H(31) - C(3) - C(4) -H(41)  -49.2(22)
H(31) - C(3) - C(4) -H(42) -166.1(22)
H(32) - C(3) - C(4) - C(5)  -45.6(16)
H(32) - C(3) - C(4) -H(41) -168.5(22)
H(32) - C(3) - C(4) -H(42)   74.6(22)
H(41) - C(4) - C(5) - O(3)  -53.8(15)
H(41) - C(4) - C(5) - O(4)  126.2(15)
H(42) - C(4) - C(5) - O(3)   58.7(15)
H(42) - C(4) - C(5) - O(4) -121.2(15)
C(4) - C(5) - O(3) -H(103) -174.0(17)
O(4) - C(5) - O(3) -H(103)   5.9(17)
```

As is easily visible in the three unit cell projections overleaf, the main packing force in this structure is hydrogen bonding. Each molecule is a donor for four hydrogen bonds and an acceptor for five, with the water molecule accepting one hydrogen bond and donating protons for two (see table of distances below).



PROJECTION DOWN THE XAXIS

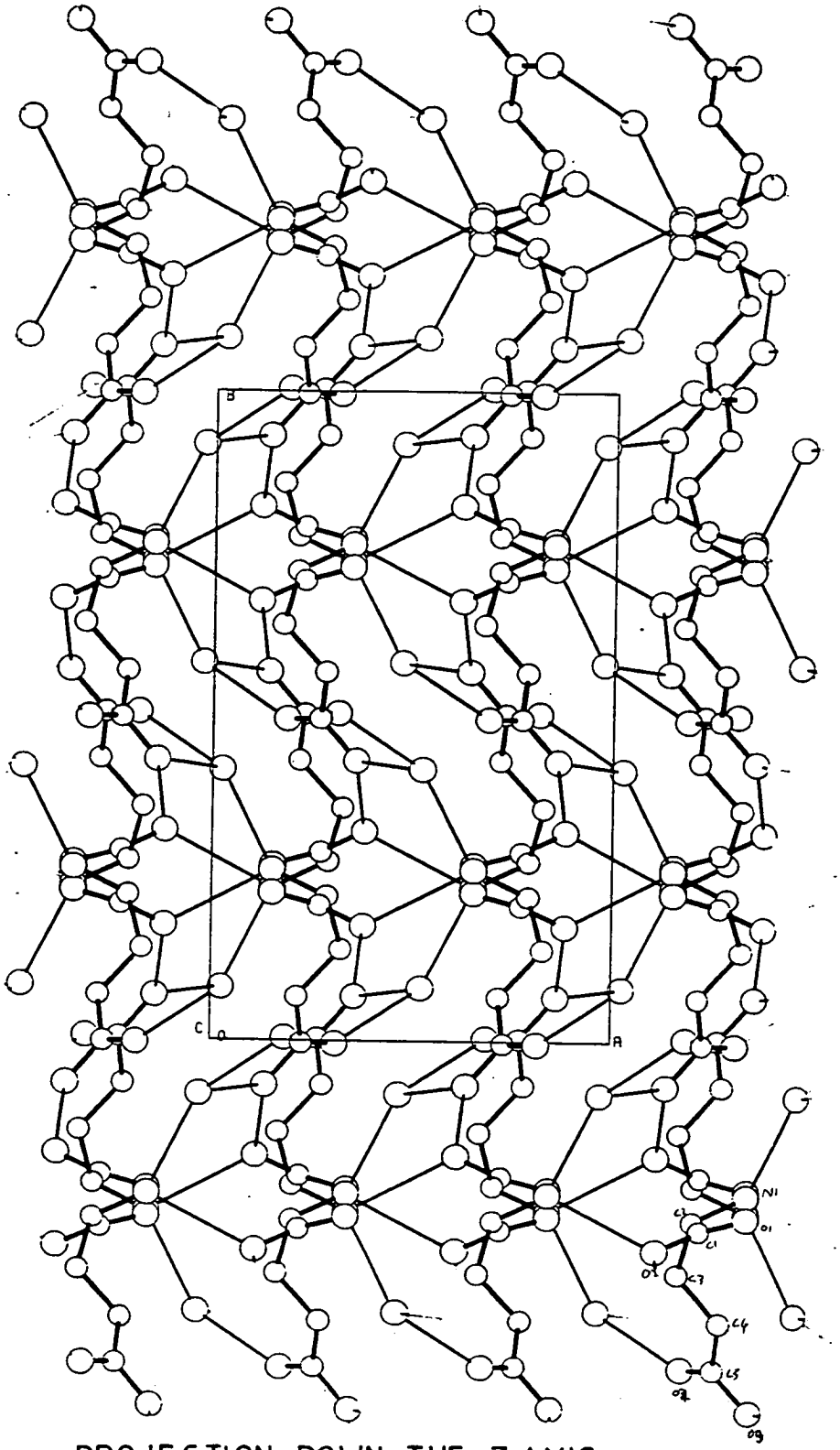
DL-GLUTAMIC ACID MONOHYDRATE



PROJECTION DOWN THE

Y AXIS

DL-GLUTAMIC ACID MONOHYDRATE



PROJECTION DOWN THE Z AXIS

DL-GLUTAMIC ACID MONOHYDRATE

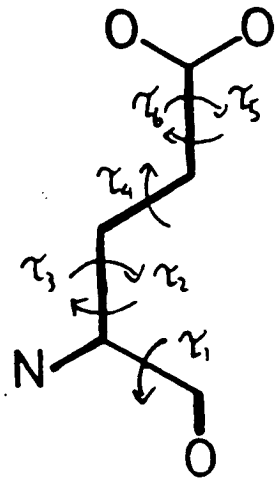
O1	2.764 (2)	N1	(X,1/2-Y,1/2+Z)
O2	2.574 (2)	O3	(1/2-X,1 -Y,1/2+Z)
O2	2.827 (2)	N1	(1/2+X,Y,1/2-Z)
O3	2.980 (3)	O1W1	(-X,1/2+Y,1/2-Z)
O4	2.833 (3)	O1W1	(1/2+X,1/2-Y,-Z)
O1W1	2.817 (3)	N1	(X,Y,Z)
O1	1.817 (24)	H3N1	(X,1/2-Y,1/2+Z)
O2	2.027 (25)	H1N1	(1/2+X,Y,1/2-Z)
O2	1.703 (24)	H1O3	(1/2-X,1 -Y,1/2+Z)
O3	2.17 (4)	H1OW	(-X,1/2+Y,1/2-Z)
O4	2.08 (4)	H2OW	(1/2+X,1/2-Y,-Z)
O1W1	1.918 (24)	H2N1	(X,Y,Z)

The table below shows the non-hydrogen bonding contacts.

O1	3.347 (2)	C2	(-1/2+X,Y,1/2-Z)
O1	3.315 (3)	C3	(-1/2+X,Y,1/2-Z)
O2	3.336 (3)	C5	(1/2-X,1 -Y,1/2+Z)
O2	3.300 (2)	O4	(1/2-X,1 -Y,1/2+Z)
O3	3.437 (2)	C1	(1/2-X,1 -Y,-1/2+Z)
O3	3.309 (2)	N1	(1/2-X,1/2+Y,Z)
O3	3.497 (3)	O1W1	(1/2-X,1/2+Y,Z)
O4	3.440 (3)	O1W1	(1/2-X,1/2+Y,Z)
O1W1	3.436 (3)	C4	(1/2-X,-1/2+Y,Z)
O1W1	3.188 (3)	C5	(1/2-X,-1/2+Y,Z)
N1	3.490 (3)	C1	(X,1/2-Y,-1/2+Z)
O1	2.697 (23)	H21	(-1/2+X,Y,1/2-Z)
O1	2.723 (24)	H31	(-1/2+X,Y,1/2-Z)
O1	2.872 (24)	H32	(X,1/2-Y,1/2+Z)
O1	2.984 (24)	H1O3	(1/2-X,1 -Y,1/2+Z)
O2	2.590 (24)	H42	(1/2+X,Y,1/2-Z)
O2	2.812 (24)	H3N1	(X,1/2-Y,1/2+Z)
O3	2.825 (23)	H21	(1/2-X,1/2+Y,Z)
O3	2.979 (24)	H42	(-X,1 -Y,-Z)
O3	2.961 (24)	H2N1	(1/2-X,1/2+Y,Z)
O4	2.621 (24)	H41	(1/2-X,1 -Y,-1/2+Z)
O1W1	2.996 (24)	H41	(1/2-X,-1/2+Y,Z)
C1	2.907 (25)	H1N1	(1/2+X,Y,1/2-Z)
C1	2.587 (24)	H3N1	(X,1/2-Y,1/2+Z)
C1	2.586 (24)	H1O3	(1/2-X,1 -Y,1/2+Z)
H2N1	2. 44 (5)	H1OW	(X,Y,Z)
H2OW	2.40 (5)	H2N1	(X,Y,Z)

Comparison with other structures

There have been crystal structure determinations carried out for several glutamic acid derivatives. The table below contains information on the conformations of 24 independent glutamate residues from twenty crystal structure determinations. As the bond lengths and angles were all within the usual ranges, only the torsion angles were tabulated.



τ_1 is χ(O-C'-Cα-Cβ)
τ_2 is χ(C'-Cα-Cβ-Cγ)
τ_3 is χ(N-Cα-Cβ-Cγ)
τ_4 is χ(Cα-Cβ-Cγ-Cδ)
τ_5 is χ(Cβ-Cγ-Cδ-Oε1)
τ_6 is χ(Cβ-Cγ-Cδ-Oε2)

The diagram above shows a glutamate residue with the appropriate torsion angles indicated. It is not really possible, as it is for the cystine amino acids, to divide the structures into two classes on the basis of their side chain conformation. This implies that several possible conformations of comparable energy may exist and that the actual conformation in any given crystal structure depends on the molecular environment. The only molecules of similar conformation to DL-glutamic acid in the table below are leucyl glutamic acid, glutamyl glycine and the N-terminal residue of glutamyl glutamic acid. Neither the α nor the β modifications of L-glutamic acid are of similar conformation to the DL acid, which is as expected because in the case of the racemic acid, each molecule is surrounded by molecules of the opposite hand to those in the optically active compound.

CODE	τ_1	τ_2	τ_3	τ_4	τ_5	τ_6
BOFZOL	106.31	-63.29	59.05	171.15	-139.23	37.55
	-70.80					
MGHPHE20	-80.47	168.17	-65.11	-56.54	-30.71	152.62
AMBZGU10	147.41	-176.53	-54.22	74.29	40.94	-142.10
	-33.09					
ANCBGL	121.55	-174.86	-61.70	175.92	12.85	-166.87
	-56.70					
ARGGLU10	-70.99	-179.34	-56.83	-172.05	-2.58	-179.91
	107.59					
BALGLH	-78.91	179.38	59.25	62.26	-155.67	25.14
	99.49					
BCYTGA	88.52	-176.80	-51.88	-164.04	176.18	-3.16
	-92.95					
	76.39	179.48	-55.63	-174.10	-174.18	7.45
	-103.89					
BELCUQ	93.70	-58.14	60.31	-174.55	174.20	-4.20
	88.19	179.35	-57.83	-131.06	36.02	-145.50
	-91.53					
BRCPDG	135.03	90.60	-144.72	68.91	-162.45	16.88
	-44.65					
	-12.27	173.60	-61.31	-65.44	154.30	-25.39
	172.53					
BZGEGA	-9.47	170.52	-65.56	171.89	-171.81	4.45
	158.65					
	60.24	56.48	-178.80	-167.66	6.31	-171.21
CAGLCL10	89.58	67.33	-173.03	170.16	12.28	-170.84
	-89.43					
CAGLUT10	101.86	58.15	179.40	174.72	12:30	-168.94
	-76.18					

CTBGLU	76.53	-162.11	77.07	81.33	-6.89	177.65
	-106.91					
CTSGLM	-143.94	-179.15	52.82	-176.18	-172.25	10.43
	39.41					
DLGLAC	77.01	-169.08	69.59	171.78	-17.65	164.12
	-103.37					
GLUGLY	108.45	-47.76	72.10	-166.70	24.33	-157.62
LGLUAC03	68.90	59.22	178.19	68.30	74.21	-104.65
	-110.56					
LGLUAC11	78.72	-171.06	-51.79	-73.10	18.80	-160.70
	-97.99					
LGLUCA	105.58	-179.57	-56.00	-160.72	15.64	-166.40
	-72.59					
LGLUTA	-76.31	171.06	-68.91	-173.13	14.95	-167.00
	101.30					
LGPYRG	-91.01	-175.03	-55.90	-65.20	-173.29	3.79
	86.05					

Key to compounds in the above table

BOFZOL α-L-leucyl-L-glutamic acid

D. S. Eggleston, D. J. Hodgson, Acta Cryst., C39, 75, 1983

MGHPHE20 L-methionyl-L-α-glutamyl-L-histidyl-L-phenylalanine monohydrate

G. Admiraal, A. Vos, Acta Cryst., C39, 82, 1983

AMBZGU10 N-(p-aminobenzoyl)-L-glutamic acid hydrochloride

C. Chatterjee, J. K. Dattagupta, N. N. Saha, Acta Cryst., B38, 2086, 1982

ANCBGL N-carboxy-η-benzyl-L-glutamate anhydride

H. Kanazawa, T. Kawai, Y. Ohashi, Y. Sasada, Bull. Chem. Soc. Jpn., **51**, 2200, 1978

ARGGLU10 L-arginine L-glutamate monohydrate

T. N. Bhat, M. Vijayan, Acta Cryst., **B33**, 1754, 1977

BALGLH T-butoxycarbonyl-D-alanyl-D-glutamic acid monohydrate

O. Dideberg, J. Lamotte, L. Dupont, L. Christiaens, Acta Cryst., **B37**, 1150, 1981

BCYTGA 5-bromocytosine N-tosyl-L-glutamic acid

M. Ohki, A. Takenaka, H. Shimanouchi, Y. Sasada, Bull. Chem. Soc. Jpn., **49**, 3493, 1976

BELCUQ α-L-glutamyl-L-glutamic acid

D. S. Eggleston, D. J. Hodgson, Acta Cryst., **B38**, 1216, 1982

BRCPDG 5-bromocytosine-phthaloyl-DL-glutamic acid hemihydrate

M. Ohki, A. Takenaka, H. Shimanouchi, Y. Sasada, Bull. Chem. Soc. Jpn., **50**, 90, 1977

BZGEGA N-benzyloxycarbonyl-(η-ethyl)-L-glutamyl-(η-ethyl) -L-glutamic acid ethyl ester

E. Benedetti, B. Di Blasio, V. Pavone, C. Pedone, G. Germain, M. Goodman, Biopolymers, **18**, 517, 1979

CAGLCL10 Calcium L-glutamate chloride monohydrate

H. Einspahr, G. L. Gartland, C. E. Bugg, Acta Cryst., **B33**, 3385, 1977

CAGLUT10 Calcium L-glutamate trihydrate

H. Einspahr, C. E. Bugg, Acta Cryst., **B30**, 1037, 1974

CTBGLU cytidine - N-carbenzoxyglutamic acid dihydrate

T. Hata, M. Yoshikawa, S. Sato, C. Tamura, Acta Cryst., **B31**, 312, 1975

CTSGLM cytosine N, N-phthaloyl-DL-glutamic acid dihydrate

A. Takenaka, M. Ohki, Y. Sasada, Bull. Chem. Soc. Jpn., **53**, 2724, 1980

DLGLAC DL-glutamic acid hydrochloride

B. Dawson, Acta Cryst., **6**, 81, 1953

GLUGLY α-L-glutamyl-glycine

D. S. Eggleston, E. J. Valente, D. J. Hodgson, Acta Cryst., **B37**, 1430, 1981

LGLUAC03 L-glutamic acid, α-form
(Neutron study)

M. S. Lehmann, A. C. Nunes, Acta Cryst., **B36**, 1621, 1980

LGLUAC11 L-glutamic acid, β-form
(Neutron study)

M. S. Lehmann, T. F. Koetzle, W. C. Hamilton, J. Cryst. Mol. Struct., **2**, 225, 1972

LGLUCA Calcium di-L-glutamate tetrahydrate

H. Einspahr, C. E. Bugg, Acta Cryst., **B35**, 316, 1979

LGLUTA L-glutamic acid hydrochloride
(Neutron study)

A. Sequeira, H. Rajagopal, R. Chidambaram, Acta Cryst., **B28**, 2514, 1972

LGPYRG L-glutamic acid L-pyroglutamic acid monohydrate

Z. Taira, W. H. Watson, Acta Cryst., B33, 3823, 1977

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(1)	0.27108(21)	0.29127(12)	0.31289(18)	0.0218
C(2)	0.28919(20)	0.27690(12)	0.17183(18)	0.0201
C(3)	0.32520(22)	0.35903(13)	0.09922(20)	0.0241
C(4)	0.21697(23)	0.43228(13)	0.12160(21)	0.0272
C(5)	0.23023(23)	0.50375(14)	0.02771(18)	0.0266
O(1)	0.15284(15)	0.27155(11)	0.36258(13)	0.0336
O(2)	0.38100(15)	0.32293(10)	0.36890(14)	0.0292
O(3)	0.13978(17)	0.56863(10)	0.05101(15)	0.0333
O(4)	0.31303(22)	0.50443(12)	-0.06080(17)	0.0515
N(1)	0.15257(18)	0.23623(11)	0.12208(16)	0.0216
O(1W1)	0.02525(24)	0.08193(14)	0.20825(19)	0.0536
H(21)	0.367(3)	0.2338(15)	0.1624(22)	0.0337
H(31)	0.419(3)	0.3794(15)	0.1244(22)	0.0337
H(32)	0.328(3)	0.3430(15)	0.0114(23)	0.0337
H(41)	0.228(3)	0.4549(15)	0.2027(22)	0.0337
H(42)	0.118(3)	0.4136(15)	0.1179(22)	0.0337
H(1N1)	0.082(3)	0.2683(16)	0.1409(23)	0.0337
H(2N1)	0.134(3)	0.1824(16)	0.1600(22)	0.0337
H(3N1)	0.156(3)	0.2271(15)	0.0333(23)	0.0337
H(103)	0.142(3)	0.6075(16)	-0.0091(23)	0.0337
H(10W)	-0.007(4)	0.077(3)	0.280(4)	0.0903
H(20W)	-0.020(4)	0.057(3)	0.158(4)	0.0903

Thermal Vibration Parameters with Standard Deviations

	U11	U22	U33	U23	U13	U12
C(1)	0.0223(9)	0.0229(9)	0.0201(9)	0.0008(8)	-0.0018(8)	0.0062(8)
C(2)	0.0170(9)	0.0242(9)	0.0193(9)	-0.0011(8)	0.0001(7)	0.0017(7)
C(3)	0.0216(10)	0.0259(10)	0.0248(10)	0.0026(8)	0.0031(8)	-0.0010(8)
C(4)	0.0266(11)	0.0269(11)	0.0279(10)	0.0054(9)	0.0056(9)	0.0018(8)
C(5)	0.0262(10)	0.0279(10)	0.0258(10)	0.0034(8)	-0.0014(8)	0.0022(8)
O(1)	0.0233(7)	0.0589(11)	0.0187(7)	0.0009(7)	0.0020(6)	-0.0042(7)
O(2)	0.0209(7)	0.0367(8)	0.0300(7)	-0.0109(7)	-0.0062(6)	0.0039(6)
O(3)	0.0376(9)	0.0303(8)	0.0319(8)	0.0102(7)	0.0071(7)	0.0102(7)
O(4)	0.0609(12)	0.0515(11)	0.0421(10)	0.0191(9)	0.0266(9)	0.0214(9)
N(1)	0.0202(8)	0.0256(9)	0.0189(8)	-0.0007(7)	-0.0001(7)	-0.0009(7)
O(1W1)	0.0625(13)	0.0576(12)	0.0406(11)	-0.0005(9)	0.0035(10)	-0.0302(11)

CHAPTER SEVEN
STRYCHNINE

STRYCHNINE

Introduction

The indole alkaloid strychnine is obtained from the seeds of *strychnos nux vomica*, and in smaller yields from related species. Although it is no longer considered to be of therapeutic value (it was once used as a "tonic" stimulant and as a laxative), its mechanism of action is better understood than that of almost any other stimulant compound.

The main effect of strychnine on the central nervous system consists of the stimulation, followed by the depression, of reflexes. The drug increases the motor effects of spinal reflexes and diminishes the latent period. This leads to reflexes becoming more general and, after large doses, small sensory stimuli will send all the voluntary muscles in the body into violent convulsions. It is known that strychnine does not excite directly, but inhibits the inhibition of spinal motor neurone signals by glycine. Strychnine has an independent excitatory action on the medulla oblongata, where it stimulates the vasomotor and vagal centres. It also has an enhancing effect on all five senses, although its bitter taste tends to mask the enhancement of flavour. This same bitter taste increases the appetite. A high dose of strychnine causes restlessness and small stimuli will cause the limbs to jerk. This leads to generalised convulsions with the trunk and limbs rigidly extended and the face distorted into a rictus. Each convulsion is very painful and lasts a few minutes. After five or six convulsions, each of which is followed by a longer period of exhaustion, respiration fails to return and death by asphyxia results. It is possible to counteract the effect of strychnine after absorption and stop the convulsions by reducing sensory stimulation to a minimum and administrating a general anaesthetic. Barbiturates or mephenesin (a muscle relaxant and interneuronal blocker) may be used as an antidote.

Although crystals of strychnine were examined at Edinburgh as long ago as 1944, the crystal structure was not determined as it was too complex a problem for the methods available at the time. After the

publication of the structures of strychnine hydrobromide (Robertson and Beevers, 1951) and strychnine sulphate (Bokhoven, Schoone and Bijvoet, 1951) interest in the pure base waned, although the crystal structures of some other strychnine salts have been determined.

Crystallisation

The crystals of strychnine used in this determination were grown from a solution of equimolar quantities of strychnine and N-acetyl-L-phenylalanine dissolved in the minimum of hot water and allowed to cool slowly to room temperature. In general, strychnine crystals were found in most attempted crystallisations of molecular complexes of strychnine with N-acyl amino acids. This could be because not all of the strychnine was in solution as strychninium cations.

Crystal Data

Formula $C_{21}H_{22}N_2O_2$ Mol.Wt. 334.41 F(000)=712

Space group $P2_12_12_1$ Int. Tab. No. 19

Cell dimensions a = 11.267(2) b = 11.89(1) c = 12.105(4)
V = 1621.9 A^3 Z = 4 Dc = 1.369 gcm^{-3}
Radiation MoKα λ = 0.71069 A μ = 0.83cm^{-1}
Final R = 0.0370 based on 1195 independent data
h_{max} = 13 k_{max} = 14 l_{max} = 14

θ_{max} = 25° $\sin\theta_{max}/\lambda$ = 0.5947
Intensity control -1 -1 4
0.988 < drift curve < 1.016
1648 reflections measured
453 reflections considered unobserved ($F < 4\sigma(F)$)
Weighting scheme : $W = 0.1625/(\sigma^2(F)+0.006531(F^2))$
314 parameters refined
Final difference Fourier max. 0.1699 eA^{-3} min. -0.2131 eA^{-3}
Max. Shift/σ in final cycle 0.005

Space group determination

The space group was uniquely determined to be $P2_12_12_1$ by the systematic absences (h00, h odd; 0k0, k odd; 00l, l odd).

Data collection and reduction

The data was collected on the CAD4 and corrected for Lorentz and polarisation effects by the program CADABS.

Solution and Refinement

The structure was solved automatically with the MULTAN 77 system. The first electron density map output revealed the entire structure except for one carbon atom.

The program SHELX was used for least-squares refinement, the progress of which is summarised below:

R	Model
0.2598	All non H but for one carbon
0.2385	All non H atoms included
0.0941	14 hydrogen added in calculated positions with free isotropic thermal parameters
0.0819	Another 7 hydrogen atoms added
0.0476	All hydrogen atoms included in model and all non-hydrogen atoms with anisotropic thermal parameters
0.0411	Weighting scheme applied
0.0370	Final convergence.*

Description of structure

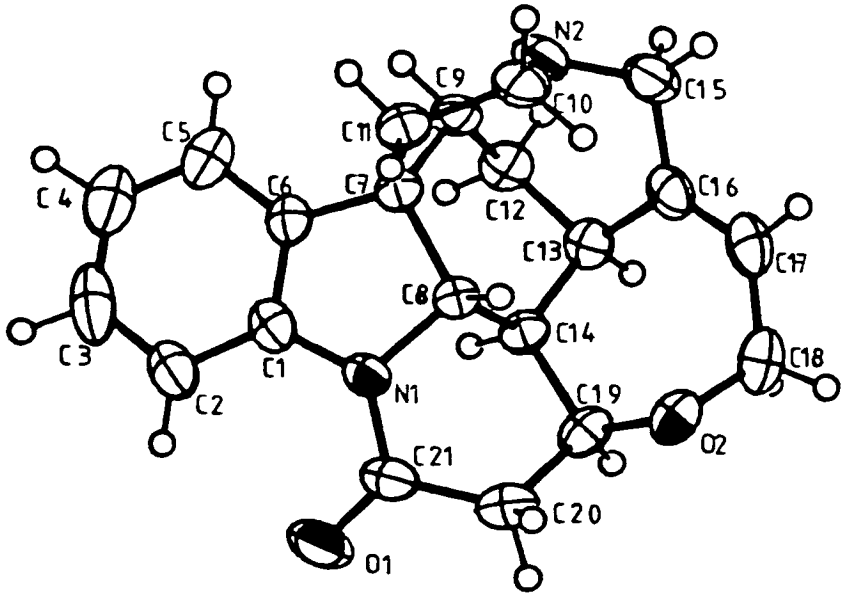
It could be claimed that strychnine has a more complex structure than any other compound with a similar number of skeletal atoms. This results in a very rigid structure with a greater than usual deviation of skeletal bond lengths and angles from the ideal values. There is a difference of 12 e.s.d's between the longest and the shortest carbon carbon bonds,* and a difference of 61 e.s.d's between the largest and smallest angles at "tetrahedral" carbon.

C(7)-C(8)	1.556(5) A	C(17)-C(18)	1.497(5) A
mean C-C	1.523(19) A		

* No constraints were applied to the hydrogen positions or thermal parameters

* C(17)-C(18) is so short because it is a bond between an sp^2 and an sp^3 carbon atom.

C(9)-C(7)-C(11)	100.6(3)°	C(13)-C(14)-C(19)	118.9(3)°
mean C-C-C	111(5)°		



The tables below list the bonds, angles and torsion angles of strychnine, using the numbering system illustrated above.

C(1) - C(2)	1.395(6)	C(10) - N(2)	1.471(6)
C(1) - C(6)	1.386(5)	C(12) -C(13)	1.522(6)
C(1) - N(1)	1.408(5)	C(13) -C(14)	1.530(5)
C(2) - C(3)	1.396(8)	C(13) -C(16)	1.520(6)
C(3) - C(4)	1.375(8)	C(14) -C(19)	1.543(5)
C(4) - C(5)	1.380(7)	C(15) -C(16)	1.506(6)
C(5) - C(6)	1.386(6)	C(15) - N(2)	1.464(6)
C(6) - C(7)	1.498(5)	C(16) -C(17)	1.321(6)
C(7) - C(8)	1.556(5)	C(17) -C(18)	1.497(7)
C(7) - C(9)	1.542(5)	C(18) - O(2)	1.435(6)
C(7) -C(11)	1.545(5)	C(19) -C(20)	1.528(6)
C(8) -C(14)	1.531(5)	C(19) - O(2)	1.425(5)
C(8) - N(1)	1.489(5)	C(20) -C(21)	1.498(6)
C(9) -C(12)	1.516(6)	C(21) - N(1)	1.371(5)
C(9) - N(2)	1.493(5)	C(21) - O(1)	1.221(6)
C(10) -C(11)	1.515(6)		

C(2) - C(1) - C(6)	121.1(4)	C(12) -C(13) -C(16)	109.4(3)
C(2) - C(1) - N(1)	128.8(4)	C(14) -C(13) -C(16)	114.8(3)
C(6) - C(1) - N(1)	110.1(3)	C(8) -C(14) -C(13)	112.8(3)
C(1) - C(2) - C(3)	117.1(4)	C(8) -C(14) -C(19)	107.1(3)
C(2) - C(3) - C(4)	122.4(5)	C(13) -C(14) -C(19)	118.9(3)
C(3) - C(4) - C(5)	119.4(5)	C(16) -C(15) - N(2)	111.6(4)
C(4) - C(5) - C(6)	119.9(4)	C(13) -C(16) -C(15)	113.9(4)
C(1) - C(6) - C(5)	120.0(4)	C(13) -C(16) -C(17)	122.3(4)
C(1) - C(6) - C(7)	110.7(3)	C(15) -C(16) -C(17)	123.8(4)
C(5) - C(6) - C(7)	129.1(4)	C(16) -C(17) -C(18)	122.7(4)
C(6) - C(7) - C(8)	102.6(3)	C(17) -C(18) - O(2)	111.8(4)
C(6) - C(7) - C(9)	116.6(3)	C(14) -C(19) -C(20)	109.9(3)
C(6) - C(7) -C(11)	111.9(3)	C(14) -C(19) - O(2)	114.7(3)
C(8) - C(7) - C(9)	114.2(3)	C(20) -C(19) - O(2)	105.5(3)
C(8) - C(7) -C(11)	111.2(3)	C(19) -C(20) -C(21)	117.3(4)
C(9) - C(7) -C(11)	100.6(3)	C(20) -C(21) - N(1)	115.6(4)
C(7) - C(8) -C(14)	116.8(3)	C(20) -C(21) - O(1)	122.6(4)
C(7) - C(8) - N(1)	104.3(3)	N(1) -C(21) - O(1)	121.8(4)
C(14) - C(8) - N(1)	106.5(3)	C(1) - N(1) - C(8)	109.3(3)
C(7) - C(9) -C(12)	114.2(3)	C(1) - N(1) -C(21)	125.2(3)
C(7) - C(9) - N(2)	105.3(3)	C(8) - N(1) -C(21)	118.1(3)
C(12) - C(9) - N(2)	111.3(3)	C(9) - N(2) -C(10)	108.1(3)
C(11) -C10) - N(2)	105.2(3)	C(9) - N(2) -C(15)	113.5(3)
C(7) -C(11) -C(10)	101.6(3)	C(10) - N(2) -C(15)	113.4(3)
C(9) -C(12) -C(13)	108.9(3)	C(18) - O(2) -C(19)	114.6(3)
C(12) -C(13) -C(14)	106.5(3)		

C(6) - C(1) - C(2) - C(3)	1.5(6)
N(1) - C(1) - C(2) - C(3)	-178.5(4)
C(2) - C(1) - C(6) - C(5)	-1.7(6)
C(2) - C(1) - C(6) - C(7)	174.0(4)
N(1) - C(1) - C(6) - C(5)	178.3(4)
N(1) - C(1) - C(6) - C(7)	-6.0(4)
C(2) - C(1) - N(1) - C(8)	174.2(4)
C(2) - C(1) - N(1) -C(21)	25.0(6)
C(6) - C(1) - N(1) - C(8)	-5.8(4)
C(6) - C(1) - N(1) -C(21)	-155.1(4)
C(1) - C(2) - C(3) - C(4)	0.1(8)
C(2) - C(3) - C(4) - C(5)	-1.5(8)
C(3) - C(4) - C(5) - C(6)	1.3(8)
C(4) - C(5) - C(6) - C(1)	0.2(7)
C(4) - C(5) - C(6) - C(7)	-174.6(4)
C(1) - C(6) - C(7) - C(8)	14.5(4)

C(1)	C(6)	C(7)	C(9)	140.1(3)
C(1)	C(6)	C(7)	-C(11)	-104.9(4)
C(5)	C(6)	C(7)	C(8)	-170.4(4)
C(5)	C(6)	C(7)	C(9)	-44.7(6)
C(5)	C(6)	C(7)	-C(11)	70.3(5)
C(6)	C(7)	C(8)	-C(14)	100.3(3)
C(6)	C(7)	C(8)	N(1)	-16.9(3)
C(9)	C(7)	C(8)	-C(14)	-26.9(4)
C(9)	C(7)	C(8)	N(1)	-144.1(3)
C(11)	C(7)	C(8)	-C(14)	-139.9(3)
C(11)	C(7)	C(8)	N(1)	102.9(3)
C(6)	C(7)	C(9)	-C(12)	-83.6(4)
C(6)	C(7)	C(9)	N(2)	154.0(3)
C(8)	C(7)	C(9)	-C(12)	36.0(4)
C(8)	C(7)	C(9)	N(2)	-86.5(4)
C(11)	C(7)	C(9)	-C(12)	155.2(3)
C(11)	C(7)	C(9)	N(2)	32.8(4)
C(6)	C(7)	-C(11)	-C(10)	-167.5(3)
C(8)	C(7)	-C(11)	-C(10)	78.4(4)
C(9)	C(7)	-C(11)	-C(10)	-43.0(4)
C(7)	C(8)	-C(14)	-C(13)	40.1(4)
C(7)	C(8)	-C(14)	-C(19)	172.8(3)
N(1)	C(8)	-C(14)	-C(13)	156.0(3)
N(1)	C(8)	-C(14)	-C(19)	-71.2(3)
C(7)	C(8)	N(1)	C(1)	14.5(4)
C(7)	C(8)	N(1)	-C(21)	166.2(3)
C(14)	C(8)	N(1)	C(1)	-109.6(3)
C(14)	C(8)	N(1)	-C(21)	42.1(4)
C(7)	C(9)	-C(12)	-C(13)	-58.3(4)
N(2)	C(9)	-C(12)	-C(13)	60.8(4)
C(7)	C(9)	N(2)	-C(10)	-9.9(4)
C(7)	C(9)	N(2)	-C(15)	116.7(4)
C(12)	C(9)	N(2)	-C(10)	-134.2(3)
C(12)	C(9)	N(2)	-C(15)	-7.5(5)
N(2)	-C(10)	-C(11)	C(7)	38.2(4)
C(11)	-C(10)	N(2)	C(9)	-17.8(4)
C(11)	-C(10)	N(2)	-C(15)	-144.6(4)
C(9)	-C(12)	-C(13)	-C(14)	69.2(4)
C(9)	-C(12)	-C(13)	-C(16)	-55.4(4)
C(12)	-C(13)	-C(14)	C(8)	-60.3(4)
C(12)	-C(13)	-C(14)	-C(19)	173.1(3)
C(16)	-C(13)	-C(14)	C(8)	61.0(4)
C(16)	-C(13)	-C(14)	-C(19)	-65.7(5)
C(12)	-C(13)	-C(16)	-C(15)	0.4(5)
C(12)	-C(13)	-C(16)	-C(17)	177.8(4)

C(14)	-C(13)	-C(16)	-C(15) -119.2(4)
C(14)	-C(13)	-C(16)	-C(17) 58.2(6)
C(8)	-C(14)	-C(19)	-C(20) 43.8(4)
C(8)	-C(14)	-C(19)	- O(2) -74.9(4)
C(13)	-C(14)	-C(19)	-C(20) 173.1(3)
C(13)	-C(14)	-C(19)	- O(2) 54.4(5)
N(2)	-C(15)	-C(16)	-C(13) 52.5(5)
N(2)	-C(15)	-C(16)	-C(17) -124.8(5)
C(16)	-C(15)	- N(2)	- C(9) -48.2(5)
C(16)	-C(15)	- N(2)	-C(10) 75.7(5)
C(13)	-C(16)	-C(17)	-C(18) -2.5(7)
C(15)	-C(16)	-C(17)	-C(18) 174.7(4)
C(16)	-C(17)	-C(18)	- O(2) -65.2(6)
C(17)	-C(18)	- O(2)	-C(19) 88.2(4)
C(14)	-C(19)	-C(20)	-C(21) 10.1(5)
O(2)	-C(19)	-C(20)	-C(21) 134.3(4)
C(14)	-C(19)	- O(2)	-C(18) -66.1(4)
C(20)	-C(19)	- O(2)	-C(18) 172.8(3)
C(19)	-C(20)	-C(21)	- N(1) -41.7(5)
C(19)	-C(20)	-C(21)	- O(1) 139.6(4)
C(20)	-C(21)	- N(1)	- C(1) 159.9(4)
C(20)	-C(21)	- N(1)	- C(8) 13.1(5)
O(1)	-C(21)	- N(1)	- C(1) -21.3(6)
O(1)	-C(21)	- N(1)	- C(8) -168.1(4)

The bonds, angles and torsion angles involving hydrogen are tabulated below.

C(2)	-H(021)	0.97(4)	C(12)	-H(122)	1.03(5)
C(3)	-H(031)	0.93(6)	C(13)	-H(131)	0.99(5)
C(4)	-H(041)	1.05(6)	C(14)	-H(141)	1.03(4)
C(5)	-H(051)	0.98(5)	C(15)	-H(151)	1.09(6)
C(8)	-H(081)	0.98(4)	C(15)	-H(152)	1.06(5)
C(9)	-H(091)	1.01(3)	C(17)	-H(171)	0.99(5)
C(10)	-H(101)	1.04(5)	C(18)	-H(181)	0.96(6)
C(10)	-H(102)	1.00(5)	C(18)	-H(182)	1.04(5)
C(11)	-H(111)	0.96(4)	C(19)	-H(191)	0.95(4)
C(11)	-H(112)	0.92(4)	C(20)	-H(201)	0.98(7)
C(12)	-H(121)	0.93(5)	C(20)	-H(202)	1.00(5)

C(1) - C(2) -H(021)	120.4(25)	H(122)-C(12) -C(13)	109.8(26)
H(021)- C(2) - C(3)	122.5(25)	C(12) -C(13) -H(131)	115 (3)
C(2) - C(3) -H(031)	117 (4)	H(131)-C(13) -C(14)	108 (3)
H(031)- C(3) - C(4)	120 (4)	H(131)-C(13) -C(16)	103 (3)
C(3) - C(4) -H(041)	117 (4)	C(8) -C(14) -H(141)	105.7(25)
H(041)- C(4) - C(5)	123 (3)	C(13) -C(14) -H(141)	100.2(25)
C(4) - C(5) -H(051)	118 (3)	H(141)-C(14) -C(19)	111.4(25)
H(051)- C(5) - C(6)	122 (3)	H(151)-C(15) -H(152)	110 (4)
C(7) - C(8) -H(081)	107.2(23)	H(151)-C(15) -C(16)	109 (3)
H(081)- C(8) -C(14)	112.2(23)	H(151)-C(15) - N(2)	109 (3)
H(081)- C(8) - N(1)	109.4(23)	H(152)-C(15) -C(16)	112.2(27)
C(7) - C(9) -H(091)	108.1(20)	H(152)-C(15) - N(2)	105.7(27)
H(091)- C(9) -C(12)	109.2(20)	C(16) -C(17) -H(171)	118 (3)
H(091)- C(9) - N(2)	108.5(20)	H(171)-C(17) -C(18)	119 (3)
H(101)-C(10) -H(102)	108 (4)	C(17) -C(18) -H(181)	113 (4)
H(101)-C(10) -C(11)	113.7(26)	C(17) -C(18) -H(182)	113.7(27)
H(101)-C(10) - N(2)	109.5(26)	H(181)-C(18) -H(182)	98 (4)
H(102)-C(10) -C(11)	107.2(27)	H(181)-C(18) - O(2)	109 (4)
H(102)-C(10) - N(2)	113.1(27)	H(182)-C(18) - O(2)	111.0(27)
C(7) -C(11) -H(111)	107.1(26)	C(14) -C(19) -H(191)	108.8(24)
C(7) -C(11) -H(112)	112.4(24)	H(191)-C(19) -C(20)	111.0(24)
C(10) -C(11) -H(111)	114.6(26)	H(191)-C(19) - O(2)	106.9(24)
C(10) -C(11) -H(112)	112.4(24)	C(19) -C(20) -H(201)	111 (4)
H(111)-C(11) -H(112)	109 (4)	C(19) -C(20) -H(202)	106 (3)
C(9) -C(12) -H(121)	100 (3)	H(201)-C(20) -H(202)	92(5)
C(9) -C(12) -H(122)	111.7(26)	H(201)-C(20) -C(21)	109 (4)
H(121)-C(12) -H(122)	111 (4)	H(202)-C(20) -C(21)	118 (3)
H(121)-C(12) -C(13)	116 (3)		

C(6) - C(1) - C(2) -H(021)	-180 (3)
N(1) - C(1) - C(2) -H(021)	1 (3)
C(1) - C(2) - C(3) -H(031)	-170 (4)
H(021)- C(2) - C(3) -H(031)	11 (5)
H(021)- C(2) - C(3) - C(4)	-179 (3)
C(2) - C(3) - C(4) -H(041)	177 (4)
H(031)- C(3) - C(4) -H(041)	-13 (6)
H(031)- C(3) - C(4) - C(5)	168 (4)
C(3) - C(4) - C(5) -H(051)	-179 (3)
H(041)- C(4) - C(5) -H(051)	3 (5)
H(041)- C(4) - C(5) - C(6)	-177 (4)
H(051)- C(5) - C(6) - C(1)	-180 (3)
H(051)- C(5) - C(6) - C(7)	5 (3)
C(6) - C(7) - C(8) -H(081)	-132.8(24)
C(9) - C(7) - C(8) -H(081)	100.0(24)

C(11) - C(7) - C(8) -H(081)	-13.0(24)
C(6) - C(7) - C(9) -H(091)	38.1(21)
C(8) - C(7) - C(9) -H(091)	157.7(21)
C(11) - C(7) - C(9) -H(091)	-83.1(21)
C(6) - C(7) -C(11) -H(111)	-47 (3)
C(6) - C(7) -C(11) -H(112)	72.1(26)
C(8) - C(7) -C(11) -H(111)	-161.1(27)
C(8) - C(7) -C(11) -H(112)	-42.0(26)
C(9) - C(7) -C(11) -H(111)	77.5(27)
C(9) - C(7) -C(11) -H(112)	-163.4(26)
C(7) - C(8) -C(14) -H(141)	-68.4(26)
H(081)- C(8) -C(14) -C(13)	-84.3(25)
H(081)- C(8) -C(14) -H(141)	167 (4)
H(081)- C(8) -C(14) -C(19)	48.5(25)
N(1) - C(8) -C(14) -H(141)	47.6(26)
H(081)- C(8) - N(1) - C(1)	128.9(25)
H(081)- C(8) - N(1) -C(21)	-79.4(25)
C(7) - C(9) -C(12) -H(121)	63 (3)
C(7) - C(9) -C(12) -H(122)	-180 (3)
H(091)- C(9) -C(12) -H(121)	-58 (4)
H(091)- C(9) -C(12) -H(122)	59 (4)
H(091)- C(9) -C(12) -C(13)	-179.4(21)
N(2) - C(9) -C(12) -H(121)	-178 (3)
N(2) - C(9) -C(12) -H(122)	-61 (3)
H(091)- C(9) - N(2) -C(10)	105.6(21)
H(091)- C(9) - N(2) -C(15)	-127.7(21)
H(101)-C(10) -C(11) - C(7)	-82 (3)
H(101)-C(10) -C(11) -H(111)	163 (4)
H(101)-C(10) -C(11) -H(112)	39 (4)
H(102)-C(10) -C(11) - C(7)	159 (3)
H(102)-C(10) -C(11) -H(111)	44 (4)
H(102)-C(10) -C(11) -H(112)	-81 (4)
N(2) -C(10) -C(11) -H(111)	-77 (3)
N(2) -C(10) -C(11) -H(112)	158.6(26)
H(101)-C(10) - N(2) - C(9)	104.8(27)
H(101)-C(10) - N(2) -C(15)	-22.0(27)
H(102)-C(10) - N(2) - C(9)	-135 (3)
H(102)-C(10) - N(2) -C(15)	99 (3)
C(9) -C(12) -C(13) -H(131)	-171 (3)
H(121)-C(12) -C(13) -H(131)	78 (5)
H(121)-C(12) -C(13) -C(14)	-42 (4)
H(121)-C(12) -C(13) -C(16)	-167 (4)
H(122)-C(12) -C(13) -H(131)	-49 (4)
H(122)-C(12) -C(13) -C(14)	-168.2(27)
H(122)-C(12) -C(13) -C(16)	67.1(27)

C(12)	-C(13)	-C(14)	-H(141)	51.6(26)
H(131)	-C(13)	-C(14)	- C(8)	176 (3)
H(131)	-C(13)	-C(14)	-H(141)	-72 (4)
H(131)	-C(13)	-C(14)	-C(19)	49 (3)
C(16)	-C(13)	-C(14)	-H(141)	172.9(26)
H(131)	-C(13)	-C(16)	-C(15)	123 (3)
H(131)	-C(13)	-C(16)	-C(17)	-59 (3)
C(8)	-C(14)	-C(19)	-H(191)	165.6(25)
C(13)	-C(14)	-C(19)	-H(191)	-65.2(26)
H(141)	-C(14)	-C(19)	-H(191)	51 (4)
H(141)	-C(14)	-C(19)	-C(20)	-71.3(27)
H(141)	-C(14)	-C(19)	- O(2)	170.1(27)
H(151)	-C(15)	-C(16)	-C(13)	-67 (3)
H(151)	-C(15)	-C(16)	-C(17)	116 (3)
H(152)	-C(15)	-C(16)	-C(13)	171 (3)
H(152)	-C(15)	-C(16)	-C(17)	-6 (3)
H(151)	-C(15)	- N(2)	- C(9)	72 (3)
H(151)	-C(15)	- N(2)	-C(10)	-165 (3)
H(152)	-C(15)	- N(2)	- C(9)	-171 (3)
H(152)	-C(15)	- N(2)	-C(10)	-47 (3)
C(13)	-C(16)	-C(17)	-H(171)	-177 (3)
C(15)	-C(16)	-C(17)	-H(171)	0 (3)
C(16)	-C(17)	-C(18)	-H(181)	172 (4)
C(16)	-C(17)	-C(18)	-H(182)	61 (3)
H(171)	-C(17)	-C(18)	-H(181)	-14 (5)
H(171)	-C(17)	-C(18)	-H(182)	-124 (5)
H(171)	-C(17)	-C(18)	- O(2)	109 (3)
H(181)	-C(18)	- O(2)	-C(19)	-146 (4)
H(182)	-C(18)	- O(2)	-C(19)	-40 (3)
C(14)	-C(19)	-C(20)	-H(201)	-117 (4)
C(14)	-C(19)	-C(20)	-H(202)	144 (3)
H(191)	-C(19)	-C(20)	-H(201)	123 (5)
H(191)	-C(19)	-C(20)	-H(202)	24 (4)
H(191)	-C(19)	-C(20)	-C(21)	-110.3(26)
O(2)	-C(19)	-C(20)	-H(201)	8 (4)
O(2)	-C(19)	-C(20)	-H(202)	-92 (3)
H(191)	-C(19)	- O(2)	-C(18)	54.6(25)
H(201)	-C(20)	-C(21)	- N(1)	86 (4)
H(201)	-C(20)	-C(21)	- O(1)	-93 (4)
H(202)	-C(20)	-C(21)	- N(1)	-170 (3)
H(202)	-C(20)	-C(21)	- O(1)	11 (3)

Unit cell projections down all three axes are shown on the next three pages following this section, and they demonstrate clearly both that the molecular packing is dominated entirely by hydrophobic interactions and that there are no hydrogen bonding contacts at all. There are two carbon oxygen contacts and one carbon nitrogen contact less than 3.5 angstroms.

C(4)	O(1)	3.310	(-1-x, 1/2+y, -1/2-z)
C(12)	O(1)	3.456(6)	(1/2+x, -1/2-y, -1-z)
C(20)	N(2)	3.372(6)	(-x, -1/2-y, -1/2-z)

There are five oxygen hydrogen and one nitrogen hydrogen contacts closer than 3 angstroms.

O(1)	H(041)	2.41(6)	(-1-x, -1/2+y, -1/2-z)
O(1)	H(112)	2.79(4)	(-1/2-x, -1-y, -1/2-z)
O(1)	H(122)	2.61(5)	(-1/2+x, -1/2-y, -1-z)
O(2)	H(051)	2.87(5)	(-x, -1/2+y, -1/2-z)
O(2)	H(091)	2.98(4)	(-x, -1/2+y, -1/2-z)
N(2)	H(201)	2.53(7)	(-x, 1/2+y, -1/2-z)

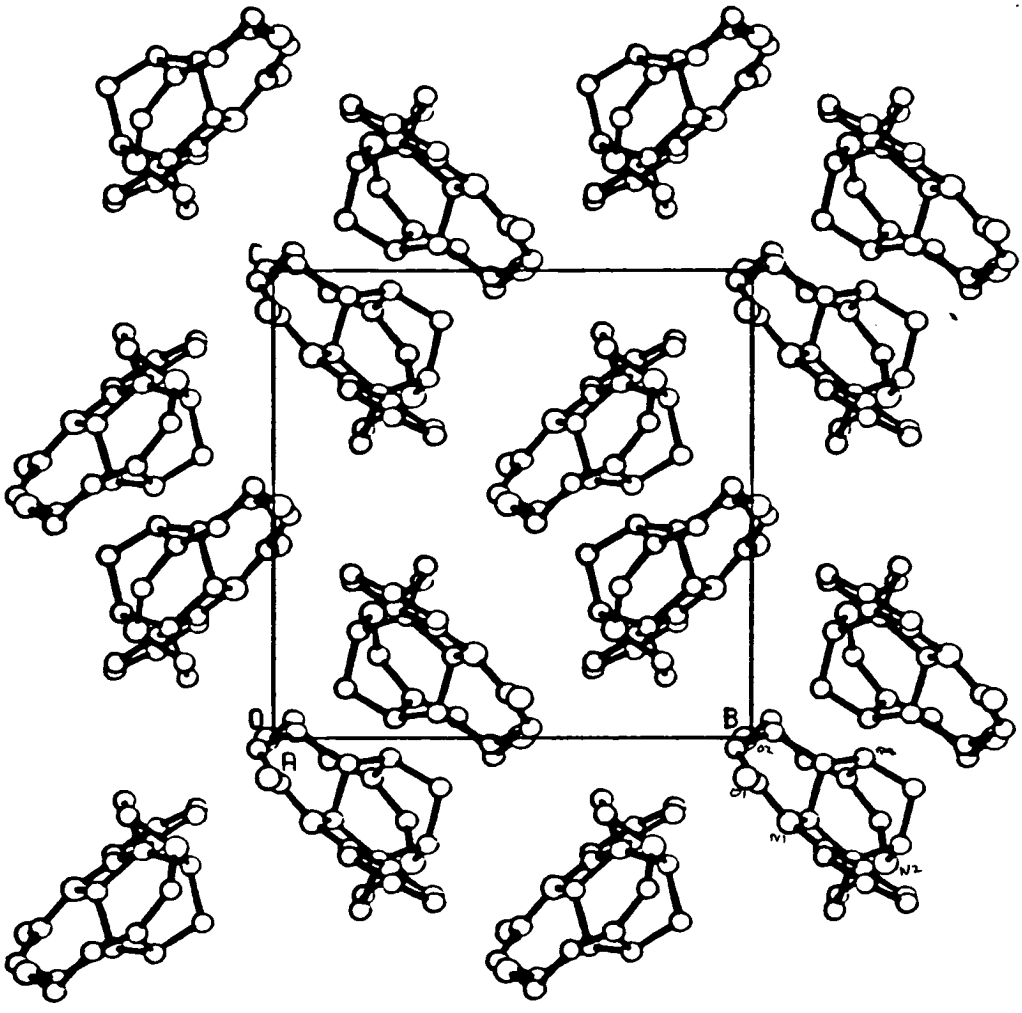
With one exception the carbon to hydrogen contacts may be divided into those involving the carbons of the aromatic ring, and those involving the double bond carbon atoms in the ether ring, which may be a result of an interaction between hydrogen atoms and a cloud of π electrons. The contacts between the aromatic ring and protons above or below the ring plane are of a type interpreted as an energetically favoured interaction between the π-cloud of the ring system and neighbouring protons when they occurred in solid benzene (Beevers, 1976).

C(1)	H(202)	2.83(5)	(-1/2-x, -1-y, 1/2+z)
C(2)	H(131)	2.83(5)	(-1/2+x, -1/2-y, -1-z)
C(2)	H(202)	2.92(5)	(-1/2-x, -1-y, 1/2+z)
C(4)	H(102)	2.99(5)	(-1/2+x, -1/2-y, -z)

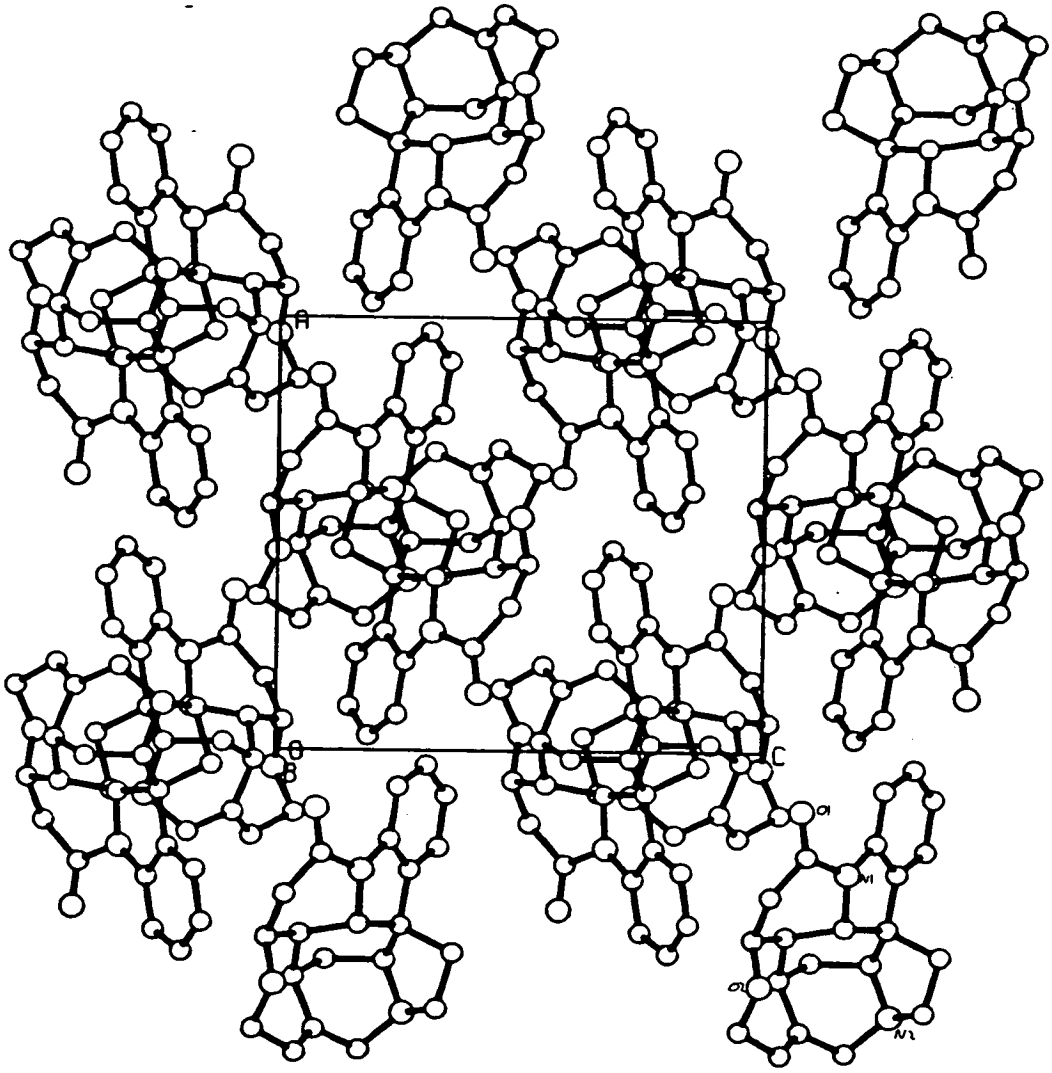
C(16)	H(141)	2.92(5)	(1/2+x,	-1/2-y, -1-z)
C(17)	H(051)	2.86(5)	(-x,	-1/2+y, -1/2-z)
C(17)	H(121)	2.90(5)	(1/2+x,	-1/2-y, -1-z)
C(17)	H(141)	2.89(5)	(1/2+x,	-1/2-y, -1-z)
C(18)	H(051)	2.88(5)	(-x,	-1/2+y, -1/2-z)
C(10)	H(201)	2.84(7)	(-x,	1/2+y, -1/2-z)

There are only three hydrogen to hydrogen contacts less than 2.5 A.

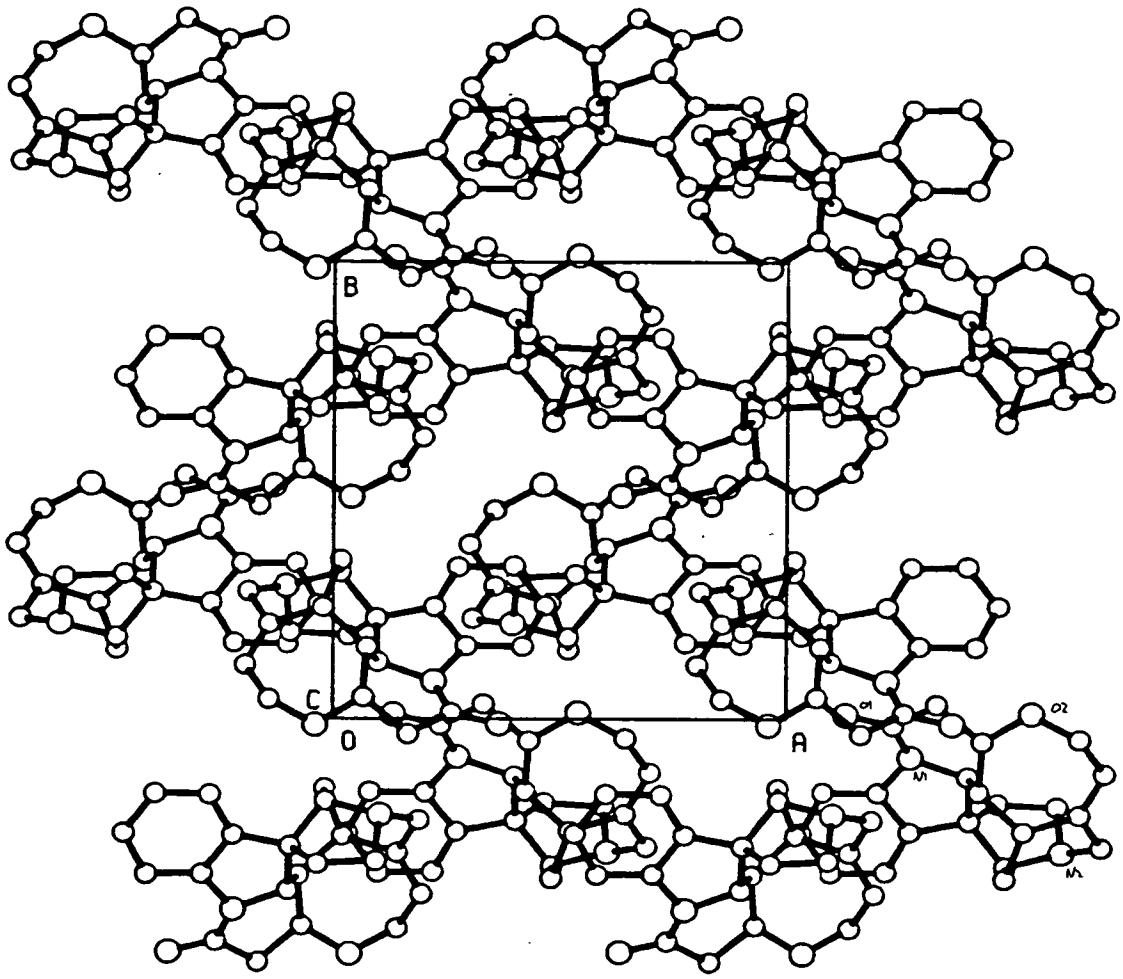
H(081)	H(091)	2.35(5)	(-x,	-1/2+y, -1/2-z)
H(102)	H(201)	2.47(8)	(-x,	1/2+y, -1/2-z)
H(112)	H(122)	2.36(6)	(-x,	-1/2+y, -1/2-z)



PROJECTION DOWN THE X AXIS



PROJECTION DOWN THE Y AXIS



PROJECTION DOWN THE Z AXIS

Comparison with the strychnine salts

A superficial comparison of the structure of the strychnine molecule with that of the average strychninium cation reveals little in the way of systematic structural differences. This may even be taken as self-evident, given the rigid nature of the molecular skeleton, but there is one significant difference between the two molecules. The bonds to the tertiary amine nitrogen in the free base are uniformly shorter than the equivalent bonds (to the tertiary ammonium nitrogen) in the idealised cation by about 5σ.

bond	base	ideal cation	Δ
C(9)-N(2)	1.493(5)	1.537(3)	0.044
C(10)-N(2)	1.470(6)	1.501(3)	0.031
C(15)-N(2)	1.464(6)	1.445(3)	0.031

A possible rationalisation for this difference is that the lone pair on the nitrogen in the neutral molecule is free to overlap the bonding orbitals of the adjacent carbons and thus shorten the bonds, while the equivalent electron density in the cation is used to form a dative bond to the proton. There are no other significant differences between the two molecules. The packing of the strychnine molecules in the unit cell has no similarity at all to the packing of the strychnine salts, as it is governed solely by the need to fill space as efficiently as possible untempered by any consideration of electrostatic interactions.

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(1)	-0.2946(3)	-0.3397(3)	-0.2632(3)	0.0374
C(2)	-0.4172(4)	-0.3407(5)	-0.2471(3)	0.0526
C(3)	-0.4661(5)	-0.2507(6)	-0.1887(4)	0.0671
C(4)	-0.3982(5)	-0.1640(5)	-0.1481(4)	0.0630
C(5)	-0.2768(4)	-0.1663(4)	-0.1629(4)	0.0507
C(6)	-0.2247(4)	-0.2542(3)	-0.2203(3)	0.0360
C(7)	-0.0957(3)	-0.2788(3)	-0.2384(3)	0.0322
C(8)	-0.0992(3)	-0.3752(3)	-0.3253(3)	0.0302
C(9)	-0.0165(4)	-0.1785(3)	-0.2713(3)	0.0368
C(10)	0.0958(4)	-0.3102(4)	-0.1635(4)	0.0445
C(11)	-0.0338(4)	-0.3172(4)	-0.1310(3)	0.0382
C(12)	-0.0176(4)	-0.1515(4)	-0.3937(4)	0.0416
C(13)	0.0226(4)	-0.2549(3)	-0.4576(4)	0.0384
C(14)	-0.0761(4)	-0.3424(3)	-0.4456(3)	0.0339
C(15)	0.1912(4)	-0.2205(4)	-0.3229(4)	0.0525
C(16)	0.1434(4)	-0.2923(4)	-0.4152(3)	0.0430
C(17)	0.1979(4)	-0.3825(4)	-0.4538(4)	0.0510
C(18)	0.1437(4)	-0.4579(4)	-0.5390(4)	0.0548
C(19)	-0.0679(4)	-0.4525(3)	-0.5127(3)	0.0393
C(20)	-0.1668(4)	-0.5328(4)	-0.4776(4)	0.0469
C(21)	-0.2619(4)	-0.4867(4)	-0.4038(3)	0.0434
N(1)	-0.2234(3)	-0.41769(25)	-0.3205(3)	0.0355
N(2)	0.1052(3)	-0.2091(3)	-0.2329(3)	0.0450
O(1)	-0.3666(3)	-0.5103(3)	-0.4153(3)	0.0679
O(2)	0.03974(25)	-0.51362(24)	-0.49798(23)	0.0453

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(021)	-0.465(4)	-0.401(3)	-0.277(3)	0.0369
H(031)	-0.545(6)	-0.258(4)	-0.168(5)	0.0809
H(041)	-0.443(6)	-0.098(5)	-0.109(5)	0.0849
H(051)	-0.230(4)	-0.105(4)	-0.132(4)	0.0535
H(081)	-0.045(3)	-0.434(3)	-0.300(3)	0.0351
H(091)	-0.044(3)	-0.111(3)	-0.229(3)	0.0193
H(101)	0.126(4)	-0.380(4)	-0.208(4)	0.0470
H(102)	0.143(4)	-0.305(4)	-0.094(4)	0.0474
H(111)	-0.057(4)	-0.266(4)	-0.074(4)	0.0426
H(112)	-0.056(4)	-0.389(3)	-0.110(3)	0.0274
H(121)	-0.097(5)	-0.132(4)	-0.402(4)	0.0621
H(122)	0.037(4)	-0.085(4)	-0.412(4)	0.0475
H(131)	0.036(4)	-0.242(4)	-0.538(4)	0.0527
H(141)	-0.148(4)	-0.295(4)	-0.469(4)	0.0455
H(151)	0.211(5)	-0.137(5)	-0.356(5)	0.0694
H(152)	0.269(5)	-0.256(4)	-0.287(4)	0.0553
H(171)	0.274(4)	-0.404(4)	-0.420(4)	0.0629
H(181)	0.198(4)	-0.513(5)	-0.567(5)	0.0840
H(182)	0.126(4)	-0.418(4)	-0.614(4)	0.0552
H(191)	-0.074(3)	-0.435(3)	-0.589(4)	0.0323
H(201)	-0.135(5)	-0.601(6)	-0.445(6)	0.0884
H(202)	-0.192(4)	-0.573(4)	-0.546(4)	0.0551

Thermal Vibration Parameters with Standard Deviations

	U or U11	U22	U33	U23	U13	U12
C(1)	0.0354(21)	0.0489(21)	0.0280(17)	0.0120(18)	0.0009(15)	-0.0031(18)
C(2)	0.0372(24)	0.0861(34)	0.0347(22)	0.0071(24)	-0.0011(17)	-0.0079(25)
C(3)	0.0354(25)	0.1237(49)	0.0423(25)	0.0178(31)	0.0071(22)	0.0245(28)
C(4)	0.0574(29)	0.0886(39)	0.0431(22)	0.0020(27)	0.0050(23)	0.0247(31)
C(5)	0.0599(28)	0.0534(26)	0.0388(21)	0.0007(21)	0.0057(22)	0.0173(24)
C(6)	0.0353(20)	0.0446(21)	0.0280(16)	0.0050(17)	0.0003(15)	0.0042(17)
C(7)	0.0356(19)	0.0312(19)	0.0298(18)	0.0008(15)	-0.0014(15)	-0.0004(16)
C(8)	0.0333(18)	0.0288(17)	0.0284(16)	0.0011(15)	-0.0021(16)	0.0004(16)
C(9)	0.0411(22)	0.0273(19)	0.0421(20)	-0.0025(18)	-0.0002(17)	-0.0075(16)
C(10)	0.0389(23)	0.0529(25)	0.0419(22)	-0.0033(21)	-0.0129(19)	0.0002(19)
C(11)	0.0454(24)	0.0383(24)	0.0311(19)	0.0012(17)	-0.0047(17)	0.0007(19)
C(12)	0.0481(24)	0.0317(19)	0.0450(23)	0.0053(19)	0.0022(21)	-0.0024(20)
C(13)	0.0423(21)	0.0387(19)	0.0341(18)	0.0065(17)	0.0045(18)	-0.0013(18)
C(14)	0.0397(21)	0.0342(19)	0.0280(18)	0.0005(16)	-0.0023(15)	-0.0004(18)
C(15)	0.0424(23)	0.0617(29)	0.0535(25)	-0.0047(24)	-0.0014(22)	-0.0165(22)
C(16)	0.0361(21)	0.0525(23)	0.0405(21)	0.0085(19)	0.0093(17)	-0.0093(20)
C(17)	0.0382(25)	0.0673(29)	0.0475(24)	0.0112(24)	0.0127(22)	-0.0004(21)
C(18)	0.0527(27)	0.0635(29)	0.0481(25)	0.0036(24)	0.0184(22)	0.0111(25)
C(19)	0.0527(24)	0.0407(21)	0.0246(18)	0.0012(16)	-0.0008(16)	0.0068(19)
C(20)	0.0592(26)	0.0382(22)	0.0433(22)	-0.0045(19)	-0.0056(20)	-0.0075(21)
C(21)	0.0469(25)	0.0452(22)	0.0382(18)	0.0011(19)	-0.0054(18)	-0.0161(18)
N(1)	0.0336(15)	0.0381(16)	0.0350(16)	0.0027(15)	-0.0016(14)	-0.0093(14)
N(2)	0.0386(18)	0.0501(18)	0.0465(19)	0.0022(17)	-0.0036(16)	-0.0120(17)
O(1)	0.0548(20)	0.0879(24)	0.0611(19)	-0.0116(20)	-0.0061(16)	-0.0342(19)
O(2)	0.0533(17)	0.0408(15)	0.0419(15)	-0.0025(14)	0.0029(14)	0.0104(13)

CHAPTER EIGHT
STRYCHNINE NITRATE

STRYCHNINE NITRATE

Introduction

Although the inorganic salts of strychnine have varying degrees of hydration and crystallise in several different space groups, the actual molecular packing in each structure determined so far is quite similar (Gould *et al*,1981). This implies that the recognition taking place between the strychnine cations depends neither on the degree of solvation, nor on the counter-ion present.

To test this hypothesis, and to provide more information for a comparative study of the strychnine salts, the X-ray structure of strychnine nitrate was examined. The table below summarises the similarities between the salts already examined.

Acid	HI	HCl	HBr	$0.5(H_2SO_4)$
a/A	7.750	7.833	7.70	7.84
b/A	16.19	32.22	33.20	34.34
c/A	7.580	7.558	7.64	7.56
α/°	90	90	90	90
β/°	90	90.82	90	90
γ/°	92.57	90	90	94.75
Space Group	$P2_1$	$P2_1$	$P2_12_12_1$	I2
Z	2	4	4	4
Hydration	1	1.5	2	2.5

Crystallisation

The crystals were grown by dissolving the salt in the minimum possible quantity of hot water, and allowing the solution to cool slowly to ambient temperature. The crystal used for diffraction was grown by Miss Rosemary Kelly as part of an Honours project on drug-receptor interactions.

Crystal Data

Formula $C_{21}H_{23}N_2O_2^+ NO_3^-$ M = 397.41 F(000) = 840

Space Group $P2_12_12_1$ Int. Tab. No. 19

Cell dimensions a = 7.371(4) b = 31.020(10) c = 8.056(4) A
V = 1842.0 A^3 Z = 4 Dc = 1.433gcm^{-3}
Radiation MoKα λ = 0.71069 A μ = 0.97cm^{-1}
θ_{max} = 25° $Sin\theta_{max}/\lambda$ = 0.5947

h_{max} = 8 k_{max} = 36 l_{max} = 8

Final R = 0.0555 based on 1076 independent reflections
1865 reflections measured (1859 after sorting)
765 reflections considered unobserved ($F<5\sigma(F)$)
332 parameters refined
Max. shift/σ in final cycle 0.048
Difference Fourier map max. 0.2141 eA^{-3} min. -0.2299 eA^{-3}

Data collection and reduction

The data was collected on the STADI-2 two circle diffractometer and the program STOEABS was used to correct for Lorentz and polarisation effects.

Solution and refinement

The structure was solved using the MULTAN77 system. The program NORMAL was used to calculate structure factors based on a Debye curve derived from the spherically averaged molecular scattering factors of a strychnine cation (from the strychnine hydrochloride structure). The electron density map revealed almost the entire strychnine cation and the resultant atomic coordinates were refined using the program SHELX-76. The table below summarises the course of the refinement.

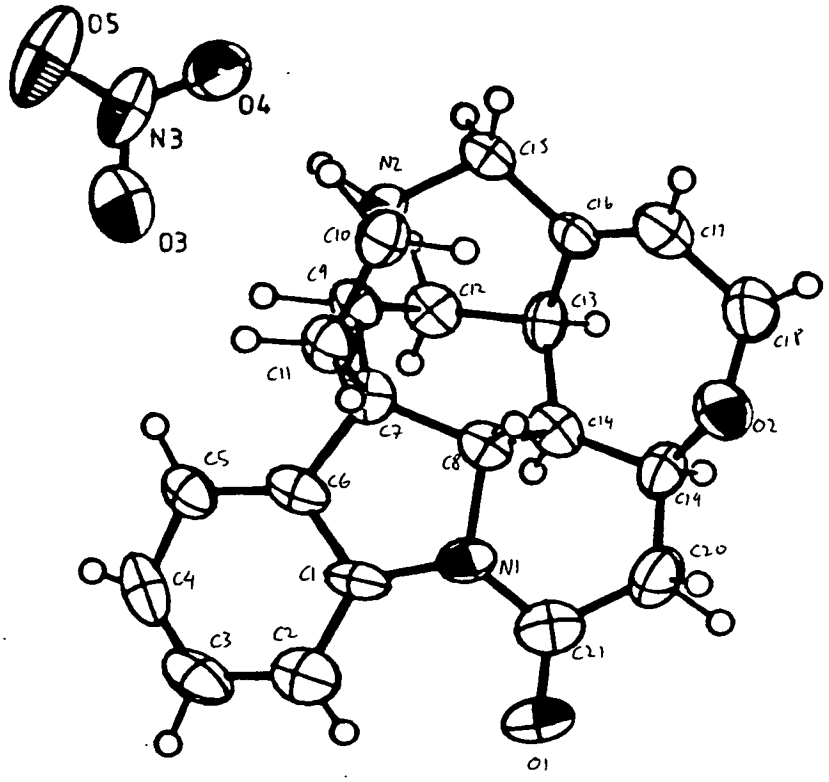
R	Model
0.3433	All but one carbon atom of cation input
0.3366	Nitrate group and other carbon atom located
0.1820	All non-Hydrogen atoms input
0.1274	Thermal parameters allowed to refine
0.1158	Hydrogens input in calculated positions
0.0989	All non-Hydrogen atoms with anisotropic thermal parameters
0.0621	Convergence and removal of constraints to H positions
0.0555	Final convergence

All hydrogen thermal parameters were constrained to be equal, but no positional restraints were employed

At one stage during the refinement, the fractional coordinates of a strychnine cation (from strychnine hydrochloride) were input to SHELX to investigate whether they would refine towards the correct coordinates. This was not found to be the case, but this may have been due to the difference in length of the long axes of the unit cells and the difference in relative position in the unit cell for the two molecules (This was approximately 1/3 of a unit cell in all directions, as can be seen from the packing diagrams on the succeeding pages).

Structure Description

There are no significant differences between the strychnine nitrate cation and the idealised strychnine cation (see below). This is almost certainly attributable to the rigid conformation of the molecule and the similarity of molecular environment over the series of strychnine salts used in the idealistaion process. As would be expected, there are no significant differences between the strychnine nitrate cation and the cations in any of the other salt structures. Structural parameters for all the strychnine salts studied at Edinburgh are tabulated at the end of the section on the calculation of the idealised cation.



A comparison of the strychnine cation with the neutral molecule shows only slight differences in lengths of bonds to the protonated nitrogen and of the ether ring double bond which are not very significant and the perspective plot of the cation shows that this similarity extends to the thermal motions of the atoms.

Bond	$StrH^+$	Str	Idealised
C9 N2	1.543(11)	1.493(5)	1.53(4)
C10 N2	1.518(12)	1.470(6)	1.50(4)
C15 N2	1.498(12)	1.464(6)	1.49(3)
C16 C17	1.277(14)	1.322(6)	1.314(16)

There are also slight differences in torsion angle around the amide and ether rings, but as this is the only part of the molecule where the geometry is not rigidly constrained, it is to be expected that some small variation in this region will occur between the free base and the cation depnding on the different environments in the base or in the salt.

Molecular packing

The molecular packing in all the inorganic salts of strychnine examined so far consists of bilayers of strychnine cations separated by sheets containing the counter-ions. With the exception of the strychnine sulphate sesterhydrate (Bokhoven *et al*, 1951), the bilayers in each structure appear superficially similar, but the finer detail of the intermolecular interactions within the bilayer varies with the symmetry operators applied in each space group. The anhydrous nitrate salt and the bromide dihydrate both crystallise in $P2_12_12_1$, with alternate bilayers related to one another by a twofold screw axis, and the molecules within each bilayer are related to each other by a combination of unit cell translations and twofold screw axes. Although the iodide monohydrate and the chloride sesquihydrate both crystallise in $P2_1$, there are two strychnine cations in the asymmetric unit of the chloride salt, related by a non-crystallographic twofold screw axis. The bilayers are related by the crystallographic screw axis. In the iodide salt however, the bilayers are related only by a unit cell translation and the twofold screw axis relates molecules within the bilayer. The sulphate sesterhydrate contains molecules related by rotation and by the body centre of the lattice. This results in a distinctive shift of half a unit cell translation between the bilayers and a striking change in relative orientation within them as compared to the rest of the structures. It is particularly noticeable that the alternation of orientation of the cations between layers no longer occurs in the bilayers.

The layers of anions contain varying amounts of water of crystallisation in the different structures, ranging from none in the nitrate, to the sulphate sesterhydrate which has some solvent disorder. Where present, this water allows the formation of infinite hydrogen bonded sheets running parallel to the bilayers. This can be contrasted with the nitrate structure in which each anion interacts specifically with only one cation.

It is a measure of the stability of the bilayer packing arrangement

that these salts are so insoluble compared with the strychnine: amino acid complexes, which tend to have less close contacts between strychnine molecules, although some vestiges of the bilayer system are detectable in the strychnine:benzoylalanine complex (Gould *et al*, 1984).

The tables below show the close contacts found in the strychnine nitrate structure. It is immediately noticeable that there are proportionately far more contacts between the strychnine cation and the nitrate than occur between cations. This is due to the relative weakness of van der Waals forces as compared to electrostatic interactions.

There is only the one hydrogen bonded contact:

O(4)	2.695	(10)	N(2)	(x,y,z)
H(1N2)	1.82	(8)	O(4)	(x,y,z)

Other contacts to the nitrate ion are:

O(2)	3.315	(10)	O(4)	(x,y,1 +z)
O(3)	3.195	(12)	N(2)	(x,y,z)
O(3)	3.194	(13)	C(9)	(x,y,z)
O(3)	3.497	(14)	C(11)	(x,y,z)
O(3)	3.476	(16)	C(18)	(-1 +x,y,-1 +z)
O(4)	3.455	(11)	C(9)	(x,y,z)
O(4)	3.455	(12)	C(15)	(x,y,z)
O(4)	3.241	(12)	C(15)	(2 1/2-x,-y,-1/2+z)
O(4)	3.479	(13)	C(17)	(2 1/2-x,-y,-1/2+z)
O(5)	3.156	(14)	C(10)	(x,y,-1 +z)
O(5)	3.366	(14)	C(10)	(1 1/2-x,-y,-1/2+z)
O(5)	3.485	(13)	C(11)	(x,y,-1 +z)
N(2)	3.385	(12)	N(3)	(x,y,z)

H(5)	2.91	(8)	O(3)	(x,y,z)
H(8)	2.96	(8)	O(5)	(x,y,1 +z)
H(9)	2.31	(7)	O(3)	(x,y,z)
H(101)	2.96	(7)	O(4)	(x,y,z)
H(101)	2.53	(7)	O(5)	(1 1/2-x,-y,1/2+z)
H(102)	2.38	(8)	O(5)	(x,y,1 +z)
H(112)	2.86	(7)	O(3)	(x,y,z)
H(121)	3.00	(8)	O(3)	(1 +x,y,1 +z)
H(152)	2.40	(7)	O(4)	(2 1/2-x,-y,1/2+z)
H(171)	2.78	(7)	O(4)	(2 1/2-x,-y,1/2+z)
H(1N2)	2.52	(8)	O(3)	(x,y,z)
H(1N2)	2.53	(8)	N(3)	(x,y,z)

The rest of the contacts are between strychnine cations in the same bilayer:

O(1)	3.396	(14)	C(20)	(-1/2+x,-1/2-y,2 -z)
C(4)	3.440	(16)	C(12)	(-1 +x,y,z)
H(202)	2.80	(7)	O(1)	(1/2+x,-1/2-y,2 -z)
H(3)	2.87	(8)	C(1)	(-1/2+x,-1/2-y,1 -z)
H(3)	2.92	(8)	C(6)	(-1/2+x,-1/2-y,1 -z)
H(9)	2.97	(7)	C(20)	(x,y,-1 +z)
H(111)	2.95	(8)	C(16)	(-1 +x,y,z)
H(111)	2.79	(8)	C(17)	(-1 +x,y,z)
H(13)	2.81	(8)	C(1)	(1 +x,y,z)
H(13)	2.79	(8)	C(2)	(1 +x,y,z)
H(13)	2.97	(8)	C(6)	(1 +x,y,z)
H(201)	2.94	(8)	C(9)	(x,y,1 +z)
H(2)	2.31	(11)	H(202)	(-1/2+x,-1/2-y,2 -z)
H(9)	2.07	(11)	H(201)	(x,y,-1 +z)

Comparison of molecular interactions in strychnine bilayers

The molecular interactions in these structures can be divided into four classes:

a) Interactions within the hydrophilic sheets.

b) Interactions between the hydrophilic sheets and the strychnine cations.

c) Interactions between strychnine cations in the same half of a bilayer. (*i.e* separated by a unit cell translation)

d) Interactions between strychnine cations on opposite sides of the bilayer.

An examination of these last two groups should provide the most useful information on the structural similarities and intermolecular interactions in the series of salts. The first two groups of interactions depend mostly on the counter-ion and degree of hydration of the salt. In general, the closest contacts within a bilayer are those involving a unit cell translation corresponding to the x direction in the nitrate salt (*i.e* x direction in the chloride and bromide or y direction in the iodide or sulphate). The table below shows those contacts of this type closer than 4 A in the nitrate structure, with the equivalent distances from the other structures for comparison. It can be seen that although the precise numerical figures might change, these contacts all involve the same regions of the cation (C12-C17 to the aromatic ring and C11).

contact	NIT	CL	CL'	I	BR	SUL
C4-C12	3.439	3.476	3.490	3.481	3.569	3.678
C11-C16	3.605	3.788	3.818	3.877	3.787	3.751
C2-C13	3.627	3.637	3.607	3.600	3.523	3.892
C3-C13	3.640	3.632	3.633	3.539	3.516	3.985
C11-C17	3.669	3.791	3.903	3.859	3.767	4.013
C5-C12	3.694	3.831	3.855	3.855	4.006	3.576
C3-C12	3.712	3.694	3.648	3.673	3.683	3.734
C4-C13	3.717	3.793	3.853	3.706	3.699	4.270
C1-C13	3.734	3.778	3.745	3.759	3.749	3.660
C6-C13	3.769	3.924	3.967	3.933	3.873	3.988
C5-C13	3.774	3.929	4.011	3.864	3.953	3.968
C2-C14	3.932	4.106	4.147	4.180	4.142	4.676
C11-C15	3.956	4.043	4.008	4.055	4.155	4.166

The next table shows the contacts between the strychnine cations separated by a unit cell translation equivalent to the z direction in the strychnine nitrate structure.(*i.e* by z in all but the bromide, which has y). Again it can be seen that the contacts involve the same general regions of the molecule. Although the exact orientation is not totally maintained, the interaction is in each case between the face of the molecule with the C4-C5 and C9-C12 bonds and that with the oxygen containing ring and carbonyl group.

contact	NIT	CL	CL'	I	BR	SUL
O2-C12	3.605	3.295	3.313	3.269	3.356	3.317
O1-C4	3.739	3.614	3.655	3.607	3.693	3.284
O2-C9	3.745	3.679	3.680	3.610	3.764	3.575
O1-C5	3.757	4.055	4.088	3.967	3.901	4.206
C12-C20	3.796	4.043	4.051	3.904	4.009	4.127
C9-C20	3.840	4.247	4.255	4.082	4.167	4.170
C5-C21	3.921	3.894	3.930	3.833	3.926	3.858
C5-C20	3.940	3.702	3.714	3.712	3.727	3.617

There are no contacts between strychnine cations involving translation in both of the short unit cell directions. For most of the structures examined, the contacts across a bilayer involve the carbonyl group and the aromatic ring. These contacts are not close enough to be considered as any form of ring stacking, but they do seem as if the non-polar end of each molecule forms a sort of hydrophobic pocket for the aromatic group across the bilayer from it.

contact	NIT	CL	CL'	I	BR	SUL
O1-O1	3.939	4.165	4.115	4.135	4.183	4.306
O1-C20	3.397	3.152	3.159	3.240	3.153	3.851
O1-C19	3.621	3.326	3.391	3.400	3.479	4.902
O1-C4	3.939	3.604	3.633	3.625	3.655	3.608
O1-C21	3.954	4.014	4.034	4.068	4.071	4.831
O1-C2	3.955	4.122	4.812	4.792	4.842	3.170
C1-C3	3.680	3.814	3.821	3.796	3.759	>5
C2-C3	3.750	3.756	3.760	3.731	3.777	4.953
C3-C6	3.838	4.442	4.469	4.417	4.435	>5
C3-C3	3.881	4.346	4.346	4.333	4.372	>5
C2-C20	3.975	4.294	4.426	4.384	4.343	3.094
C3-C4	3.993	4.914	4.935	4.878	4.770	>5

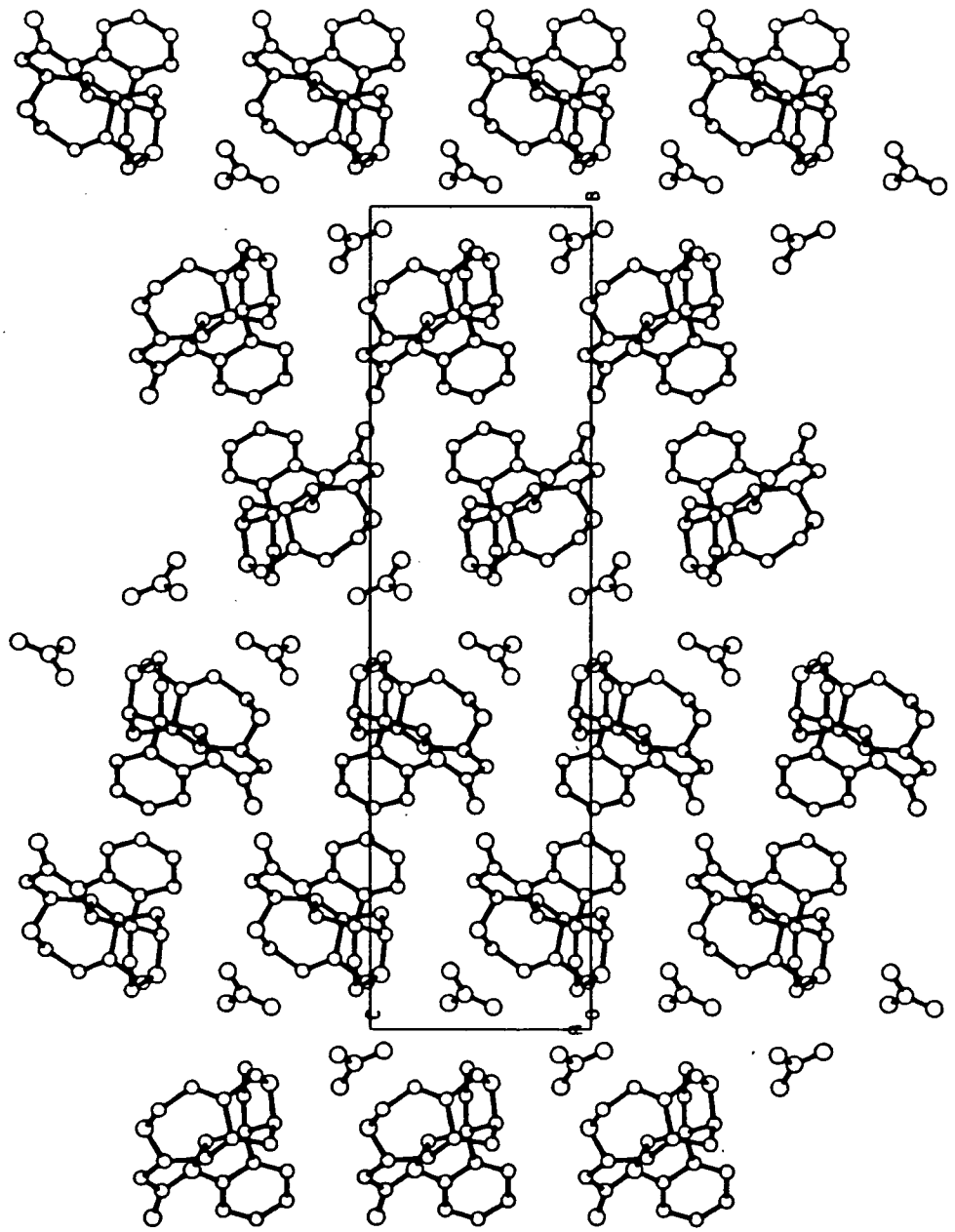
It is quite clear that the contacts listed for the strychnine sulphate cation show that it has a very different interaction at this part of the bilayer. The greatest similarities lie between the four halide structure cations, although their interactions are not extremely different from those of the nitrate. In the halide salts, the carbonyl oxygen is close to the aromatic ring, while in the nitrate, the carbonyl oxygen seems to prefer an interaction with the carbonyl carbon of a neighbouring cation. In none of the strychnine salts is there any sign of hydrogen bonding to this carbonyl group. This could be because the

nitrogen of the amide allows formation of a continuous π-system with the aromatic ring, leading to loss of electron density on the oxygen.

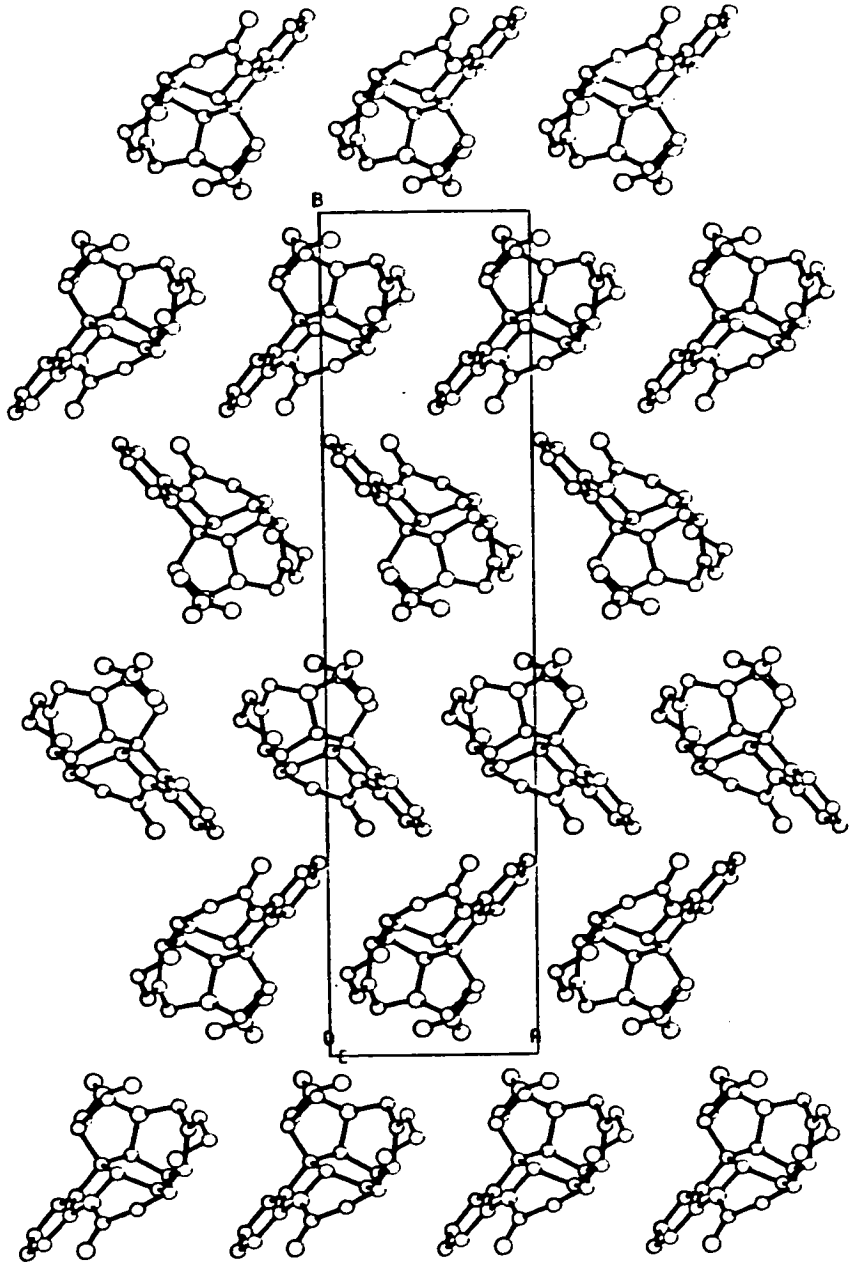
The table below shows the rest of the close contacts for the strychnine sulphate salt not listed above.

C2-C21	3.251
C4-C21	3.363
C3-C20	3.408
C3-C21	3.450
C3-C19	3.473
C2-C19	3.643
C3-C14	3.882

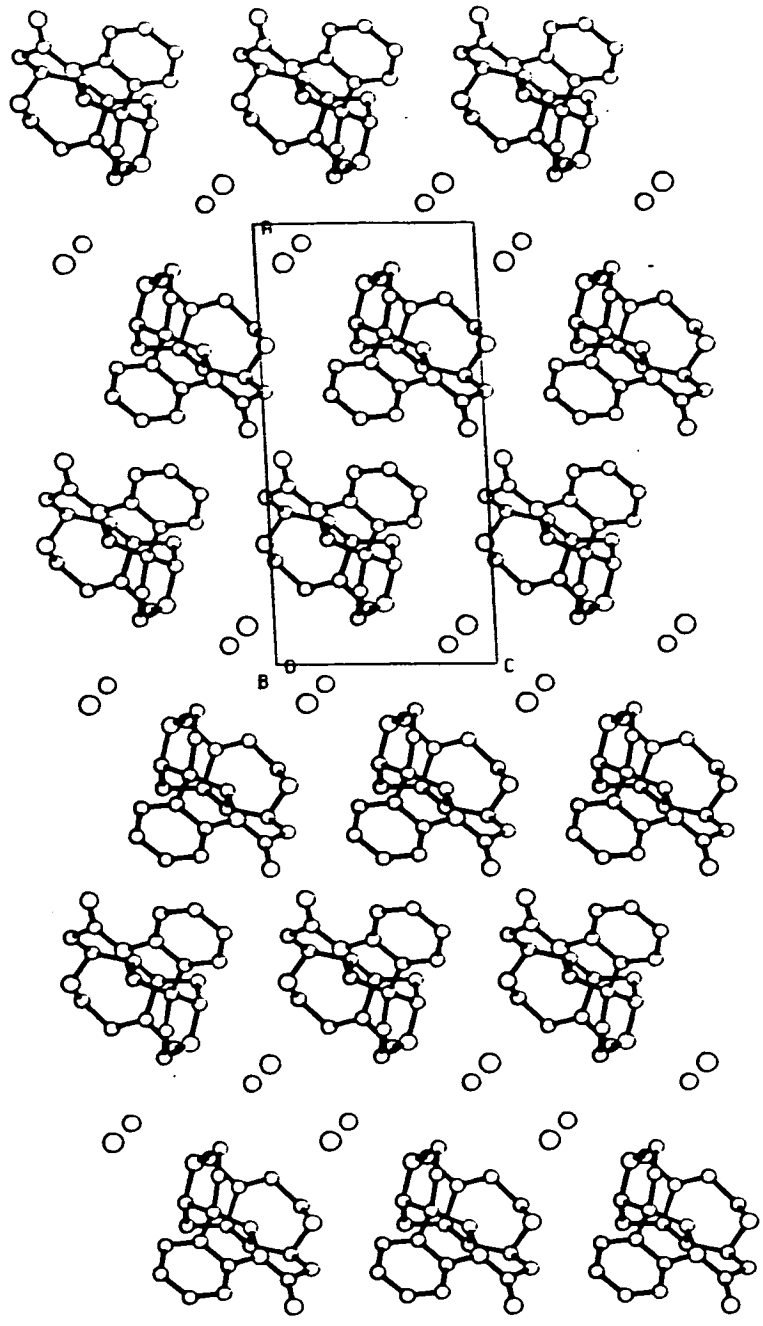
In this case it is clear that there is still an hydrophobic interaction involving the aromatic ring, but it no longer involves the carbonyl group.



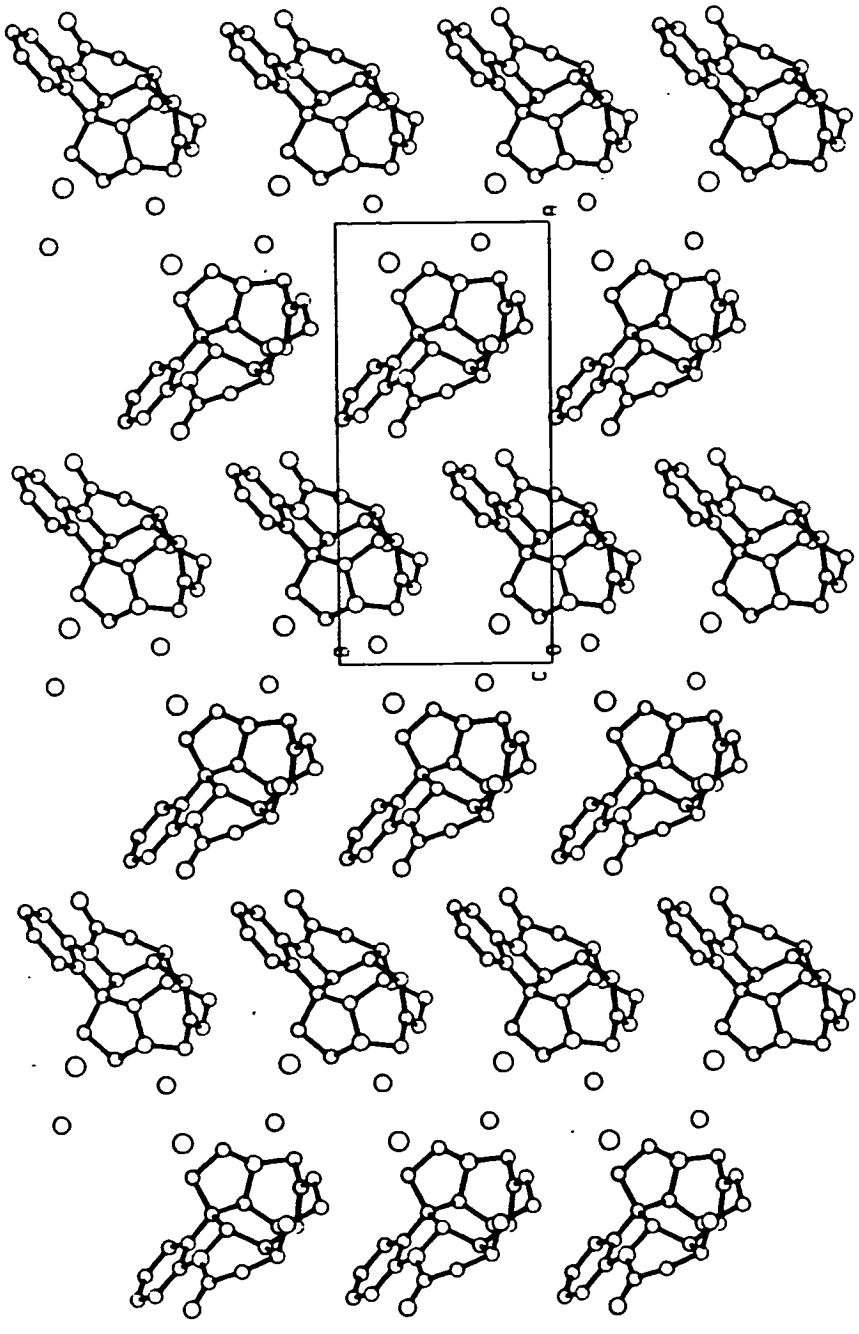
NITRATE VIEW DOWN X AXIS



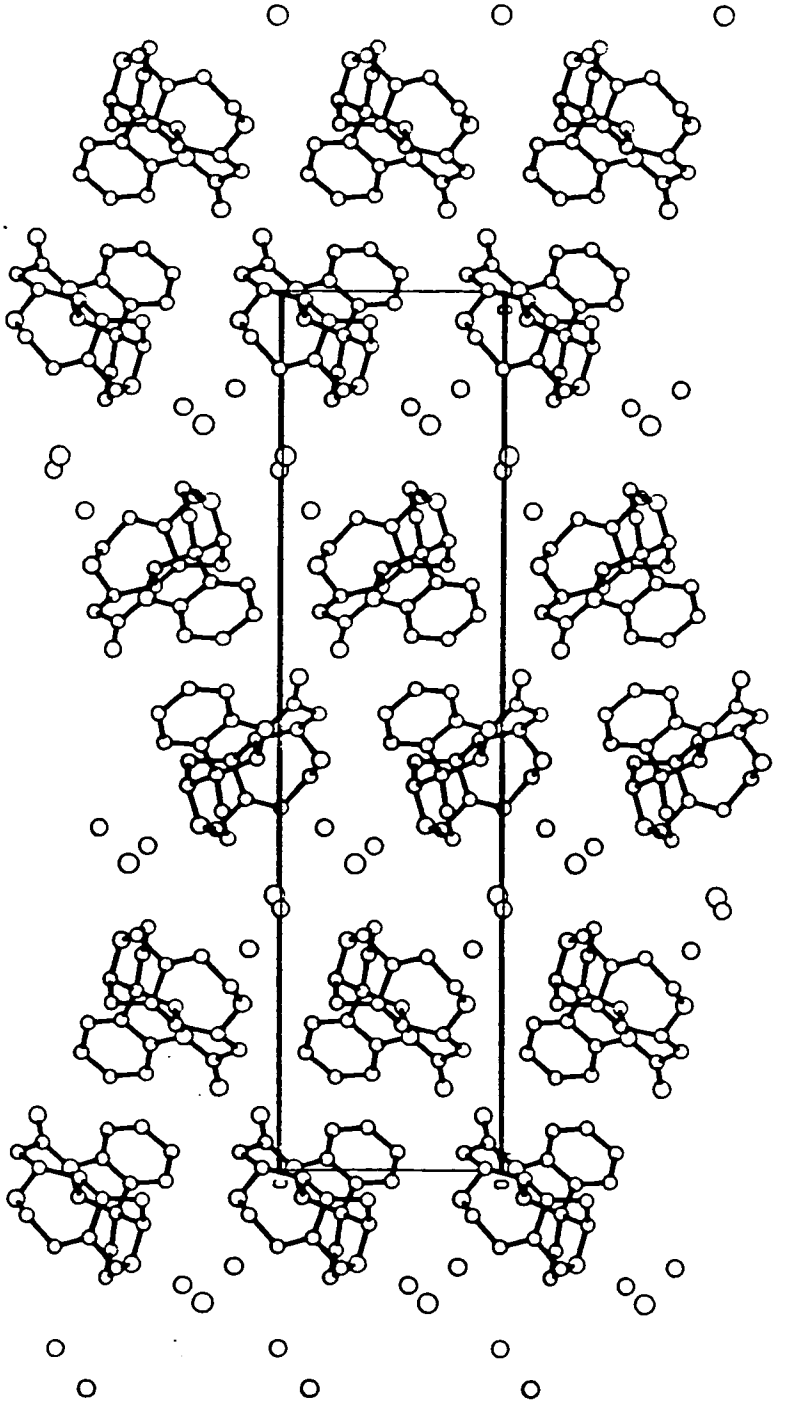
NITRATE VIEW DOWN Z AXIS



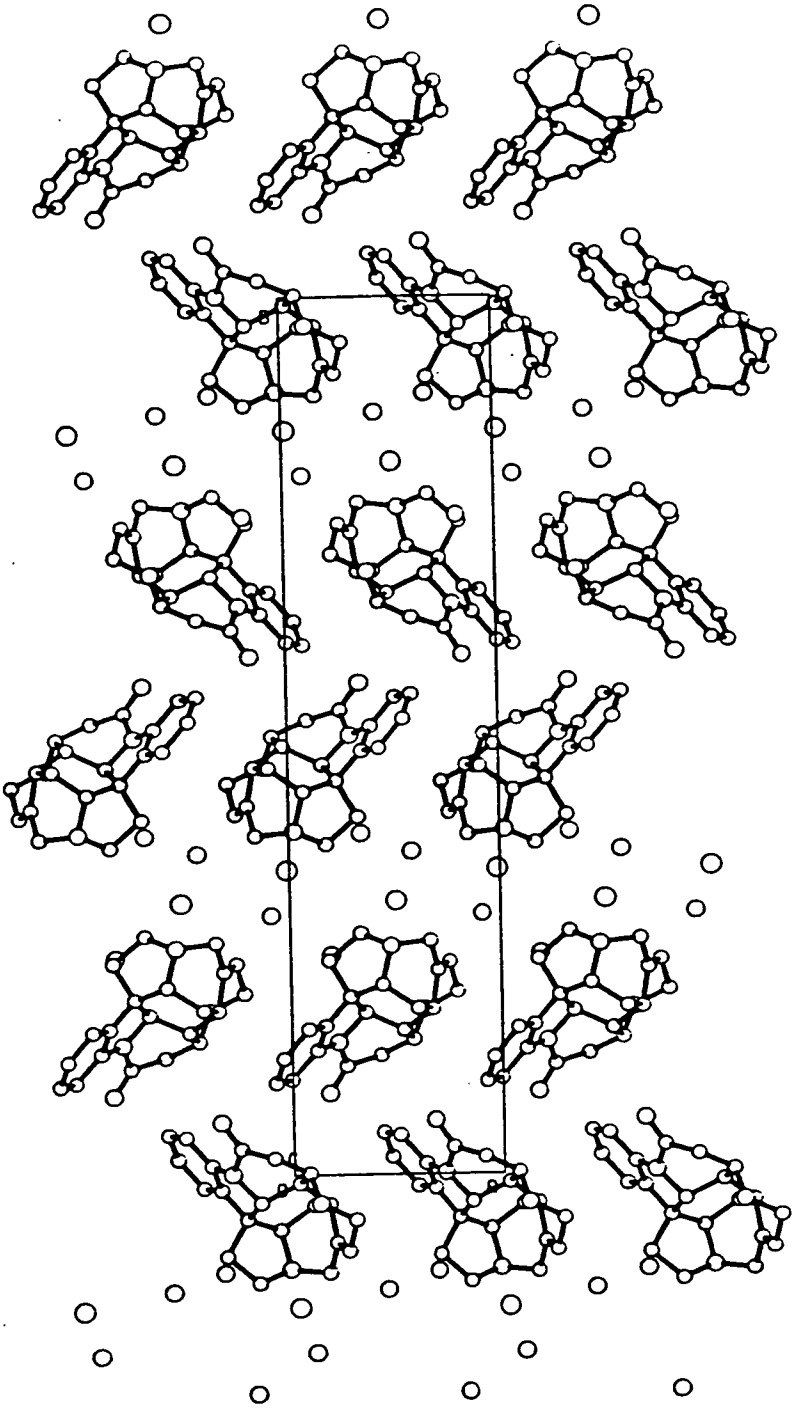
IODIDE VIEW DOWN YAXIS



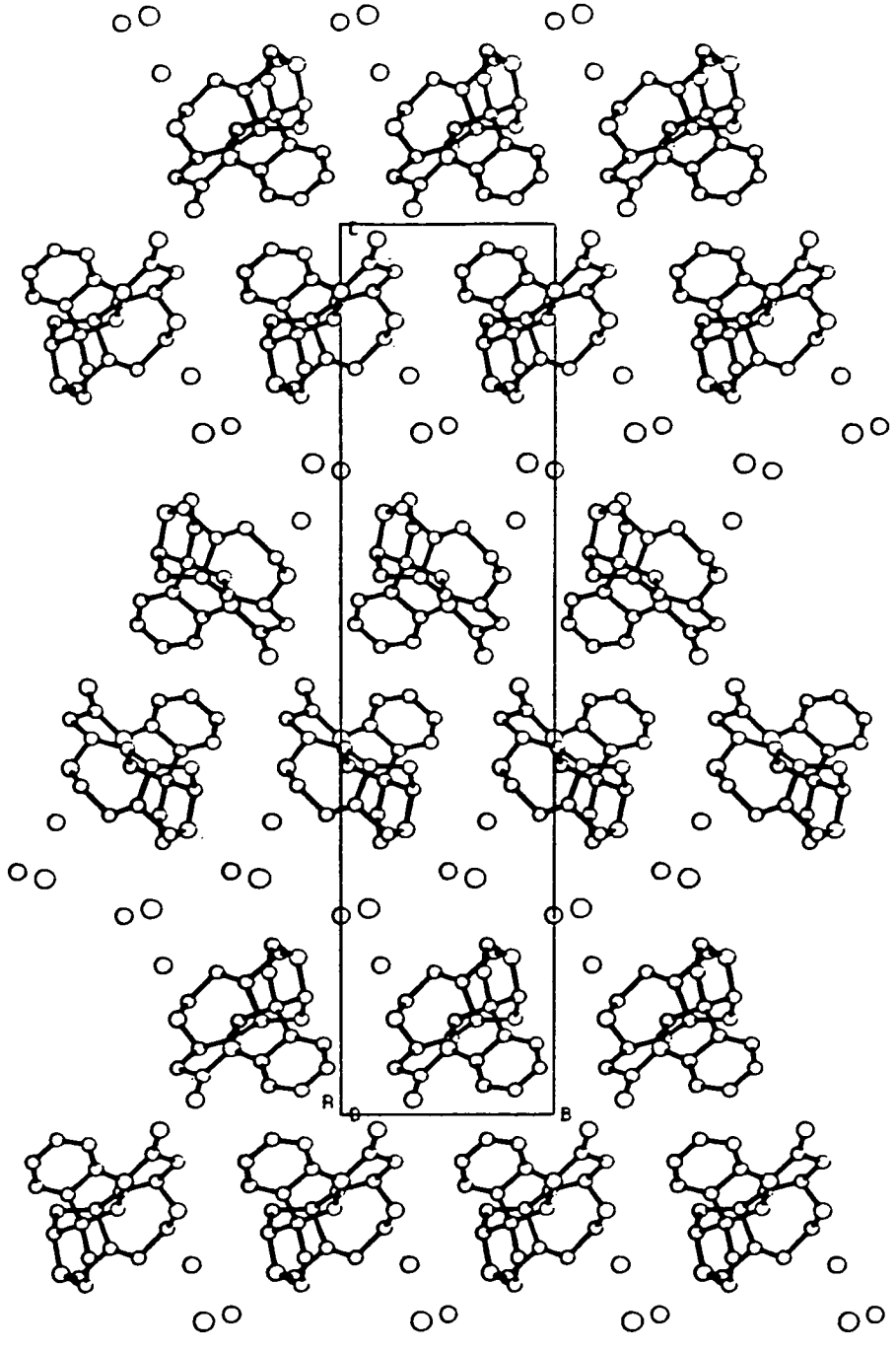
IODIDE VIEW DOWN ZAXIS



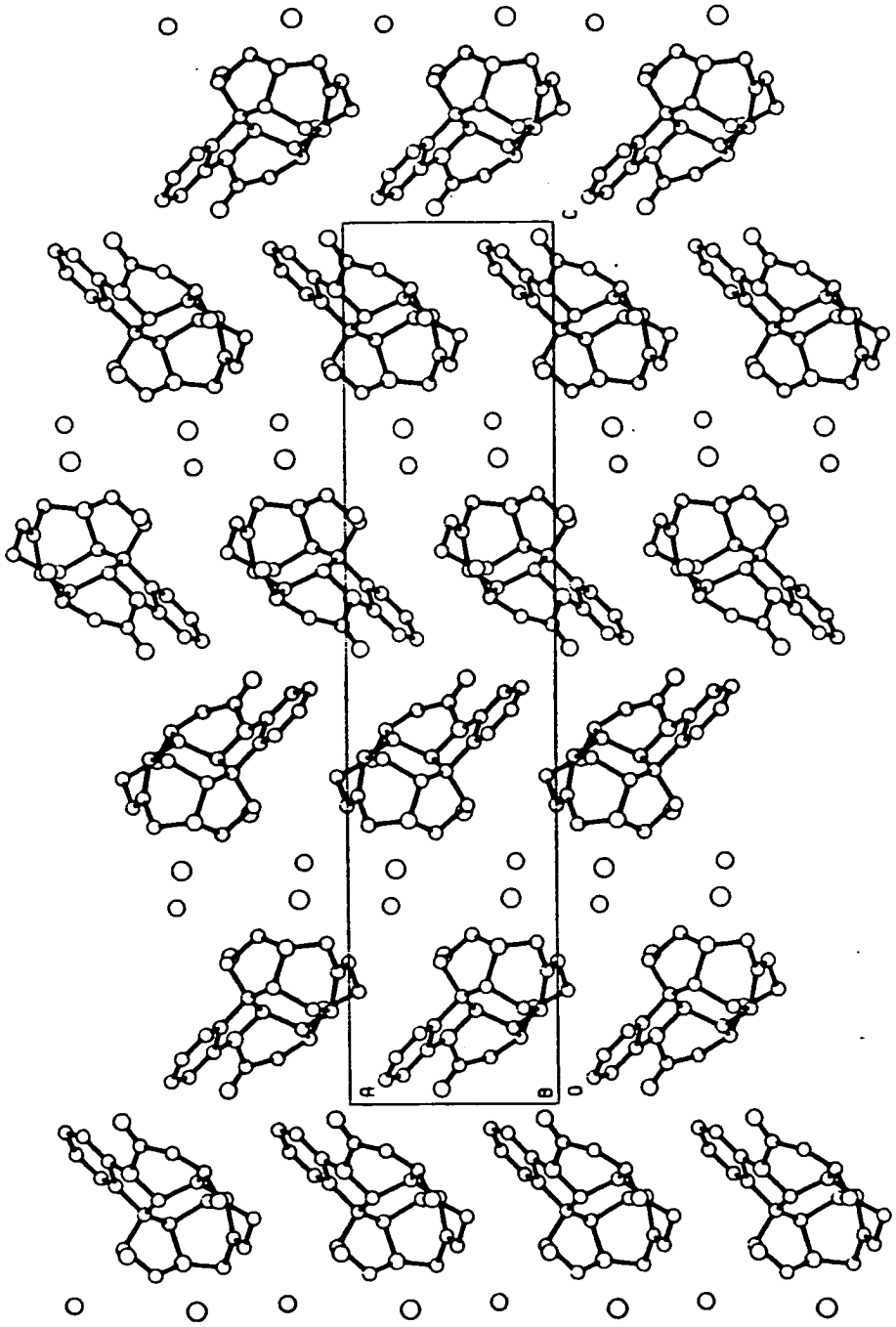
CHLORIDE VIEW DOWN X AXIS



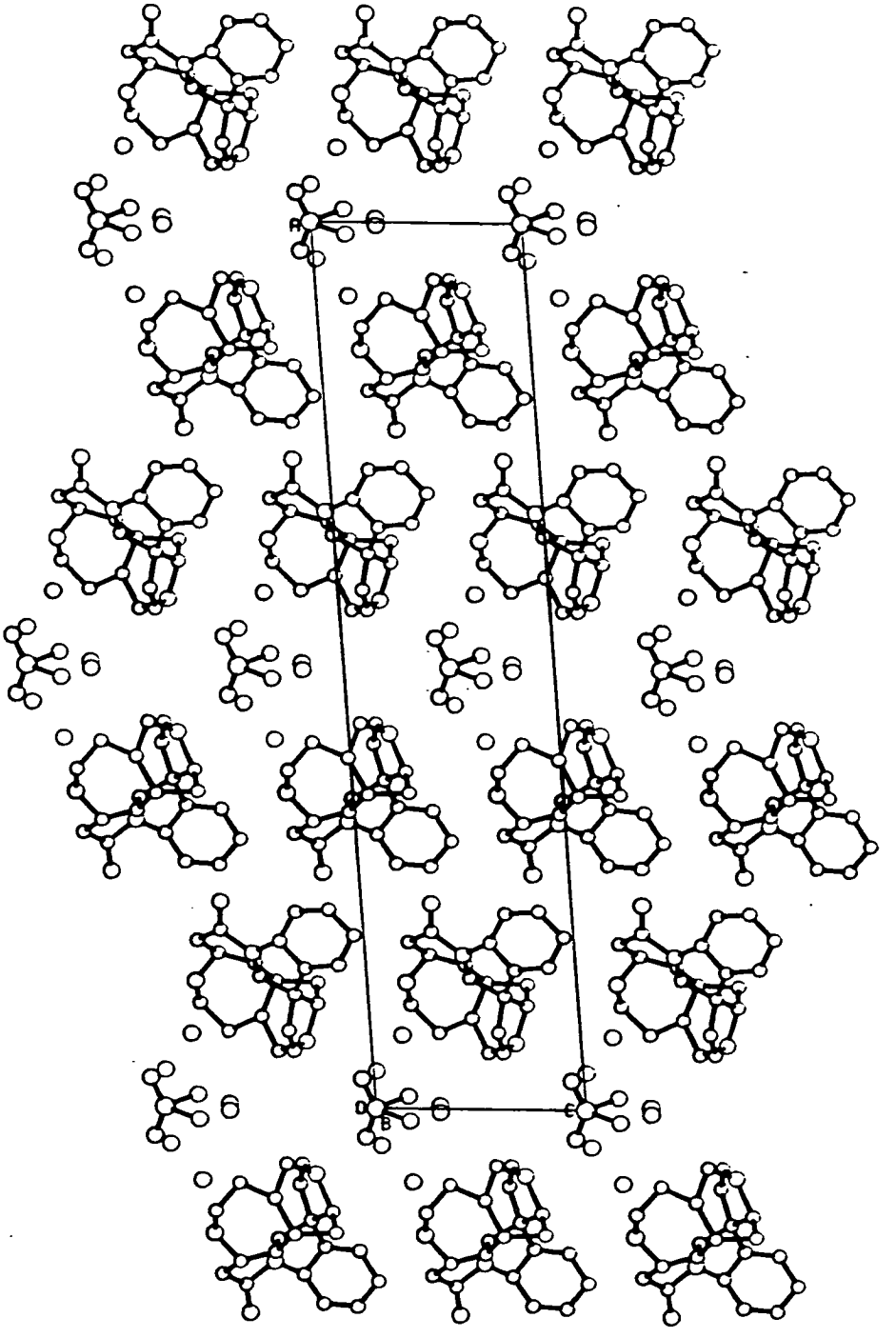
CHLORIDE VIEW DOWN Z AXIS



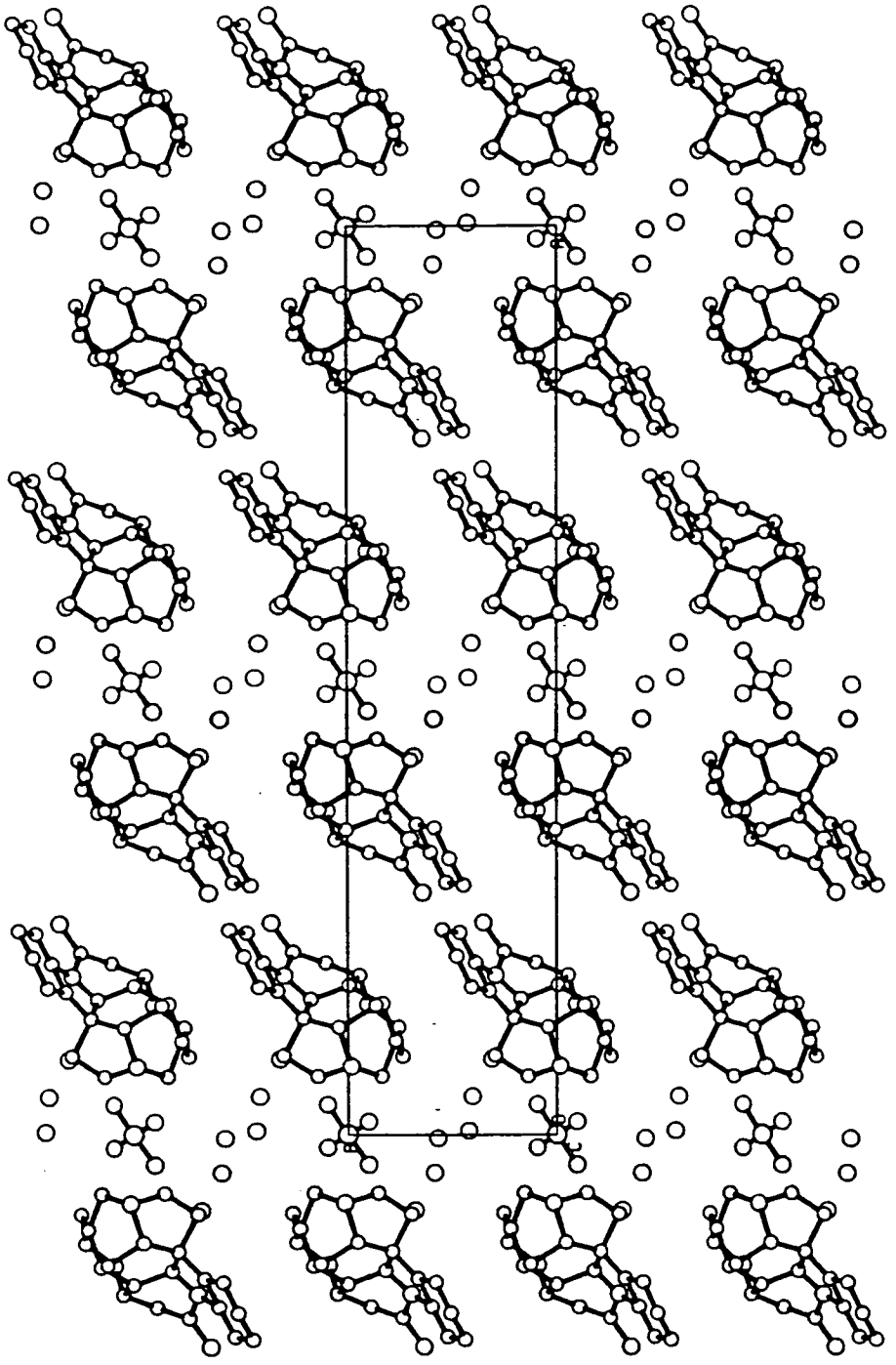
BROMIDE VIEW DOWN X AXIS



BROMIDE VIEW DOWN Y AXIS



SULPHATE VIEW DOWN Y AXIS



SULPHATE VIEW DOWN Z AXIS

Calculation of an idealised strychnine cation

As the strychnine cation is of almost identical geometry and environment in each of the salts studied so far, it was thought that an 'average strychnine' would be useful both for comparison with other strychnine containing structures, and as an aid for structure solution (the molecule being used either as a basis for the calculation of a Debye curve or for use with a rotation search of a Patterson map)

An idealised strychnine molecule, produced using the positional e.s.d's of the different strychnine cations whose structures are known, could possibly yield more accurate geometrical information than is normally available from a single crystal structure determination.

Two different methods were used to calculate the ideal geometry from the available sets of coordinates. The first method used was to transform the sets of coordinates to be idealised to the same coordinate system and then calculate a weighted mean of each positional parameter, which could then be used to calculate the geometry. The other method used was that of calculating a weighted mean of each bond length, angle and torsion in each of the input molecules.

Calculation of ideal molecule

The IDEAL link in the CALC program was used to produce a best fit of each input molecule onto a starting set of coordinates, which were then converted into orthogonal angstrom units and combined with their positional root mean square e.s.d's. The program below was then used to calculate the ideal molecule.

```
      DIMENSION F(3,10),E(10),V(10),S(3,10),S4(3,10),TOT(3),
     *RECIPV(3),SUM(3),SUM4(3),FID(3),EID2(3)
C SETS UP ARRAYS FOR COORDINATES, ESD'S  VARIANCES + THOSE USED
C IN CALCULATING WEIGHTED MEANS
      CHARACTER*4 L1
      READ(11,*)J
C  READS IN FROM TERMINAL THE NUMBER OF MOLECULES TO AVERAGE
```

```
 1     DO 10 K=1,3
       TOT(K)=0
       RECIPV(K)=0
       SUM(K)=0
       SUM4(K)=0
 10    CONTINUE
C     THIS INITIALISES THE ARRAY CONTENTS TO 0 BEFORE
C     READING EACH SET OF FIGURES
       DO 20I=1,J
C      ONCE FOR EACH MOLECULE
       RFAD(I,999)L1,(F(K,I),K=1,3),E(I)
       IF(L1.EQ.'    ')STOP
C FILE ENDING OF A BLANK LINE STOPS THE PROGRAM
       V(I)=1.0/(E(I)**2)
C RECIPROCAL VARIANCE
       DO 30 M=1,3
C FOR EACH OF X Y Z COORDS PER MOLECULE
       TOT(M)=TOT(M)+(F(M,I)*V(I))
C  WEIGHTED SUMS OF COORDS
       RECIPV(M)=RECIPV(M)+V(I)
C SUMS OF RECIPROCAL VARIANCE FOR EACH MOLECULE
       FID(M)=TOT(M)/RECIPV(M)
C  WEIGHTED COORD
 30    CONTINUE
 20    CONTINUE
       EID1=SQRT(1.0/RECIPV(1))
C  UNWEIGHTED ESD
       DO 40 I=1,J
       DO 50 K=1,3
C  CALCULATES WEIGHTED ESD OR SIGMA2
       S(K,I)=((FID(K)-F(K,I))*V(I))**2
       SUM(K)=SUM(K)+S(K,I)
       S4(K,I)= V(I)**2
       SUM4(K)=SUM4(K)+S4(K,I)
       EID2(K)=SQRT(SUM(K)/SUM4(K))
 50    CONTINUE
 40    CONTINUE
       EIDISO=SQRT((EID2(1)**2+(EID2(2)**2)+(EID2(3)**2))/3)
       EID=AMAX1(EID1,EIDISO)
       WRITE(12,1000)L1,(FID(K),K=1,3),(EID2(K),K=1,3)
      *EIDISO,EID1,EID
       GO TO 1
 999   FORMAT(A4,4X,4F10.5)
 1000  FORMAT(' ',A4,4X,9F10.5)
       END
```

Average X coord $X_m = \Sigma(X_i/(\sigma_i^2)) / (\Sigma(\sigma_i^{-2}))$

First e.s.d $\sigma_1 = \sqrt{(1/(\Sigma(\sigma_i^{-2})))}$

"Isotropic e.s.d" $\sigma_{iso} = \sqrt{(((\sigma_X^2)+(\sigma_Y^2)+(\sigma_Z^2))/3)}$

WHERE $\sigma_X = \sqrt{\ \Sigma((X_i-X_m)/(\sigma_i^2))/(\Sigma(\ 1/(\sigma_i^2)^2))}$

The ideal molecule with e.s.d's was read into CALC and used to produce a list of bonds, angles and torsions (column headed ID(I) in table below).

The second method involved using a modification of the above program on each molecule's ZIP output from CALC. (ZIP is the link of the CALC program that produces an output list of bonds, angles and torsion angles. The list may contain e.s.d's if requested and the e.s.d's of the atomic coordinates are supplied.)

```
      DIMENSION F(10),E(10),V(10),S(10),S4(10)
      CHARACTER*6 L1,L2,L3,L4
      READ(11,*)J
    1 TOT=0
      RECIPV=0
      SUM=0
      SUM4=0
      DO 20I=1,J
      READ(I,999)L1,L2,L3,L4,F(I),E(I)
      IF(L1.EQ.'      ')STOP
      V(I)=1.0/(E(I)**2)
      TOT=TOT+(F(I)*V(I))
      RECIPV=RECIPV+V(I)
      FID=TOT/RECIPV
   20 CONTINUE
      EID1=SQRT(1.0/RECIPV)
      DO 40 I=1,J
      S(I)=((FID-F(I))*V(I))**2
      SUM=SUM+S(I)
      S4(I)= V(I)**2
      SUM4=SUM4+S4(I)
      EID2=SQRT(SUM/SUM4)
   40 CONTINUE
```

```
      EID=AMAX1(EID1,EID2)
      WRITE(12,1000)L1,L2,L3,L4,FID,EID2,EID1,EID
      GO TO 1
999   FORMAT(4A6,2F10.4)
1000  FORMAT(' ',4A6,4F10.4)
      END
```

The result of this program is tabulated as ID(BAT) and has fractionally better e.s.d's than the ID(I) output. This is most noticeable around the oxepin ring, where there is most conformational difference between individual structures. As the bond lengths and angles are nearly the same in each case, the ideal parameters calculated on this basis have small weighted e.s.d's. For the parameters calculated from the averaged coordinates however, the e.s.d's are a lot larger, as there is a greater difference in actual atomic position relative to the origin of the coordinate system here than in any other part of the molecule and the average position for the atoms of this ring are closer to the molecular centre, leading to distorted, shorter bond lengths.

LABEL	CL1	CL2	I	NIT	AL	ID(I)	ID(BAT)
C1 C2	1.386(5)	1.376(5)	1.375(14)	1.401(14)	1.376(8)	1.386(13)	1.381(5)
C1 C6	1.391(5)	1.382(5)	1.402(13)	1.368(12)	1.385(7)	1.391(17)	1.386(5)
C1 N1	1.410(5)	1.406(5)	1.404(12)	1.396(11)	1.432(7)	1.410(13)	1.411(8)
C2 C3	1.399(6)	1.366(6)	1.382(16)	1.396(16)	1.377(8)	1.398(20)	1.382(15)
C3 C4	1.365(7)	1.384(7)	1.365(17)	1.328(17)	1.399(9)	1.36(3)	1.376(14)
C4 C5	1.376(6)	1.388(7)	1.364(16)	1.399(16)	1.382(9)	1.38(4)	1.382(5)
C5 C6	1.384(5)	1.383(5)	1.379(14)	1.398(13)	1.394(8)	1.38(3)	1.386(4)
C6 C7	1.495(5)	1.510(5)	1.488(12)	1.515(12)	1.512(7)	1.495(20)	1.504(8)
C7 C8	1.554(5)	1.542(5)	1.552(12)	1.553(12)	1.569(7)	1.554(15)	1.552(8)
C7 C9	1.526(5)	1.534(5)	1.524(12)	1.533(12)	1.515(7)	1.53(3)	1.527(6)
C7 C11	1.529(5)	1.527(5)	1.514(12)	1.559(13)	1.539(7)	1.53(3)	1.531(5)
C8 C14	1.533(5)	1.515(5)	1.479(12)	1.541(13)	1.531(7)	1.534(14)	1.524(10)
C8 N1	1.473(4)	1.483(4)	1.493(11)	1.483(11)	1.500(6)	1.473(12)	1.483(8)
C9 C12	1.504(5)	1.505(5)	1.476(13)	1.530(13)	1.516(8)	1.50(3)	1.506(5)
C9 N2	1.530(5)	1.543(5)	1.545(12)	1.543(11)	1.538(7)	1.53(4)	1.538(7)
C10 C11	1.512(5)	1.510(5)	1.499(13)	1.478(14)	1.528(8)	1.51(4)	1.511(6)
C10 N2	1.491(5)	1.507(5)	1.484(13)	1.518(12)	1.491(8)	1.50(4)	1.498(8)
C12 C13	1.541(5)	1.521(5)	1.540(13)	1.518(13)	1.516(8)	1.541(24)	1.529(10)
C13 C14	1.513(5)	1.521(5)	1.546(12)	1.524(13)	1.546(8)	1.513(19)	1.523(10)
C13 C16	1.512(5)	1.517(5)	1.526(12)	1.518(12)	1.510(8)	1.512(17)	1.515(3)
C14 C15	1.525(5)	1.535(5)	1.547(12)	1.512(13)	1.520(8)	1.52(3)	1.529(6)
C15 C16	1.491(5)	1.479(5)	1.473(14)	1.503(13)	1.514(9)	1.493(19)	1.490(10)
C15 N2	1.497(5)	1.490(5)	1.509(14)	1.498(12)	1.494(8)	1.49(3)	1.494(4)
C16 C17	1.314(5)	1.315(5)	1.327(14)	1.277(14)	1.315(8)	1.314(16)	1.313(4)
C17 C18	1.485(6)	1.488(6)	1.495(16)	1.488(16)	1.507(9)	1.48(3)	1.491(6)
C18 O2	1.415(5)	1.423(5)	1.412(14)	1.432(14)	1.429(8)	1.41(3)	1.421(5)
C19 C20	1.501(5)	1.523(6)	1.535(14)	1.522(16)	1.531(9)	1.50(4)	1.516(12)
C19 O2	1.426(5)	1.424(5)	1.415(12)	1.435(12)	1.440(7)	1.42(3)	1.427(5)
C20 C21	1.504(5)	1.481(6)	1.489(15)	1.512(16)	1.521(9)	1.50(4)	1.498(14)
C21 O1	1.216(5)	1.213(5)	1.194(14)	1.216(12)	1.206(7)	1.22(4)	1.212(4)
C21 N1	1.368(5)	1.373(5)	1.365(13)	1.373(12)	1.363(7)	1.37(3)	1.369(4)

LABEL	CL1	CL2	I	NIT	AL	ID(I)	ID(BAT)
C2 C1 C6	121.2(3)	121.6(3)	121.7(9)	119.5(9)	122.0(5)	121.2(9)	121.4(4)
C2 C1 N1	128.6(3)	128.5(3)	128.4(9)	128.2(8)	127.9(5)	128.6(8)	128.38(23)
C6 C1 N1	110.3(3)	109.9(3)	109.9(8)	112.3(8)	110.1(4)	110.3(9)	110.2(3)
C1 C2 C3	117.3(4)	117.7(4)	117.3(10)	116.2(10)	117.9(5)	117.3(10)	117.6(3)
C2 C3 C4	121.9(4)	121.4(4)	121.3(11)	121.2(11)	121.3(6)	121.8(17)	121.6(3)
C3 C4 C5	120.3(4)	121.0(4)	121.6(11)	122.3(11)	120.0(6)	120.2(24)	120.6(4)
C4 C5 C6	119.5(4)	117.3(4)	116.9(10)	116.6(9)	119.0(6)	119.6(23)	118.4(11)
C1 C6 C5	119.9(3)	120.8(3)	119.1(9)	121.9(8)	119.7(5)	119.8(16)	120.3(5)
C1 C6 C7	109.6(3)	110.6(3)	110.5(8)	109.1(7)	111.4(4)	109.6(11)	110.3(6)
C5 C6 C7	130.4(3)	128.3(3)	130.0(8)	128.8(8)	128.8(5)	130.4(16)	129.2(10)
C6 C7 C8	103.4(3)	102.1(3)	102.5(7)	102.6(7)	102.1(4)	103.4(10)	102.6(7)
C6 C7 C9	115.2(3)	114.5(3)	113.3(7)	114.4(7)	116.5(4)	115.1(13)	115.0(6)
C6 C7 C11	112.6(3)	112.6(3)	114.2(7)	111.6(7)	111.4(4)	112.6(14)	112.4(4)
C8 C7 C9	113.5(3)	114.3(3)	113.4(7)	116.8(7)	113.8(4)	113.6(12)	114.0(5)
C8 C7 C11	111.1(3)	112.3(3)	111.6(7)	111.1(7)	111.2(4)	111.0(13)	111.6(6)
C9 C7 C11	101.4(3)	101.5(3)	102.2(7)	100.8(7)	102.2(4)	101.5(15)	101.58(25)
C7 C8 C14	117.3(3)	116.7(3)	118.3(7)	115.2(7)	116.6(4)	117.3(8)	116.9(4)
C7 C8 N1	104.0(3)	104.7(3)	104.3(6)	104.6(7)	105.0(4)	104.1(7)	104.5(4)
C14 C8 N1	105.4(3)	106.3(3)	106.4(7)	106.6(7)	105.7(4)	105.4(7)	105.9(4)
C7 C9 C12	115.5(3)	116.3(3)	116.8(7)	114.2(7)	116.6(5)	115.6(16)	115.9(5)
C7 C9 N2	104.9(3)	104.1(3)	104.4(7)	104.7(6)	105.4(4)	105.0(18)	104.7(4)
C12 C9 N2	110.9(3)	109.8(3)	109.9(7)	108.8(7)	110.2(4)	110.7(19)	110.2(6)
C11 C10 N2	104.3(3)	104.1(3)	104.9(7)	105.4(8)	105.1(5)	104.2(21)	104.4(3)
C7 C11 C10	103.5(3)	103.9(3)	105.3(7)	104.4(8)	102.9(4)	103.5(18)	103.7(4)
C9 C12 C13	108.2(3)	108.6(3)	109.8(8)	108.9(7)	107.8(5)	108.3(15)	108.4(3)
C12 C13 C14	106.4(3)	107.1(3)	105.4(7)	107.2(7)	106.5(4)	106.4(12)	106.6(4)
C12 C13 C16	110.1(3)	108.9(3)	107.9(7)	110.5(7)	111.1(5)	110.1(12)	109.7(7)
C14 C13 C16	114.0(3)	113.7(3)	112.8(7)	113.5(7)	113.6(4)	114.0(11)	113.74(24)
C8 C14 C13	112.0(3)	113.5(3)	113.3(7)	111.9(7)	112.4(4)	112.0(10)	112.7(7)
C8 C14 C15	107.7(3)	107.9(3)	109.3(7)	106.5(7)	108.3(4)	107.7(12)	107.9(3)
C13 C14 C15	119.1(3)	118.6(3)	116.7(7)	119.1(8)	118.9(4)	119.1(13)	118.7(3)
C16 C15 N2	110.2(3)	110.4(3)	109.3(8)	110.3(7)	109.8(5)	110.2(15)	110.19(21)
C13 C16 C15	115.4(3)	116.2(3)	116.1(8)	113.4(7)	114.4(5)	115.5(10)	115.5(6)
C13 C16 C17	123.0(3)	122.6(3)	120.8(8)	122.8(9)	123.5(5)	123.1(10)	122.8(4)
C15 C16 C17	121.5(3)	121.2(3)	123.0(9)	123.8(9)	122.0(5)	121.5(11)	121.7(4)
C16 C17 C18	121.6(4)	121.7(4)	123.5(10)	124.9(10)	121.8(6)	121.6(14)	121.9(4)
C17 C18 O2	111.2(4)	112.1(4)	110.8(9)	112.2(9)	111.4(5)	111.3(20)	111.6(4)
C14 C19 C20	110.8(3)	110.3(3)	108.3(8)	111.0(8)	110.1(5)	110.8(20)	110.4(4)
C14 C19 O2	114.5(3)	113.8(3)	113.6(7)	116.3(8)	114.8(5)	114.5(19)	114.3(5)
C20 C19 O2	103.8(3)	103.3(3)	103.0(7)	102.7(8)	104.6(5)	103.9(21)	103.7(4)
C19 C20 C21	117.0(3)	117.3(3)	118.0(9)	118.0(9)	115.6(5)	117.0(24)	117.0(5)
C20 C21 O1	123.2(4)	122.9(4)	122.7(10)	121.7(9)	123.0(5)	123 (3)	122.96(22)
C20 C21 N1	114.9(3)	114.9(3)	115.1(9)	115.1(9)	113.6(5)	114.9(20)	114.7(4)
O1 C21 N1	121.9(3)	122.2(4)	122.1(10)	123.2(9)	123.4(5)	122 (3)	122.3(4)
C18 O2 C19	116.1(3)	115.0(3)	114.5(8)	112.7(8)	115.8(4)	116.4(21)	115.4(6)
C1 N1 C8	109.6(3)	109.3(3)	108.6(7)	108.6(7)	108.7(4)	109.6(8)	109.2(3)
C1 N1 C21	125.7(3)	124.6(3)	124.7(8)	124.8(8)	124.1(4)	125.7(13)	124.9(6)
C8 N1 C21	118.5(3)	118.6(3)	118.5(8)	119.2(7)	118.9(4)	118.6(13)	118.63(17)
C9 N2 C10	107.6(3)	107.5(3)	107.8(7)	106.9(6)	107.2(4)	107.5(22)	107.48(17)
C9 N2 C15	113.9(3)	113.7(3)	113.2(7)	113.7(6)	114.1(5)	114.3(21)	113.79(17)
C10 N2 C15	112.5(3)	113.3(3)	112.9(8)	113.2(7)	113.7(5)	112.1(22)	113.1(4)

LABEL				CL1	CL2	I	NIT	AL	ID(I)	ID(BAT)
C6	C1	C2	C3	0 9(6)	0 1(6)	0.7(15)	4.7(15)	3.5(8)	1.0(16)	1.3(12)
N1	C1	C2	C3	181.6(4)	181.5(4)	179.9(10)	184.0(9)	182 9(5)	-178.2(11)	181.9(6)
C2	C1	C6	C5	-0 6(6)	1.3(6)	0.8(14)	-2.9(14)	-2 4(8)	-0.6(21)	-0 3(13)
C2	C1	C6	C7	175 2(3)	175.9(3)	174.8(9)	172 9(8)	174.0(5)	175.2(10)	175 1(7)
N1	C1	C6	C5	178 7(3)	180 2(3)	181 4(8)	177 7(8)	178 1(5)	178 7(16)	179.2(8)
N1	C1	C6	C7	-5 4(4)	-5.3(4)	-4 6(10)	-6.5(10)	-5.4(6)	-5.5(14)	-5.36(24)
C2	C1	N1	C8	172 8(4)	171.4(4)	171.5(9)	175 8(9)	174.9(5)	172.9(8)	172 8(12)
C2	C1	N1	C21	21 2(6)	22 4(6)	23.8(15)	26 5(15)	27.2(8)	21.1(20)	23.2(20)
C6	C1	N1	C8	-6.9(4)	-7.4(4)	-9.2(10)	-4.9(10)	-5.7(6)	-6.4(12)	-6.7(6)
C6	C1	N1	C21	-158 2(3)	-156 3(3)	-156 9(9)	-154 2(8)	-153 4(5)	-158 2(16)	-156.4(15)
C1	C2	C3	C4	0 0(7)	0.4(7)	-0.2(17)	-2 8(17)	-2 9(9)	0 0(24)	-0.6(11)
C2	C3	C4	C5	-1 0(7)	-2 4(7)	-1 7(19)	-1.0(19)	1.1(10)	-1 (4)	-1.1(12)
C3	C4	C5	C6	1.3(7)	3 7(7)	3.2(17)	2 8(17)	0 0(9)	2 (4)	2.1(14)
C4	C5	C6	C1	-0 5(6)	-3.2(6)	-2.7(15)	-0.9(14)	0.6(8)	-1 (3)	-1.4(15)
C4	C5	C6	C7	-175.3(4)	-176.6(4)	-175.3(10)	-175 7(9)	-175.2(5)	-175.4(20)	-175.8(7)
C1	C6	C7	C8	14.1(4)	14.8(4)	15.5(9)	14.3(9)	13.4(5)	14.2(13)	14.4(5)
C1	C6	C7	C9	138.6(3)	138.9(3)	138.1(8)	141.7(8)	138.1(5)	138.6(14)	138.8(4)
C1	C6	C7	C11	-105.6(3)	-105.8(3)	-105.4(9)	-104.7(8)	-105.3(5)	-105.7(16)	-105.63(22)
C5	C6	C7	C8	-170.6(4)	-171.1(4)	-171.4(10)	-170.3(9)	-170.5(5)	-170.6(19)	-170.8(3)
C5	C6	C7	C9	-46.1(5)	-47.1(5)	-46.8(13)	-42.9(12)	-45.8(8)	-46 (3)	-46 4(7)
C5	C6	C7	C11	69.5(5)	68.2(5)	67.7(12)	70.7(11)	70.8(7)	69.5(25)	69.2(9)
C6	C7	C8	C14	98.9(3)	99.0(3)	98.1(8)	100.3(8)	100.7(5)	98.9(11)	99.3(6)
C6	C7	C8	N1	-17 1(3)	-18.1(3)	-19.8(8)	-16.4(8)	-15.9(5)	-17.1(11)	-17.4(8)
C9	C7	C8	C14	-26.6(4)	-25.3(4)	-24.4(10)	-25.7(11)	-25.7(6)	-26.6(15)	-25.8(7)
C9	C7	C8	N1	-142.6(3)	-142.4(3)	-142.4(7)	-142.3(7)	-142.3(4)	-142.5(12)	-142.43(17)
C11	C7	C8	C14	-140.1(3)	-140.2(3)	-139.2(8)	-140.4(8)	-140.4(4)	-140.1(13)	-140.18(18)
C11	C7	C8	N1	103.9(3)	102.7(3)	102.8(8)	102.9(8)	103.0(4)	103.9(13)	103.2(6)
C6	C7	C9	C12	-83 4(4)	-83.9(4)	-84.3(9)	-87.8(9)	-83.6(6)	-83.4(19)	-84.0(6)
C6	C7	C9	N2	154.2(3)	155.2(3)	154.1(7)	153.3(7)	153.9(4)	154.3(16)	154.5(6)
C8	C7	C9	C12	35.6(4)	33.3(4)	32.0(10)	32.0(10)	34.9(6)	35.6(21)	34.2(12)
C8	C7	C9	N2	-86.8(3)	-87.5(3)	-89.6(8)	-86.9(8)	-87.7(5)	-86.7(18)	-87.4(5)
C11	C7	C9	C12	154.8(3)	154.5(3)	152.3(8)	152 4(7)	154.8(5)	154.7(18)	154.4(4)
C11	C7	C9	N2	32 4(3)	33.6(3)	30.7(8)	33.6(8)	32.2(5)	32.4(20)	32.8(7)
C6	C7	C11	C10	-166.6(3)	-166.8(3)	-162.8(7)	-164.5(7)	-166.7(4)	-166.6(16)	-166.4(6)
C8	C7	C11	C10	78.0(3)	78.6(3)	81.6(8)	81.7(9)	80.2(5)	78.0(18)	79.0(9)
C9	C7	C11	C10	-43.0(3)	-43.9(3)	-40.0(8)	-42.7(9)	-41.6(5)	-43.0(20)	-42.9(8)
C7	C8	C14	C13	41.0(4)	40.2(4)	40.3(10)	41.4(10)	40.0(6)	41.0(13)	40.5(4)
C7	C8	C14	C15	173.8(3)	173.8(3)	172 3(7)	173.1(7)	173.4(4)	173.8(12)	173.59(22)
N1	C8	C14	C13	156.2(3)	156.5(3)	157.2(7)	156.9(7)	156.1(4)	156.2(10)	156.37(19)
N1	C8	C14	C15	-71.0(3)	-70.0(3)	-70.8(8)	-71.4(9)	-70.4(5)	-71.0(13)	-70.6(5)
C7	C8	N1	C1	14.9(3)	16 4(3)	18.4(9)	13.6(9)	13.8(5)	14.8(10)	15.4(10)
C7	C8	N1	C21	168.9(3)	167.9(3)	168.4(8)	164.9(7)	163.4(4)	168.9(14)	167.2(16)

LABEL				CL1	CL2	I	NIT	AL	ID(I)	ID(BAT)
C14	CE	N1	C1	-109.2(3)	-107.7(3)	-107.5(8)	-108.9(8)	-110.0(4)	-109.2(9)	-108.7(9)
C14	C8	N1	C21	44.8(4)	43.5(4)	42.6(10)	42.5(10)	39.6(6)	44.9(19)	43.3(19)
C7	C9	C12	C13	-57.7(4)	-54.9(4)	-55.0(10)	-53.8(10)	-57.7(6)	-57.7(21)	-56.3(14)
N2	C9	C12	C13	61.4(4)	62.9(4)	63.6(9)	62.6(9)	62.3(6)	61.6(22)	62.3(8)
C7	C9	N2	C10	-10.4(4)	-11.8(4)	-11.2(9)	-13.8(8)	-10.9(5)	-10.4(25)	-11.3(7)
C7	C9	N2	C15	115.1(3)	114.5(3)	114.4(8)	111.9(7)	115.9(5)	114.7(21)	114.7(5)
C12	C9	N2	C10	-135.7(3)	-136.9(3)	-137.2(8)	-136.3(7)	-137.5(5)	-135.8(20)	-136.6(7)
C12	C9	N2	C15	-10.3(4)	-10.6(4)	-11.6(10)	-10.6(9)	-10.6(6)	-11 (3)	-10.55(24)
N2	C10	C11	C7	37.0(4)	36.7(4)	33.3(9)	34.7(9)	35.3(5)	37.0(24)	36.3(7)
C11	C10	N2	C9	-16.4(4)	-15.2(4)	-13.3(9)	-13.2(9)	-15.3(6)	-16 (3)	-15.4(8)
C11	C10	N2	C15	-142.7(3)	-141.7(3)	-139.0(8)	-139.1(8)	-142.3(5)	-142.8(21)	-141.8(7)
C9	C12	C13	C14	69.3(3)	66.7(4)	66.4(9)	69.1(9)	68.7(6)	69.2(17)	68.1(12)
C9	C12	C13	C16	-54.7(4)	-56.6(4)	-54.3(9)	-55.0(9)	-55.5(6)	-54.8(17)	-55.5(9)
C12	C13	C14	CE	-61.0(3)	-60.5(4)	-59.6(9)	-63.2(9)	-61.3(6)	-60.9(14)	-60.9(4)
C12	C13	C14	C15	172.2(3)	171.3(3)	172.1(7)	171.8(8)	170.7(5)	172.2(15)	171.6(5)
C16	C13	C14	CE	60.6(4)	59.8(4)	57.9(10)	59.1(10)	61.4(6)	60.6(14)	60.2(6)
C16	C13	C14	C15	-66.3(4)	-68.4(4)	-70.4(10)	-65.9(11)	-66.6(6)	-66.3(18)	-67.4(11)
C12	C13	C16	C15	-2.1(4)	-1.0(4)	-6.1(11)	-4.2(10)	-1.9(7)	-2.0(16)	-2.0(8)
C12	C13	C16	C17	179.2(4)	178.5(3)	177.0(9)	176.8(9)	180.1(6)	179.2(13)	178.8(6)
C14	C13	C16	C15	-121.6(3)	-120.3(3)	-122.1(9)	-124.7(8)	-122.0(5)	-121.5(12)	-121.4(9)
C14	C13	C16	C17	59.7(5)	59.2(5)	61.0(11)	56.4(12)	60.0(7)	59.7(16)	59.4(4)
CE	C14	C19	C20	41.0(4)	40.9(4)	42.6(9)	46.6(10)	41.5(6)	41.0(22)	41.4(7)
CE	C14	C19	O2	-76.0(4)	-74.6(4)	-71.2(9)	-70.3(10)	-76.2(5)	-76.1(20)	-75.0(10)
C13	C14	C19	C20	169.9(3)	171.7(3)	172.8(8)	174.2(8)	171.4(5)	169.9(17)	171.2(10)
C13	C14	C19	O2	-52.9(4)	56.1(4)	59.0(10)	57.3(11)	53.7(7)	52.8(24)	54.8(17)
N2	C15	C16	C13	52.8(4)	52.4(4)	56.0(11)	56.0(10)	53.1(7)	52.3(18)	53.1(7)
N2	C15	C16	C17	-126.4(4)	-127.1(4)	-127.2(10)	-125.0(10)	-128.8(6)	-128.8(17)	-127.7(7)
C16	C15	N2	C9	-45.9(4)	-45.2(4)	-46.0(10)	-47.2(9)	-45.7(6)	-45.3(25)	-45.7(4)
C16	C15	N2	C10	77.0(4)	78.0(4)	76.8(10)	75.0(9)	77.7(6)	77.3(23)	77.3(6)
C13	C16	C17	C18	-2.7(6)	-1.9(6)	-3.5(15)	-1.2(16)	-3.7(9)	-2.8(21)	-2.5(6)
C15	C16	C17	C18	178.6(4)	177.6(4)	179.8(10)	179.9(10)	178.4(6)	178.4(16)	178.3(6)
C16	C17	C18	O2	-65.6(5)	-66.2(5)	-65.1(13)	-65.6(14)	-64.6(8)	-65.2(24)	-65.6(5)
C17	C18	O2	C19	88.2(4)	88.3(4)	89.4(10)	84.4(10)	87.1(6)	87.9(25)	87.9(6)
C14	C19	C20	C21	13.4(5)	13.6(5)	12.1(12)	4.6(13)	15.5(7)	13 (3)	13.3(10)
O2	C19	C20	C21	136.8(3)	135.6(4)	132.6(9)	129.6(9)	139.4(5)	136.8(24)	136.2(13)
C14	C19	O2	C18	-64.6(4)	-65.7(4)	-69.8(10)	-65.7(10)	-64.9(6)	-64 (3)	-65.4(8)
C20	C19	O2	C18	174.5(3)	174.7(3)	173.3(8)	172.9(8)	174.2(5)	174.7(22)	174.4(3)
C19	C20	C21	N1	-42.1(5)	-42.6(5)	-41.3(13)	-35.7(13)	-47.9(7)	-42 (3)	-42.9(18)
C19	C20	C21	O1	137.4(4)	138.3(4)	137.0(11)	142.5(10)	133.2(6)	138 (3)	137.3(14)
C20	C21	N1	C1	159.4(3)	158.0(3)	156.9(9)	156.2(9)	162.1(5)	159.3(17)	159.1(12)
C20	C21	N1	CE	9.9(5)	11.7(5)	12.0(13)	9.8(12)	17.4(7)	10 (3)	11.9(22)
O1	C21	N1	C1	-20.2(6)	-22.8(6)	-21.5(16)	-22.1(15)	-19.1(9)	-21 (4)	-21.1(14)
O1	C21	N1	CE	-169.6(3)	-169.2(3)	-166.3(10)	-168.4(9)	-163.8(5)	-170.0(25)	-168.3(17)

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(1)	0.7460(11)	-0.18600(25)	0.6686(13)	0.0423
C(2)	0.6199(15)	-0.2198(3)	0.6665(17)	0.0566
C(3)	0.5402(14)	-0.2308(3)	0.5151(18)	0.0599
C(4)	0.5763(16)	-0.2088(4)	0.3776(17)	0.0606
C(5)	0.6931(13)	-0.1733(3)	0.3764(13)	0.0450
C(6)	0.7774(10)	-0.16296(25)	0.5268(12)	0.0405
C(7)	0.8999(11)	-0.1252(3)	0.5668(11)	0.0392
C(8)	0.9734(12)	-0.1370(3)	0.7416(12)	0.0349
C(9)	1.0412(11)	-0.11502(25)	0.4327(11)	0.0378
C(10)	0.9334(13)	-0.0482(3)	0.5638(14)	0.0486
C(11)	0.7921(12)	-0.0819(3)	0.5714(14)	0.0447
C(12)	1.2189(12)	-0.1401(3)	0.4509(12)	0.0459
C(13)	1.2930(13)	-0.1332(3)	0.6247(11)	0.0417
C(14)	1.1659(12)	-0.1564(3)	0.7443(12)	0.0414
C(15)	1.2688(12)	-0.0571(3)	0.5151(13)	0.0463
C(16)	1.3130(11)	-0.0854(3)	0.6611(12)	0.0394
C(17)	1.3651(14)	-0.0710(3)	0.8018(14)	0.0551
C(18)	1.4095(17)	-0.0982(4)	0.9483(15)	0.0690
C(19)	1.2194(15)	-0.1602(3)	0.9249(12)	0.0483
C(20)	1.0672(18)	-0.1802(4)	1.0268(14)	0.0707
C(21)	0.9001(14)	-0.1958(3)	0.9360(13)	0.0534
N(1)	0.8493(10)	-0.17117(20)	0.8022(9)	0.0429
N(2)	1.0812(9)	-0.06649(20)	0.4532(10)	0.0386
O(1)	0.8205(11)	-0.22837(21)	0.9776(10)	0.0799
O(2)	1.2539(9)	-0.12056(19)	1.0110(8)	0.0588
N(3)	0.9126(15)	-0.0425(3)	0.0735(11)	0.0672
O(3)	0.8160(12)	-0.0708(3)	0.1399(13)	0.1098
O(4)	1.0521(12)	-0.03187(21)	0.1474(10)	0.0742
O(5)	0.8679(14)	-0.0270(3)	-0.0577(10)	0.1097

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(2)	0.598(11)	-0.2335(25)	0.759(10)	0.0323
H(3)	0.453(10)	-0.2537(24)	0.524(10)	0.0323
H(4)	0.544(12)	-0.206(3)	0.294(11)	0.0323
H(5)	0.711(10)	-0.1522(22)	0.292(10)	0.0323
H(8)	0.955(10)	-0.1136(23)	0.811(9)	0.0323
H(9)	0.979(10)	-0.1192(23)	0.300(9)	0.0323
H(101)	0.903(9)	-0.0218(22)	0.486(9)	0.0323
H(102)	0.988(11)	-0.0469(22)	0.679(10)	0.0323
H(111)	0.713(11)	-0.0848(23)	0.675(10)	0.0323
H(112)	0.709(10)	-0.0802(22)	0.479(9)	0.0323
H(121)	1.193(11)	-0.1681(22)	0.428(10)	0.0323
H(122)	1.307(10)	-0.1308(21)	0.339(10)	0.0323
H(13)	1.405(10)	-0.1460(22)	0.652(10)	0.0323
H(14)	1.165(10)	-0.1845(23)	0.703(9)	0.0323
H(151)	1.350(10)	-0.0605(22)	0.410(10)	0.0323
H(152)	1.263(10)	-0.0249(21)	0.538(9)	0.0323
H(171)	1.369(10)	-0.0419(22)	0.829(9)	0.0323
H(121)	1.426(10)	-0.0756(22)	1.037(10)	0.0323
H(122)	1.514(10)	-0.1190(22)	0.906(10)	0.0323
H(19)	1.332(10)	-0.1771(21)	0.929(10)	0.0323
H(201)	1.041(11)	-0.1656(23)	1.124(11)	0.0323
H(202)	1.120(10)	-0.1992(22)	1.120(10)	0.0323
H(1N2)	1.051(11)	-0.0539(23)	0.357(10)	0.0323

Thermal Vibration Parameters with Standard Deviations

	U or U11	U22	U33	U23	U13	U12
C(1)	0.0301(52)	0.0278(44)	0.0691(67)	0.0077(45)	0.0100(51)	-0.0030(42)
C(2)	0.0422(61)	0.0436(61)	0.0839(87)	0.0043(58)	0.0023(67)	0.0105(55)
C(3)	0.0439(59)	0.0289(47)	0.1071(110)	-0.0088(63)	-0.0025(73)	-0.0011(49)
C(4)	0.0439(67)	0.0668(71)	0.0710(90)	-0.0238(71)	-0.0219(64)	0.0059(61)
C(5)	0.0299(49)	0.0396(49)	0.0654(71)	-0.0039(47)	-0.0036(51)	0.0040(48)
C(6)	0.0192(41)	0.0371(47)	0.0652(66)	-0.0045(45)	0.0113(48)	0.0040(39)
C(7)	0.0389(47)	0.0374(46)	0.0414(50)	-0.0063(44)	0.0002(46)	0.0112(43)
C(8)	0.0358(53)	0.0278(47)	0.0411(53)	-0.0109(39)	0.0066(43)	0.0030(41)
C(9)	0.0286(44)	0.0380(47)	0.0467(54)	-0.0065(46)	0.0005(44)	0.0017(41)
C(10)	0.0459(60)	0.0510(56)	0.0488(61)	0.0056(51)	-0.0076(53)	0.0078(50)
C(11)	0.0289(50)	0.0497(55)	0.0555(59)	0.0018(54)	-0.0057(49)	0.0085(47)
C(12)	0.0407(56)	0.0446(52)	0.0523(66)	-0.0071(48)	0.0034(55)	0.0059(49)
C(13)	0.0328(51)	0.0477(52)	0.0445(56)	0.0000(45)	-0.0089(45)	0.0171(47)
C(14)	0.0381(56)	0.0311(46)	0.0551(62)	-0.0137(44)	0.0025(50)	0.0072(43)
C(15)	0.0366(52)	0.0485(56)	0.0539(64)	0.0015(50)	-0.0009(52)	-0.0120(45)
C(16)	0.0269(45)	0.0408(46)	0.0505(59)	-0.0005(48)	-0.0004(45)	-0.0059(44)
C(17)	0.0479(60)	0.0566(58)	0.0608(71)	-0.0072(57)	0.0036(56)	-0.0114(59)
C(18)	0.0712(79)	0.0870(88)	0.0489(72)	-0.0026(62)	-0.0104(70)	-0.0171(71)
C(19)	0.0584(63)	0.0513(58)	0.0351(53)	0.0016(46)	-0.0058(54)	0.0037(55)
C(20)	0.1031(95)	0.0697(77)	0.0393(67)	0.0083(58)	-0.0161(72)	-0.0146(74)
C(21)	0.0622(65)	0.0370(50)	0.0612(66)	-0.0025(55)	0.0163(62)	0.0088(52)
N(1)	0.0539(50)	0.0263(35)	0.0486(49)	0.0036(35)	0.0042(42)	0.0004(36)
N(2)	0.0381(41)	0.0345(39)	0.0432(49)	0.0118(36)	-0.0074(41)	0.0017(36)
O(1)	0.0985(56)	0.0603(39)	0.0807(56)	0.0327(40)	-0.0015(53)	-0.0259(44)
O(2)	0.0663(45)	0.0551(38)	0.0549(42)	-0.0089(33)	-0.0041(39)	-0.0097(39)
N(3)	0.0852(79)	0.0751(67)	0.0414(57)	0.0001(51)	-0.0103(58)	0.0364(65)
O(3)	0.0763(61)	0.1391(80)	0.1141(82)	0.0281(68)	-0.0331(61)	-0.0291(64)
O(4)	0.1054(64)	0.0573(44)	0.0599(49)	0.0074(39)	-0.0114(52)	-0.0118(47)
O(5)	0.1519(90)	0.1298(72)	0.0475(50)	0.0058(52)	-0.0141(60)	0.0630(69)

CHAPTER NINE
BRUCINE ETHANOLATE DIHYDRATE

BRUCINE ETHANOL DIHYDRATE

Introduction

Brucine is an indole alkaloid of the strychnine family isolated from the seeds of *strychnos nux vomica*. The ring system in brucine is identical to that of strychnine both in connectivity and in stereochemistry, with the only difference between the molecules being the presence of two methoxy groups on the aromatic ring. This alteration results in a great difference in physiological effect between strychnine and brucine. Although the main effect of brucine is a paralysis caused by a blockage of the neuromuscular junction, it does have strychnine-like effects on the central nervous system when it is present in sufficiently high concentration. This would imply that the addition of the methoxy groups does not completely prevent binding of brucine to the strychnine receptor. Perhaps the most important use of brucine and strychnine is in the optical resolution of optically active acids or alcohols (Fischer, 1899; Toda and Tanaka, 1981) by selective crystallisation of the diastereomeric salts and subsequent alkaline treatment to afford the desired enantiomer as the free acid. Although strychnine and brucine have the same configuration at all of their chiral centres, there is a distinct difference in the relative solubilities of their D and L amino acid salts. In the majority of cases the brucine salt of the D amino acid is far less soluble than the corresponding L amino acid salt, while the opposite tends to hold true for the strychnine salts (Greenstein and Winitz, 1961). As well as providing information on the mechanisms involved in chiral recognition, study of the solid state structures of these complexes provide a useful model system for the examination of drug receptor interactions.

Crystallisation

Brucine ethanol dihydrate crystals were grown from a solution of brucine tetrahydrate in absolute ethanol that was allowed to evaporate slowly at room temperature. The crystals were in the form of transparent cuboids which crumbled to an opaque powder after about thirty minutes exposure to air, so it was necessary to mount the

crystal used for data collection in a glass tube containing a small amount of mother liquor to prevent its decomposition.

Crystal Data

Formula $C_{23}H_{26}N_2O_4.C_2H_5OH.2H_2O$ Mol. Wt. 476.5 F(000) = 1024

Space group $P2_12_12_1$ Int. Tab. No. 19

Cell dimensions a = 7.723(1) b = 12.337(1) c = 25.212(2) A
V = 2402.8 A^3 Z = 4 Dc = 1.317 gcm^{-3}
Radiation MoKα λ = 0.71069 A μ = 0.90 cm^{-1}
Final R = 0.0387 based on 1684 independent data
h_{max} = 9 k_{max} = 14 l_{max} = 29

θ_{max} = 25° $\sin\theta_{max}/\lambda$ = 0.5947

2 intensity controls (0 6 1 and 0 0 12)
Drift curve min. 0.981 max. 1.049
2459 reflections measured (26 systematic absences)
749 reflections considered unobserved ($F<4\sigma(F)$)
Weighting scheme: $W = 1.5864/(\sigma^2(F)+0.001733F^2)$
401 parameters refined
Final difference Fourier max. 0.1439 min. -0.1436 eA^{-3}
Max. shift/σ = 0.176

Data collection and reduction

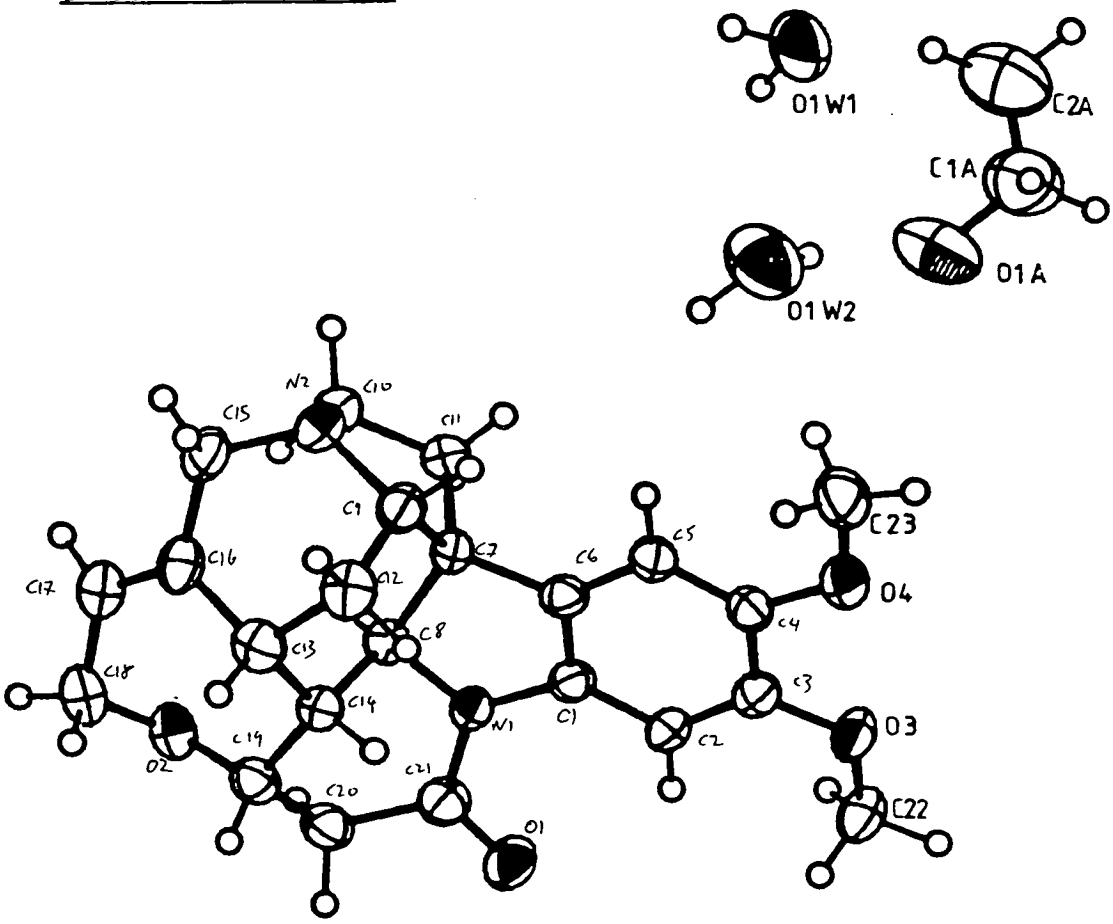
The data was collected on the CAD4 and corrected for Lorentz and polarisation effects using the program CADABS.

Solution and refinement

The structure was solved automatically using the MULTAN77 system of programs. The first electron density map to be examined contained the entire brucine skeleton with the exception of two carbon atoms. Difference Fourier maps calculated by the program SHELX were used to locate the remaining atoms and to refine the structure. The course of the least-squares refinement is summarised below.

R	Model
0.3954	$C_{21}N_2O_4$ as input fragment
0.1775	$C_{23}N_2O_4.2O.C_2O$ (all non-H atoms)
0.1057	All atoms isotropic, 14 H atoms input
0.0647	All non-H atoms anisotropic, 23 H atoms included
0.0489	33 H atoms included
0.0384	All H atoms included. H thermal parameters tied to separate free variables for brucine and for solvent molecules. Ethanol H atoms in calculated positions 793 reflections unobserved (F<5σ(F))
0.0401	F<4σ(F) 749 reflections unobserved
0.0387	Final Convergence*

Structure description



The diagram above shows the additional numbering scheme used for this complex, carrying on from the scheme used to number the strychnine

*The only hydrogens to be constrained to calculated positions were those on the ethanol molecule. The thermal parameters were constrained in two sets, one for the brucine hydrogens, and one for the solvent hydrogens.

molecule and cation. All bond lengths and angles are within the expected ranges for their types, the range of carbon carbon single bonds in the brucine molecule being from 1.561(6) angstroms for C(7)-C(8) to 1.488(6) angstroms for C(17)-C(18) (a difference of 12 σ)) although the carbon carbon bond in the ethanol is actually slightly shorter than the carbon oxygen bond (1.437(11) vs. 1.454(9) A). The range of values for the angles at nominally tetrahedral carbon is also rather broad (101.2(3)° for C(9)-C(7)-C(11) *versus* 119.3(4) for C(19)-C(20)-C(21) - a difference of almost 60 σ). The bonds from the aromatic ring to the methoxy groups are shorter than the usual carbon oxygen single bond length, possibly because of an overlap between the lone pairs on the oxygens with the π cloud of the aromatic ring. There is a pronounced alternation of bond lengths around the aromatic ring, which is likely to be caused either by the steric effect of the two oxygen substituents being in such close contact (O(3)-O(4) 2.587(4) angstroms compared with usual hydrogen bonded contact distance of 2.75 angstroms), or as a result of a possible canonical form of the structure with a double bond to one of the oxygens (this explanation also rationalises the difference between the two C-O bonds, as it would be possible for one of the two resonance hybrids to be fractionally stabilised by the amide group.). The other carbon oxygen bonds and the carbon nitrogen are within the expected ranges for their types as can be seen from the tables below.

C(1) - C(2)	1.407(6)	C(12) -C(13)	1.540(7)
C(1) - C(6)	1.371(6)	C(13) -C(14)	1.533(6)
C(1) - N(1)	1.416(5)	C(13) -C(16)	1.524(6)
C(2) - C(3)	1.375(6)	C(14) -C(19)	1.536(6)
C(3) - C(4)	1.425(6)	C(15) -C(16)	1.505(7)
C(3) - O(3)	1.353(5)	C(15) - N(2)	1.485(6)
C(4) - C(5)	1.372(6)	C(16) -C(17)	1.319(6)
C(4) - O(4)	1.374(5)	C(17) -C(18)	1.488(7)
C(5) - C(6)	1.394(6)	C(18) - O(2)	1.427(6)
C(6) - C(7)	1.512(6)	C(19) -C(20)	1.528(7)
C(7) - C(8)	1.561(6)	C(19) - O(2)	1.432(5)
C(7) - C(9)	1.550(6)	C(20) -C(21)	1.511(6)
C(7) -C(11)	1.539(6)	C(21) - N(1)	1.366(5)
C(8) -C(14)	1.522(6)	C(21) - O(1)	1.231(6)
C(8) - N(1)	1.485(5)	C(22) - O(3)	1.433(6)

C(9) -C(12)	1.508(6)	C(23) - O(4)	1.420(7)
C(9) - N(2)	1.494(6)	C(1A) -C(2A)	1.437(11)
C(10) -C(11)	1.512(7)	C(1A) -O(1A)	1.454(9)
C(10) - N(2)	1.474(6)		

C(2) - C(1) - C(6)	121.5(4)	C(12) -C(13) -C(14)	105.9(4)
C(2) - C(1) - N(1)	127.8(4)	C(12) -C(13) -C(16)	109.3(4)
C(6) - C(1) - N(1)	110.7(3)	C(14) -C(13) -C(16)	115.2(4)
C(1) - C(2) - C(3)	118.2(4)	C(8) -C(14) -C(13)	112.7(3)
C(2) - C(3) - C(4)	120.3(4)	C(8) -C(14) -C(19)	107.6(3)
C(2) - C(3) - O(3)	124.3(4)	C(13) -C(14) -C(19)	118.1(4)
C(4) - C(3) - O(3)	115.3(4)	C(16) -C(15) - N(2)	111.8(4)
C(3) - C(4) - C(5)	120.2(4)	C(13) -C(16) -C(15)	114.1(4)
C(3) - C(4) - O(4)	115.1(4)	C(13) -C(16) -C(17)	122.2(4)
C(5) - C(4) - O(4)	124.7(4)	C(15) -C(16) -C(17)	123.5(4)
C(4) - C(5) - C(6)	119.4(4)	C(16) -C(17) -C(18)	123.9(4)
C(1) - C(6) - C(5)	120.3(4)	C(17) -C(18) - O(2)	112.0(4)
C(1) - C(6) - C(7)	110.4(3)	C(14) -C(19) -C(20)	109.8(4)
C(5) - C(6) - C(7)	129.2(4)	C(14) -C(19) - O(2)	113.7(3)
C(6) - C(7) - C(8)	102.0(3)	C(20) -C(19) - O(2)	106.0(3)
C(6) - C(7) - C(9)	115.9(3)	C(19) -C(20) -C(21)	119.3(4)
C(6) - C(7) -C(11)	113.4(3)	C(20) -C(21) - N(1)	116.0(4)
C(8) - C(7) - C(9)	113.9(3)	C(20) -C(21) - O(1)	121.3(4)
C(8) - C(7) -C(11)	110.8(3)	N(1) -C(21) - O(1)	122.6(4)
C(9) - C(7) -C(11)	101.2(3)	C(1) - N(1) - C(8)	108.9(3)
C(7) - C(8) -C(14)	117.2(3)	C(1) - N(1) -C(21)	125.6(3)
C(7) - C(8) - N(1)	104.4(3)	C(8) - N(1) -C(21)	119.6(3)
C(14) - C(8) - N(1)	106.5(3)	C(9) - N(2) -C(10)	108.5(3)
C(7) - C(9) -C(12)	114.4(4)	C(9) - N(2) -C(15)	112.9(3)
C(7) - C(9) - N(2)	105.2(3)	C(10) - N(2) -C(15)	112.7(4)
C(12) - C(9) - N(2)	112.1(4)	C(18) - O(2) -C(19)	115.0(3)
C(11) -C(10) - N(2)	105.5(4)	C(3) - O(3) -C(22)	115.9(3)
C(7) -C(11) -C(10)	102.7(4)	C(4) - O(4) -C(23)	117.3(4)
C(9) -C(12) -C(13)	108.7(4)	C(2A) -C(1A) -O(1A)	112.8(6)

C(6) - C(1) - C(2) - C(3)	2.4(6)
N(1) - C(1) - C(2) - C(3)	-176.5(4)
C(2) - C(1) - C(6) - C(5)	-3.5(6)
C(2) - C(1) - C(6) - C(7)	172.9(4)
N(1) - C(1) - C(6) - C(5)	175.6(4)
N(1) - C(1) - C(6) - C(7)	-8.1(5)
C(2) - C(1) - N(1) - C(8)	174.2(4)
C(2) - C(1) - N(1) -C(21)	21.5(6)
C(6) - C(1) - N(1) - C(8)	-4.8(4)
C(6) - C(1) - N(1) -C(21)	-157.5(4)
C(1) - C(2) - C(3) - C(4)	0.9(6)
C(1) - C(2) - C(3) - O(3)	-179.7(4)
C(2) - C(3) - C(4) - C(5)	-3.2(6)
C(2) - C(3) - C(4) - O(4)	177.1(4)
O(3) - C(3) - C(4) - C(5)	177.3(4)
O(3) - C(3) - C(4) - O(4)	-2.4(5)
C(2) - C(3) - O(3) -C(22)	4.0(6)
C(4) - C(3) - O(3) -C(22)	-176.5(4)
C(3) - C(4) - C(5) - C(6)	2.2(6)
O(4) - C(4) - C(5) - C(6)	-178.1(4)
C(3) - C(4) - O(4) -C(23)	-176.6(4)
C(5) - C(4) - O(4) -C(23)	3.7(6)
C(4) - C(5) - C(6) - C(1)	1.1(6)
C(4) - C(5) - C(6) - C(7)	-174.5(4)
C(1) - C(6) - C(7) - C(8)	16.6(4)
C(1) - C(6) - C(7) - C(9)	140.9(4)
C(1) - C(6) - C(7) -C(11)	-102.6(4)
C(5) - C(6) - C(7) - C(8)	-167.5(4)
C(5) - C(6) - C(7) - C(9)	-43.2(6)
C(5) - C(6) - C(7) -C(11)	73.3(5)
C(6) - C(7) - C(8) -C(14)	99.2(4)
C(6) - C(7) - C(8) - N(1)	-18.3(4)
C(9) - C(7) - C(8) -C(14)	-26.5(5)
C(9) - C(7) - C(8) - N(1)	-144.0(3)
C(11) - C(7) - C(8) -C(14)	-139.8(4)
C(11) - C(7) - C(8) - N(1)	102.7(4)
C(6) - C(7) - C(9) -C(12)	-82.3(5)
C(6) - C(7) - C(9) - N(2)	154.2(3)
C(8) - C(7) - C(9) -C(12)	35.6(5)
C(8) - C(7) - C(9) - N(2)	-87.8(4)
C(11) - C(7) - C(9) -C(12)	154.5(4)
C(11) - C(7) - C(9) - N(2)	31.1(4)
C(6) - C(7) -C(11) -C(10)	-165.5(4)
C(8) - C(7) -C(11) -C(10)	80.5(4)
C(9) - C(7) -C(11) -C(10)	-40.6(4)

C(7) - C(8) -C(14) -C(13)	40.5(5)
C(7) - C(8) -C(14) -C(19)	172.5(3)
N(1) - C(8) -C(14) -C(13)	156.9(3)
N(1) - C(8) -C(14) -C(19)	-71.1(4)
C(7) - C(8) - N(1) - C(1)	14.9(4)
C(7) - C(8) - N(1) -C(21)	169.5(3)
C(14) - C(8) - N(1) - C(1)	-109.7(3)
C(14) - C(8) - N(1) -C(21)	44.8(5)
C(7) - C(9) -C(12) -C(13)	-58.4(5)
N(2) - C(9) -C(12) -C(13)	61.3(5)
C(7) - C(9) - N(2) -C(10)	-9.8(4)
C(7) - C(9) - N(2) -C(15)	115.9(4)
C(12) - C(9) - N(2) -C(10)	-134.6(4)
C(12) - C(9) - N(2) -C(15)	-8.9(5)
N(2) -C(10) -C(11) - C(7)	35.8(5)
C(11) -C(10) - N(2) - C(9)	-16.3(5)
C(11) -C(10) - N(2) -C(15)	-142.1(4)
C(9) -C(12) -C(13) -C(14)	69.7(4)
C(9) -C(12) -C(13) -C(16)	-55.0(5)
C(12) -C(13) -C(14) - C(8)	-60.7(5)
C(12) -C(13) -C(14) -C(19)	172.8(4)
C(16) -C(13) -C(14) - C(8)	60.2(5)
C(16) -C(13) -C(14) -C(19)	-66.3(5)
C(12) -C(13) -C(16) -C(15)	-0.2(5)
C(12) -C(13) -C(16) -C(17)	176.0(4)
C(14) -C(13) -C(16) -C(15)	-119.3(4)
C(14) -C(13) -C(16) -C(17)	56.9(6)
C(8) -C(14) -C(19) -C(20)	46.9(5)
C(8) -C(14) -C(19) - O(2)	-71.7(4)
C(13) -C(14) -C(19) -C(20)	175.9(4)
C(13) -C(14) -C(19) - O(2)	57.3(5)
N(2) -C(15) -C(16) -C(13)	52.4(5)
N(2) -C(15) -C(16) -C(17)	-123.8(5)
C(16) -C(15) - N(2) - C(9)	-47.3(5)
C(16) -C(15) - N(2) -C(10)	76.1(5)
C(13) -C(16) -C(17) -C(18)	-2.5(7)
C(15) -C(16) -C(17) -C(18)	173.4(5)
C(16) -C(17) -C(18) - O(2)	-62.8(6)
C(17) -C(18) - O(2) -C(19)	87.5(5)
C(14) -C(19) -C(20) -C(21)	1.9(6)
O(2) -C(19) -C(20) -C(21)	125.1(4)
C(14) -C(19) - O(2) -C(18)	-69.1(5)
C(20) -C(19) - O(2) -C(18)	170.2(4)
C(19) -C(20) -C(21) - N(1)	-30.0(6)
C(19) -C(20) -C(21) - O(1)	148.6(4)

C(20) -C(21) - N(1) - C(1)	154.8(4)
C(20) -C(21) - N(1) - C(8)	4.8(5)
O(1) -C(21) - N(1) - C(1)	-23.8(6)
O(1) -C(21) - N(1) - C(8)	-173.8(4)

The next group of tables give the bonds, angles and torsion angles involving hydrogen atoms. As can be seen from the lower positional e.s.d's, the ethyl alcohol hydrogens were positionally constrained.

C(2) -H(021)	0.97(5)	C(20) -H(201)	1.02(5)
C(5) -H(051)	1.02(5)	C(20) -H(202)	0.92(5)
C(8) -H(081)	1.02(5)	C(22) -H(221)	1.03(5)
C(9) -H(091)	1.10(4)	C(22) -H(222)	1.00(5)
C(10)-H(101)	1.00(5)	C(22) -H(223)	1.04(4)
C(10) -H(102)	1.11(5)	C(23) -H(231)	0.94(5)
C(11) -H(111)	0.92(5)	C(23) -H(232)	0.99(5)
C(11) -H(112)	1.07(5)	C(23) -H(233)	0.95(5)
C(12) -H(121)	1.05(5)	O(1W1)-H(1W1)	0.92(11)
C(12) -H(122)	0.95(5)	O(1W1)-H(2W1)	0.96(11)
C(13) -H(131)	0.95(5)	O(1W2)-H(1W2)	0.96(11)
C(14) -H(141)	0.97(5)	O(1W2)-H(2W2)	1.03(11)
C(15) -H(151)	1.06(5)	C(1A) -H(1C1)	1.080(11)
C(15) -H(152)	0.98(5)	C(1A) -H(2C1)	1.080(11)
C(17) -H(171)	0.95(5)	C(2A) -H(1C2)	1.080(12)
C(18) -H(181)	0.99(5)	C(2A) -H(2C2)	1.080(12)
C(18) -H(182)	1.02(5)	C(2A) -H(3C2)	1.080(12)
C(19) -H(191)	1.00(5)	O(1A) -H(1OA)	1.05(11)

C(1) - C(2) -H(021)	120.4(27)	C(17) -C(18) -H(182)	111.0(26)
H(021)- C(2) - C(3)	121.4(27)	H(181)-C(18) -H(182)	111 (4)
C(4) - C(5) -H(051)	121.7(26)	H(181)-C(18) - O(2)	110 (3)
H(051)- C(5) - C(6)	118.9(26)	H(182)-C(18) - O(2)	105.3(26)
C(7) - C(8) -H(081)	107.0(26)	C(14) -C(19) -H(191)	110 (3)
H(081)- C(8) -C(14)	111.0(26)	H(191)-C(19) -C(20)	105 (3)
H(081)- C(8) - N(1)	110.5(26)	H(191)-C(19) - O(2)	112 (3)
C(7) - C(9) -H(091)	107.1(23)	C(19) -C(20) -H(201)	108.4(26)
H(091)- C(9) -C(12)	108.9(23)	C(19) -C(20) -H(202)	100 (3)
H(091)- C(9) - N(2)	108.9(23)	H(201)-C(20) -H(202)	116 (4)
H(101)-C(10) -H(102)	113 (4)	H(201)-C(20) -C(21)	104.9(26)
H(101)-C(10) -C(11)	110 (3)	H(202)-C(20) -C(21)	109 (3)
H(101)-C(10) - N(2)	108 (3)	H(221)-C(22) -H(222)	106 (4)
H(102)-C(10) -C(11)	113.9(24)	H(221)-C(22) -H(223)	122 (4)
H(102)-C(10) - N(2)	106.5(23)	H(221)-C(22) - O(3)	108.6(27)

C(7) -C(11) -H(111)	108 (3)	H(222)-C(22) -H(223)	102 (4)
C(7) -C(11) -H(112)	112.4(25)	H(222)-C(22) - O(3)	109.6(27)
C(10) -C(11) -H(111)	114 (3)	H(223)-C(22) - O(3)	108.6(25)
C(10) -C(11) -H(112)	112.4(25)	H(231)-C(23) -H(232)	115 (4)
H(111)-C(11) -H(112)	107 (4)	H(231)-C(23) -H(233)	101 (4)
C(9) -C(12) -H(121)	111.8(26)	H(231)-C(23) - O(4)	113 (3)
C(9) -C(12) -H(122)	111 (3)	H(232)-C(23) -H(233)	111 (4)
H(121)-C(12) -H(122)	108 (4)	H(232)-C(23) - O(4)	110.4(27)
H(121)-C(12) -C(13)	106.5(26)	H(233)-C(23) - O(4)	106 (3)
H(122)-C(12) -C(13)	111 (3)	H(1W1)-O(1W1)-H(2W1)	81 (9)
C(12) -C(13) -H(131)	111 (3)	H(1W2)-O(1W2)-H(2W2)	110 (9)
H(131)-C(13) -C(14)	108 (3)	H(1C1)-C(1A) -H(2C1)	109.5(9)
H(131)-C(13) -C(16)	108 (3)	H(1C1)-C(1A) -C(2A)	108.6(8)
C(8) -C(14) -H(141)	102.8(27)	H(1C1)-C(1A) -O(1A)	108.6(7)
C(13) -C(14) -H(141)	107.6(27)	H(2C1)-C(1A) -C(2A)	108.6(8)
H(141)-C(14) -C(19)	106.8(27)	H(2C1)-C(1A) -O(1A)	108.6(7)
H(151)-C(15) -H(152)	99 (4)	C(1A) -C(2A) -H(1C2)	109.5(8)
H(151)-C(15) -C(16)	111.6(25)	C(1A) -C(2A) -H(2C2)	109.5(8)
H(151)-C(15) - N(2)	111.0(25)	C(1A) -C(2A) -H(3C2)	109.5(8)
H(152)-C(15) -C(16)	118 (3)	H(1C2)-C(2A) -H(2C2)	109.5(10)
H(152)-C(15) - N(2)	104 (3)	H(1C2)-C(2A) -H(3C2)	109.5(10)
C(16) -C(17) -H(171)	117 (3)	H(2C2)-C(2A) -H(3C2)	109.5(10)
H(171)-C(17) -C(18)	118 (3)	C(1A) -O(1A) -H(10A)	113 (6)
C(17) -C(18) -H(181)	108 (3)		

C(6) - C(1) - C(2) -H(021)	180 (3)
N(1) - C(1) - C(2) -H(021)	1 (3)
H(021)- C(2) - C(3) - C(4)	-177 (3)
H(021)- C(2) - C(3) - O(3)	3 (3)
C(3) - C(4) - C(5) -H(051)	-179 (3)
O(4) - C(4) - C(5) -H(051)	1 (3)
H(051)- C(5) - C(6) - C(1)	-178 (3)
H(051)- C(5) - C(6) - C(7)	7 (3)
C(6) - C(7) - C(8) -H(081)	-136 (3)
C(9) - C(7) - C(8) -H(081)	99 (3)
C(11) - C(7) - C(8) -H(081)	-14 (3)
C(6) - C(7) - C(9) -H(091)	38.4(25)
C(8) - C(7) - C(9) -H(091)	156.4(24)
C(11) - C(7) - C(9) -H(091)	-84.7(25)
C(6) - C(7) -C(11) -H(111)	-44 (3)
C(6) - C(7) -C(11) -H(112)	73.4(27)
C(8) - C(7) -C(11) -H(111)	-158 (3)
C(8) - C(7) -C(11) -H(112)	-40.6(27)
C(9) - C(7) -C(11) -H(111)	81 (3)
C(9) - C(7) -C(11) -H(112)	-161.7(27)

C(7) - C(8) -C(14) -H(141)	-75 (3)
H(081)- C(8) -C(14) -C(13)	-83 (3)
H(081)- C(8) -C(14) -H(141)	162 (4)
H(081)- C(8) -C(14) -C(19)	49 (3)
N(1) - C(8) -C(14) -H(141)	41 (3)
H(081)- C(8) - N(1) - C(1)	130 (3)
H(081)- C(8) - N(1) -C(21)	-76 (3)
C(7) - C(9) -C(12) -H(121)	59 (3)
C(7) - C(9) -C(12) -H(122)	180 (3)
H(091)- C(9) -C(12) -H(121)	-61 (4)
H(091)- C(9) -C(12) -H(122)	60 (4)
H(091)- C(9) -C(12) -C(13)	-178.1(25)
N(2) - C(9) -C(12) -H(121)	179 (3)
N(2) - C(9) -C(12) -H(122)	-61 (3)
H(091)- C(9) - N(2) -C(10)	104.7(25)
H(091)- C(9) - N(2) -C(15)	-129.5(25)
H(101)-C(10) -C(11) - C(7)	-80 (3)
H(101)-C(10) -C(11) -H(111)	164 (4)
H(101)-C(10) -C(11) -H(112)	41 (4)
H(102)-C(10) -C(11) - C(7)	152.3(26)
H(102)-C(10) -C(11) -H(111)	36 (4)
H(102)-C(10) -C(11) -H(112)	-87 (4)
N(2) -C(10) -C(11) -H(111)	-81 (3)
N(2) -C(10) -C(11) -H(112)	156.9(27)
H(101)-C(10) - N(2) - C(9)	101 (3)
H(101)-C(10) - N(2) -C(15)	-25 (3)
H(102)-C(10) - N(2) - C(9)	-137.6(24)
H(102)-C(10) - N(2) -C(15)	96.6(25)
C(9) -C(12) -C(13) -H(131)	-174 (3)
H(121)-C(12) -C(13) -H(131)	66 (4)
H(121)-C(12) -C(13) -C(14)	-50.9(27)
H(121)-C(12) -C(13) -C(16)	-175.6(27)
H(122)-C(12) -C(13) -H(131)	-51 (4)
H(122)-C(12) -C(13) -C(14)	-168 (3)
H(122)-C(12) -C(13) -C(16)	67 (3)
C(12) -C(13) -C(14) -H(141)	52 (3)
H(131)-C(13) -C(14) - C(8)	-180 (3)
H(131)-C(13) -C(14) -H(141)	-67 (4)
H(131)-C(13) -C(14) -C(19)	54 (3)
C(16) -C(13) -C(14) -H(141)	173 (3)
H(131)-C(13) -C(16) -C(15)	121 (3)
H(131)-C(13) -C(16) -C(17)	-63 (3)
C(8) -C(14) -C(19) -H(191)	162 (3)
C(13) -C(14) -C(19) -H(191)	-69 (3)
H(141)-C(14) -C(19) -H(191)	52 (4)

H(141)-C(14) -C(19) -C(20)	-63 (3)
H(141)-C(14) -C(19) - O(2)	179 (3)
H(151)-C(15) -C(16) -C(13)	-72.6(27)
H(151)-C(15) -C(16) -C(17)	111.2(27)
H(152)-C(15) -C(16) -C(13)	173 (3)
H(152)-C(15) -C(16) -C(17)	-3 (3)
H(151)-C(15) - N(2) - C(9)	78.0(27)
H(151)-C(15) - N(2) -C(10)	-158.6(27)
H(152)-C(15) - N(2) - C(9)	-176 (3)
H(152)-C(15) - N(2) -C(10)	-53 (3)
C(13) -C(16) -C(17) -H(171)	-172 (3)
C(15) -C(16) -C(17) -H(171)	4 (3)
C(16) -C(17) -C(18) -H(181)	58 (3)
C(16) -C(17) -C(18) -H(182)	179.8(27)
H(171)-C(17) -C(18) -H(181)	-133 (4)
H(171)-C(17) -C(18) -H(182)	-11 (4)
H(171)-C(17) -C(18) - O(2)	107 (3)
H(181)-C(18) - O(2) -C(19)	-32 (3)
H(182)-C(18) - O(2) -C(19)	-151.8(26)
C(14) -C(19) -C(20) -H(201)	121.7(27)
C(14) -C(19) -C(20) -H(202)	-117 (3)
H(191)-C(19) -C(20) -H(201)	4 (4)
H(191)-C(19) -C(20) -H(202)	125 (4)
H(191)-C(19) -C(20) -C(21)	-116 (3)
O(2) -C(19) -C(20) -H(201)	-115.1(27)
O(2) -C(19) -C(20) -H(202)	6 (3)
H(191)-C(19) - O(2) -C(18)	56 (3)
H(201)-C(20) -C(21) - N(1)	-151.6(27)
H(201)-C(20) -C(21) - O(1)	26.9(27)
H(202)-C(20) -C(21) - N(1)	84 (3)
H(202)-C(20) -C(21) - O(1)	-98 (3)
H(221)-C(22) - O(3) - C(3)	52 (3)
H(222)-C(22) - O(3) - C(3)	-64 (3)
H(223)-C(22) - O(3) - C(3)	-173.8(26)
H(231)-C(23) - O(4) - C(4)	-59 (3)
H(232)-C(23) - O(4) - C(4)	171 (3)
H(233)-C(23) - O(4) - C(4)	51 (3)
H(1C1)-C(1A) -C(2A) -H(1C2)	-59.5(11)
H(1C1)-C(1A) -C(2A) -H(2C2)	-179.5(9)
H(1C1)-C(1A) -C(2A) -H(3C2)	60.5(11)
H(2C1)-C(1A) -C(2A) -H(1C2)	59.5(11)
H(2C1)-C(1A) -C(2A) -H(2C2)	-60.5(11)
H(2C1)-C(1A) -C(2A) -H(3C2)	179.5(9)
O(1A) -C(1A) -C(2A) -H(1C2)	-180.0(8)
O(1A) -C(1A) -C(2A) -H(2C2)	60.0(10)

O(1A) -C(1A) -C(2A) -H(3C2)	-60.0(10)
H(1C1)-C(1A) -O(1A) -H(10A)	-73 (7)
H(2C1)-C(1A) -O(1A) -H(10A)	168 (7)
C(2A) -C(1A) -O(1A) -H(10A)	47 (7)

Hydrogen bonding and intermolecular contacts

As can be seen in the packing diagrams overleaf, there are "infinite" channels running parallel to the b axis of the crystal. These channels contain sets of solvent molecules which form short hydrogen bonded chains between the carbonyl group of one molecule and the amine lone pair on a molecule related to the first one by the screw axis parallel to z. The hydrogen bonded contacts are shown in the table below.

O1	2.875	(6)	O1W2	(1 1/2-x,2 -y,-1/2+z)
O1W1	2.781	(6)	N2	(1/2+x,1 1/2-y,1 -z)
O1W1	2.736	(7)	O1A	(1 1/2+x,1 1/2-y,1 -z)
O1W1	2.847	(7)	O1W2	(x,y,z)
O1W2	2.800	(8)	O1A	(1 +x,1 +y,1 +z)
H1W1	1.89	(11)	N2	(1/2+x,1 1/2-y,1 -z)
H2W1	2.24	(11)	O1W2	(x,y,z)
H1W2	1.89	(11)	O1A	(1 +x,1 +y,1 +z)
H2W2	1.85	(11)	O1	(1 1/2-x,2 -y,1/2+z)
H10A	1.72	(11)	O1W1	(-1 1/2+x,1 1/2-y,1 -z)

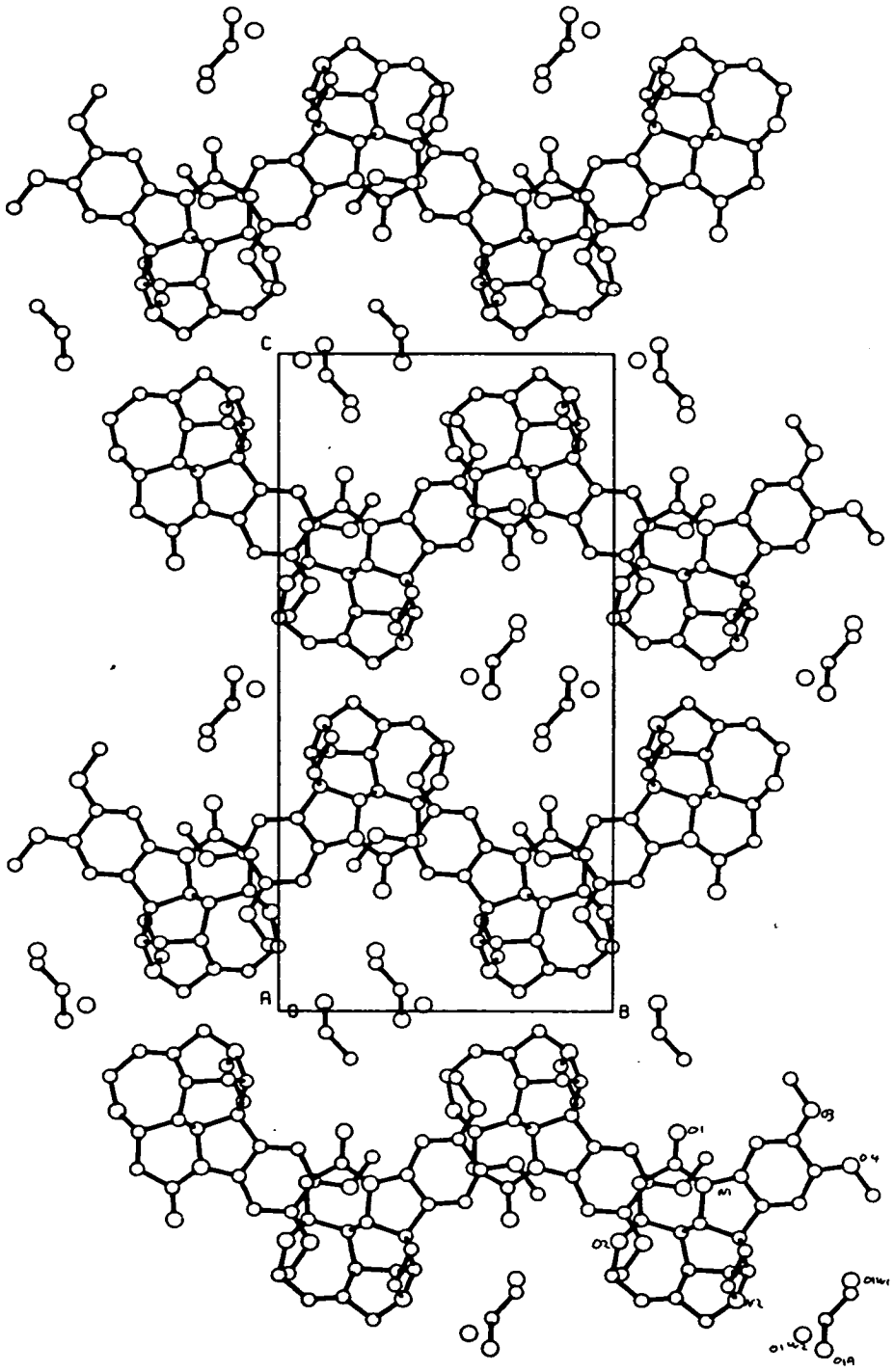
There are a number of other contact distances involving oxygen, but no carbon to carbon or nitrogen contacts less than 3.5 A. There is a possible interaction between the ether oxygen of the 7 membered ring (O(2)) and the π cloud of the aromatic ring on an adjacent brucine molecule, and another between a methoxy oxygen and the electron density on the amide nitrogen.

O2	3.488	(5)	C2	(1 -x,1/2+y,1/2-z)
O2	3.459	(5)	C3	(1 -x,1/2+y,1/2-z)
O2	3.483	(6)	C22	(1 -x,1/2+y,1/2-z)
O3	3.459	(5)	C13	(2 -x,-1/2+y,1/2-z)
O4	3.372	(4)	N1	(1 -x,-1/2+y,1/2-z)
O1W1	3.343	(6)	C9	(1/2+x,1 1/2-y,1 -z)
O1W1	3.324	(7)	C22	(2 -x,1/2+y,1 1/2-z)
O1A	3.213	(8)	C22	(1/2-x,1 -y,-1/2+z)

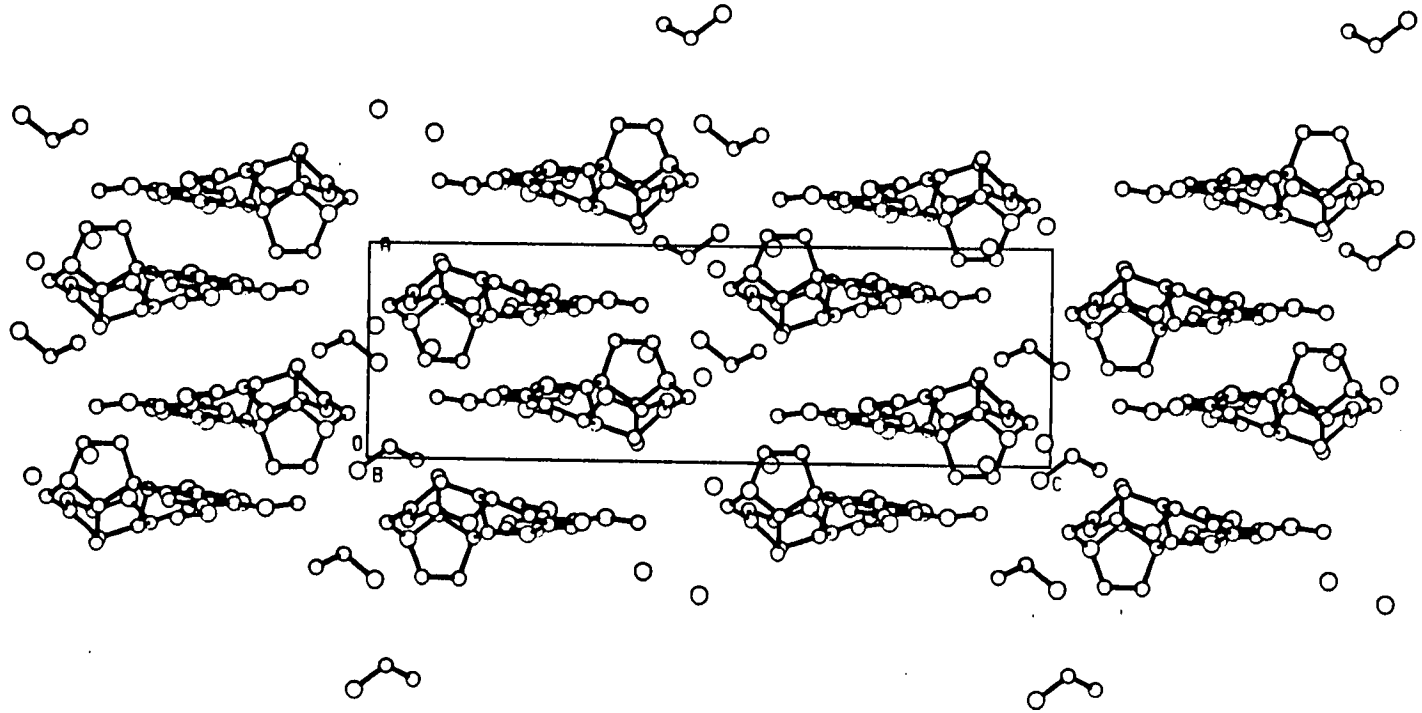
The table below shows all the hydrogen contacts less than 3 angstroms (or less than 2.5 angstroms to other hydrogens).

H051	2.99	(5)	O1W2	(-1/2+x,1 1/2-y,1 -z)
H081	2.79	(5)	O3	(1 -x,1/2+y,1/2-z)

H091	2.84	(5)	O1W1	(-1/2+x,1 1/2-y,1 -z)
H112	2.70	(5)	O4	(1 -x,1/2+y,1/2-z)
H131	2.63	(5)	O3	(2 -x,1/2+y,1/2-z)
H141	2.73	(5)	O4	(2 -x,1/2+y,1/2-z)
H171	2.99	(5)	O1A	(1/2+x,1 1/2-y,-z)
H182	2.91	(5)	O1A	(1/2+x,1 1/2-y,-z)
H191	2.74	(5)	O3	(2 -x,1/2+y,1/2-z)
H221	2.67	(5)	O2	(1 -x,-1/2+y,1/2-z)
H221	2.74	(5)	O1A	(1/2-x,1 -y,1/2+z)
H222	2.86	(5)	O1W1	(2 -x,-1/2+y,1 1/2-z)
H223	2.89	(4)	O1W1	(2 -x,-1/2+y,1 1/2-z)
H1C2	2.535	(9)	O1	(1 -x,-1/2+y,1/2-z)
H2C2	2.573	(10)	O1W1	(-1 +x,-1 +y,-1 +z)
H3C2	2.775	(10)	O1W2	(-1 1/2+x,1 1/2-y,1 -z)
H2W1	2.96	(11)	N2	(1/2+x,1 1/2-y,1 -z)
H131	2.97	(5)	C22	(2 -x,1/2+y,1/2-z)
H191	2.68	(5)	C3	(2 -x,1/2+y,1/2-z)
H191	2.99	(5)	C4	(2 -x,1/2+y,1/2-z)
H202	2.96	(5)	C3	(1 -x,1/2+y,1/2-z)
H202	2.99	(5)	C4	(1 -x,1/2+y,1/2-z)
H221	2.93	(5)	C17	(1 -x,-1/2+y,1/2-z)
H221	2.83	(5)	C18	(1 -x,-1/2+y,1/2-z)
H232	2.95	(5)	C20	(x,-1 +y,z)
H1W1	2.62	(11)	C9	(1/2+x,1 1/2-y,1 -z)
H1W1	2.64	(11)	C10	(1/2+x,1 1/2-y,1 -z)
H1W1	2.89	(11)	C15	(1/2+x,1 1/2-y,1 -z)
H1W2	2.91	(11)	C1A	(1 +x,1 +y,1 +z)
H2W2	2.87	(11)	C21	(1 1/2-x,2 -y,1/2+z)
H091	2.36	(12)	H1W1	(-1/2+x,1 1/2-y,1 -z)
H102	2.45	(12)	H1W1	(-1/2+x,1 1/2-y,1 -z)
H122	2.49	(5)	H1C1	(1 +x,y,z)
H151	2.34	(6)	H223	(1 1/2-x,1 -y,-1/2+z)
H181	2.33	(7)	H222	(2 -x,1/2+y,1/2-z)
H201	2.50	(6)	H232	(x,1 +y,z)
H1W1	2.37	(16)	H10A	(1 1/2+x,1 1/2-y,1 -z)
H2W1	2.37	(15)	H1W2	(x,y,z)
H1W2	2.42	(15)	H10A	(1 +x,1 +y,1 +z)



PROJECTION DOWN THE X AXIS



PROJECTION DOWN THE Y AXIS

Comparison with Strychnine

The most obvious difference between the brucine and strychnine molecules is the change in the aromatic ring caused by the methoxy groups. The range of bond lengths is fractionally larger in the brucine molecule, although the longest and shortest bonds are still in the same positions (implying that the difference in length essential for the molecular geometry to be maintained, and not merely random deviation), and the same is true of the largest and smallest angles. There is, of course, no hydrogen bond to the amine in the strychnine structure, which may explain the barely significant difference in length of the C(15)-N(2) bond between the two structures (1.485(6) angstroms in brucine and 1.464(6) angstroms in strychnine. In fact, the differences between the two structures are so small, that when one is fitted to the other, the root mean square deviation from an ideal fit is only 0.05 A. There is a somewhat larger deviation (ca. 0.15 A) in the position of the carbonyl group, almost certainly due to the hydrogen bonding to the carbonyl oxygen, which does not occur in the strychnine free base or in any of the salts and complexes examined so far.

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(1)	0.6761(5)	0.6174(3)	0.25061(15)	0.0343
C(2)	0.6936(6)	0.5719(3)	0.30152(16)	0.0351
C(3)	0.7208(6)	0.4620(3)	0.30538(16)	0.0378
C(4)	0.7331(6)	0.3972(3)	0.25874(16)	0.0370
C(5)	0.7096(6)	0.4431(3)	0.20973(16)	0.0369
C(6)	0.6793(5)	0.5543(3)	0.20587(15)	0.0349
C(7)	0.6355(5)	0.6214(3)	0.15753(15)	0.0327
C(8)	0.6613(6)	0.7393(3)	0.17854(15)	0.0329
C(9)	0.7405(6)	0.5956(3)	0.10670(16)	0.0399
C(10)	0.4536(7)	0.6545(4)	0.08295(19)	0.0470
C(11)	0.4491(6)	0.6045(4)	0.13770(18)	0.0404
C(12)	0.9157(6)	0.6492(4)	0.10435(20)	0.0450
C(13)	0.8909(6)	0.7729(4)	0.10821(17)	0.0418
C(14)	0.8313(6)	0.7953(3)	0.16514(16)	0.0360
C(15)	0.7007(8)	0.7198(4)	0.03016(17)	0.0491
C(16)	0.7663(7)	0.8100(3)	0.06501(16)	0.0444
C(17)	0.7139(7)	0.9115(4)	0.06097(18)	0.0481
C(18)	0.7639(8)	0.9989(4)	0.09865(20)	0.0507
C(19)	0.8165(6)	0.9139(3)	0.18324(17)	0.0405
C(20)	0.7429(7)	0.9186(3)	0.23944(19)	0.0450
C(21)	0.7030(6)	0.8141(3)	0.26826(17)	0.0420
C(22)	0.7170(8)	0.4709(4)	0.39896(17)	0.0485
C(23)	0.7918(10)	0.2225(4)	0.22218(21)	0.0609
N(1)	0.6565(4)	0.72821(25)	0.23716(12)	0.0351
N(2)	0.6267(5)	0.6293(3)	0.06173(13)	0.0437
O(1)	0.7157(6)	0.80730(24)	0.31682(12)	0.0590
O(2)	0.7030(4)	0.97786(22)	0.15108(12)	0.0459
O(3)	0.7390(4)	0.40781(22)	0.35170(10)	0.0447
O(4)	0.7685(4)	0.28946(22)	0.26745(11)	0.0481
O(1W1)	1.1081(7)	1.0689(4)	0.99056(16)	0.0790
O(1W2)	1.0067(7)	1.2123(4)	0.90774(20)	0.0975
C(1A)	0.0511(11)	0.3618(6)	0.0326(3)	0.0962
C(2A)	-0.0108(11)	0.2882(7)	0.0725(3)	0.1093
O(1A)	-0.0615(8)	0.3667(4)	-0.01355(18)	0.1006

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
H(021)	0.691(6)	0.618(4)	0.3328(17)	0.0477
H(051)	0.716(7)	0.398(4)	0.1759(17)	0.0477
H(081)	0.558(7)	0.784(4)	0.1655(16)	0.0477
H(091)	0.758(6)	0.507(4)	0.1053(16)	0.0477
H(101)	0.443(6)	0.735(4)	0.0856(17)	0.0477
H(102)	0.359(6)	0.619(3)	0.0549(17)	0.0477
H(111)	0.427(6)	0.531(4)	0.1381(18)	0.0477
H(112)	0.356(6)	0.642(4)	0.1629(18)	0.0477
H(121)	0.994(6)	0.627(4)	0.1365(18)	0.0477
H(122)	0.975(6)	0.631(4)	0.0726(18)	0.0477
H(131)	0.997(6)	0.810(4)	0.1029(17)	0.0477
H(141)	0.912(6)	0.760(4)	0.1887(17)	0.0477
H(151)	0.798(7)	0.691(3)	0.0040(16)	0.0477
H(152)	0.610(6)	0.737(4)	0.0040(20)	0.0477
H(171)	0.625(6)	0.926(3)	0.0360(18)	0.0477
H(181)	0.892(7)	1.004(4)	0.0988(18)	0.0477
H(182)	0.709(6)	1.071(4)	0.0881(16)	0.0477
H(191)	0.934(7)	0.947(4)	0.1861(17)	0.0477
H(201)	0.830(6)	0.958(4)	0.2631(18)	0.0477
H(202)	0.641(7)	0.955(4)	0.2329(18)	0.0477
H(221)	0.601(7)	0.512(4)	0.3965(18)	0.0477
H(222)	0.809(7)	0.528(4)	0.4008(17)	0.0477
H(223)	0.747(6)	0.423(3)	0.4315(18)	0.0477
H(231)	0.694(7)	0.222(4)	0.1999(19)	0.0477
H(232)	0.835(6)	0.150(4)	0.2328(16)	0.0477
H(233)	0.874(6)	0.259(4)	0.2004(19)	0.0477
H(1W1)	1.091(16)	1.004(8)	0.973(4)	0.1755
H(2W1)	1.004(14)	1.087(9)	0.972(4)	0.1755
H(1W2)	0.952(15)	1.259(8)	0.933(4)	0.1755
H(2W2)	0.932(15)	1.210(8)	0.874(4)	0.1755
H(1C1)	0.0599(11)	0.4420(6)	0.0497(3)	0.1755
H(2C1)	0.1779(11)	0.3356(6)	0.0200(3)	0.1755
H(1C2)	0.0777(11)	0.2880(7)	0.1057(3)	0.1755
H(2C2)	-0.0192(11)	0.2075(7)	0.0561(3)	0.1755
H(3C2)	-0.1372(11)	0.3138(7)	0.0858(3)	0.1755
H(10A)	-0.192(16)	0.376(9)	-0.004(4)	0.1755

Thermal Vibration Parameters with Standard Deviations

	U or U11	U22	U33	U23	U13	U12
C(1)	0.0343(22)	0.0352(21)	0.0333(21)	-0.0013(18)	-0.0049(18)	0.0010(19)
C(2)	0.0397(23)	0.0396(23)	0.0259(20)	0.0008(18)	-0.0011(19)	-0.0002(20)
C(3)	0.0368(24)	0.0443(24)	0.0322(22)	0.0016(18)	-0.0010(19)	-0.0008(21)
C(4)	0.0354(23)	0.0332(20)	0.0425(23)	0.0037(18)	-0.0047(19)	-0.0036(19)
C(5)	0.0427(25)	0.0335(21)	0.0345(22)	-0.0017(18)	-0.0001(20)	-0.0005(21)
C(6)	0.0351(23)	0.0392(23)	0.0303(20)	-0.0025(18)	0.0010(18)	-0.0042(19)
C(7)	0.0392(24)	0.0309(21)	0.0281(20)	0.0047(18)	-0.0004(19)	-0.0015(18)
C(8)	0.0336(24)	0.0316(22)	0.0333(22)	-0.0008(17)	0.0023(18)	0.0043(18)
C(9)	0.0518(29)	0.0355(22)	0.0325(21)	0.0005(18)	-0.0018(21)	0.0051(22)
C(10)	0.0527(30)	0.0484(29)	0.0400(26)	0.0027(22)	-0.0083(23)	-0.0008(25)
C(11)	0.0442(25)	0.0385(24)	0.0385(24)	0.0010(21)	-0.0073(20)	-0.0035(22)
C(12)	0.0471(29)	0.0456(26)	0.0423(27)	0.0001(23)	0.0110(24)	0.0091(23)
C(13)	0.0371(25)	0.0482(26)	0.0400(24)	-0.0020(21)	0.0118(21)	-0.0040(23)
C(14)	0.0390(25)	0.0333(22)	0.0357(22)	0.0017(18)	-0.0042(19)	-0.0007(20)
C(15)	0.0681(34)	0.0500(24)	0.0291(22)	0.0051(21)	0.0014(25)	-0.0001(28)
C(16)	0.0509(29)	0.0510(26)	0.0314(21)	0.0077(18)	0.0103(22)	0.0003(24)
C(17)	0.0540(30)	0.0472(27)	0.0432(26)	0.0123(21)	0.0026(24)	0.0005(25)
C(18)	0.0556(32)	0.0414(24)	0.0552(30)	0.0133(22)	0.0072(25)	-0.0042(23)
C(19)	0.0352(25)	0.0381(24)	0.0481(25)	0.0003(19)	-0.0018(20)	-0.0033(21)
C(20)	0.0482(29)	0.0338(22)	0.0531(28)	-0.0030(20)	-0.0084(24)	-0.0015(22)
C(21)	0.0488(28)	0.0374(22)	0.0399(26)	-0.0043(19)	-0.0020(22)	0.0054(21)
C(22)	0.0587(33)	0.0545(28)	0.0323(24)	0.0065(21)	0.0008(24)	0.0044(29)
C(23)	0.0913(45)	0.0360(25)	0.0554(30)	-0.0009(23)	0.0003(32)	0.0023(31)
N(1)	0.0448(22)	0.0322(18)	0.0283(17)	0.0020(15)	-0.0025(16)	-0.0008(16)
N(2)	0.0539(24)	0.0477(22)	0.0296(19)	0.0019(17)	-0.0016(18)	-0.0032(19)
O(1)	0.0969(29)	0.0470(17)	0.0330(17)	-0.0045(13)	-0.0082(18)	0.0030(21)
O(2)	0.0481(19)	0.0378(15)	0.0517(18)	0.0068(14)	0.0041(16)	0.0032(15)
O(3)	0.0566(19)	0.0452(15)	0.0323(15)	0.0083(13)	-0.0019(15)	0.0059(15)
O(4)	0.0630(21)	0.0340(15)	0.0474(17)	0.0027(13)	-0.0056(17)	0.0040(16)
O(1W1)	0.1119(36)	0.0646(24)	0.0605(24)	-0.0182(20)	-0.0127(24)	0.0092(28)
O(1W2)	0.1064(40)	0.1008(34)	0.0853(31)	-0.0009(29)	-0.0211(28)	0.0248(31)
C(1A)	0.1214(61)	0.0921(47)	0.0750(42)	0.0061(40)	0.0004(45)	0.0066(50)
C(2A)	0.1226(65)	0.1292(60)	0.0761(44)	0.0103(48)	0.0096(46)	0.0195(58)
O(1A)	0.1193(40)	0.1201(37)	0.0623(25)	0.0121(26)	-0.0003(28)	0.0272(36)

CHAPTER TEN
STRYCHNINE N-ACETYL TYROSINE TETRAHYDRATE

STRYCHNINE:N-ACETYL-L-TYROSINE TETRAHYDRATE

Introduction

This salt was prepared as part of a study of molecular recognition in amino acid:alkaloid complexes. These complexes provide information about recognition phenomena on two levels. They provide direct information on chiral recognition, as they have long been used as a means of resolving amino acids via diastereomeric salt formation and they provide indirect information on drug:receptor interaction as the alkaloids used are biologically active and would be expected to interact with a receptor protein in the same ways as they interact with an acyl amino acid. The interactions possible in this salt may be divided into hydrogen bonding and hydrophobic interactions. The strychninium cation has one possible hydrogen bond donor (the proton on the quaternary nitrogen) and one possible hydrogen bond acceptor (the carbonyl oxygen). The N-acetyl-L-tyrosine anion has two possible hydrogen bond donors (the amide and phenol hydrogens) and four possible hydrogen bond acceptors (the carboxylate group, the amide carbonyl and the phenol oxygen). There are three possible hydrophobic interactions: a stacking interaction between the aromatic rings, formation of a 'hydrophobic pocket' for one of the molecules by the packing of the other, or an interaction between the π-cloud of an aromatic ring, and the lone pair electrons of a nearby oxygen atom. A charge transfer interaction between the two aromatic systems is not impossible, but was considered to be rather unlikely.

Although tyrosine has been resolved by the method of diastereomeric salt formation on previous occasions, this is the only complex between strychnine and a tyrosine derivative to have been prepared, the earlier workers having used brucine or cinchonine. Fischer (1900) resolved N-benzoyl tyrosine by using brucine to precipitate the L-isomer and cinchonine to precipitate the D-form. Because hydrolysis of the benzoyl group required reaction conditions strong enough to epimerise the chiral centre, a resolution system using a more easily removed acyl group was sought. Both N-formyl tyrosine (Abderhalden and Sichel, 1923)

and N-acetyl tyrosine (Sealock, 1946) were resolved with brucine, the D-isomer in both cases being the first to precipitate.

It was decided to attempt the preparation of a complex of N-acetyl L-tyrosine with strychnine because this is directly comparable with the strychnine: N-benzoylalanine dihydrate complex previously determined (Gould and Walkinshaw, 1984) in terms of hydrogen bonding ability, but with the opposite arrangement of steric bulk around the Cα. As most resolutions using strychnine precipitate the L-isomer whereas brucine resolutions precipitate the D-isomer almost irrespective of the size of the functional groups (Greenstein, 1961) it was thought that hydrogen bonding ability rather than molecular shape would be the determining factor in formation of a crystalline complex.

The high degree of solvation of this complex is similar to that found in the benzoyl alanine complexes, but the positional disorder in the acetyl tyrosine moiety is a feature of this complex not repeated in any of the others examined so far. It is likely that this disorder, and the concomitant difficulties involved in crystallising this complex, provide the reason why this complex was not employed in resolution.

Crystallisation

Crystals of Strychnine: N-Acetyl-L-tyrosine were prepared by dissolving equal parts (1 mM) of strychnine and acetyl tyrosine in excess (5 ml) hot aqueous ethanol (50% v/v) and allowing the solution to cool slowly to room temperature. Needle-like crystals which turned opaque and crumbled when exposed to air were produced as the solvent evaporated. The crystal used for preliminary photography and data collection was sealed in a glass tube with some mother liquor in order to prevent its decomposition in this way.

Crystal data

Formula $C_{21}H_{22}N_2O_2 \cdot C_{11}H_{13}NO_4 \cdot 4H_2O$ Mol. Wt. = 629.66 F(000) = 672

Space group $P2_1$ Int. Tab. No. 4
Cell dimensions a = 16.544(2) b = 7.866(3) c = 15.384(2) A
β = 115.714(12)°

V = 1803.8 A^3 Z = 2 D_c = 1.159 gcm^{-3} D_m = 1.174 gcm^{-3}

Radiation MoKα λ = 0.71069 A μ = 0.81 cm^{-1}
Final R = 0.1040 based on 1162 independent reflections
h_{range} -17 15 k_{max} 8 l_{max} 14

θ_{max} = 22° $Sin(\theta_{max})/\lambda$ = 0.5271

Drift curve min. 0.991 max. 1.011
2452 reflections measured (2413 unique)
1251 reflections considered unobserved ($F<4\sigma(F)$)
Merging R = 0.0154
351 Parameters refined
Max. shift/e.s.d in last cycle 0.632
Weighting scheme: $W = 1.2705/(\sigma^2(F)+0.009138F^2)$
Final difference Fourier min. -0.30 eA^{-3} max. 0.353 eA^{-3}

Data Collection and Reduction

The data was collected on the CAD4 automated 4-circle diffractometer and corrected for Lorentz and polarisation effects by the program CADABS.

Structure Solution and Refinement

The initial attempt at solving the structure using a totally automated run of the MULTAN system was unsuccessful. The data set was renormalised using a Debye curve calculated for spherically averaged molecules of strychnine and N-acetyl tyrosine. This changed the E-values of enough reflections to result in MULTAN choosing a new starting set of reflections, but it did not lead to a solution of the structure. The data was then renormalised on the basis of a Wilson plot calculated from twice the cell contents, but the electron density

maps output by SEARCH showed no sign of recognisable molecular fragments. A final run of the program NORMAL with the thermal parameter B input as 7.0 rather than calculated from a Wilson plot changed the E-values sufficiently for the choice of a new starting set. The Fourier map calculated from this run of MULTAN contained a recognisable strychnine molecule and a fragment of the N-acetyl-tyrosine residue. The table below shows the starting sets with final phases and figures of merit from each run of MULTAN. (the code number referred to is the position in order of decreasing E-value and the letter 'E' is used to mark enantiomorph fixing reflections) The program SHELX was used to generate a difference Fourier phased on this fragment, but it soon became obvious that the rest of the tyrosine moiety was not going to appear as a chemically reasonable fragment. The strychnine molecule was used as an input to the DIRDIF system, but no additional peaks beyond the original C_5O fragment of the aromatic ring were found in the output electron density map. This result was a strong implication that the tyrosine was positionally disordered. When dealing with such a structure it is not possible to locate atoms by means of a peak search routine applied to a difference map, as it is possible that the overlap between two adjacent disordered atoms may produce a higher peak than the 'true' positions of the atoms. The program FMAPO was used to scale and interpolate the full difference maps output by SHELX so that they could be contoured and examined for reasonable fragments of the molecule.

Origin fixing Reflections					Other reflections in starting set					Figure of merit	
code	h	k	l	angle	code	h	k	l	angle	Abs	Ψ(0)	R
Run 1 B = 5.85												
4	8	3	7	0	1	0	2	0	270			
8	7	3	8	45E	18	2	1	-1	0			
9	3	2	0	±45	43	3	3	11	90			
					45	6	4	-1	270	1.228	2.448	37.49
Run 2 B = 6.55												
3	13	0	3	0	1	0	2	0	153E			
4	8	3	7	0	8	3	2	0	193			
113	11	0	-6	0	9	7	3	8	233			
					231	4	3	-7	313			
					371	4	2	-3	74			
					55	5	1	9	275			
					77	6	3	-2	236	1.3623	0.553	21.76
Run 3 B = 5.93												
4	8	3	7	0	1	0	2	0	11			
7	7	3	8	45E	9	3	2	0	203			
71	6	0	9	0	25	2	1	-1	34			
					261	4	3	-7	56			
					146	1	6	-10	270	1.4006	2.360	41.52
Run 4 B = 7.00 (set)												
3	8	3	7	0	8	14	2	-6	234			
60	7	0	8	0	23	7	1	8	29			
88	8	0	7	0	25	4	3	8	340			
					28	7	2	8	79E			
					47	2	1	-1	86			
					204	10	1	-13	142	1.1368	1.371	24.7

Starting with a map phased on the strychnine parameters only, it was possible to locate two probable positions for the fragment containing the phenolic oxygen, the aromatic ring and the β-carbon, as well as two fully occupied water sites. A map phased on this additional

information revealed the position of the amide side chain (again distributed between two sites). The carboxylate groups and the α-carbons were the last to be identified in the maps.

The aromatic fragment and the amide side chain were each treated as rigid groups for the purposes of refinement and distance constraints were used to hold both fragments in position relative to the Cα and C' carbons. The carboxylate group carbon to oxygen bond distances were allowed to refine normally. A difference map phased on this model showed several peaks of electron density which may be interpreted as positionally disordered water molecules.

Description of Structure

The model used for this structure contains a non-disordered strychninium cation and an N-acetyltyrosine anion distributed over two equally occupied locations.* There are four molecules of water of crystallisation per asymmetric unit, of which two molecules are in fully occupied sites and the other two are each associated with one possible position for the tyrosine moiety and are distributed over three sites. None of the contact distances between possibly occupied sites are close enough to force the crystal structure into any form of long range order or 'super cell', although there are of course positions that may not be occupied simultaneously.

As would be expected from the rigidity of the strychninium cation, none of its bond lengths or angles are significantly different from the average strychninium cation from the strychnine salt series of structures, even though the environment of the cation is not strictly comparable. There is also no significant difference in molecular geometry between this strychnine and the strychnine in the benzoylalanine complex.

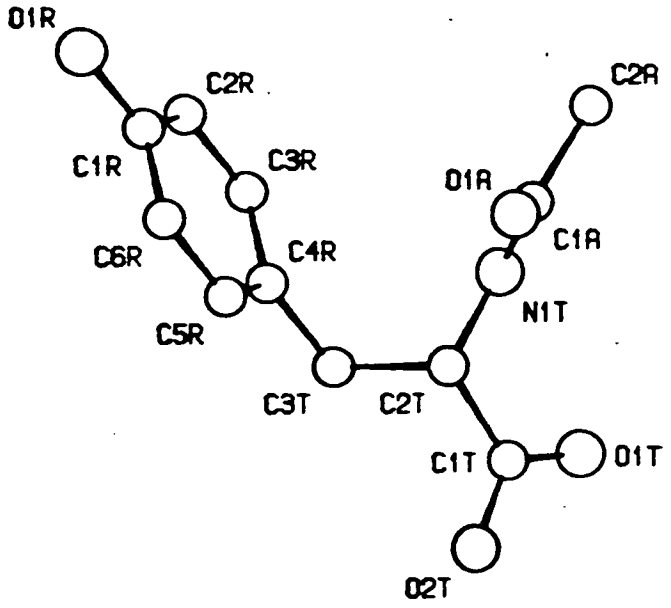
All bond lengths and angles in the N-acetyl tyrosine molecules are within three e.s.d's or less of their usual values, with no significant differences between the two possible molecules, but this to be

* **No hydrogen atoms were positioned on the disordered acetyl tyrosine moiety, but the hydrogen atoms on the strychnine cation were totally unconstrained. No hydrogen atoms were placed on the solvent molecules.**

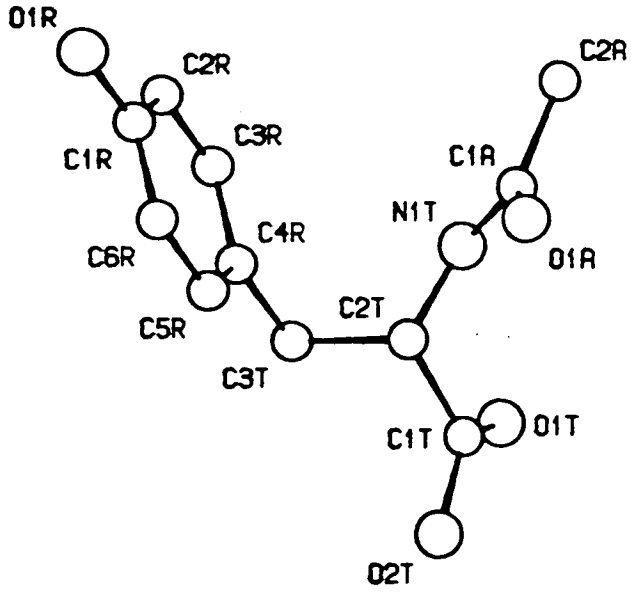
expected because the constraints used in refinement held all bond lengths and angles, with the exception of those involving the carboxylate oxygens, close to the ideal values. There is a difference between the bond lengths of the carboxylate oxygens, but it is not quite large enough to be considered significant.

bond	B site	L site
C(1T)-O(1T)	1.20(5)	1.46(5)
C(1T)-O(2T)	1.30(5)	1.43(6)

The two tyrosine molecules are illustrated below.



B SITE



L SITE

Both molecules take up a similar conformation, with the only significant difference being in the two torsion angles involving O(1T) of the carboxylate group.

C(3T)-C(2T)-C(1T)-O(1T)	115.9(41)	85.9(30)
N(1T)-C(2T)-C(1T)-O(1T)	-5.6(51)	-41.7(34)

This similarity of conformation is not a result of the constraints applied during refinement, as the torsion angles which define peptide conformation (those involving the Cα) were not held at any particular preconceived values. The torsion angles that were constrained were those of the aromatic ring and the amide group, which would not be expected to deviate from planarity in any case. The tables below list all bond lengths, angles and torsion angles for both the strychninium cation and the disordered tyrosine molecules, except for those fixed during refinement.

C1-C2	1.37(3)	C10-N2	1.46(3)
C1-C6	1.372(23)	C12-C13	1.48(3)
C1-N1	1.394(20)	C13-C14	1.499(23)
C2-C3	1.31(3)	C13-C16	1.57(3)
C3-C4	1.46(4)	C14-C19	1.584(24)
C4-C5	1.40(4)	C15-C16	1.48(3)
C5-C6	1.44(3)	C15-N2	1.43(3)
C6-C7	1.481(25)	C16-C17	1.35(3)
C7-C8	1.574(24)	C17-C18	1.57(3)
C7-C9	1.52(3)	C18-O2	1.42(3)
C7-C11	1.518(25)	C19-C20	1.46(3)
C8-C14	1.512(23)	C19-O2	1.438(22)
C8-N1	1.502(22)	C20-C21	1.50(3)
C9-C12	1.55(3)	C21-N1	1.420(23)
C9-N2	1.58(3)	C21-O1	1.206(23)
C10-C11	1.56(3)		

	B	L
C1T-O1T	1.20(5)	1.46(5)
C1T-O2T	1.30(5)	1.43(6)

C2-C1-C6	125.7(16)	C12-C13-C16	108.9(15)
C2-C1-N1	126.1(15)	C14-C13-C16	115.6(14)

C6-C1-N1	108.1(14)	C8-C14-C13	112.6(13)
C1-C2-C3	116.9(20)	C8-C14-C19	106.2(13)
C2-C3-C4	122.6(24)	C13-C14-C19	121.3(13)
C3-C4-C5	120.1(25)	C16-C15-N2	110.4(17)
C4-C5-C6	115.7(21)	C13-C16-C15	113.3(17)
C1-C6-C5	118.9(16)	C13-C16-C17	122.4(19)
C1-C6-C7	113.0(15)	C15-C16-C17	124.3(20)
C5-C6-C7	128.0(17)	C16-C17-C18	118.5(19)
C6-C7-C8	102.6(14)	C17-C18-O2	111.7(17)
C6-C7-C9	115.3(15)	C14-C19-C20	110.4(15)
C6-C7-C11	112.8(15)	C14-C19-O2	110.3(14)
C8-C7-C9	112.8(14)	C20-C19-O2	107.8(15)
C8-C7-C11	111.4(14)	C19-C20-C21	118.9(16)
C9-C7-C11	102.3(14)	C20-C21-N1	116.4(15)
C7-C8-C14	117.5(14)	C20-C21-O1	123.5(17)
C7-C8-N1	101.8(13)	N1-C21-O1	119.9(16)
C14-C8-N1	107.1(13)	C1-N1-C8	111.6(12)
C7-C9-C12	116.2(16)	C1-N1-C21	126.8(14)
C7-C9-N2	105.1(14)	C8-N1-C21	114.9(13)
C12-C9-N2	109.3(15)	C9-N2-C10	107.0(15)
C11-C10-N2	105.2(16)	C9-N2-C15	111.6(15)
C7-C11-C10	103.7(15)	C10-N2-C15	117.5(16)
C9-C12-C13	107.3(15)	C18-O2-C19	113.9(14)
C12-C13-C14	108.4(14)		

	R	L
C4R-C3T-C2T	112.6(16)	105.2(16)
C2T-N1T-C1A	119.1(21)	121.3(19)
C3T-C2T-N1T	109.4(19)	113.1(18)
C3T-C2T-C1T	112.3(24)	111.6(21)
N1T-C2T-C1T	109.1(24)	111.6(22)
C2T-C1T-O1T	122 (4)	109 (3)
C2T-C1T-O2T	116 (4)	122 (3)
O1T-C1T-O2T	122 (4)	123 (4)

C6-C1-C2-C3	-2 (3)
N1-C1-C2-C3	177.6(19)
C2-C1-C6-C5	-1.1(28)
C2-C1-C6-C7	175.9(17)
N1-C1-C6-C5	179.6(16)
N1-C1-C6-C7	-3.4(19)
C2-C1-N1-C8	172.2(17)
C2-C1-N1-C21	22.9(26)

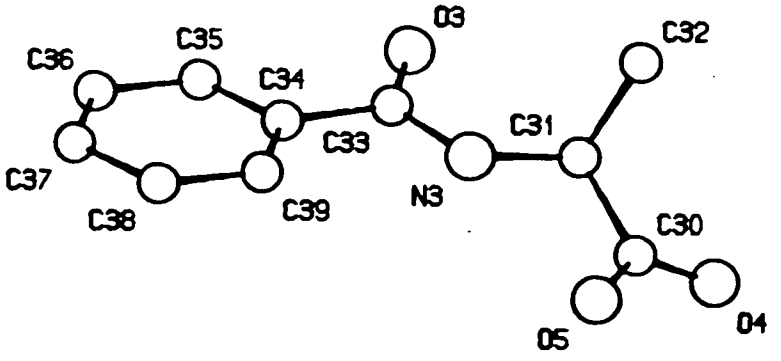
C6-C1-N1-C8	-8.5(18)
C6-C1-N1-C21	-157.9(16)
C1-C2-C3-C4	4 (4)
C2-C3-C4-C5	-4 (4)
C3-C4-C5-C6	1 (4)
C4-C5-C6-C1	1 (3)
C4-C5-C6-C7	-175.4(21)
C1-C6-C7-C8	12.8(19)
C1-C6-C7-C9	135.8(16)
C1-C6-C7-C11	-107.2(17)
C5-C6-C7-C8	-170.5(18)
C5-C6-C7-C9	-47.5(26)
C5-C6-C7-C11	69.5(24)
C6-C7-C8-C14	100.6(16)
C6-C7-C8-N1	-16.0(16)
C9-C7-C8-C14	-24.1(21)
C9-C7-C8-N1	-140.7(14)
C11-C7-C8-C14	-138.5(15)
C11-C7-C8-N1	104.9(15)
C6-C7-C9-C12	-84.6(20)
C6-C7-C9-N2	154.4(15)
C8-C7-C9-C12	32.8(22)
C8-C7-C9-N2	-88.2(17)
C11-C7-C9-C12	152.6(16)
C11-C7-C9-N2	31.6(17)
C6-C7-C11-C10	-165.4(15)
C8-C7-C11-C10	79.8(17)
C9-C7-C11-C10	-41.0(17)
C7-C8-C14-C13	38.7(20)
C7-C8-C14-C19	173.6(14)
N1-C8-C14-C13	152.3(13)
N1-C8-C14-C19	-72.8(15)
C7-C8-N1-C1	15.6(16)
C7-C8-N1-C21	168.9(14)
C14-C8-N1-C1	-108.3(14)
C14-C8-N1-C21	45.0(18)
C7-C9-C12-C13	-56.2(21)
N2-C9-C12-C13	62.5(18)
C7-C9-N2-C10	-10.3(19)
C7-C9-N2-C15	119.5(17)
C12-C9-N2-C10	-135.7(16)
C12-C9-N2-C15	-5.9(21)
N2-C10-C11-C7	35.2(18)
C11-C10-N2-C9	-14.9(19)
C11-C10-N2-C15	-141.3(17)

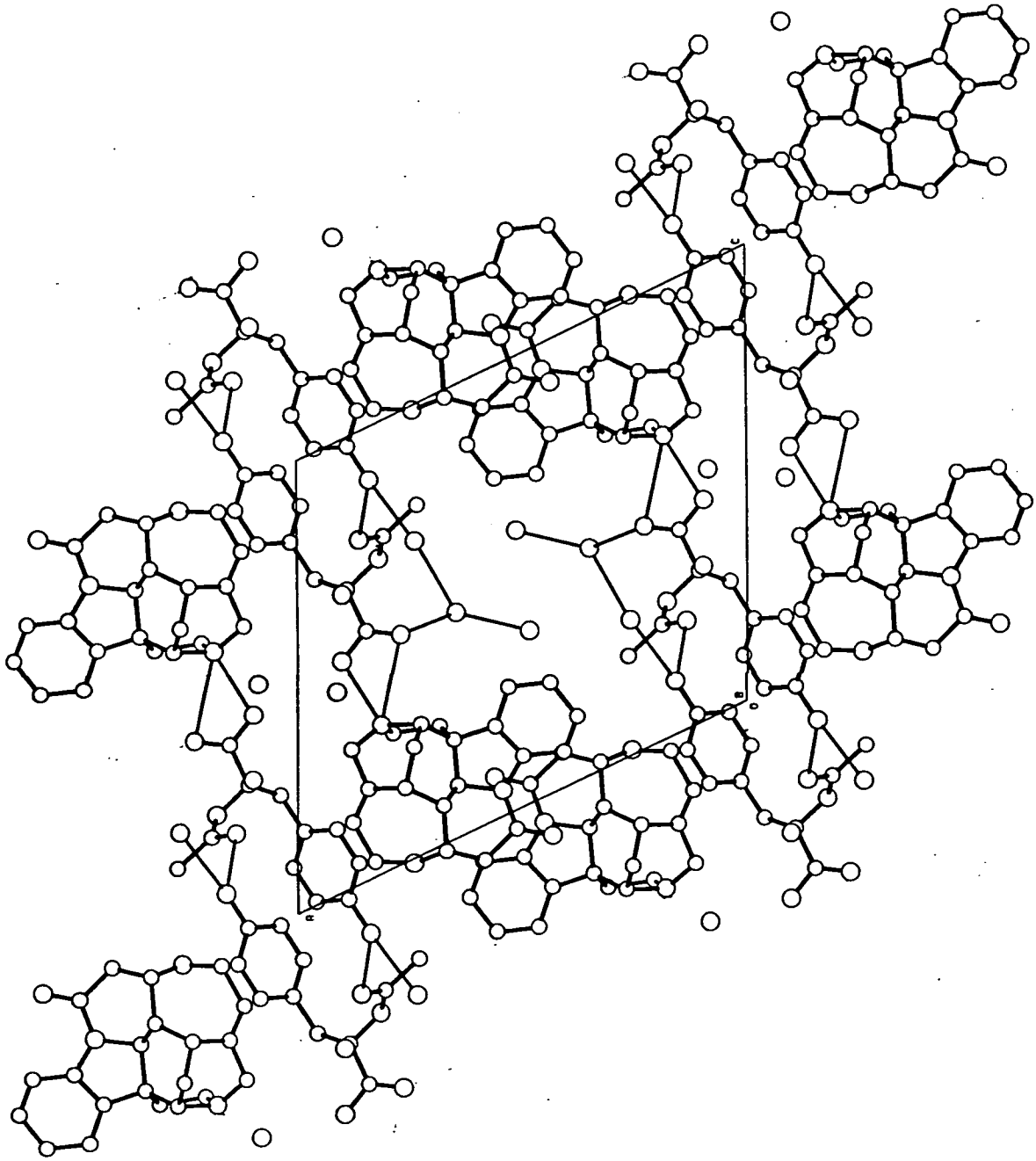
C9-C12-C13-C14	69.1(18)
C9-C12-C13-C16	-57.5(19)
C12-C13-C14-C8	-62.0(18)
C12-C13-C14-C19	170.7(15)
C16-C13-C14-C8	60.6(19)
C16-C13-C14-C19	-66.7(20)
C12-C13-C16-C15	-0.3(23)
C12-C13-C16-C17	-179.9(20)
C14-C13-C16-C15	-122.6(18)
C14-C13-C16-C17	57.9(26)
C8-C14-C19-C20	44.2(18)
C8-C14-C19-O2	-74.8(16)
C13-C14-C19-C20	174.3(15)
C13-C14-C19-O2	55.3(20)
N2-C15-C16-C13	58.5(22)
N2-C15-C16-C17	-122.0(23)
C16-C15-N2-C9	-52.7(21)
C16-C15-N2-C10	71.4(22)
C13-C16-C17-C18	-3 (3)
C15-C16-C17-C18	177.4(19)
C16-C17-C18-O2	-66.4(25)
C17-C18-O2-C19	95.4(19)
C14-C19-C20-C21	9.7(23)
O2-C19-C20-C21	130.2(17)
C14-C19-O2-C18	-68.9(18)
C20-C19-O2-C18	170.6(16)
C19-C20-C21-N1	-39.8(24)
C19-C20-C21-O1	145.6(19)
C20-C21-N1-C1	158.6(15)
C20-C21-N1-C8	10.1(21)
O1-C21-N1-C1	-26.6(26)
O1-C21-N1-C8	-175.1(16)

	B	L
C2T-C3T-C4R-C3R	99.5(21)	100.5(20)
C2T-C3T-C4R-C5R	-80.4(22)	-79.5(21)
C4R-C3T-C2T-N1T	-67.3(22)	-73.2(21)
C4R-C3T-C2T-C1T	171.4(24)	160.0(21)
C3T-C2T-N1T-C1A	115.7(24)	128.1(21)
C1T-C2T-N1T-C1A	-121 (3)	-105.1(27)
C2T-N1T-C1A-O1A	-3 (4)	5 (3)
C2T-N1T-C1A-C2A	178.6(21)	-169.4(20)
C3T-C2T-C1T-O1T	116 (4)	86 (3)
C3T-C2T-C1T-O2T	-64 (4)	-67 (4)

N1T-C2T-C1T-O1T	-6	(5)		-42	(3)
N1T-C2T-C1T-O2T	175	(3)		166	(3)

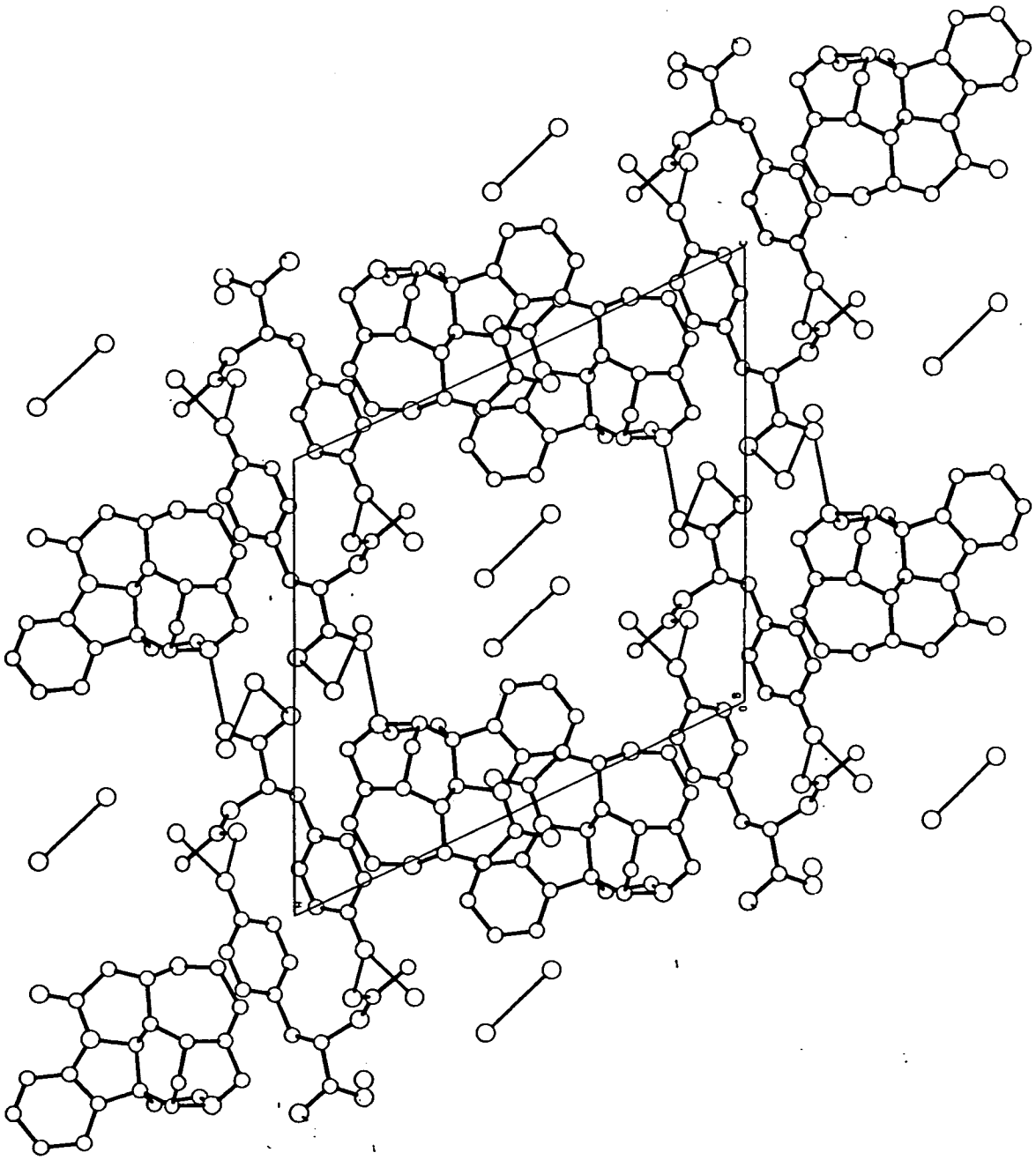
The next four pages show projections down the b axis for two possible arrangements of the strychnine tyrosine complex and projections down the a and c axes of the strychnine alanine complex. The numbering scheme used for the benzoyl alanine molecule is shown below.





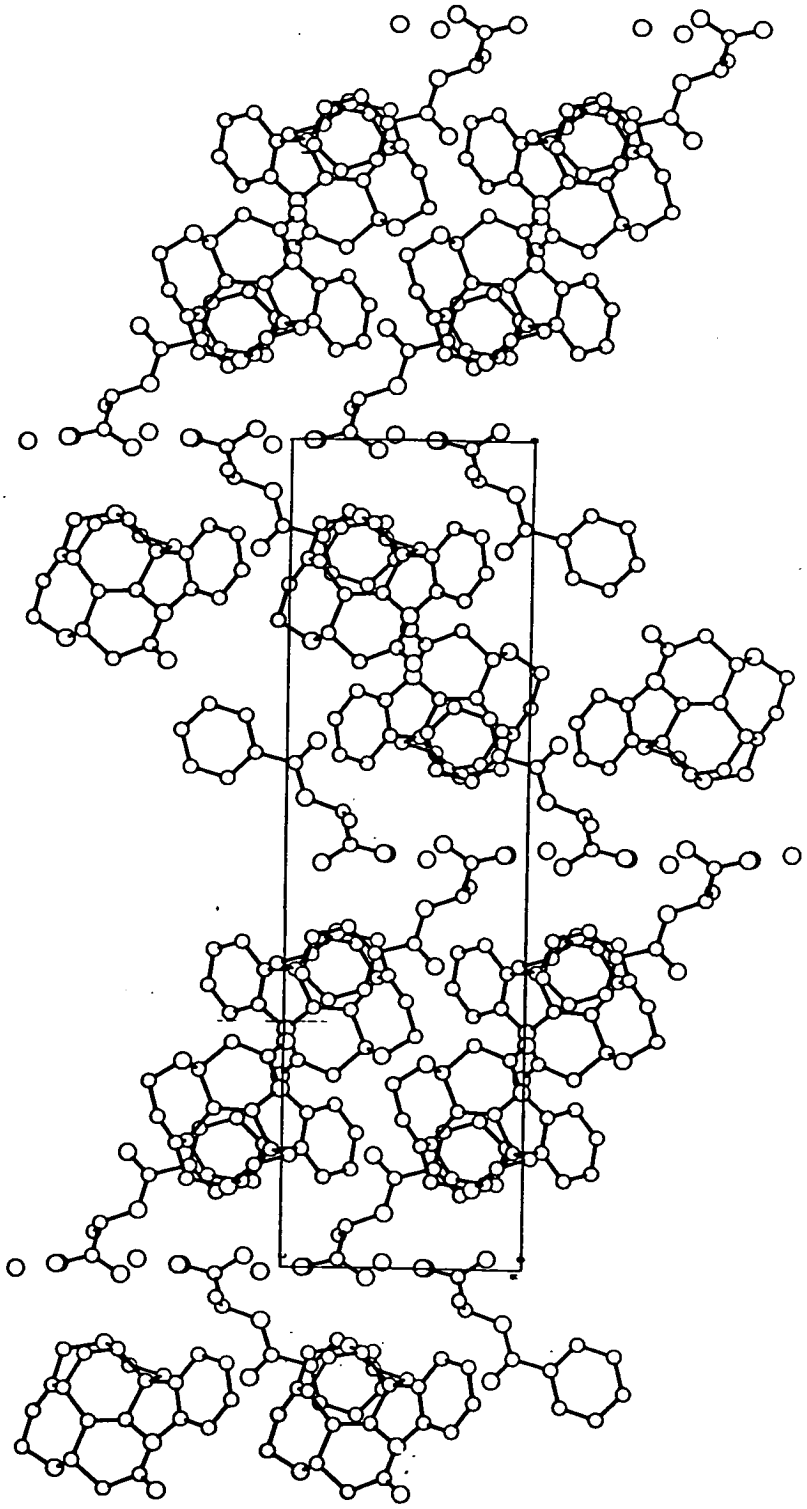
PROJECTION DOWN Y AXIS FOR 'B' SITE

STRYCHNINE : N-ACETYL-L-TYROSINE TETRAHYDRATE



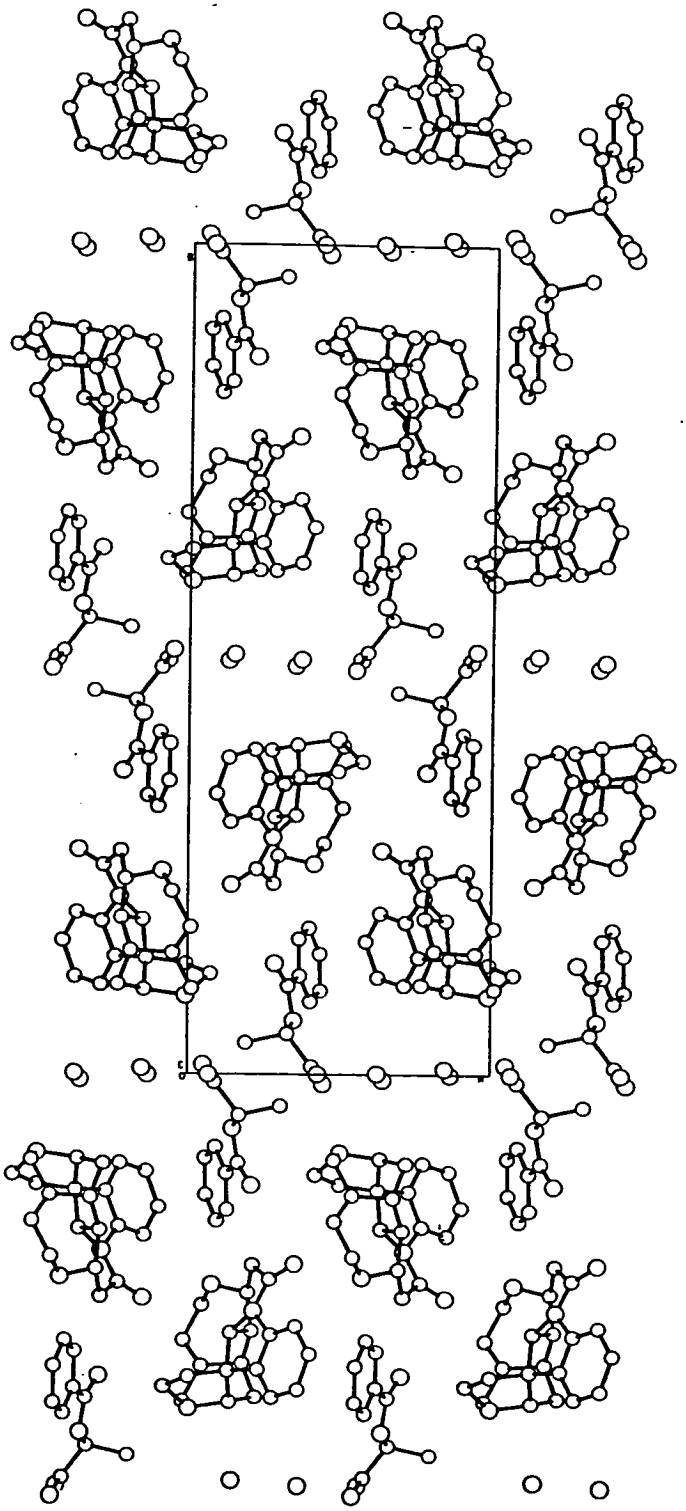
PROJECTION DOWN Y AXIS FOR 'L' SITE

STRYCHNINE : N-ACETYL-L-TYROSINE TETRAHYDRATE



PROJECTION DOWN THE X AXIS

STRYCHNINE : N-BENZOYL-L-ALANINE DIHYDRATE



PROJECTION DOWN THE Z AXIS

STRYCHNINE : N-BENZOYL-L-ALANINE DIHYDRATE

The only close contact between adjacent strychnine molecules involves the carbonyl oxygen O(1) and the methene carbon of the pyrrole ring C(11)

C11	3.393 (23)	01	(1 -x,-1/2+y,-z)

There are eleven contacts closer than 3.5 angstroms between the strychnine and the tyrosine molecules. It is noticeable that the aromatic residue of the amino acid makes contact with the oxepin ring, the carboxylate group with the immediate surroundings of the quaternary nitrogen and the phenol oxygen comes close to the aromatic ring of the strychnine.

C3	3.49 (3)	O1RB	(1 +x,y,z)
C9	3.29 (3)	O1TB	(x,y,-1 +z)
C9	3.48 (4)	O2TB	(x,y,-1 +z)
C9	3.40 (4)	O1TL	(x,y,-1 +z)
C10	3.38 (4)	O2TB	(x,y,-1 +z)
C1	3.470 (5)	O2TL	(-x,-1/2+y,-z)
C15	3.07 (4)	O2TB	(x,y,-1 +z)
C17	3.463 (28)	C5RL	(-x,-1/2+y,-z)
C18	3.47 (3)	C3RL	(-x,1/2+y,-z)
N2	3.26 (5)	C1TB	(x,y,-1 +z)
O2	3.352 (22)	C6RL	(-x,-1/2+y,-z)

These may be compared with the equivalent close contacts to strychnine in the strychnine: N-benzoylalanine complex. There is a similar contact between O(1) and C(11) and again it can be seen that the carboxylate group is in close contact with the protonated nitrogen (C30 is the C', O4 and O5 are the carboxylate oxygens and O3 is the benzoyl group oxygen). The benzoyl oxygen, which does not take part in hydrogen bonding, has three close contacts to hydrophobic regions of the strychnine molecule.

O2	3.408 (7)	C2	(1/2+x,1/2-y,-z)
O1	3.497 (7)	C8	(-1/2+x,1/2-y,-z)
O1	3.450 (8)	C10	(-1/2+x,1/2-y,-z)
O1	3.329 (7)	C11	(-1/2+x,1/2-y,-z)

C30	3.385 (7)	N2	(1 -x,-1/2+y,1/2-z)
O3	3.427 (7)	C3	(1/2+x,1/2-y,1 -z)
O3	3.420 (7)	C17	(-1/2+x,1/2-y,-z)
O3	3.263 (7)	C20	(x,y,1 +z)
O5	3.453 (7)	C9	(1 -x,-1/2+y,1/2-z)
O5	3.365 (7)	C10	(1 -x,-1/2+y,1/2-z)
O5	3.475 (8)	C15	(1 -x,-1/2+y,1/2-z)
O4	3.230 (8)	C15	(1 -x,-1/2+y,1/2-z)

The close contacts between tyrosine molecules are mostly between the aromatic ring and nearby oxygen atoms and there is one close contact between two aromatic rings. The carbon atoms of one of the amide side chains have close contact with the phenolic oxygens, but this may be a consequence of the amide oxygen: aromatic ring interaction, and not an actual interaction in itself.

C3RB	3.376 (26)	O1RB	(-x,-1/2+y,-z)
C1RL	3.204 (28)	O1AL	(-x,-1/2+y,-z)
C2RL	3.306 (28)	O1AL	(-x,-1/2+y,-z)
C1AL	3.152 (28)	O1RL	(-x,1/2+y,-z)
C2AL	3.221 (28)	O1RL	(-x,1/2+y,-z)
C1RB	3.477 (28)	O1AL	(-x,-1/2+y,-z)
C2RB	3.434 (28)	O1AL	(-x,-1/2+y,-z)
C3RB	3.466 (26)	C1RL	(-x,-1/2+y,-z)
C3RB	3.204 (26)	O1RL	(-x,-1/2+y,-z)
O1RB	3.381 (28)	C1AL	(-x,-1/2+y,-z)
O1RB	3.273 (28)	C2AL	(-x,-1/2+y,-z)
O1AB	3.38 (3)	C1RL	(-x,1/2+y,-z)
O1AB	3.40 (3)	C2RL	(-x,1/2+y,-z)
C1AB	3.46 (3)	O1RL	(-x,1/2+y,-z)

The model used to describe this structure permits the existence of two self-consistent hydrogen bonding schemes, each of which connect the strychninium cation and the fully ocupied water positions to one of the disordered tyrosine molecules via a different set of partially occupied water molecules. There are also a number of interconnections between the two systems, allowing adjacent asymmetric units to have either possible amino acid position occupied, without causing any long-range ordering of the cells. There are only four pairs of water

sites that may not be occupied simultaneously (marked '*'), allowing a very large number of possible hydrogen bonding schemes to be drawn up, depending on which combination of disordered molecules is used.

N2		3.16	(3)	O1TB	(x,y,-1 +z)
N2		2.56	(3)	O2TB	(x,y,-1 +z)
N1TB		2.58	(4)	O1W2	(x,y,z)
O1TB		2.99	(7)	O1WB	(1 -x,-1/2+y,1 -z)
O2TB		2.86	(4)	O1W1	(x,y,z)
O1RB		2.89	(3)	O1AB	(-x,-1/2+y,-z)
O1RB		2.98	(3)	O1W2	(-x,1/2+y,-z)
O1AB		2.69	(7)	O2WB	(x,y,z)
O1W1		3.38	(7)	O2WB	(-x,-1/2+y,1 -z)
O1W2		2.83	(7)	O1WB	(1 -x,-1/2+y,1 -z)
O1W2		3.44	(7)	O2WB	(x,-1 +y,z)
O1WB		2.55	(9)	O3WB	(1 -x,-1/2+y,1 -z)
N2		2.86	(4)	O1TL	(x,y,-1 +z)
N1TL		3.27	(3)	O1W2	(x,y,z)
O2TL		2.35	(6)	O1W1	(x,y,z)
O2TL		2.77	(6)	O1W1	(-x,1/2+y,1 -z)
O1RL		2.318	(28)	O1AL	(-x,-1/2+y,-z)
O1RL		2.72	(3)	O1W2	(-x,1/2+y,-z)
O1AL		3.18	(7)	O1WL	(x,y,z)
O1W1		2.58	(7)	O1WL	(-x,-1/2+y,1 -z)
O2WL		3.18	(9)	O3WL	(x,1 +y,z)
O1TB		3.49	(7)	O1WL	(x,-1 +y,z)
O1RB		2.664	(28)	O1AL	(-x,-1/2+y,-z)
O1RL		2.60	(3)	O1AB	(-x,-1/2+y,-z)
O1AB		3.46	(7)	O1WL	(x,y,z)
O1RL		3.50	(7)	O2WB	(-x,-1/2+y,-z)
O1AL	*	2.30	(7)	O2WB	(x,y,z)
O1WB		3.20	(9)	O1WL	(1 -x,-1/2+y,1 -z)
O1WB	*	2.14	(9)	O2WL	(x,-1 +y,z)
O3WB	*	1.55	(9)	O2WL	(1 -x,-1/2+y,1 -z)
O3WB	*	1.85	(9)	O3WL	(1 -x,1/2+y,1 -z)

The hydrogen bonding scheme in the strychnine: benzoylalanine complex is comparatively simple, involving only two water molecules and three other atoms (the quaternary nitrogen and the carboxylate group of the alanine) in a 'tighter', more rigid three dimensional network.

O5	2.670	(6)	N2	(1 -x,-1/2+y,1/2-z)
O4	3.423	(7)	N2	(1 -x,-1/2+y,1/2-z)
O5	2.836	(7)	O91W	(1/2+x,1/2-y,-z)
O4	2.817	(9)	O92W	(1 -x,-1/2+y,1/2-z)
O91W	2.785	(10)	O92W	(x,y,z)
O91W	2.881	(10)	O92W	(1/2-x,1 -y,1/2+z)

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(1)	0.5002(11)	-0.06837(0)	-0.1137(12)	0.0810
C(2)	0.5909(12)	-0.0472(23)	-0.0804(16)	0.0866
C(3)	0.6212(13)	-0.022(3)	-0.1448(18)	0.1208
C(4)	0.5621(19)	-0.005(3)	-0.2476(23)	0.1640
C(5)	0.4690(12)	-0.027(3)	-0.2813(14)	0.1123
C(6)	0.4385(11)	-0.0617(23)	-0.2086(11)	0.0755
C(7)	0.3462(10)	-0.102(3)	-0.2225(9)	0.0738
C(8)	0.3548(10)	-0.0943(24)	-0.1166(10)	0.0721
C(9)	0.2736(10)	0.015(3)	-0.2912(12)	0.0866
C(10)	0.2112(11)	-0.270(3)	-0.3004(14)	0.0915
C(11)	0.3144(10)	-0.2751(24)	-0.2677(11)	0.0760
C(12)	0.2580(11)	0.182(3)	-0.2474(12)	0.0815
C(13)	0.2385(10)	0.1353(23)	-0.1656(10)	0.0701
C(14)	0.3224(9)	0.0651(20)	-0.0866(10)	0.0569
C(15)	0.1179(12)	-0.020(3)	-0.3076(13)	0.0919
C(16)	0.1551(11)	0.014(3)	-0.2030(16)	0.1016
C(17)	0.1213(12)	-0.050(3)	-0.1442(15)	0.1055
C(18)	0.1706(13)	-0.008(3)	-0.0335(15)	0.1135
C(19)	0.3287(12)	0.0279(23)	0.0173(10)	0.0753
C(20)	0.4139(12)	-0.0556(24)	0.0778(12)	0.0901
C(21)	0.4848(12)	-0.0668(22)	0.0419(12)	0.0731
N(1)	0.4547(7)	-0.1025(18)	-0.0578(9)	0.0642
N(2)	0.1847(9)	-0.095(3)	-0.3311(9)	0.0930
O(1)	0.5641(9)	-0.0591(21)	0.0940(8)	0.1177
O(2)	0.2574(7)	-0.0837(18)	0.0105(7)	0.0872
H(2)	0.6339(12)	-0.0477(23)	-0.0038(16)	0.2771
H(3)	0.6927(13)	-0.012(3)	-0.1212(18)	0.1714
H(4)	0.5884(19)	0.016(3)	-0.2998(23)	0.2324
H(5)	0.4221(12)	-0.013(3)	-0.3562(14)	0.0811
H(8)	0.3144(10)	-0.1948(24)	-0.1080(10)	0.0893
H(9)	0.2916(10)	0.055(3)	-0.3477(12)	0.0255
H(101)	0.1953(11)	-0.302(3)	-0.2413(14)	0.0474
H(102)	0.1771(11)	-0.356(3)	-0.3596(14)	0.0429
H(111)	0.3439(10)	-0.3753(24)	-0.2154(11)	0.0590
H(112)	0.3299(10)	-0.2958(24)	-0.3283(11)	0.0575
H(121)	0.2042(11)	0.256(3)	-0.3009(12)	0.1611
H(122)	0.3192(11)	0.256(3)	-0.2205(12)	0.0055
H(13)	0.2172(10)	0.2416(23)	-0.1362(10)	0.1352
H(14)	0.3659(9)	0.1728(20)	-0.0774(10)	0.0636
H(151)	0.0639(12)	-0.111(3)	-0.3254(13)	0.0775
H(152)	0.0922(12)	0.095(3)	-0.3479(13)	0.0648
H(17)	0.0631(12)	-0.131(3)	-0.1730(15)	1.0535
H(181)	0.1303(13)	-0.056(3)	0.0005(15)	2.7469
H(182)	0.1780(13)	0.128(3)	-0.0234(15)	0.1406
H(19)	0.3204(12)	0.1469(23)	0.0475(10)	0.0765
H(201)	0.3991(12)	-0.1843(24)	0.0907(12)	0.1091
H(202)	0.4431(12)	0.0128(24)	0.1453(12)	0.1493
H(1N2)	0.1503(9)	-0.094(3)	-0.4089(9)	1.4424

Table Fractional Coordinates of Atoms with Standard Deviations

	x	y	z	Ueq
C(3TB)	0.0379(12)	-0.184(3)	0.2789(9)	0.0857
C(1RB)	-0.1106(12)	-0.057(3)	-0.0217(9)	0.0736
C(2RB)	-0.0392(12)	-0.172(3)	0.0071(9)	0.0607
C(3RB)	0.0091(12)	-0.213(3)	0.1047(9)	0.0663
C(4RB)	-0.0142(12)	-0.139(3)	0.1735(9)	0.0987
C(5RB)	-0.0855(12)	-0.025(3)	0.1445(9)	0.0651
C(6RB)	-0.1338(12)	0.016(3)	0.0468(9)	0.0858
O(1RB)	-0.1584(12)	-0.017(3)	-0.1183(9)	0.0739
C(2TB)	0.1079(14)	-0.049(3)	0.3349(16)	0.0803
C(3TL)	-0.0070(12)	-0.131(3)	0.2591(9)	0.0732
C(1RL)	-0.1168(12)	-0.042(3)	-0.0471(9)	0.0906
C(2RL)	-0.0468(12)	-0.158(3)	-0.0034(9)	0.1075
C(3RL)	-0.0111(12)	-0.187(3)	0.0960(9)	0.0752
C(4RL)	-0.0454(12)	-0.100(3)	0.1517(9)	0.0631
C(5RL)	-0.1153(12)	0.017(3)	0.1080(9)	0.0731
C(6RL)	-0.1510(12)	0.046(3)	0.0085(9)	0.0943
O(1RL)	-0.1521(12)	-0.013(3)	-0.1456(9)	0.0949
C(2TL)	0.0565(13)	0.018(3)	0.3054(16)	0.0834
N(1TB)	0.1791(13)	-0.047(3)	0.3012(16)	0.0891
O(1AB)	0.1357(13)	0.224(3)	0.2396(16)	0.1275
C(1AB)	0.1901(13)	0.094(3)	0.2569(16)	0.1500
C(2AB)	0.2659(13)	0.097(3)	0.2239(16)	0.1414
C(1TB)	0.151(3)	-0.080(8)	0.4441(14)	0.1500
O(1TB)	0.2289(17)	-0.112(4)	0.4892(17)	0.1274
O(2TB)	0.0967(18)	-0.072(4)	0.4855(18)	0.1225
N(1TL)	0.1401(12)	0.008(3)	0.2935(15)	0.0802
O(1AL)	0.1248(12)	0.284(3)	0.2389(15)	0.1087
C(1AL)	0.1691(12)	0.142(3)	0.2568(15)	0.0746
C(2AL)	0.2467(12)	0.126(3)	0.2274(15)	0.1703
C(1TL)	0.0754(23)	0.041(6)	0.4111(19)	0.1500
O(1TL)	0.1496(22)	-0.069(5)	0.4706(17)	0.1163
O(2TL)	0.007(3)	0.092(7)	0.438(3)	0.2208
O(1W1)	-0.0860(14)	-0.125(4)	0.4498(17)	0.2739
O(1W2)	0.2609(15)	-0.326(4)	0.3008(17)	0.2642
O(1WB)	0.649(3)	0.100(9)	0.500(4)	0.2000
O(2WB)	0.098(4)	0.463(9)	0.341(4)	0.2000
O(3WB)	0.515(4)	0.547(9)	0.616(4)	0.2000
O(1WL)	0.149(4)	0.472(9)	0.431(4)	0.2000
O(2WL)	0.564(4)	0.943(9)	0.382(4)	0.2000
O(3WL)	0.416(3)	0.048(9)	0.452(4)	0.2000

Thermal Vibration Parameters with Standard Deviations

	U11	U22	U33	U23	U13	U12
C(1)	0.0886(130)	0.0748(134)	0.0733(112)	-0.0041(105)	0.0585(118)	0.0201(115)
C(2)	0.0683(129)	0.0619(127)	0.1075(151)	0.0029(114)	0.0349(112)	0.0043(106)
C(3)	0.0774(135)	0.1430(230)	0.1184(170)	0.0147(174)	0.0410(137)	0.0401(146)
C(4)	0.1560(219)	0.1148(200)	0.2136(288)	-0.0095(203)	0.1461(227)	0.0085(187)
C(5)	0.0716(114)	0.1416(201)	0.1115(148)	0.0094(138)	0.0571(113)	0.0204(131)
C(6)	0.0763(107)	0.0828(135)	0.0545(104)	0.0010(104)	0.0322(89)	0.0207(109)
C(7)	0.0675(109)	0.1083(163)	0.0229(79)	0.0096(107)	-0.0033(69)	0.0186(119)
C(8)	0.0690(105)	0.0580(117)	0.0680(102)	0.0002(98)	0.0195(85)	-0.0046(102)
C(9)	0.0698(120)	0.1031(162)	0.0615(108)	0.0095(116)	0.0097(97)	-0.0040(119)
C(10)	0.0705(124)	0.0873(165)	0.0914(137)	-0.0399(132)	0.0230(106)	-0.0001(118)
C(11)	0.0749(120)	0.0744(138)	0.0529(100)	-0.0345(109)	0.0073(82)	0.0017(105)
C(12)	0.0597(107)	0.0760(135)	0.0866(123)	0.0046(114)	0.0221(104)	0.0044(98)
C(13)	0.0585(99)	0.0857(133)	0.0539(100)	-0.0166(98)	0.0262(90)	-0.0066(98)
C(14)	0.0447(83)	0.0462(110)	0.0678(101)	-0.0110(83)	0.0265(76)	-0.0054(81)
C(15)	0.0634(109)	0.1041(180)	0.0732(125)	-0.0400(128)	-0.0058(88)	-0.0199(120)
C(16)	0.0694(111)	0.0853(157)	0.1253(175)	-0.0268(138)	0.0379(123)	0.0207(116)
C(17)	0.0962(129)	0.0700(138)	0.1394(164)	-0.0036(137)	0.0819(134)	0.0122(116)
C(18)	0.0957(142)	0.0931(186)	0.1309(185)	-0.0149(141)	0.0590(131)	0.0098(131)
C(19)	0.1107(134)	0.0373(95)	0.0564(103)	-0.0135(89)	0.0322(102)	0.0015(110)
C(20)	0.1063(140)	0.0539(125)	0.0772(120)	0.0199(110)	0.0180(108)	-0.0062(117)
C(21)	0.0798(118)	0.0531(114)	0.0684(120)	0.0137(101)	0.0307(103)	0.0262(105)
N(1)	0.0593(74)	0.0438(84)	0.0635(89)	0.0025(75)	0.0045(70)	0.0018(68)
N(2)	0.0684(94)	0.1209(155)	0.0612(82)	-0.0141(100)	0.0030(74)	0.0227(110)
O(1)	0.0901(86)	0.1293(128)	0.0861(86)	-0.0206(91)	-0.0126(71)	0.0319(96)
O(2)	0.0832(74)	0.0845(87)	0.0830(69)	-0.0073(75)	0.0513(62)	-0.0186(86)

REFERENCES

REFERENCES

Abderhalden, E. & Sichel, H., (1923) Z. Physiol. Chem. **131**, 277

Aberhart, D. J., (1977) Tetrahedron, **33**, 1545

Arnstein, H. R. V. & Clubb, M. E., (1958) Biochem. J. **68**, 528

Ashida, T., Hata, Y., Matsuura, Y., Tanaka, N. & Kakudo, M., (1977) Acta Cryst. **B33**, 3561-4

Baldwin, J. E. & Wan, T. S., (1981) Tetrahedron, **31**, 1589-95

Baldwin, J. E., Bahudur, G., Wan, T., Jung, M., Abraham, E. P., Huddleston, J. A. & White, R. L., (1981) J. Chem. Soc. Chem. Commun. **1981**, 1146-7

Barfield, M. & Karplus, M., (1969) J. Am. Chem. Soc. **91**, 1-10

Barfield, M. & Gearhart, H. L., (1973) J. Am. Chem. Soc. **95**(2), 641-3

Beevers, C. A., (1976) Proc. 3rd European Crystallographic Meeting, Zurich, 257.

Beevers, C. A. & Robertson, J. H., (1951) Acta Cryst. **4**, 270-5

Behr, J.-P., Lehn, J.-M. & Vierling, P., (1982) Helv. Chim. Acta, **65**(6), 1853-67

Berg, C. P., (1936) J. Biol. Chem. **115**, 9-15

Berman, H. M., Stallings, W., Carrell, H. L., Glusker, J. P., Neidle, S., Taylor, G. & Achari, A., (1979) Biopolymers, **18**, 2405

Bokhoven, C., Schoone, J. C. & Bijvoet, J. M., (1951) Acta Cryst. **4**, 275-80

Bradshaw, J. S., Jones, B. A., Davidson, R. B., Christensen, J. J., Lamb, J. D., Izatt, R. M., Morin, F. G. & Grant, D. M., (1982) J. Org. Chem. **47**, 3362-4

Bystrov, V. F., Portnova, S. L., Tsetlin, V. I., Ivanov, V. T. & Ovchinnikov, Yu. A., (1969) Tetrahedron, **25**, 493-515

Capasso, S., Mattia, C. & Mazzarella, L., (1977) Acta Cryst. **B33**, 2080-83

Capasso, S., Mazzarella, L. & Tancredi, T., (1979) Biopolymers, **18**, 1555-8

Carey, F. A. & Kuehne, M. E., (1982) J. Org. Chem. **47**(20), 3811-5

Chandrasekaran, R. & Mitra, A. K., (1977) Int. J. Peptide Protein Res. **10**, 235-9

Chandrasekaran, R. & Balasubramanian, R., (1969) Biochim. Biophys. Acta, **188**, 1-9

Ciunik, Z. & Glowiak, T., (1983) Acta Cryst. **C39**, 1271-3

Clarke, H. T., Johnson, J. R. & Robinson, R., (Eds.), (1949) "The Chemistry of Penicillins", Princeton Univ. Press, Princeton, New Jersey, 472 et seq.,

Cram, D. J., Lingenfelter, D. S. & Helgeson, R. C., (1981) J. Org. Chem. **46**, 393-406

Davis, D. G., Live, D. H., Agosta, W. C. & Cowburn, D., (1982) Org. Mag. Reson. **19**(4), 211-5

Dawson, B., (1953) Acta Cryst. **6**, 81-7

De Marco, A. & Llinas, M., (1980) J. Magn. Reson. **39**, 253-62

DeWitt, H. D. & Ingersoll, A. W., (1951) J. Am. Chem. Soc. **73**, 5782-3

Dobashi, A. & Hara, S., (1983) Tetrahedron Letters, **24**(14), 1509-10

Dunitz, J. D. & Winkler, F. K., (1975) Acta Cryst. **B31**, 251-63

Dunn, M. S. & Rockland, R. B., (1947) Adv. Protein Chem. **3**, 335-9

Endo, T. Miyazawa, K., Endo, M., Uchida, A., Ohashi, Y. & Sasada, Y., (1982) Chem. Lett. **1982**, 1989-92

Endo, T., Tasai, H., Miyazawa, K., Endo, M., Kato, K., Uchida, A., Ohashi, Y. & Sasada, Y., (1983) J. Chem. Soc. Chem. Commun. **1983**(11), 636-8

Fischer, E., (1900) Ber. **33**, 3638

Foldi, Z., (1954) Acta Chim. Acad. Sci. Hung. **5**, 187 (from Chemical Abstracts, **50**, 981i)

Fraga, S., (1983) J. Mol. Struct., **94**, 251-60 THEOCHEM

Gorman, A. A., Hamblet, I., Smith, K. & Standen, M. C., (1984) Tetrahedron Letters, **25**(5), 581-4

Gould, R. O., Cleasby, A., Moulden, N. & Walkinshaw, M. D., (1981) Acta Cryst. **A37**, C72 03. 3-11

Gould, R. O., Taylor, P. & Walkinshaw, M. D., (1984) Manuscript in preparation

Greenstein, J. P. & Winitz, M., (1961) "Chemistry of the Amino Acids", **1**, John Wiley (New York and London), 715 et seq.

Greenstein, J. P., (1954) Adv. Protein Chem, **9**, 129-202

Hall, D. L. & Sanders, J. K. M., (1980) J. Am. Chem. Soc. **102**(18), 5703-11

Hossain, M. B., Van der Helm, D., Olsen, R. K., Jones P. G., Sheldrick, G. M., Egert, E. T., Kennard, O., Waring, M. J., Viswamitra, M
J. Am. Chem. Soc. **104**, 3401

Hyatt, J. A., Duesler, E. N., Curtin, D. Y. & Paul, K., (1980) J. Org. Chem. **43**, 5074-9

Ingersoll, A. W. & DeWitt, H. D., (1951) J. Am. Chem. Soc. **73**, 3360-2

Inoue, M. Shibata, M. & Ishida, T., (1980) Biochem. Biophys. Res. Comm. **93**(2), 415-9

Inoue, M., Hurano, H., Sugiyama, K. & Ishida, T., (1982) Biochem. Biophys. Res. Comm. **108**(2), 604-9

Inoue, M., Okuda, Y., Ishida, T. & Nakagaki, M., (1983) Arch. Biochem. Biophys. **227**(1), 52-70

Inoue, M., Sakaki, T., Fujiwara, T., & Tomita, K.-I., (1978ii) Bull. Chem. Soc. Jpn. **51**(4), 1123-7

Inoue, M., Sakaki, T., Fujiwara, T. & Tomita, K.-I., (1978i) Bull. Chem. Soc. Jpn. **51**(4), 1118-22

Inoue, M., Shibata, M., Kondo, Y.-I. & Ishida, T., (1981) Biochemistry, **20**(10), 2936-45

Isenberg, N. & Grdinic, M., (1972) J. Chem. Ed. **49**(6), 392-7

Ishida, T. & Inoue, M., (1981) Acta Cryst. **B37**, 2117-2120

Ishida, T. & Inoue, M., (1979) J. Chem. Soc. Chem. Commun. 358-360

Ishida, T. & Inoue, M., (1981) Biochem. Biophys. Res. Comm. **99**(1), 149-54

Ishida, T., Inoue, M. & Tomita, K.-I., (1979) Acta Cryst. **B35**, 1642-8

Ishida, T., Inoue, M., Senda, S. & Tomita K.-I., (1979) Bull. Chem. Soc. Jpn. **52**(10), 2953-8

Ishida, T., Inoue, M., Ota, A., Kurihara, T., (1980) J. Chem. Soc. Chem. Commun. **1980**, 1074-5

Ishida, T., Mitoguchi, S., Miyamoto, Y., Tomita, K.-I. & Inoue, M., (1980) Biochim. Biophys. Acta, **609**, 158-72

Ishida, T., Miyamoto, Y., Nakamura, A. & Inoue, M., (1982) Acta Cryst. **B38**, 192-5

Ishida, T., Tomita, K.-I., Inoue, M., (1980) Arch. Biochem. Biophys. **200**(2), 492-502

Ishida, T., Usami, H., Inoue, M., Yamagata, Y. & Tomita, K.-I., (1982) Biochem. Biophys. Res. Comm. **107**(2), 746-751

Ishida, T., Yamamoto, K. & Inoue, M., (1983) Bull. Chem. Soc. Jpn. **56**, 955-956

Ishida, T., Yanagitani, T., Nakagawa, Y. & Inoue, M., (1981) Biochem. Biophys. Res. Comm. **99**(4), 1179-84

Ivanov, V. T., Laine, I. A., Abdulaev, N. D., Senyavina, L. B., Popov, E. M., Ovchinnikov, Yu. A. & Shemyakin, M. M., (1969) Biochem. Biophys. Res. Comm. **34**, 803

Karplus, M., (1959) J. Chem. Phys. **30**, 11

Karplus, M., (1963) J. Am. Chem. Soc. **85**, 2870-1

Kearley, F. J. & Ingersoll, A. W., (1951) J. Am. Chem. Soc. **73**, 5783-5

Kennard, O., Cruse, W. B. T., Viswamitra, M. A., Sheldrick, G. M. & Jones, P. G., (1980) Am. Cryst. Assoc. Ser. 2, 7, 19

Lipscomb , W. N., (1970) Accts. Chem. Res. 3, 81

Lipscomb , W. N. & Quiocho, F. A., (1971) Adv. Protein Chem. 25, 1

Lipscomb , W. N., (1972) Chem. Soc. Rev. 3, 319

Llinas, M. & Klein, M. P., (1975) J. Am. Chem. Soc. **97**, 4731-7

Llinas, M., De Marco, A. & Wuthrich, K., (1978) Biopolymers, **17**, 637-50

Llinas, M., De Marco, A. & Wuthrich, K., (1978) Biopolymers, **17**, 617-36

Llinas, M., Klein, M. P. & Neilands, J. B., (1970) J. Mol. Biol. **52**, 399-414

MacNicol, D. D., McKendrick, J. J. & Wilson, D. R., (1978) Chem. Soc. Rev. **7**, 65-87

Nakazaki, M., Yamamoto, K., Ikeda, T., Kitsuki, T. & Okamoto, Y., (1983) J. Chem. Soc. Chem. Comm, **1983**, 787-8

Nardelli, M., Bigoli, F., Lanfranchi, M., Leporati, E. & Pellinghelli, M. A., (1981) Acta Cryst. **B37**, 1258-65

Newcomb, M., Helgeson, R. C. & Cram, D. J., (1974) J. Am. Chem. Soc. **96**, 7367-9

Norrestam, R., Stensland, B. & Branden, C. I., (1975) J. Mol. Biol. **99**, 501-6

Ohnishi, M. & Urry, D. W., (1969) Biochem. Biophys. Res. Comm. **36**, 194

Ottnad, M., Hartter, P. & Jung, G., (1975) Hoppe-Seyler's Z. Physiol. Chem. **356**, 1011-25

Overby, L. R. & Ingersoll, A. W., (1951) J. Am. Chem. Soc. **73**, 3363-6

Panijpan, B., (1977) J. Chem. Ed. **54**(11), 670-2

Peerderman, A. F., (1956) Acta Cryst. **9**, 824

Ptak, M., (1973) Biopolymers, **12**, 1575-89

Queener, S. W. & Neuss, N., (1982) "Chemistry and Biology of, β-lactam Antibiotics", 3-Biochemistry 2-83 eds. Morin, R. B. & Gorman, M., (Academic Press, New York, N. Y.

Ramanchandran, G. N., Chandrasekaran, R. & Kopple, K. D., (1971) Biopolymers, **10**, 2113-31

Richards, W. G., Clarkson, R. & Ganellin, C. R., (1975) Phil. Trans. R. Soc. Lond. **B272**, 75-85

Robinson, R., (1947) Nature, **159**, 263 & ibid, **160**, 18

Sanders, J. K. M. & Mersh, J. D., (1983) "Progress in NMR Spectroscopy", **15**, 353-400 (Pergamon Press, London)

Scheraga, H. A. & Pincus, M. R., (1981) Acc. Chem. Res. **14**, 299-306

Scott, A. I. & Gordon, E. M. G., (1973) Unpublished work, 1973

Sealock, R. R., (1946) J. Biol. Chem. **166**, 1

Sealock, R. R., Speeter, M. E. & Schweet, R. S., (1951) J. Am. Chem. Soc. **73**, 5386-88 19

Shabica, A. C. & Tishler, M., (1949) J. Am. Chem. Soc. **71**, 3251

Stern, A., Gibbons, W. A. & Craig, L. C., (1968) Proc. Natl. Acad. Sci. U. S. A., **61**, 734

Stoddart, J. F., (1978) Chem. Soc. Rev. **8**, 85-151

Toda, F. & Tanaka, K., (1981) Tetrahedron Letters, **22**(46), 4669-72

Toda, F. & Tanaka, K., (1983) J. Am. Chem. Soc. **105**, 5151-2

Toda, F., Tanaka, K. & Mori, K., (1983) Chem. Lett. 827-30

Toda, F., Tanaka, K., Ueda, H., Oshima, T., (1983) J. Chem. Soc. Chem. Commun. **1983**, 743-4

Toda, F., Ward, D. L. & Hart, H., (1981) Tetrahedron Letters, **22**(39), 3865-8

Tonelli, A. E., (1978) Biopolymers, **17**, 1175-9

Toniolo, C., (1980) CRC Crit. Rev. Biochem. 1-44 (October)

Tsai, C.-C., Jain, S. C. & Sobell, H. M., (1975) Phil. Trans. R. Soc. Lond. B, **272**, 137-46

Urry, D. W., Venkatachalam, C. M., Khaled, M. A. & Sugano, H., (1981) J. Am. Chem. Soc. **103**, 2372-9

van Tamelin, E. E., Wagner, E. R., Sjoberg, B., Thelin, H. & Nathorst-Westfelt, L., (1965) Tetrahedron Letters, **4**, 281-6

Viswamitra, M. A., Kennard, O., Cruse, W. B. T., Egert, E., Sheldrick, G. M., Jones, P. G., Waring, M. J., Wakelin, L. P. G. & Olsen, Nature, **289**, 817-9

Walkinshaw, M. D. & Saenger, W., (1984) J. Eur. Biochem. submitted for publication

Williams, D. H., Hyde, E., Kalman, J. R., Reid, D. G. & Olsen, R. K., (1982) J. Chem. Soc. Perkin I, **1982**, 1041-8

Wipff, G., Kollman, P. A. & Lehn, J. M., (1983) J. Mol. Struct. **93**, 153-65 THEOCHEM

Wolfe, S., Bowers, R. J., Hasan, S. K. & Kazmaier, P. M., (1981) Can. J. Chem. **59**, 406-21

Woodward, R. B., Brehn, W. J. & Nelson, A. L., (1947) J. Am. Chem. Soc. **69**, 2250

Yang, C.-H., Brown, J. N., Kopple, K. D., (1981) J. Am. Chem. Soc. **103**(7), 1715-9

Zalkin, A., Forrester, J. D. & Templeton, D. H., (1966) J. Am. Chem. Soc. **88**, 1810-4

APPENDIX
STRUCTURE FACTOR TABLES

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	-1	-13	97	46	1	0	-9	186	184	4	-1	-8	172	157	3	-2	-7	138	137	7	-8	-6	94	85
2	-1	-12	96	84	2	0	-9	141	147	5	-1	-8	232	233	4	-2	-7	94	102	1	-7	-6	112	114
3	-1	-12	114	103	4	0	-9	102	80	6	-1	-8	83	78	5	-2	-7	83	99	4	-7	-6	90	87
6	-4	-11	99	84	0	1	-9	148	155	1	0	-8	122	124	6	-2	-7	210	205	1	-6	-6	188	181
4	-3	-11	119	108	3	1	-9	162	170	2	0	-8	223	214	8	-2	-7	100	86	2	-6	-6	116	123
1	-1	-11	130	135	4	1	-9	200	207	0	1	-8	224	226	1	-1	-7	109	120	4	-6	-6	166	168
3	-1	-11	120	133	0	2	-9	183	201	2	1	-8	142	131	2	-1	-7	140	145	5	-6	-6	81	101
1	1	-11	106	124	1	2	-9	139	124	3	1	-8	132	138	3	-1	-7	165	163	1	-5	-6	76	91
0	3	-11	125	116	2	2	-9	138	134	4	1	-8	111	119	4	-1	-7	219	224	2	-5	-6	100	124
1	-5	-10	88	84	0	4	-9	147	160	5	1	-8	188	180	5	-1	-7	181	182	3	-5	-6	159	152
6	-4	-10	112	105	1	4	-9	111	122	6	1	-8	97	92	6	-1	-7	243	236	8	-5	-6	97	115
5	-3	-10	124	128	2	5	-9	113	114	7	1	-8	96	90	7	-1	-7	138	154	9	-5	-6	86	89
1	-2	-10	107	112	3	5	-9	95	88	0	2	-8	135	141	1	0	-7	102	106	1	-4	-6	207	208
2	-2	-10	141	165	4	5	-9	89	82	2	2	-8	173	173	2	0	-7	179	170	7	-4	-6	101	85
3	-2	-10	167	167	0	6	-9	147	164	0	3	-8	151	163	3	0	-7	221	222	10	-4	-6	102	102
4	-2	-10	129	115	1	6	-9	114	103	0	4	-8	90	70	4	0	-7	84	87	1	-3	-6	80	55
6	-2	-10	92	70	2	6	-9	85	44	8	-7	-7	96	72	5	0	-7	171	168	2	-3	-6	179	178
3	-1	-10	122	137	5	-8	-8	104	112	4	-6	-7	100	88	6	0	-7	106	87	3	-3	-6	128	124
3	0	-10	180	181	4	-7	-8	96	111	1	-5	-7	111	121	0	1	-7	106	129	6	-3	-6	166	163
4	0	-10	123	116	3	-6	-8	142	139	2	-5	-7	103	110	1	1	-7	288	288	7	-3	-6	100	103
0	1	-10	89	104	5	-6	-8	96	78	4	-5	-7	120	127	2	1	-7	145	145	8	-3	-6	162	151
2	1	-10	164	137	1	-5	-8	115	115	5	-5	-7	88	83	4	1	-7	308	313	9	-3	-6	120	112
3	1	-10	133	136	6	-5	-8	150	146	6	-5	-7	90	97	6	1	-7	167	176	1	-2	-6	136	159
1	2	-10	130	110	2	-4	-8	93	95	2	-4	-7	108	131	0	2	-7	194	190	2	-2	-6	159	151
0	3	-10	88	114	7	-4	-8	85	68	6	-4	-7	81	97	1	2	-7	90	106	3	-2	-6	175	178
3	-7	-9	117	119	4	-3	-8	81	98	7	-4	-7	126	119	2	2	-7	225	213	4	-2	-6	135	135
1	-6	-9	94	101	7	-3	-8	100	100	9	-4	-7	124	119	3	2	-7	129	145	5	-2	-6	126	133
1	-5	-9	97	75	1	-2	-8	79	77	2	-3	-7	110	113	1	3	-7	146	155	6	-2	-6	164	158
5	-3	-9	198	193	3	-2	-8	130	141	4	-3	-7	91	77	0	4	-7	86	99	1	-1	-6	194	191
1	-2	-9	115	131	4	-2	-8	149	127	5	-3	-7	158	160	3	4	-7	119	107	2	-1	-6	206	215
2	-1	-9	111	114	5	-2	-8	181	190	7	-3	-7	134	161	1	5	-7	98	80	5	-1	-6	188	190
3	-1	-9	182	176	6	-2	-8	162	171	8	-3	-7	104	113	3	5	-7	104	73	6	-1	-6	208	200
4	-1	-9	228	218	2	-1	-8	114	108	1	-2	-7	115	109	0	6	-7	114	138	7	-1	-6	213	220
5	-1	-9	202	209	3	-1	-8	146	143	2	-2	-7	95	99	3	-8	-6	114	96	1	0	-6	214	213

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	-6	221	209	3	-6	-5	247	263	4	0	-5	183	195	4	-8	-4	138	128	2	-2	-4	304	296
3	0	-6	83	87	5	-6	-5	191	178	5	0	-5	197	191	5	-8	-4	157	158	3	-2	-4	316	331
4	0	-6	335	354	2	-5	-5	133	137	6	0	-5	138	138	6	-8	-4	145	164	4	-2	-4	159	151
6	0	-6	91	73	3	-5	-5	172	180	7	0	-5	161	160	9	-8	-4	132	135	5	-2	-4	134	144
0	1	-6	107	110	4	-5	-5	167	173	0	1	-5	132	142	3	-7	-4	182	164	6	-2	-4	168	161
1	1	-6	220	221	5	-5	-5	93	85	1	1	-5	281	264	4	-7	-4	90	99	7	-2	-4	145	145
2	1	-6	279	271	1	-4	-5	196	191	2	1	-5	170	172	5	-7	-4	123	125	8	-2	-4	144	149
3	1	-6	165	166	2	-4	-5	241	243	3	1	-5	183	191	7	-7	-4	92	90	9	-2	-4	101	103
4	1	-6	153	149	3	-4	-5	306	292	4	1	-5	125	117	1	-6	-4	81	96	1	-1	-4	260	276
5	1	-6	189	180	4	-4	-5	147	138	7	1	-5	214	212	3	-6	-4	155	163	3	-1	-4	392	361
6	1	-6	148	148	5	-4	-5	196	205	0	2	-5	232	225	4	-6	-4	231	238	4	-1	-4	174	171
7	1	-6	93	102	1	-3	-5	190	189	1	2	-5	549	543	5	-6	-4	152	145	8	-1	-4	124	132
0	2	-6	205	206	2	-3	-5	91	104	2	2	-5	262	253	6	-6	-4	134	136	9	-1	-4	87	78
1	2	-6	218	216	3	-3	-5	119	121	3	2	-5	173	176	7	-6	-4	82	120	1	0	-4	210	213
2	2	-6	115	114	4	-3	-5	162	156	4	2	-5	229	238	8	-6	-4	79	65	2	0	-4	218	210
4	2	-6	137	146	5	-3	-5	120	112	5	2	-5	147	152	9	-6	-4	84	101	3	0	-4	127	123
1	3	-6	214	206	7	-3	-5	130	129	6	2	-5	134	145	1	-5	-4	154	150	4	0	-4	88	88
2	3	-6	122	119	8	-3	-5	89	77	0	3	-5	482	493	2	-5	-4	240	249	5	0	-4	235	237
0	4	-6	166	164	1	-2	-5	311	318	1	3	-5	234	240	3	-5	-4	158	158	6	0	-4	219	227
1	4	-6	89	80	2	-2	-5	431	419	2	3	-5	103	107	4	-5	-4	245	240	7	0	-4	128	117
4	4	-6	95	70	3	-2	-5	251	237	0	4	-5	120	121	5	-5	-4	227	219	0	1	-4	160	167
1	5	-6	76	56	5	-2	-5	163	149	1	4	-5	162	178	7	-5	-4	143	149	1	1	-4	194	197
0	6	-6	139	133	7	-2	-5	121	119	5	4	-5	86	101	1	-4	-4	165	170	2	1	-4	316	324
1	6	-6	83	89	8	-2	-5	132	160	0	5	-5	193	181	2	-4	-4	228	213	3	1	-4	161	159
1	7	-6	90	82	1	-1	-5	306	321	3	5	-5	107	98	3	-4	-4	310	323	4	1	-4	198	193
0	8	-6	106	88	2	-1	-5	85	82	5	5	-5	85	94	4	-4	-4	66	54	5	1	-4	116	119
3	-8	-5	114	128	3	-1	-5	148	146	0	6	-5	147	159	5	-4	-4	207	195	6	1	-4	97	87
4	-8	-5	139	156	4	-1	-5	148	155	1	6	-5	236	251	1	-3	-4	436	447	7	1	-4	97	106
6	-8	-5	90	84	5	-1	-5	129	128	4	6	-5	117	105	2	-3	-4	260	291	0	2	-4	155	153
2	-7	-5	113	127	6	-1	-5	263	261	0	7	-5	179	194	3	-3	-4	311	293	1	2	-4	212	213
3	-7	-5	138	140	7	-1	-5	203	198	1	7	-5	84	112	4	-3	-4	169	181	2	2	-4	119	131
4	-7	-5	139	131	9	-1	-5	178	152	0	8	-5	188	199	5	-3	-4	100	106	3	2	-4	325	332
5	-7	-5	94	88	2	0	-5	180	177	2	8	-5	114	109	8	-3	-4	104	111	4	2	-4	125	109
1	-6	-5	86	90	3	0	-5	154	141	0	9	-5	92	76	1	-2	-4	134	134	5	2	-4	169	169

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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
0	3	-4	312	310	5	-6	-3	176	167	7	-1	-3	134	145	4	4	-3	75	71	5	-6	-2	237	264
1	3	-4	117	121	6	-6	-3	132	123	8	-1	-3	108	110	7	4	-3	115	114	6	-6	-2	246	237
2	3	-4	178	166	7	-6	-3	132	140	1	0	-3	176	175	0	5	-3	126	123	7	-6	-2	94	84
3	3	-4	252	253	1	-5	-3	82	79	2	0	-3	177	182	1	5	-3	171	163	8	-6	-2	163	159
4	3	-4	125	113	2	-5	-3	130	132	3	0	-3	143	139	2	5	-3	188	180	9	-6	-2	122	116
7	3	-4	111	118	3	-5	-3	113	123	4	0	-3	276	254	3	5	-3	98	100	1	-5	-2	166	166
0	4	-4	118	101	4	-5	-3	112	114	5	0	-3	134	131	0	6	-3	133	123	2	-5	-2	151	142
1	4	-4	175	191	5	-5	-3	284	285	6	0	-3	138	146	3	6	-3	146	142	4	-5	-2	73	77
2	4	-4	120	110	6	-5	-3	154	156	7	0	-3	147	144	4	6	-3	97	115	6	-5	-2	173	179
4	4	-4	89	89	1	-4	-3	318	318	0	1	-3	80	75	0	7	-3	107	109	9	-5	-2	82	73
0	5	-4	99	107	2	-4	-3	78	75	1	1	-3	205	198	1	7	-3	123	115	1	-4	-2	164	154
1	5	-4	161	167	3	-4	-3	251	256	2	1	-3	108	112	2	7	-3	103	94	2	-4	-2	88	84
1	6	-4	152	149	4	-4	-3	176	188	3	1	-3	335	336	0	8	-3	130	145	4	-4	-2	103	116
2	6	-4	164	163	5	-4	-3	169	162	4	1	-3	162	163	2	8	-3	109	103	5	-4	-2	329	322
4	6	-4	134	136	9	-4	-3	80	82	5	1	-3	285	286	3	8	-3	84	100	6	-4	-2	121	118
0	7	-4	164	185	1	-3	-3	65	78	6	1	-3	76	86	2-10	-2		96	79	7	-4	-2	90	107
1	7	-4	229	226	2	-3	-3	116	126	7	1	-3	101	101	6-10	-2		104	67	8	-4	-2	103	101
0	8	-4	87	73	3	-3	-3	177	183	8	1	-3	133	129	3	-9	-2	124	123	1	-3	-2	236	233
1	8	-4	110	110	4	-3	-3	274	246	0	2	-3	303	303	4	-9	-2	133	149	2	-3	-2	344	353
0	9	-4	146	137	5	-3	-3	82	80	1	2	-3	729	725	5	-9	-2	84	73	3	-3	-2	426	441
1-10	-3		109	97	6	-3	-3	98	101	2	2	-3	118	122	2	-8	-2	124	114	4	-3	-2	270	260
3	-9	-3	107	98	7	-3	-3	74	56	3	2	-3	316	312	3	-8	-2	107	109	5	-3	-2	117	107
1	-8	-3	92	91	1	-2	-3	566	555	4	2	-3	140	139	6	-8	-2	146	149	6	-3	-2	234	247
2	-8	-3	131	137	3	-2	-3	241	236	5	2	-3	103	123	7	-8	-2	114	113	1	-2	-2	392	393
3	-8	-3	96	96	4	-2	-3	478	467	6	2	-3	130	144	8	-8	-2	112	119	2	-2	-2	216	221
4	-8	-3	153	157	5	-2	-3	131	133	0	3	-3	108	111	9	-8	-2	92	74	3	-2	-2	65	72
8	-8	-3	89	67	6	-2	-3	122	124	1	3	-3	255	242	1	-7	-2	159	152	4	-2	-2	407	405
2	-7	-3	153	149	8	-2	-3	110	119	2	3	-3	365	351	3	-7	-2	111	112	5	-2	-2	317	297
3	-7	-3	104	97	9	-2	-3	132	117	3	3	-3	128	136	4	-7	-2	101	94	6	-2	-2	154	169
5	-7	-3	200	192	1	-1	-3	103	109	6	3	-3	86	92	5	-7	-2	112	131	8	-2	-2	119	127
6	-7	-3	137	127	2	-1	-3	321	313	0	4	-3	345	328	7	-7	-2	142	128	1	-1	-2	113	114
1	-6	-3	94	88	4	-1	-3	181	179	1	4	-3	403	412	1	-6	-2	142	135	3	-1	-2	203	198
3	-6	-3	192	180	5	-1	-3	210	210	2	4	-3	70	54	2	-6	-2	142	146	4	-1	-2	125	116
4	-6	-3	140	149	6	-1	-3	127	119	3	4	-3	162	159	4	-6	-2	114	117	5	-1	-2	92	97

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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
6	-1	-2	152	146	3	4	-2	109	118	5	-6	-1	142	141	1	0	-1	436	452	0	5	-1	253	251
9	-1	-2	108	131	5	4	-2	131	138	6	-6	-1	140	149	2	0	-1	253	260	1	5	-1	175	173
1	0	-2	156	160	7	4	-2	110	115	7	-6	-1	89	83	3	0	-1	399	407	4	5	-1	115	129
2	0	-2	268	262	0	5	-2	153	156	2	-5	-1	177	174	4	0	-1	171	163	1	6	-1	136	143
3	0	-2	185	192	1	5	-2	121	118	3	-5	-1	287	259	5	0	-1	129	138	0	7	-1	127	130
4	0	-2	128	125	2	5	-2	202	189	5	-5	-1	279	273	6	0	-1	178	180	3	7	-1	92	107
5	0	-2	146	138	3	5	-2	87	101	7	-5	-1	159	172	8	0	-1	127	133	2-10		0	69	98
6	0	-2	86	88	0	6	-2	198	207	8	-5	-1	124	119	9	0	-1	98	105	3-10		0	96	110
7	0	-2	206	221	1	6	-2	88	95	1	-4	-1	257	250	0	1	-1	379	380	5	-9	0	101	103
0	1	-2	412	418	3	6	-2	97	73	2	-4	-1	201	200	1	1	-1	287	289	6	-9	0	93	96
2	1	-2	199	195	4	6	-2	143	158	3	-4	-1	127	111	3	1	-1	130	134	4	-8	0	170	164
3	1	-2	354	344	0	7	-2	87	118	5	-4	-1	384	361	4	1	-1	148	152	5	-8	0	101	91
4	1	-2	96	88	2	7	-2	108	107	6	-4	-1	199	208	6	1	-1	173	184	6	-8	0	121	118
5	1	-2	263	274	3	7	-2	134	118	7	-4	-1	72	74	7	1	-1	206	196	7	-8	0	100	93
6	1	-2	187	193	1	8	-2	124	143	1	-3	-1	638	628	0	2	-1	461	455	9	-8	0	99	98
0	2	-2	162	163	2	9	-2	100	106	2	-3	-1	369	347	1	2	-1	207	200	10	-8	0	89	86
1	2	-2	159	152	3-10		-1	150	147	3	-3	-1	315	321	2	2	-1	219	224	2	-7	0	78	71
2	2	-2	282	280	4-10		-1	87	69	4	-3	-1	390	398	3	2	-1	233	219	5	-7	0	229	211
3	2	-2	342	338	7-10		-1	85	54	5	-3	-1	145	143	4	2	-1	211	215	6	-7	0	111	97
4	2	-2	138	157	5	-9	-1	93	87	6	-3	-1	111	99	5	2	-1	277	259	7	-7	0	81	74
5	2	-2	124	136	1	-8	-1	96	111	1	-2	-1	471	470	6	2	-1	204	220	1	-6	0	408	389
6	2	-2	80	84	2	-8	-1	146	153	2	-2	-1	580	572	8	2	-1	125	106	2	-6	0	141	144
7	2	-2	134	133	3	-8	-1	156	136	3	-2	-1	485	470	0	3	-1	302	297	3	-6	0	129	123
8	2	-2	101	117	4	-8	-1	129	142	5	-2	-1	190	182	1	3	-1	174	170	4	-6	0	106	93
0	3	-2	98	90	7	-8	-1	124	121	6	-2	-1	124	127	2	3	-1	92	76	7	-6	0	179	178
1	3	-2	165	134	8	-8	-1	80	82	7	-2	-1	120	109	3	3	-1	147	140	9	-6	0	104	104
2	3	-2	373	359	1	-7	-1	105	107	1	-1	-1	290	305	4	3	-1	207	214	10	-6	0	87	85
3	3	-2	317	314	2	-7	-1	74	81	2	-1	-1	88	87	6	3	-1	106	131	1	-5	0	222	223
4	3	-2	91	84	5	-7	-1	138	139	3	-1	-1	163	164	0	4	-1	191	191	2	-5	0	97	98
5	3	-2	163	176	6	-7	-1	159	150	4	-1	-1	397	402	1	4	-1	80	73	3	-5	0	189	187
6	3	-2	106	94	10	-7	-1	127	117	6	-1	-1	134	161	2	4	-1	188	186	4	-5	0	104	84
0	4	-2	335	324	1	-6	-1	213	205	8	-1	-1	125	125	3	4	-1	111	110	5	-5	0	227	229
1	4	-2	296	287	2	-6	-1	121	121	9	-1	-1	122	134	4	4	-1	140	164	8	-5	0	130	139
2	4	-2	106	105	3	-6	-1	190	181	10	-1	-1	93	83	5	4	-1	138	142	1	-4	0	434	423

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	-4	0	276	269	9	0	0	106	106	0	7	0	92	82	1	-3	1	198	192	6	2	1	109	119
3	-4	0	151	145	0	1	0	49	36	0	8	0	92	53	2	-3	1	311	301	7	2	1	125	127
4	-4	0	302	282	1	1	0	187	189	0	10	0	89	93	3	-3	1	142	147	0	3	1	314	312
6	-4	0	224	208	3	1	0	100	98	3-10	1		90	65	4	-3	1	120	113	1	3	1	114	113
9	-4	0	117	116	4	1	0	67	72	4-10	1		130	116	5	-3	1	331	336	2	3	1	146	149
1	-3	0	211	206	5	1	0	217	214	5-10	1		136	146	6	-3	1	272	288	4	3	1	163	166
2	-3	0	736	740	6	1	0	108	111	6	-9	1	103	99	7	-3	1	174	178	0	4	1	326	333
3	-3	0	143	154	7	1	0	146	156	3	-8	1	131	138	1	-2	1	1004	1027	1	4	1	108	107
4	-3	0	250	252	8	1	0	79	84	4	-8	1	177	189	2	-2	1	289	279	2	4	1	65	68
5	-3	0	221	218	0	2	0	1109	1144	6	-8	1	135	126	3	-2	1	168	176	3	4	1	124	134
8	-3	0	98	105	1	2	0	353	352	7	-8	1	99	106	4	-2	1	375	387	4	4	1	141	146
10	-3	0	87	78	2	2	0	138	132	1	-7	1	101	97	5	-2	1	325	324	6	4	1	84	69
1	-2	0	675	686	3	2	0	162	161	3	-7	1	137	143	6	-2	1	96	108	0	5	1	161	149
2	-2	0	253	262	4	2	0	66	61	4	-7	1	115	91	2	-1	1	525	537	1	5	1	91	121
3	-2	0	373	379	5	2	0	140	150	7	-7	1	127	149	3	-1	1	633	625	2	5	1	151	156
4	-2	0	361	375	6	2	0	162	148	1	-6	1	149	136	4	-1	1	253	270	4	5	1	100	79
5	-2	0	57	56	7	2	0	75	73	2	-6	1	208	193	5	-1	1	143	148	0	6	1	109	102
6	-2	0	228	214	0	3	0	85	93	3	-6	1	193	193	1	0	1	667	718	1	6	1	89	89
7	-2	0	67	67	1	3	0	203	201	5	-6	1	99	100	2	0	1	642	657	2	6	1	91	74
8	-2	0	150	151	2	3	0	69	68	7	-6	1	192	187	3	0	1	147	157	1	7	1	148	161
1	-1	0	408	420	4	3	0	249	250	1	-5	1	142	140	4	0	1	144	138	0	8	1	177	185
2	-1	0	823	854	5	3	0	181	187	2	-5	1	225	217	5	0	1	87	63	2-10	2		118	116
3	-1	0	292	280	0	4	0	185	180	3	-5	1	69	58	6	0	1	226	236	3	-8	2	94	89
4	-1	0	456	450	2	4	0	320	324	4	-5	1	145	133	7	0	1	77	75	1	-7	2	140	136
5	-1	0	188	179	3	4	0	185	183	5	-5	1	104	107	0	1	1	187	184	3	-7	2	103	95
7	-1	0	72	81	0	5	0	150	148	6	-5	1	86	108	1	1	1	207	209	4	-7	2	91	110
8	-1	0	112	101	1	5	0	183	183	7	-5	1	210	225	2	1	1	161	167	5	-7	2	82	99
10	-1	0	100	91	2	5	0	112	124	9	-5	1	93	92	3	1	1	370	383	7	-7	2	110	92
1	0	0	211	218	3	5	0	71	62	1	-4	1	93	72	4	1	1	192	207	1	-6	2	74	70
2	0	0	82	72	4	5	0	131	133	2	-4	1	331	319	0	2	1	545	564	2	-6	2	102	104
3	0	0	374	379	0	6	0	153	138	3	-4	1	157	150	1	2	1	371	359	3	-6	2	343	336
4	0	0	273	279	2	6	0	120	117	4	-4	1	279	274	2	2	1	224	224	4	-6	2	123	107
6	0	0	149	148	3	6	0	93	96	5	-4	1	254	251	3	2	1	136	133	5	-6	2	178	181
7	0	0	148	151	6	6	0	87	75	7	-4	1	183	182	4	2	1	215	210	9	-6	2	88	116

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	2	273	283	4	-1	2	443	435	2	5	2	156	167	1	-3	3	125	114	6	2	3	154	169
2	-5	2	83	68	5	-1	2	78	91	3	5	2	128	120	2	-3	3	266	260	1	3	3	279	273
3	-5	2	192	189	6	-1	2	169	183	5	5	2	109	93	3	-3	3	103	94	4	3	3	78	71
4	-5	2	145	145	0	0	2	316	326	1	6	2	215	214	4	-3	3	68	69	5	3	3	89	111
5	-5	2	107	102	1	0	2	441	446	2	6	2	122	120	5	-3	3	101	107	6	3	3	93	96
6	-5	2	78	91	2	0	2	961	991	4	6	2	89	101	6	-3	3	78	78	7	3	3	96	101
7	-5	2	210	218	3	0	2	442	459	0	7	2	174	175	7	-3	3	159	153	0	4	3	208	208
10	-5	2	105	118	4	0	2	132	128	3	7	2	109	98	8	-3	3	90	76	2	4	3	100	91
1	-4	2	227	220	5	0	2	301	299	2-10		3	99	76	1	-2	3	285	286	3	4	3	128	141
2	-4	2	335	329	6	0	2	93	82	6-10		3	103	84	2	-2	3	90	84	5	4	3	163	146
3	-4	2	94	97	0	1	2	794	798	7-10		3	114	105	3	-2	3	267	248	6	4	3	112	118
4	-4	2	92	98	1	1	2	487	478	1	-8	3	124	134	5	-2	3	161	159	0	5	3	74	66
5	-4	2	96	109	2	1	2	176	172	4	-8	3	141	147	6	-2	3	302	307	1	5	3	212	220
6	-4	2	185	182	3	1	2	242	237	5	-8	3	127	115	1	-1	3	119	110	4	5	3	159	160
7	-4	2	78	79	6	1	2	100	107	8	-8	3	86	80	2	-1	3	169	175	0	6	3	135	136
8	-4	2	140	140	7	1	2	79	85	2	-7	3	135	139	3	-1	3	83	97	1	6	3	155	154
9	-4	2	127	112	0	2	2	201	204	5	-7	3	119	131	4	-1	3	174	181	2	6	3	131	120
1	-3	2	141	151	1	2	2	568	574	7	-7	3	110	142	5	-1	3	201	184	3	6	3	85	86
2	-3	2	71	68	2	2	2	95	106	1	-6	3	101	120	6	-1	3	160	165	1	7	3	125	116
3	-3	2	117	120	4	2	2	164	170	3	-6	3	164	174	0	0	3	350	334	4	7	3	94	81
4	-3	2	324	313	5	2	2	80	87	5	-6	3	98	97	1	0	3	256	246	2	8	3	116	116
5	-3	2	152	148	7	2	2	111	106	6	-6	3	83	68	2	0	3	688	705	1	-8	4	133	139
6	-3	2	211	199	0	3	2	68	62	9	-6	3	108	99	3	0	3	270	259	2	-8	4	87	90
7	-3	2	153	165	1	3	2	65	78	10	-6	3	93	85	4	0	3	208	213	4	-8	4	126	138
1	-2	2	240	246	2	3	2	87	83	1	-5	3	310	303	5	0	3	142	132	5	-8	4	98	99
2	-2	2	331	331	3	3	2	174	167	2	-5	3	301	309	6	0	3	152	157	7	-8	4	85	72
3	-2	2	459	460	4	3	2	84	94	4	-5	3	92	81	0	1	3	246	231	2	-7	4	100	100
4	-2	2	78	109	6	3	2	138	119	7	-5	3	170	168	1	1	3	635	619	4	-7	4	92	101
5	-2	2	331	344	0	4	2	178	169	1	-4	3	186	167	4	1	3	131	142	6	-7	4	137	143
6	-2	2	151	144	1	4	2	122	110	3	-4	3	233	229	7	1	3	86	77	7	-7	4	105	105
8	-2	2	100	90	2	4	2	118	137	4	-4	3	91	73	0	2	3	148	148	1	-6	4	145	141
1	-1	2	198	196	3	4	2	109	113	5	-4	3	93	97	1	2	3	207	216	2	-6	4	138	169
2	-1	2	69	57	4	4	2	123	97	6	-4	3	165	158	2	2	3	240	235	3	-6	4	148	133
3	-1	2	257	269	1	5	2	80	87	7	-4	3	173	169	5	2	3	98	97	4	-6	4	167	165

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	-6	4	197	194	2	2	4	215	217	1	-3	5	139	137	2	-8	6	165	170	4	2	6	85	94
1	-5	4	116	104	3	2	4	147	147	3	-3	5	95	101	6	-8	6	118	118	0	3	6	187	192
3	-5	4	119	123	4	2	4	120	117	4	-3	5	113	104	7	-8	6	91	85	1	3	6	203	199
5	-5	4	129	113	6	2	4	91	96	6	-3	5	134	123	1	-7	6	85	76	2	3	6	180	194
6	-5	4	150	160	7	2	4	96	81	2	-2	5	211	218	3	-6	6	106	115	3	3	6	132	136
7	-5	4	91	104	0	3	4	259	265	4	-2	5	110	92	6	-6	6	117	114	0	4	6	122	116
2	-4	4	102	101	1	3	4	362	363	5	-2	5	155	169	4	-5	6	115	112	1	4	6	184	200
3	-4	4	113	111	2	3	4	109	109	1	-1	5	199	192	2	-4	6	75	79	2	4	6	153	144
4	-4	4	165	171	3	3	4	134	132	4	-1	5	142	137	3	-4	6	172	177	3	4	6	89	99
5	-4	4	94	94	0	4	4	211	227	6	-1	5	174	173	2	-3	6	111	108	0	5	6	270	271
2	-3	4	151	152	1	4	4	97	103	7	-1	5	100	104	4	-3	6	108	105	3	-8	7	106	95
5	-3	4	117	108	2	4	4	91	95	0	0	5	125	133	1	-2	6	235	253	2	-7	7	114	103
6	-3	4	204	219	4	4	4	113	99	1	0	5	204	200	2	-2	6	134	137	3	-7	7	100	79
8	-3	4	153	138	1	5	4	142	153	4	0	5	310	296	3	-2	6	103	102	6	-7	7	103	78
1	-2	4	136	135	4	5	4	88	110	5	0	5	149	157	4	-2	6	92	96	2	-6	7	107	113
2	-2	4	179	177	0	7	4	79	81	6	0	5	130	128	6	-2	6	126	148	1	-5	7	121	125
4	-2	4	93	83	1	7	4	117	120	0	1	5	207	195	1	-1	6	211	219	2	-5	7	146	150
6	-2	4	144	147	4-10		5	130	109	1	1	5	330	319	4	-1	6	228	236	3	-5	7	135	144
1	-1	4	155	153	1	-9	5	101	111	2	1	5	203	202	5	-1	6	88	66	5	-5	7	154	147
2	-1	4	447	429	1	-8	5	130	136	3	1	5	228	226	7	-1	6	114	99	6	-5	7	110	122
5	-1	4	236	240	2	-8	5	89	91	4	1	5	120	125	1	0	6	91	103	1	-4	7	165	163
6	-1	4	174	164	1	-7	5	139	145	1	2	5	353	361	2	0	6	290	302	4	-4	7	119	107
0	0	4	490	495	2	-7	5	79	78	2	2	5	188	180	3	0	6	280	283	6	-4	7	101	99
1	0	4	418	416	4	-7	5	138	139	3	2	5	140	121	4	0	6	93	109	1	-3	7	138	148
3	0	4	102	101	5	-7	5	114	128	4	2	5	151	154	5	0	6	113	111	2	-3	7	243	252
4	0	4	322	332	6	-7	5	87	103	0	3	5	224	222	6	0	6	139	148	3	-3	7	138	141
5	0	4	135	126	2	-6	5	90	98	1	3	5	69	69	0	1	6	131	148	4	-3	7	98	98
6	0	4	99	84	1	-5	5	199	191	2	3	5	113	93	1	1	6	184	192	5	-3	7	93	82
0	1	4	453	457	4	-5	5	203	205	0	4	5	126	116	2	1	6	204	197	1	-2	7	269	277
1	1	4	297	288	1	-4	5	196	189	1	4	5	91	109	3	1	6	122	119	4	-2	7	104	99
2	1	4	339	316	2	-4	5	350	354	0	6	5	82	82	4	1	6	179	182	6	-2	7	91	103
5	1	4	175	186	3	-4	5	114	110	1	6	5	118	128	0	2	6	176	188	1	-1	7	196	188
0	2	4	223	221	4	-4	5	173	178	2	6	5	130	112	2	2	6	271	282	3	-1	7	114	115
1	2	4	314	322	8	-4	5	94	101	0	7	5	137	126	3	2	6	124	140	0	0	7	106	119

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CYCLIC SULPHIDE

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	7	176	178	2	3	7	154	163	2	-3	8	87	77	3	3	8	96	97	2	-1	9	100	70
3	0	7	137	145	4	3	7	100	57	4	-3	8	123	135	1	4	8	118	108	1	0	9	88	78
4	0	7	149	122	5	3	7	93	83	1	-2	8	211	219	3	5	8	87	70	1	2	9	124	100
7	0	7	93	98	0	4	7	168	163	4	-2	8	115	115	1	-8	9	98	102	1	4	9	119	98
0	1	7	146	134	0	5	7	112	102	2	-1	8	79	78	2	-5	9	87	99	3	-5	10	91	96
3	1	7	163	183	1	-7	8	93	78	3	-1	8	81	88	5	-5	9	94	110	3	-2	10	86	94
4	1	7	111	132	1	-6	8	173	180	0	0	8	123	125	1	-4	9	96	120	4	-2	10	98	73
0	2	7	173	176	2	-6	8	100	126	1	0	8	163	164	5	-4	9	109	102	0	0	10	139	154
1	2	7	136	133	3	-6	8	95	105	7	0	8	92	76	1	-3	9	208	211	1	5	10	113	102
2	2	7	80	92	5	-6	8	105	117	0	2	8	88	81	4	-3	9	133	130	1	-2	11	89	86
3	2	7	87	83	6	-6	8	135	115	4	2	8	94	106	1	-2	9	126	131	2	-2	11	108	93
4	2	7	88	98	8	-4	8	147	153	0	3	8	101	127	2	-2	9	118	122	2	-1	11	100	78
0	3	7	92	91	1	-3	8	86	36	2	3	8	83	74	1	-1	9	245	239	0	1	11	91	87
1	3	7	151	163																				

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	2	0	1372	1379	5	9	0	227	179	13	13	0	366	397	1	22	0	308	242
2	3	0	556	555	7	9	0	254	286	1	14	0	336	353	1	2	1	1039	1148
1	4	0	1848	1780	8	9	0	157	179	3	14	0	219	123	2	2	1	3080	3075
2	4	0	3457	3349	9	9	0	381	375	4	14	0	424	427	0	3	1	273	168
3	4	0	1044	1032	0	10	0	1802	1782	5	14	0	174	132	1	3	1	686	691
4	4	0	2349	2316	1	10	0	1593	1477	7	14	0	176	168	2	3	1	1329	1489
1	5	0	1765	2106	2	10	0	394	384	8	14	0	189	89	3	3	1	663	632
2	5	0	313	229	3	10	0	870	710	10	14	0	195	59	0	4	1	5730	5749
3	5	0	1036	1243	4	10	0	341	305	13	14	0	318	234	1	4	1	1912	2040
4	5	0	1029	948	6	10	0	714	730	14	14	0	183	123	2	4	1	2231	1902
5	5	0	1385	1233	9	10	0	339	391	1	15	0	116	183	3	4	1	709	729
0	6	0	6408	5859	10	10	0	394	337	2	15	0	113	155	4	4	1	3929	3823
1	6	0	2837	2723	1	11	0	318	268	3	15	0	194	133	0	5	1	1025	1070
2	6	0	2574	2339	2	11	0	1134	1103	8	15	0	183	279	1	5	1	1243	1129
3	6	0	174	221	4	11	0	160	106	11	15	0	688	672	2	5	1	586	623
4	6	0	396	411	5	11	0	907	832	13	15	0	752	766	3	5	1	525	622
5	6	0	763	751	6	11	0	623	593	0	16	0	224	118	4	5	1	2444	2335
6	6	0	1815	1859	8	11	0	210	209	1	16	0	1013	1057	5	5	1	245	251
1	7	0	217	242	9	11	0	426	316	2	16	0	218	121	0	6	1	1091	1153
2	7	0	436	482	11	11	0	237	337	5	16	0	610	624	1	6	1	1563	1476
3	7	0	459	438	0	12	0	1951	1943	6	16	0	159	111	2	6	1	1108	1107
5	7	0	271	225	1	12	0	1846	1792	8	17	0	302	406	3	6	1	1377	1483
7	7	0	693	746	2	12	0	748	755	9	17	0	592	509	4	6	1	2740	2651
0	8	0	1342	1215	3	12	0	650	617	11	17	0	659	631	5	6	1	1539	1280
3	8	0	1729	1785	4	12	0	914	773	1	18	0	992	984	6	6	1	1020	1002
4	8	0	614	646	6	12	0	778	782	3	18	0	448	472	0	7	1	244	64
5	8	0	1477	1410	7	12	0	251	165	4	18	0	385	318	1	7	1	591	604
6	8	0	566	486	9	12	0	342	407	5	18	0	250	199	2	7	1	317	295
7	8	0	943	615	11	12	0	370	366	12	18	0	142	60	4	7	1	578	615
8	8	0	623	648	12	12	0	293	244	1	19	0	133	5	5	7	1	263	222
1	9	0	1086	1234	4	13	0	575	583	3	20	0	181	290	6	7	1	679	622
2	9	0	333	206	6	13	0	156	87	4	20	0	197	24	0	8	1	1001	990
3	9	0	636	672	8	13	0	247	100	5	20	0	264	233	1	9	1	1184	1104
4	9	0	495	440	11	13	0	187	136	5	21	0	180	196	2	8	1	335	351
3	8	1	483	497
4	8	1	1030	1053
5	8	1	657	661
6	8	1	882	962
7	8	1	210	90
8	8	1	318	325
0	9	1	467	474
1	9	1	496	511
2	9	1	120	170
3	9	1	515	469
4	9	1	333	287
5	9	1	506	559
6	9	1	482	492
7	9	1	347	298
8	9	1	532	506
1	9	1	371	351
0	10	1	310	259
1	10	1	1000	1124
2	10	1	819	841
3	10	1	1418	1335
4	10	1	1341	1328
5	10	1	844	926
6	10	1	319	349
7	10	1	338	346
8	10	1	499	485
9	10	1	453	435
0	11	1	556	445
1	11	1	556	547
2	11	1	518	394
3	11	1	336	366
4	11	1	921	842
5	11	1	655	675
6	11	1	354	303
8	11	1	309	361

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
9	11	1	310	338	1	15	1	267	304	2	5	2	1505	1556	9	9	2	842	870
0	12	1	913	976	2	15	1	335	299	3	5	2	197	196	0	10	2	997	978
1	12	1	504	584	5	15	1	291	284	4	5	2	322	361	1	10	2	828	845
2	12	1	1044	990	6	15	1	305	230	5	5	2	1079	1139	2	10	2	644	680
3	12	1	1462	1378	9	15	1	250	260	0	6	2	3044	2835	3	10	2	1311	1235
4	12	1	956	876	11	15	1	460	437	1	6	2	365	375	4	10	2	401	376
5	12	1	702	769	13	15	1	283	302	2	6	2	1412	1438	5	10	2	1054	1053
6	12	1	294	268	3	16	1	119	74	3	6	2	1994	2035	6	10	2	496	450
7	12	1	496	522	7	16	1	155	236	4	6	2	1943	1876	7	10	2	723	736
9	12	1	399	408	8	16	1	215	189	5	6	2	803	838	8	10	2	361	390
10	12	1	374	395	9	16	1	341	330	6	6	2	257	203	9	10	2	643	689
12	12	1	319	260	10	17	1	267	266	0	7	2	421	438	1	11	2	602	638
1	13	1	228	291	1	18	1	432	422	1	7	2	470	459	2	11	2	459	401
2	13	1	188	155	2	18	1	257	143	2	7	2	953	915	4	11	2	340	359
3	13	1	343	278	3	18	1	411	383	3	7	2	967	1021	5	11	2	270	263
4	13	1	187	117	5	18	1	319	327	4	7	2	324	330	7	11	2	196	326
5	13	1	558	630	9	18	1	144	203	6	7	2	421	382	9	11	2	214	301
6	13	1	318	259	13	18	1	148	74	7	7	2	343	348	11	11	2	808	739
7	13	1	384	292	7	19	1	159	71	0	8	2	169	263	0	12	2	748	756
8	13	1	238	301	4	20	1	158	154	1	8	2	1468	1278	1	12	2	431	386
11	13	1	361	428	1	22	1	150	88	2	8	2	2738	2603	2	12	2	467	423
12	13	1	287	264	2	22	1	235	147	3	8	2	2058	2150	3	12	2	344	341
13	13	1	482	507	0	2	2	911	1115	4	8	2	1298	1285	4	12	2	436	464
0	14	1	516	576	1	2	2	878	902	5	8	2	560	545	6	12	2	427	440
1	14	1	564	584	2	2	2	3668	3886	6	8	2	238	288	7	12	2	204	292
2	14	1	415	296	0	3	2	1276	1138	8	8	2	977	940	8	12	2	328	323
3	14	1	468	541	2	3	2	1172	1128	0	9	2	985	829	9	12	2	388	321
4	14	1	378	361	3	3	2	282	336	1	9	2	497	520	10	12	2	209	247
5	14	1	698	696	0	4	2	310	29	2	9	2	442	422	11	12	2	205	196
6	14	1	454	463	1	4	2	1000	935	3	9	2	333	432	12	12	2	263	312
7	14	1	179	175	2	4	2	1695	1777	4	9	2	562	486	1	13	2	159	158
8	14	1	205	195	3	4	2	320	316	5	9	2	287	238	3	13	2	176	139
9	14	1	324	342	4	4	2	1002	860	7	9	2	422	457	4	13	2	203	255
11	14	1	229	243	0	5	2	1704	1860	8	9	2	422	486	5	13	2	333	386
6	13	2	374	363
7	13	2	279	282
8	13	2	392	401
9	13	2	921	991
10	13	2	862	812
13	13	2	207	267
14	13	2	504	606
1	14	2	774	682
2	14	2	648	655
3	14	2	756	776
4	14	2	889	826
5	14	2	374	336
6	14	2	326	349
7	14	2	621	975
8	14	2	314	349
9	14	2	977	620
12	14	2	303	334
13	14	2	342	341
1	15	2	214	352
7	15	2	903	881
8	15	2	210	253
9	15	2	629	724
11	15	2	303	439
12	15	2	257	270
15	15	2	224	132
0	16	2	348	311
1	16	2	181	191
3	16	2	346	344
6	16	2	210	182
7	16	2	232	263
9	16	2	306	469
10	16	2	145	148
12	16	2	219	101
2	6	3	922	883
2	18	3	290	362
3	18	3	436	392
5	18	3	182	199
6	18	3	140	221
7	18	3	190	203
9	18	3	261	213
10	18	3	153	35
11	18	3	294	190
7	19	3	305	263
10	19	3	159	119
2	20	3	280	215
3	20	3	190	151
5	20	3	269	256
9	21	3	146	155
6	22	3	203	111
0	0	4	193	117
0	1	4	656	772
1	1	4	453	416
0	2	4	1100	1062
1	2	4	1122	1143
2	2	4	588	492
0	3	4	230	246
1	3	4	439	475
2	3	4	436	449
3	3	4	449	463
0	4	4	233	222
1	4	4	927	893
2	4	4	2026	2061
3	4	4	1270	1290
4	4	4	1066	1062
0	5	4	676	573
1	5	4	951	1109
2	5	4	647	649

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
13	16	2	211	167	0	6	3	1176	1043	4	10	3	520	522	11	13	3	444	393
14	16	2	192	112	1	6	3	2799	2683	5	10	3	979	1007	12	13	3	434	426
0	17	2	128	155	2	6	3	3096	2858	6	10	3	279	339	0	14	3	337	283
4	17	2	144	172	3	6	3	1606	1670	7	10	3	728	719	1	14	3	635	788
5	17	2	399	399	4	6	3	1081	1019	8	10	3	208	297	2	14	3	344	393
7	17	2	460	467	5	6	3	1412	1361	9	10	3	242	272	3	14	3	489	460
2	10	2	412	466	0	7	3	959	960	1	11	3	147	320	4	14	3	481	470
3	18	2	367	301	1	7	3	737	678	3	11	3	575	574	5	14	3	807	791
4	18	2	210	235	2	7	3	604	598	4	11	3	325	404	6	14	3	456	479
5	18	2	411	401	3	7	3	534	612	5	11	3	285	292	7	14	3	509	577
6	18	2	140	99	4	7	3	923	944	6	11	3	241	197	9	14	3	210	244
8	18	2	332	290	5	7	3	322	286	9	11	3	228	246	11	14	3	224	298
0	20	2	154	209	6	7	3	869	831	10	11	3	338	390	14	14	3	228	139
2	20	2	297	239	7	7	3	451	414	0	12	3	790	834	4	15	3	143	212
5	20	2	217	238	0	8	3	862	785	1	12	3	1333	1295	5	15	3	511	562
0	1	3	432	499	1	8	3	176	117	2	12	3	550	538	6	15	3	323	380
1	1	3	498	585	2	8	3	941	985	3	12	3	1053	1091	7	15	3	280	293
0	2	3	1328	1308	3	8	3	852	827	4	12	3	269	239	8	15	3	148	131
2	2	3	890	734	4	8	3	1038	1176	5	12	3	767	800	9	15	3	220	237
0	3	3	720	812	5	8	3	417	420	6	12	3	442	419	11	15	3	335	335
1	3	3	973	1152	6	8	3	431	471	7	12	3	406	324	12	15	3	304	348
2	3	3	1018	1206	7	8	3	411	451	8	12	3	137	166	15	15	3	336	252
3	3	3	540	650	8	8	3	358	370	9	12	3	203	201	1	16	3	450	380
4	3	3	3982	4035	0	9	3	296	260	10	12	3	328	350	3	16	3	355	389
1	4	3	705	641	2	9	3	370	321	11	12	3	266	273	4	16	3	196	205
2	4	3	3833	3739	3	9	3	397	423	12	12	3	311	392	5	16	3	376	339
3	4	3	1461	1304	5	9	3	611	602	1	13	3	422	372	11	16	3	272	199
4	4	3	1764	1633	6	9	3	506	534	4	13	3	135	226	3	17	3	228	248
0	5	3	2176	2277	8	9	3	151	81	5	13	3	302	280	4	17	3	228	313
1	5	3	747	714	9	9	3	401	403	6	13	3	552	545	7	17	3	343	308
2	5	3	987	1090	0	10	3	251	303	7	13	3	500	487	9	17	3	257	278
3	5	3	907	958	1	10	3	1785	1706	8	13	3	298	290	12	17	3	206	94
4	5	3	1795	1823	2	10	3	514	468	9	13	3	193	217	15	17	3	225	186
5	5	3	112	58	3	10	3	1494	1405	10	13	3	383	375	0	18	3	621	622
0	5	4	1555	1568
3	6	5	689	802
5	6	5	590	634
0	7	5	1513	1598
1	7	5	136	217
2	7	5	895	859
3	7	5	633	660
4	7	5	310	319
1	8	5	110	26
6	7	5	669	569
7	7	5	518	484
0	8	5	731	739
1	8	5	905	1031
2	8	5	1129	1027
3	8	5	1029	1043
4	8	5	1188	1106
5	8	5	1097	1126
6	8	5	814	493
7	8	5	1198	1234
8	8	5	129	177
0	9	5	291	262
1	9	5	628	571
2	9	5	969	982
3	9	5	564	903
4	9	5	819	739
5	9	5	644	590
6	9	5	481	581
7	9	5	306	280
8	9	5	247	244
9	9	5	473	498
0	10	5	1438	1329
1	10	5	715	614
2	10	5	1435	1405
3	10	5	306	308

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
3	5	4	657	685	1	10	4	875	912	10	13	4	400	407	8	17	4	175	214
4	5	4	949	951	2	10	4	739	765	11	13	4	388	412	9	17	4	230	265
5	5	4	611	498	4	10	4	704	660	12	13	4	208	191	11	17	4	128	296
1	6	4	1490	1476	5	10	4	627	549	0	14	4	525	538	0	18	4	294	263
2	6	4	2533	2165	6	10	4	799	840	1	14	4	262	159	1	18	4	368	351
3	6	4	1125	937	7	10	4	833	829	2	14	4	482	507	3	18	4	351	305
4	6	4	368	318	8	10	4	212	152	3	14	4	427	463	5	18	4	381	376
5	6	4	667	714	9	10	4	276	291	4	14	4	160	185	7	18	4	293	245
6	6	4	1002	1109	10	10	4	485	487	5	14	4	455	447	11	18	4	131	135
0	7	4	336	332	0	11	4	929	1007	6	14	4	312	371	5	19	4	140	192
2	7	4	679	661	1	11	4	158	154	7	14	4	389	368	0	20	4	237	185
3	7	4	471	536	2	11	4	288	245	8	14	4	293	248	1	21	4	151	118
4	7	4	829	731	3	11	4	255	273	1	15	4	396	393	5	22	4	162	186
5	7	4	412	437	4	11	4	460	428	2	15	4	194	169	0	1	5	261	337
6	7	4	762	706	5	11	4	550	499	6	15	4	130	157	1	1	5	399	390
7	7	4	370	283	6	11	4	454	483	7	15	4	336	299	0	2	5	4220	4181
0	8	4	1106	834	7	11	4	402	403	8	15	4	399	427	1	2	5	1013	924
1	8	4	331	395	10	11	4	407	439	9	15	4	308	222	2	2	5	2025	1872
2	8	4	411	452	0	12	4	149	96	10	15	4	249	249	0	3	5	893	657
3	8	4	595	483	1	12	4	728	694	11	15	4	336	426	1	3	5	1129	1073
4	8	4	825	693	3	12	4	352	357	12	15	4	299	213	2	3	5	356	397
5	8	4	671	646	5	12	4	405	499	14	15	4	168	185	3	3	5	646	693
6	8	4	573	608	6	12	4	310	317	1	16	4	428	434	0	4	5	2288	2259
7	8	4	170	351	7	12	4	409	369	2	16	4	163	179	1	4	5	1065	1238
8	8	4	256	161	8	12	4	308	316	4	16	4	399	421	2	4	5	1155	1232
0	9	4	568	593	9	12	4	235	196	5	16	4	419	409	3	4	5	929	949
2	9	4	530	580	10	12	4	155	81	6	16	4	314	284	0	5	5	943	981
3	9	4	649	603	11	12	4	431	442	7	16	4	385	337	1	5	5	625	583
4	9	4	855	870	0	13	4	524	498	10	16	4	228	180	2	5	5	753	778
5	9	4	128	8	3	13	4	273	301	0	17	4	291	221	3	5	5	752	754
6	9	4	799	777	4	13	4	262	309	2	17	4	138	96	4	5	5	532	598
7	9	4	572	558	7	13	4	197	203	4	17	4	238	271	5	5	5	301	351
9	9	4	374	398	8	13	4	473	438	6	17	4	317	218	0	6	5	1374	1346
0	10	4	609	499	9	13	4	215	229	7	17	4	455	495	1	6	5	738	738

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

PAGE 5

H	K	L	10FO	10FC
4	10	5	999	1028
5	10	5	949	866
6	10	5	320	381
7	10	5	665	635
8	10	5	541	585
9	10	5	432	409
10	10	5	323	341
1	11	5	307	311
2	11	5	419	484
3	11	5	368	318
4	11	5	600	632
5	11	5	379	364
6	11	5	114	167
7	11	5	400	428
8	11	5	450	529
9	11	5	668	666
10	11	5	588	603
11	11	5	714	703
0	12	5	820	875
1	12	5	341	306
2	12	5	693	703
3	12	5	374	334
4	12	5	406	414
5	12	5	248	266
7	12	5	291	242
10	12	5	183	249
0	13	5	300	168
1	13	5	405	387
5	13	5	256	265
6	13	5	110	94
7	13	5	350	357
8	13	5	219	300
9	13	5	323	351
10	13	5	262	188
13	13	5	475	419
1	14	5	298	300
2	14	5	373	338
3	14	5	467	488
5	14	5	260	305
9	14	5	250	332
11	14	5	259	311
0	15	5	207	160
11	15	5	549	459
1	16	5	325	327
3	16	5	334	404
13	16	5	208	111
1	17	5	254	205
3	17	5	146	113
6	17	5	157	98
9	17	5	285	270
1	18	5	358	433
3	18	5	327	304
4	18	5	241	194
0	19	5	208	168
5	20	5	199	152
3	21	5	171	90
0	1	6	337	280
1	1	6	230	208
0	2	6	9993	5617
5	2	6	1979	1830
1	2	6	9670	5618
2	2	6	446	362
0	3	6	572	570
1	3	6	363	426
2	3	6	823	816
3	3	6	403	362
4	3	6	1722	1624
2	4	6	3013	2703
3	4	6	2576	2627
4	4	6	2379	2276
0	5	6	854	799
1	5	6	569	567
2	5	6	895	869
3	5	6	807	798
4	5	6	971	957
0	6	6	3211	3019
1	6	6	1349	1273
2	6	6	856	877
3	6	6	719	688
4	6	6	650	658
5	6	6	670	703
6	6	6	1199	1167
1	7	6	252	327
2	7	6	669	795
3	7	6	207	339
5	7	6	319	298
7	7	6	481	417
7	7	6	230	248
0	8	6	2319	2034
1	8	6	1232	1264
2	8	6	1803	1664
3	8	6	308	335
5	8	6	609	601
6	8	6	534	527
7	8	6	815	874
8	8	6	918	887
0	9	6	277	315
1	9	6	572	570
2	9	6	360	343
3	9	6	762	805
4	9	6	316	408
6	9	6	280	222
7	9	6	485	493
8	9	6	427	346
9	9	6	652	646
10	9	6	438	431
7	10	6	668	657
8	10	6	385	345
9	10	6	448	480
10	10	6	325	380
1	11	6	602	552
4	11	6	260	254
5	11	6	322	330
6	11	6	234	206
7	11	6	320	272
8	11	6	243	204
9	11	6	246	303
1	12	6	998	972
2	12	6	230	313
3	12	6	663	708
4	12	6	320	398
5	12	6	472	449
6	12	6	229	257
4	13	6	358	347
5	13	6	230	216
6	13	6	220	293
7	13	6	365	329
9	13	6	900	271
11	13	6	210	229
13	13	6	543	530
0	14	6	623	565
1	14	6	588	559
2	14	6	134	136
3	14	6	422	442
4	14	6	326	308
7	14	6	328	351
8	14	6	204	258
0	15	6	148	216
3	15	6	235	238
5	15	6	374	430
6	15	6	387	390
7	15	6	318	353
9	15	6	260	320
11	15	6	315	336
13	15	6	433	469
14	15	6	125	155
3	16	6	439	473
5	16	6	316	341
6	16	6	396	446
13	16	6	132	13
0	17	6	316	270
1	17	6	206	222
3	17	6	220	237
4	17	6	207	236
5	17	6	325	254
9	17	6	600	516
14	17	6	201	91
1	18	6	346	277
6	18	6	152	93
7	18	6	307	276
14	18	6	130	40
3	19	6	156	112
4	19	6	142	63
1	20	6	124	43
2	20	6	280	267

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

PAGE 7

H	K	L	10FO	10FC
6	10	8	489	427
7	10	8	373	323
8	10	8	517	519
9	10	8	197	181
0	11	8	536	496
1	11	8	390	419
3	11	8	507	510
4	11	8	377	356
6	11	8	256	298
8	11	8	486	492
10	11	8	338	319
0	12	8	916	825
1	12	8	533	517
2	12	8	645	651
5	12	8	514	471
6	12	8	219	203
7	12	8	221	199
8	12	8	392	432
10	12	8	148	86
12	12	8	153	35
1	13	8	393	440
3	13	8	360	339
4	13	8	200	249
6	13	8	355	338
7	13	8	222	187
12	13	8	168	112
0	14	8	217	305
1	14	8	169	222
2	14	8	500	462
3	14	8	458	459
4	14	8	501	546
5	14	8	264	330
11	14	8	201	211
14	14	8	140	14
2	15	8	258	275
3	15	8	135	247
5	15	8	208	172
7	15	8	373	368
9	15	8	258	232
0	16	8	423	478
1	16	8	330	277
2	16	8	259	236
4	16	8	259	227
7	16	8	271	277
8	16	8	176	144
13	16	8	176	137
5	17	8	150	170
9	17	8	285	269
1	18	8	281	254
2	18	8	207	279
3	18	8	342	460
4	18	8	218	220
5	18	8	130	223
7	18	8	197	167
8	18	8	237	167
10	18	8	144	94
7	19	8	154	173
1	20	8	306	296
2	22	8	184	122
0	1	9	746	678
0	2	9	230	136
1	2	9	548	561
0	3	9	262	354
1	3	9	527	568
2	3	9	1581	1661
0	4	9	2717	2465
2	4	9	1077	1004
3	4	9	626	638
4	4	9	831	693
1	5	9	417	426
2	5	9	997	954
3	5	9	272	301
5	5	9	283	230
0	6	9	411	379
1	6	9	380	437
2	6	9	239	209
3	6	9	1206	1119
4	6	9	317	439
6	6	9	686	676
5	7	9	738	684
6	7	9	675	662
7	7	9	1055	1129
0	8	9	340	311
1	8	9	413	358
2	8	9	664	594
3	8	9	959	868
4	8	9	1116	883
5	8	9	1916	1770
6	8	9	755	659
7	8	9	658	601
8	8	9	216	234
0	9	9	294	288
1	9	9	391	252
2	9	9	498	514
3	9	9	380	391
4	9	9	1148	1094
5	9	9	818	707
6	9	9	452	395
7	9	9	282	319
8	9	9	508	476
0	10	9	329	278
1	10	9	735	641
2	10	9	879	833
3	10	9	745	667
4	10	9	727	770
5	10	9	714	756
6	10	9	309	309
7	10	9	277	336
8	10	9	201	200
9	10	9	453	423
0	11	9	452	459
1	11	9	389	329
3	11	9	409	461
4	11	9	511	514
7	11	9	403	413
11	11	9	244	291
1	12	9	466	459
3	12	9	358	365
4	12	9	259	315
5	12	9	200	266
6	12	9	278	272
7	12	9	671	598
8	12	9	234	189
9	12	9	319	350
12	12	9	173	100
1	13	9	162	66
5	13	9	334	306
6	13	9	297	315
7	13	9	234	179
8	13	9	252	249
0	14	9	419	430
1	14	9	495	623
3	14	9	314	361
4	14	9	184	202
5	14	9	282	265
7	14	9	130	198
9	14	9	140	139
2	15	9	325	353
4	15	9	173	330
6	15	9	303	301
13	15	9	169	216
0	16	9	345	414
1	16	9	161	208
2	16	9	186	182
3	16	9	412	413
14	16	9	203	77
1	17	9	121	153
4	17	9	282	283
7	17	9	200	141
1	18	9	486	486
3	18	9	487	439
4	18	9	158	182
9	18	9	155	131
5	19	9	243	173
2	20	9	184	133
6	21	9	193	159
0	2	10	1184	1018
1	2	10	735	789
0	3	10	1136	1087
1	3	10	804	829
2	3	10	1267	1273
3	3	10	676	700
0	4	10	651	545
1	4	10	936	987
2	4	10	720	743
3	4	10	744	748
4	4	10	1001	905
0	5	10	442	441
1	5	10	442	522

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

PAGE 6

H	K	L	10FO	10FC
3	20	6	194	172
5	20	6	205	217
11	20	6	194	80
0	1	7	1837	1590
1	1	7	537	510
0	2	7	970	857
1	2	7	1412	1292
2	2	7	3015	2927
0	3	7	1083	1003
1	3	7	840	910
2	3	7	1662	1651
0	4	7	745	669
1	4	7	969	979
2	4	7	348	319
3	4	7	612	558
4	4	7	484	586
0	5	7	686	638
1	5	7	511	487
2	5	7	336	272
4	5	7	866	845
5	5	7	722	714
0	6	7	1347	1623
1	6	7	333	302
2	6	7	1734	1742
3	6	7	338	394
4	6	7	1065	1003
5	6	7	482	465
6	6	7	1027	943
0	7	7	637	701
1	7	7	161	180
2	7	7	361	462
3	7	7	302	245
4	7	7	586	546
5	7	7	634	607
6	7	7	1814	1047
7	7	7	781	709
8	7	7	2220	2247
1	8	7	1346	1348
2	8	7	1080	1013
3	8	7	882	982
4	8	7	1270	1173
5	8	7	696	689
6	8	7	425	409
8	8	7	388	429
0	9	7	297	294
1	9	7	407	490
3	9	7	341	289
4	9	7	547	509
5	9	7	1098	969
6	9	7	302	324
7	9	7	353	375
8	9	7	125	217
0	10	7	1260	1078
1	10	7	732	588
2	10	7	873	775
3	10	7	1199	1197
4	10	7	941	975
5	10	7	1130	1045
6	10	7	187	290
7	10	7	261	245
8	10	7	200	211
0	11	7	176	142
1	11	7	584	579
3	11	7	278	262
4	11	7	371	370
6	11	7	611	590
7	11	7	337	288
9	11	7	310	486
0	12	7	538	502
1	12	7	237	223
2	12	7	201	183
3	12	7	152	162
4	12	7	640	686
5	12	7	501	466
6	12	7	387	324
7	12	7	259	324
8	12	7	213	226
10	12	7	204	244
11	12	7	174	248
0	13	7	743	784
2	13	7	196	153
3	13	7	222	258
4	13	7	318	253
5	13	7	230	176
6	13	7	211	272
8	13	7	152	210
9	13	7	147	181
11	13	7	306	259
13	13	7	370	277
0	14	7	315	243
2	14	7	377	357
4	14	7	389	420
5	14	7	181	201
6	14	7	331	315
7	14	7	275	276
8	14	7	248	281
0	15	7	606	634
1	15	7	217	250
2	15	7	447	451
3	15	7	218	239
4	15	7	403	403
5	15	7	489	517
0	16	7	176	155
2	16	7	319	347
0	6	8	1634	1478
1	6	8	504	427
2	6	8	1488	1290
3	6	8	1007	895
4	6	8	644	573
5	6	8	769	890
6	6	8	646	541
0	7	8	470	456
1	7	8	499	411
2	7	8	301	303
4	7	8	219	205
5	7	8	240	220
7	7	8	730	766
0	8	8	674	695
1	8	8	611	675
2	8	8	1430	1503
3	8	8	812	745
4	8	8	973	958
5	8	8	1228	1298
6	8	8	479	417
7	8	8	309	243
8	8	8	163	211
0	9	8	279	338
1	9	8	385	352
2	9	8	808	798
3	9	8	678	659
4	9	8	433	381
5	9	8	501	516
7	9	8	245	272
0	10	8	164	143
1	10	8	538	454
2	10	8	253	299
3	10	8	797	737
4	10	8	554	616
5	10	8	942	973

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

PAGE 8

H	K	L	10FO	10FC
4	10	10	456	545
5	10	10	881	885
6	10	10	273	221
7	10	10	205	237
10	10	10	160	160
1	11	10	118	83
3	11	10	320	369
4	11	10	111	181
5	11	10	304	311
8	11	10	377	364
9	11	10	311	375
10	11	10	245	182
11	11	10	280	229
0	12	10	302	356
1	12	10	448	544
6	12	10	119	130
9	12	10	258	335
1	13	10	114	96
5	13	10	182	160
9	13	10	337	408
10	13	10	276	297
11	13	10	202	275
1	14	10	454	421
2	14	10	290	293
3	14	10	560	563
5	14	10	391	431
7	14	10	248	264
13	14	10	252	259
0	15	10	135	213
3	15	10	125	173
4	15	10	186	224
5	15	10	300	293
6	15	10	286	263
9	15	10	402	424
10	15	10	214	199
13	15	10	183	194
0	16	10	199	124
3	16	10	305	334
4	16	10	246	215
5	16	10	294	282
7	16	10	277	284
9	16	10	166	252
1	17	10	175	97
5	17	10	327	323
9	17	10	220	345
1	20	10	320	248
9	20	10	150	93
0	1	11	1230	1174
0	2	11	828	897
1	2	11	1038	1078
0	3	11	254	196
1	3	11	511	540
2	3	11	380	416
3	3	11	626	516
0	4	11	1739	1794
1	4	11	463	510
2	4	11	1562	1293
3	4	11	631	653
4	4	11	650	563
0	5	11	291	278
1	5	11	483	526
2	5	11	426	446
3	5	11	549	539
5	5	11	245	272
0	6	11	1232	1183
1	6	11	1183	1100
2	6	11	842	843
3	6	11	669	734
4	6	11	904	946
5	6	11	443	372
6	6	11	372	420
0	7	11	475	486
1	7	11	301	330
2	7	11	180	282
3	7	11	112	37
4	7	11	408	521
5	7	11	349	376
6	7	11	381	364
0	8	11	567	517
1	8	11	487	461
2	8	11	505	430
3	8	11	589	606
5	8	11	480	490
6	8	11	151	83
7	8	11	461	490
8	8	11	223	195
0	9	11	321	289
1	9	11	310	242
2	9	11	549	584
3	9	11	413	452
4	9	11	572	632
5	9	11	335	356
8	9	11	204	191
9	9	11	280	341
0	10	11	549	508
1	10	11	565	595
2	10	11	543	506
3	10	11	452	471
4	10	11	474	443
5	10	11	549	534
7	10	11	346	404
8	10	11	403	465
10	10	11	236	189
4	11	11	168	184
5	11	11	148	183
6	11	11	448	482
9	11	11	327	329
10	11	11	187	237
0	12	11	962	970
1	12	11	284	304
2	12	11	212	244
7	12	11	378	371
8	12	11	296	313
1	13	11	209	181
4	13	11	226	188
9	13	11	207	239
10	13	11	173	198
12	13	11	162	144
1	14	11	262	247
8	14	11	129	21
9	14	11	265	224
10	14	11	147	98
11	14	11	204	206
1	15	11	287	200
14	15	11	168	62
1	16	11	379	329
2	16	11	278	363
5	16	11	283	275
9	16	11	125	184
4	17	11	129	83
5	17	11	159	189
6	17	11	181	83
9	17	11	140	150
11	17	11	240	260
13	17	11	130	109
5	18	11	165	139

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC
1	19	11	206	194
5	19	11	139	76
1	21	11	148	46
0	0	12	1097	2153
0	1	12	516	605
1	1	12	357	492
0	2	12	1986	1643
1	2	12	338	376
0	3	12	501	503
1	3	12	340	374
2	3	12	529	562
0	4	12	1386	1344
2	4	12	1256	1151
3	4	12	603	551
4	4	12	1250	1089
0	5	12	344	357
1	5	12	483	557
3	5	12	662	605
4	5	12	559	504
5	5	12	291	306
0	6	12	707	728
1	6	12	1181	1226
2	6	12	1218	1122
3	6	12	1139	1172
4	6	12	325	396
5	6	12	499	478
0	7	12	573	558
4	7	12	384	363
5	7	12	279	298
6	7	12	216	126
7	7	12	357	298
1	8	12	623	628
2	8	12	391	409
3	8	12	331	259
8	8	12	843	772
7	8	12	432	460
1	9	12	389	426
3	9	12	358	305
4	9	12	275	266
5	9	12	417	346
6	9	12	427	455
9	9	12	126	170
0	10	12	544	592
4	10	12	328	313
1	10	12	340	374
2	10	12	494	492
3	10	12	248	290
9	10	12	399	372
0	11	12	552	514
1	11	12	184	201
2	11	12	415	419
3	11	12	328	294
5	11	12	388	302
6	11	12	220	229
8	11	12	367	392
9	11	12	127	133
10	11	12	299	347
11	11	12	960	930
0	12	12	543	499
1	12	12	445	441
3	12	12	421	355
5	12	12	357	351
7	12	12	385	231
0	13	12	471	499
1	13	12	186	186
2	13	12	834	892
6	13	12	367	340
11	13	12	377	388
13	13	12	186	173
2	14	12	285	244
5	14	12	158	184
6	14	12	256	227
1	15	12	141	182
2	15	12	180	162
3	15	12	255	271
4	15	12	165	97
1	16	12	302	298
3	16	12	237	258
4	16	12	243	252
5	16	12	222	267
6	16	12	208	279
7	16	12	247	284
9	16	12	200	167
14	16	12	141	101
3	17	12	150	146
9	17	12	205	226
10	17	12	210	121
13	17	12	174	72
1	18	12	297	272
2	18	12	259	301
7	18	12	299	274
4	18	12	933	888
6	18	12	185	161
9	18	12	178	125
0	19	12	167	199
3	21	12	180	86
0	2	13	2024	1890
2	2	13	2200	1987
0	3	13	113	70
1	3	13	299	266
2	3	13	196	229
3	3	13	334	257
1	4	13	1347	1310
2	4	13	1247	1220
3	4	13	189	170
4	4	13	1770	1716
0	5	13	400	372
1	5	13	205	196
2	5	13	272	279
3	5	13	291	296
5	5	13	120	50
0	6	13	767	618
1	6	13	748	805
2	6	13	1019	1043
3	6	13	450	486
4	6	13	343	415
5	6	13	572	453
6	6	13	328	309
1	7	13	531	512
2	7	13	232	277
4	7	13	376	309
5	7	13	321	308
6	7	13	407	430
7	7	13	361	422
0	8	13	865	840
1	8	13	497	484
2	8	13	629	634
3	8	13	296	233
4	8	13	683	614
5	8	13	663	543
7	8	13	816	857
1	9	13	386	299
2	9	13	422	360
3	9	13	298	287
4	9	13	490	454
5	9	13	366	304
6	9	13	298	238
7	9	13	456	436
8	9	13	426	419
0	10	13	235	279
1	10	13	201	197
2	10	13	810	743
3	10	13	722	685
4	10	13	622	568
5	10	13	368	369
6	10	13	236	266
1	11	13	241	257
2	11	13	120	150
7	11	13	180	169
9	11	13	327	302
0	12	13	295	198
1	12	13	906	866
3	12	13	584	667
4	12	13	295	345
6	12	13	173	136
7	12	13	418	475
9	12	13	431	481
1	13	13	148	192
3	13	13	239	213
5	13	13	193	210
6	13	13	266	317
7	13	13	432	442
8	13	13	337	320
11	13	13	255	268
12	13	13	241	175
13	13	13	358	323
1	14	13	614	692
2	14	13	251	286
3	14	13	370	324
4	14	13	328	343

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC
3	13	15	252	187
4	13	15	245	239
5	13	15	379	374
6	13	15	303	275
7	13	15	333	335
12	13	15	280	311
2	14	15	153	214
3	14	15	444	510
4	14	15	151	193
5	14	15	615	659
7	14	15	377	376
2	15	15	179	95
3	15	15	274	290
4	15	15	181	178
5	15	15	372	304
7	15	15	325	344
11	15	15	271	255
1	16	15	300	335
2	16	15	284	215
3	16	15	802	516
5	16	15	394	471
13	16	15	185	136
3	17	15	185	163
5	17	15	296	288
7	17	15	128	128
9	17	15	251	171
0	18	15	310	308
2	18	15	140	103
3	18	15	150	128
1	20	15	160	146
0	0	16	1607	1708
1	1	16	661	591
0	2	16	1167	1038
1	3	16	260	280
3	3	16	177	276
0	4	16	2311	2230
1	4	16	790	777
2	4	16	1019	1085
3	4	16	595	495
0	5	16	615	552
5	5	16	432	468
0	6	16	1394	1714
1	6	16	811	791
2	6	16	1019	1049
3	6	16	713	680
4	6	16	470	464
5	6	16	418	404
6	6	16	853	961
1	7	16	304	312
2	7	16	367	329
4	7	16	684	681
5	7	16	304	265
6	7	16	566	622
0	8	16	171	105
1	8	16	396	355
2	8	16	120	220
3	8	16	668	670
5	8	16	214	129
8	8	16	259	230
4	8	16	544	560
0	9	16	469	408
1	9	16	298	260
2	9	16	207	235
4	9	16	367	415
6	9	16	396	319
7	9	16	315	381
8	9	16	167	222
1	10	16	734	702
9	10	16	660	727
3	10	16	363	452
6	10	16	537	555
7	10	16	231	139
8	10	16	408	381
9	10	16	197	229
2	11	16	225	299
3	11	16	241	275
4	11	16	287	240
5	11	16	259	241
6	11	16	359	336
7	11	16	399	436
8	11	16	397	337
0	12	16	522	583
1	12	16	844	860
2	12	16	339	328
3	12	16	347	419
5	12	16	662	643
7	12	16	386	400
9	12	16	226	241
11	12	16	342	330
2	13	16	176	215
3	14	16	241	199
7	14	16	259	245
9	14	16	192	148
9	15	16	232	193
12	15	16	199	275
1	16	16	689	659
5	16	16	395	348
6	16	16	221	217
7	16	16	285	306
0	17	16	136	71
5	17	16	216	176
12	17	16	217	193
5	18	16	330	328
7	18	16	323	220
10	18	16	140	139
0	20	16	131	41
1	20	16	276	202
4	20	16	141	172
0	1	17	478	599
0	2	17	1817	1835
2	2	17	1530	1402
1	3	17	246	268
5	3	17	573	571
3	3	17	508	520
0	4	17	1990	1988
1	4	17	783	825
2	4	17	662	643
3	4	17	675	670
4	4	17	577	514
5	5	17	186	260
2	5	17	399	222
3	5	17	449	459
1	6	17	577	555
2	6	17	554	582
4	6	17	574	569
4	6	17	393	463
6	6	17	548	444
0	7	17	287	498
1	7	17	246	249
2	7	17	371	351
3	7	17	213	243
5	7	17	333	257
6	7	17	780	728
0	8	17	626	617
1	8	17	327	262
2	8	17	274	179
3	8	17	721	764
4	8	17	694	684
5	8	17	831	975
6	9	17	327	369
7	8	17	654	723
1	9	17	157	109
2	9	17	326	352
3	9	17	251	169
4	9	17	357	416
5	9	17	334	367
6	9	17	513	496
7	9	17	462	529
9	9	17	218	188
2	10	17	589	539
4	10	17	650	719
6	10	17	562	571
8	10	17	449	411
9	10	17	118	165
10	10	17	210	113
3	11	17	254	274
4	11	17	370	362
5	11	17	197	209
7	11	17	186	148
8	11	17	291	259
0	12	17	331	410
1	12	17	523	518
2	12	17	194	191
3	12	17	320	285
4	12	17	119	191
5	12	17	332	327
7	12	17	327	271
8	12	17	235	262

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC
5	14	13	275	227
6	14	13	281	300
7	14	13	372	416
9	14	13	258	317
10	14	13	228	232
0	15	13	184	261
6	15	13	269	280
7	15	13	164	132
11	15	13	295	233
15	15	13	189	69
1	16	13	269	208
3	16	13	392	399
4	16	13	150	77
14	16	13	182	128
4	18	13	226	186
5	18	13	293	324
3	19	13	222	137
8	19	13	131	137
5	20	13	217	220
0	1	14	302	495
0	2	14	1131	1103
1	2	14	695	578
2	2	14	1613	1511
0	3	14	594	567
1	4	14	566	589
2	4	14	760	787
3	4	14	1170	1178
0	5	14	418	409
1	5	14	321	348
2	5	14	252	253
4	5	14	479	544
0	6	14	903	770
2	6	14	525	543
3	6	14	306	231
4	6	14	218	202
5	6	14	768	722
6	6	14	572	528
0	7	14	310	249
1	7	14	402	320
2	7	14	596	667
3	7	14	363	272
5	7	14	606	619
7	7	14	355	313
0	8	14	780	745
1	8	14	645	691
2	8	14	1025	996
3	8	14	958	974
4	8	14	193	173
5	8	14	418	312
6	8	14	487	465
7	8	14	772	772
8	8	14	1136	1043
0	9	14	494	506
2	9	14	248	284
3	9	14	335	349
5	9	14	157	219
6	9	14	414	448
7	9	14	383	384
8	9	14	756	820
9	9	14	496	529
1	10	14	658	581
3	10	14	494	509
6	10	14	162	232
7	10	14	522	524
9	10	14	334	380
2	11	14	265	213
4	11	14	309	331
5	11	14	185	181
6	11	14	203	248
7	11	14	446	438
8	11	14	233	261
0	12	14	388	383
1	12	14	150	8
2	12	14	288	297
3	12	14	280	338
5	12	14	286	351
6	12	14	167	146
2	13	14	323	356
3	13	14	243	325
0	14	14	932	945
1	14	14	908	917
2	14	14	1113	1102
3	14	14	1024	1027
4	14	14	1126	1114
0	15	15	810	866
1	15	15	426	392
2	15	15	414	355
3	15	15	393	368
4	15	15	1161	1116
5	15	15	477	487
6	15	15	1095	1028
7	15	15	1550	1437
5	15	15	639	549
6	15	15	317	277
7	15	15	270	310
7	15	15	240	242
8	15	15	628	580
5	15	15	369	405
7	15	15	737	692
7	15	15	307	181
0	16	15	530	601
1	16	15	513	452
2	16	15	410	364
3	16	15	260	256
4	16	15	575	495
5	16	15	439	368
6	16	15	330	263
9	16	15	285	325
1	17	15	829	899
2	17	15	403	163
9	17	15	259	284
0	10	15	373	319
1	10	15	1166	1080
2	10	15	410	372
3	10	15	526	528
4	10	15	287	278
6	10	15	204	225
7	10	15	328	247
8	10	15	299	350
0	11	15	291	274
1	11	15	161	249
3	11	15	177	242
4	11	15	420	415
6	11	15	366	380
8	11	15	207	274
9	11	15	329	267
10	11	15	288	314
1	12	15	529	600
2	12	15	276	287
3	12	15	643	640
4	12	15	478	546
5	12	15	286	271
6	12	15	398	522
7	12	15	440	461
0	13	15	274	191
2	13	15	178	250
2	8	15	410	364
3	8	15	260	256
4	8	15	575	495
5	8	15	439	368
6	8	15	330	263
7	8	15	285	325
8	8	15	245	271
9	8	15	215	239
2	9	15	259	284
3	9	15	343	325
4	9	15	902	945
5	9	15	810	866
6	9	15	426	392
7	9	15	414	355
8	9	15	393	368
9	9	15	1161	1116
10	9	15	477	487
11	9	15	1095	1028
12	9	15	1550	1437
13	9	15	639	549
14	9	15	317	277
15	9	15	270	310
16	9	15	240	242
17	9	15	628	580
18	9	15	369	405
19	9	15	737	692
20	9	15	307	181
21	9	15	530	601
22	9	15	513	452

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

H	K	L	10FO	10FC
9	12	17	499	481
10	12	17	220	196
0	13	17	238	293
3	13	17	444	518
5	13	17	272	214
6	13	17	336	370
9	13	17	181	139
1	14	17	218	212
2	14	17	326	311
7	14	17	204	220
9	14	17	283	277
1	15	17	258	192
3	15	17	321	253
4	15	17	179	164
5	15	17	190	196
12	15	17	197	171
3	16	17	248	254
4	16	17	252	193
4	17	17	159	96
7	17	17	234	146
1	18	17	169	163
2	18	17	208	268
5	18	17	237	302
7	18	17	244	227
7	19	17	194	140
2	20	17	209	160
4	20	17	164	160
1	1	18	188	169
1	2	18	302	489
2	2	18	813	839
0	3	18	724	704
1	3	18	646	669
3	3	18	209	117
1	4	18	697	678
2	4	18	1447	1469
3	4	18	543	602
4	4	18	868	829
5	5	18	620	612
3	5	18	264	306
4	5	18	415	462
1	6	18	518	400
2	6	18	487	432
3	6	18	644	599
4	6	18	357	415
5	6	18	302	226
6	6	18	497	525
0	7	18	293	371
2	7	18	462	413
3	7	18	292	344
4	7	18	371	358
6	7	18	159	296
0	8	18	514	560
1	8	18	162	294
2	8	18	1037	1080
3	8	18	545	502
4	8	18	638	610
5	8	18	308	352
6	8	18	429	460
7	8	18	523	564
8	8	18	383	398
1	9	18	315	262
4	9	18	318	259
5	9	18	260	306
7	9	18	208	203
8	9	18	324	351
9	9	18	287	242
0	10	18	556	592
2	10	18	501	440
3	10	18	383	383
5	10	18	307	336
7	10	18	408	382
8	10	18	311	316
9	10	18	346	369
10	10	18	328	298
2	11	18	459	411
3	11	18	411	367
4	11	18	337	322
5	11	18	223	277
8	11	18	235	249
9	11	18	176	209
1	12	18	279	262
2	12	18	374	364
7	12	18	217	227
8	12	18	149	98
9	12	18	267	274
10	12	18	200	112
12	12	18	148	116
2	13	18	177	228
0	14	18	352	435
1	14	18	320	297
2	14	18	254	297
2	15	18	243	289
4	15	18	343	307
7	15	18	194	181
2	17	18	194	191
3	17	18	249	218
2	18	18	220	194
3	18	18	194	191
7	18	18	271	218
0	20	18	173	245
3	20	18	230	199
1	1	19	261	288
0	2	19	1734	1801
2	2	19	467	498
0	3	19	552	471
0	4	19	345	469
2	4	19	454	526
3	4	19	431	436
4	4	19	378	386
0	5	19	690	729
4	5	19	457	423
3	5	19	215	65
0	6	19	388	440
1	6	19	447	477
2	6	19	989	995
3	6	19	475	520
4	6	19	691	683
5	6	19	659	617
6	6	19	462	518
1	7	19	363	369
4	7	19	290	323
7	7	19	360	315
6	7	19	630	706
7	7	19	179	152
0	8	19	919	943
1	8	19	530	524
3	8	19	592	579
3	8	19	165	233
4	8	19	267	272
6	8	19	636	594
9	9	19	190	233
2	10	19	261	336
3	10	19	414	475
5	10	19	168	228
10	10	19	297	363
0	11	19	481	486
3	11	19	219	222
4	11	19	477	446
5	11	19	255	231
6	11	19	348	371
8	11	19	186	168
9	11	19	149	227
10	11	19	347	295
0	12	19	305	279
1	12	19	270	187
2	12	19	709	680
3	12	19	388	413
4	12	19	326	269
6	12	19	310	346
7	12	19	279	210
1	13	19	196	165
5	13	19	343	349
11	13	19	171	212
0	14	19	527	621
1	14	19	448	389
2	14	19	416	443
3	14	19	353	293
5	14	19	477	515
6	14	19	261	292
7	14	19	355	321
0	15	19	243	189
3	15	19	204	226
4	15	19	162	105
9	15	19	214	190
4	16	19	339	345
6	17	19	204	147
10	17	19	234	202
4	18	19	247	220
0	0	20	962	983

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

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	1	20	385	468	8	9	20	328	326	0	2	21	339	449	6	11	21	384	318	4	6	22	730	792
0	2	20	329	126	0	10	20	339	289	2	2	21	370	506	7	11	21	284	254	5	6	22	823	805
1	2	20	508	649	1	10	20	373	326	2	3	21	260	337	8	11	21	199	213	1	7	22	864	175
2	2	20	892	838	2	10	20	253	232	0	4	21	505	529	9	11	21	239	248	2	7	22	310	325
0	3	20	554	608	3	10	20	361	347	1	4	21	700	746	10	11	21	176	148	3	7	22	216	236
0	4	20	739	697	4	10	20	284	292	2	4	21	760	736	1	12	21	584	547	4	7	22	408	498
2	4	20	466	434	6	10	20	354	333	4	4	21	1034	881	3	12	21	465	564	5	7	22	280	325
3	4	20	258	212	8	10	20	264	256	0	5	21	284	298	4	12	21	164	142	6	7	22	139	76
4	4	20	429	524	10	10	20	479	445	2	5	21	337	337	6	12	21	171	209	1	8	22	454	502
1	5	20	423	436	0	11	20	301	294	4	5	21	351	348	9	12	21	193	183	2	8	22	617	656
2	5	20	288	284	2	11	20	325	237	0	6	21	411	355	4	13	21	241	256	3	8	22	404	429
3	5	20	337	434	3	11	20	142	125	1	6	21	410	387	7	13	21	219	247	6	8	22	373	355
5	5	20	296	123	5	11	20	250	336	2	6	21	651	644	13	13	21	214	190	1	9	22	198	142
0	6	20	573	574	6	11	20	158	152	3	6	21	417	401	1	14	21	240	302	6	9	22	331	301
1	6	20	276	140	7	11	20	373	308	4	6	21	602	616	2	14	21	289	337	7	9	22	377	424
2	6	20	241	252	8	11	20	322	390	6	6	21	319	338	3	14	21	434	464	9	9	22	544	561
3	6	20	138	235	9	11	20	167	206	0	7	21	303	246	4	14	21	268	355	0	10	22	224	237
4	6	20	339	273	10	11	20	228	264	2	7	21	333	293	5	14	21	437	468	1	10	22	734	778
5	6	20	503	417	0	12	20	147	170	1	8	21	275	238	10	15	21	203	148	3	10	22	284	272
6	6	20	1005	1092	1	12	20	238	264	3	8	21	577	596	11	15	21	148	135	6	10	22	308	283
0	7	20	585	625	4	12	20	266	261	4	8	21	239	234	3	16	21	322	291	7	10	22	326	304
2	7	20	221	219	5	12	20	289	368	6	8	21	268	230	5	16	21	173	173	9	10	22	350	360
4	7	20	402	425	6	12	20	329	357	0	9	21	162	144	9	16	21	162	137	5	11	22	302	299
6	7	20	563	444	10	12	20	193	215	1	9	21	263	289	1	17	21	171	125	6	11	22	227	313
1	8	20	492	545	3	13	20	190	101	3	9	21	165	295	1	2	22	278	311	7	11	22	284	254
3	8	20	383	441	12	13	20	135	90	7	9	21	223	238	1	4	22	472	443	8	11	22	173	196
5	8	20	431	445	1	15	20	192	193	0	10	21	255	222	3	4	22	317	294	1	12	22	144	134
7	8	20	275	308	4	15	20	160	114	2	10	21	467	441	4	4	22	608	505	5	12	22	254	296
0	9	20	392	368	12	15	20	154	139	4	10	21	321	344	0	5	22	416	339	12	13	22	193	73
1	9	20	429	391	1	16	20	281	353	6	10	21	419	436	4	5	22	394	445	0	14	22	224	233
2	9	20	403	373	6	16	20	279	225	8	10	21	340	370	5	5	22	345	309	2	14	22	235	204
3	9	20	158	201	10	16	20	224	207	10	10	21	220	282	1	6	22	422	475	9	14	22	174	205
5	9	20	130	37	8	17	20	173	135	0	11	21	362	324	2	6	22	485	523	0	16	22	228	238
6	9	20	291	265	0	1	21	285	364	1	11	21	363	241	3	6	22	465	467	0	17	22	144	170

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

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	17	22	237	147	5	10	23	428	344	0	9	24	355	357	5	8	25	320	223	2	2	27	473	531
5	17	22	133	126	7	10	23	167	169	6	9	24	343	356	7	9	25	278	307	0	4	27	238	257
3	18	22	177	177	2	11	23	397	398	7	9	24	288	204	0	10	25	466	710	4	4	27	328	266
0	1	23	473	439	3	11	23	314	274	0	10	24	238	205	4	11	25	204	161	3	6	27	244	174
0	3	23	381	605	4	11	23	446	455	3	10	24	396	318	5	11	25	191	236	4	6	27	229	255
1	3	23	168	117	5	11	23	410	501	4	10	24	136	312	6	11	25	163	196	1	7	27	156	143
2	3	23	182	175	6	11	23	165	151	7	11	24	156	235	0	12	25	272	240	4	7	27	461	378
0	4	23	265	349	0	12	23	290	175	8	11	24	184	48	1	12	25	211	307	0	8	27	259	225
1	4	23	330	371	1	12	23	552	634	11	11	24	146	106	2	12	25	299	279	9	9	27	172	163
0	5	23	474	449	3	12	23	277	439	1	12	24	217	237	4	12	25	251	296	0	10	27	181	172
1	5	23	237	170	4	12	23	286	330	7	13	24	160	137	5	13	25	188	170	1	11	27	140	66
2	5	23	344	257	9	13	23	183	26	9	13	24	150	103	0	14	25	145	91	1	12	27	216	195
5	5	23	211	167	1	14	23	168	100	2	14	24	197	177	3	14	25	201	168	9	12	27	179	120
3	6	23	276	291	5	14	23	137	109	5	14	24	183	140	0	16	25	239	176	5	13	27	162	194
4	6	23	417	477	7	14	23	145	96	3	15	24	201	211	0	6	26	278	271	8	13	27	198	85
5	6	23	216	170	0	15	23	159	100	4	15	24	138	185	2	6	26	296	352	0	0	28	852	818
6	6	23	379	527	1	15	23	193	202	7	15	24	189	103	4	6	26	352	366	0	6	28	377	298
6	7	23	254	289	4	17	23	139	120	4	17	24	152	73	0	7	26	151	213	2	6	28	348	3[illegible]
0	8	23	213	135	5	17	23	223	100	1	1	25	299	331	2	[illegible]	26	154	196	4	6	28	259	
1	8	23	427	418	0	18	23	172	54	2	2	25	338	300	[illegible]	6	26	363	346	5	6	28	[illegible]	
2	8	23	221	270	0	0	24	1110	801	2	4	25	144	[illegible]	1	8	26	229	128	1	7	28	[illegible]	
3	8	23	373	355	1	1	24	141	124	4	4	[illegible]	[illegible]	[illegible]	[illegible]	8	26	447	383	4	7	28	242	[illegible]
4	8	23	372	362	2	2	24	367	248	0	5	25	[illegible]	[illegible]	[illegible]	[illegible]	26	416	414	7	7	28	201	157
5	8	23	648	601	0	4	24	579	593	1	5	25	187	[illegible]	[illegible]	1	26	181	358	0	8	28	248	278
6	8	23	317	296	0	5	24	291	283	0	6	25	193	174	0	10	26	191	55	2	8	28	433	390
2	9	23	326	329	0	6	24	399	430	4	6	25	367	413	1	10	26	343	324	4	8	28	332	217
4	9	23	309	303	2	6	24	326	339	1	7	25	156	71	3	10	26	380	329	2	9	28	173	123
5	9	23	454	494	4	6	24	205	133	4	7	25	144	182	4	10	26	275	294	7	9	28	181	195
7	9	23	385	418	0	7	24	310	210	6	7	25	391	355	9	10	26	144	93	5	10	28	167	180
0	10	23	442	401	1	7	24	265	257	0	8	25	699	745	9	11	26	215	108	6	10	28	216	62
1	10	23	783	776	0	8	24	552	655	1	8	25	217	241	0	12	26	178	183	0	11	28	179	129
2	10	23	372	383	1	8	24	371	294	2	8	25	373	404	4	13	26	173	113	5	11	28	241	212
3	10	23	779	709	3	8	24	294	286	3	8	25	195	137	1	16	26	252	264	8	11	28	249	276
4	10	23	198	76	4	8	24	335	345	4	8	25	160	234	0	1	27	182	160	1	12	28	245	177

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR DIMERIC DISULPHIDE

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
9	12	28	232	140	0	9	29	238	183	7	7	30	410	414	3	8	31	298	250	4	8	32	243	187
0	1	29	204	301	3	10	29	325	363	1	8	30	232	273	5	8	31	355	418	0	9	32	216	280
2	2	29	265	324	6	10	29	204	115	2	8	30	273	249	2	9	31	204	172	6	9	32	244	300
4	4	29	534	607	9	10	29	169	105	7	8	30	218	201	3	10	31	388	396	4	10	32	152	213
0	5	29	169	132	1	11	29	207	230	9	9	30	218	303	5	10	31	468	437	2	6	33	284	204
1	5	29	201	168	0	12	29	225	76	0	10	30	217	217	0	11	31	165	190	1	7	33	154	72
1	6	29	313	275	3	12	29	362	378	9	10	30	233	189	4	12	31	232	154	2	8	33	215	229
4	6	29	515	547	8	12	29	154	74	1	12	30	217	188	0	0	32	593	581	2	10	33	180	135
6	6	29	490	413	3	14	29	230	267	1	2	31	191	178	0	2	32	379	381	1	2	34	233	192
4	7	29	336	334	3	4	30	350	353	0	5	31	201	259	2	2	32	270	296	0	5	34	174	134
0	8	29	447	412	1	5	30	284	335	2	5	31	169	231	6	6	32	157	97	4	8	34	163	117
3	8	29	436	427	5	5	30	271	398	0	6	31	207	172	0	7	32	234	223	1	5	35	156	90
4	8	29	271	246	0	6	30	618	515	1	6	31	337	359	7	7	32	207	192	4	5	35	279	282
5	8	29	405	319	2	6	30	297	282	6	6	31	359	381	2	8	32	247	190	0	0	36	479	459
7	8	29	307	221	4	7	30	246	239															

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR N-ACETYL METHIONINE

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	0	0	118	114	1	9	0	159	155	1	0	1	159	160	-6	3	1	36	33	2	5	1	119	118
2	0	0	89	86	2	9	0	148	148	2	0	1	176	174	-5	3	1	32	32	3	5	1	66	62
3	0	0	74	78	3	9	0	38	37	3	0	1	187	187	-4	3	1	46	45	4	5	1	57	56
4	0	0	20	18	4	9	0	66	64	4	0	1	37	38	-3	3	1	47	45	5	5	1	47	47
5	0	0	32	30	5	9	0	38	39	5	0	1	23	24	-2	3	1	152	143	-5	6	1	36	35
6	0	0	50	47	6	9	0	29	30	6	0	1	43	43	-1	3	1	68	62	-4	6	1	14	15
7	0	0	21	21	0	6	0	165	159	-7	1	1	26	26	0	3	1	315	347	-3	6	1	30	32
2	1	0	120	120	1	6	0	38	39	-6	1	1	43	43	1	3	1	267	272	-2	6	1	76	76
3	1	0	139	132	2	6	0	50	51	-5	1	1	67	71	2	3	1	109	103	-1	6	1	102	102
4	1	0	150	145	3	6	0	62	62	-4	1	1	45	47	3	3	1	119	113	0	6	1	101	105
5	1	0	92	90	4	6	0	46	43	-3	1	1	135	137	4	3	1	148	150	1	6	1	175	176
6	1	0	30	32	5	6	0	38	38	-2	1	1	182	174	5	3	1	70	69	2	6	1	48	51
7	1	0	15	14	1	7	0	19	17	-1	1	1	312	336	6	3	1	35	36	3	6	1	98	98
0	2	0	372	370	2	7	0	82	87	0	1	1	251	296	-6	4	1	21	25	4	6	1	29	29
1	2	0	259	296	3	7	0	21	22	1	1	1	120	112	-5	4	1	68	64	5	6	1	38	33
2	2	0	162	166	4	7	0	33	34	2	1	1	107	101	-4	4	1	62	64	-5	7	1	35	37
3	2	0	165	165	5	7	0	36	35	3	1	1	48	49	-3	4	1	67	66	-4	7	1	48	46
4	2	0	70	61	0	8	0	49	51	4	1	1	120	118	-2	4	1	85	91	-3	7	1	26	27
5	2	0	126	121	1	8	0	41	43	5	1	1	89	90	-1	4	1	21	19	-2	7	1	39	39
6	2	0	64	60	3	8	0	39	37	6	1	1	42	42	0	4	1	185	176	-1	7	1	79	77
7	2	0	14	18	1	9	0	31	33	7	1	1	15	18	1	4	1	143	131	0	7	1	58	61
1	3	0	204	202	2	9	0	65	68	-6	2	1	37	40	2	4	1	92	85	2	7	1	105	106
2	3	0	191	186	3	9	0	17	15	-5	2	1	16	18	3	4	1	20	18	3	7	1	39	42
3	3	0	123	120	4	9	0	27	26	-4	2	1	70	72	4	4	1	72	75	4	7	1	41	42
4	3	0	72	70	0	10	0	21	18	-3	2	1	77	73	5	4	1	39	42	5	7	1	25	24
5	3	0	40	39	1	10	0	20	24	-2	2	1	100	92	6	4	1	84	81	-4	8	1	48	46
6	3	0	22	24	2	10	0	27	30	-1	2	1	164	165	-6	5	1	39	38	-3	8	1	91	92
0	4	0	133	146	-7	0	1	21	20	0	2	1	330	375	-5	5	1	41	38	-1	8	1	25	24
1	4	0	87	91	-6	0	1	113	116	1	2	1	290	277	-4	5	1	56	54	0	8	1	17	17
2	4	0	114	114	-4	0	1	11	10	2	2	1	98	96	-3	5	1	79	82	1	8	1	57	58
3	4	0	175	174	-3	0	1	22	21	3	2	1	96	93	-2	5	1	38	36	2	8	1	25	24
4	4	0	70	71	-2	0	1	281	274	4	2	1	68	72	-1	5	1	169	162	3	8	1	67	69
5	4	0	50	48	-1	0	1	172	181	5	2	1	83	83	0	5	1	66	61	4	8	1	14	16
6	4	0	38	40	0	0	1	44	42	6	2	1	28	27	1	5	1	69	69	-3	9	1	77	77

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR N-ACETYL METHIONINE

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	9	1	14	13	-3	3	2	223	216	3	4	2	28	26	-3	8	2	47	48	0	1	3	138	138
-1	9	1	33	32	-2	3	2	243	237	-6	5	2	24	22	-2	8	2	24	24	1	1	3	291	295
0	9	1	27	29	-1	3	2	329	320	-5	5	2	42	44	-1	8	2	51	53	2	1	3	195	183
2	9	1	40	38	0	3	2	342	337	-4	5	2	38	36	0	8	2	38	37	3	1	3	129	143
3	9	1	16	13	1	3	2	235	234	-3	5	2	48	47	1	8	2	59	63	4	1	3	21	22
4	9	1	23	24	2	3	2	216	206	-2	5	2	91	88	2	8	2	37	36	5	1	3	46	48
-2	10	1	46	46	3	3	2	90	80	-1	5	2	114	118	3	8	2	23	23	6	1	3	36	37
0	10	1	25	24	4	3	2	54	53	0	5	2	78	81	4	8	2	14	15	-6	2	3	43	45
-6	0	2	84	84	5	3	2	60	56	1	5	2	94	81	-3	9	2	21	22	-5	2	3	45	43
-5	0	2	101	100	6	3	2	70	68	2	5	2	69	69	-2	9	2	39	40	-4	2	3	73	72
-4	0	2	160	160	-6	3	2	30	45	3	5	2	72	72	0	9	2	32	33	-3	2	3	70	68
-3	0	2	47	45	-5	3	2	38	34	4	5	2	40	40	1	9	2	22	21	-2	2	3	49	47
-2	0	2	59	59	-4	3	2	66	66	5	5	2	25	24	3	9	2	26	26	-1	2	3	235	230
-1	0	2	65	70	-3	3	2	138	137	6	5	2	35	31	-2	10	2	18	18	0	2	3	191	180
0	0	2	174	173	-2	3	2	217	211	-5	6	2	47	42	-1	10	2	33	33	1	2	3	295	290
2	0	2	238	247	-1	3	2	75	66	-4	6	2	52	57	1	10	2	14	13	2	2	3	136	138
3	0	2	146	141	0	3	2	145	140	-3	6	2	91	89	2	10	2	24	24	3	2	3	36	34
4	0	2	159	163	1	3	2	373	414	-2	6	2	64	65	-6	0	3	25	23	4	2	3	78	78
6	0	2	18	18	2	3	2	191	181	-1	6	2	85	86	-4	0	3	31	34	5	2	3	107	110
-7	1	2	24	23	3	3	2	98	96	0	6	2	39	43	-2	0	3	281	282	-6	3	3	31	33
-6	1	2	53	51	4	3	2	21	21	1	6	2	76	77	-1	0	3	91	77	-5	3	3	25	27
-5	1	2	98	102	5	3	2	71	73	2	6	2	48	51	0	0	3	45	42	-5	3	3	86	83
-4	1	2	44	44	6	3	2	36	39	3	6	2	106	106	1	0	3	143	140	-4	3	3	30	29
-3	1	2	219	204	-6	4	2	20	16	4	6	2	25	26	2	0	3	380	399	-3	3	3	51	50
-2	1	2	215	214	-5	4	2	48	51	5	6	2	23	26	3	0	3	49	46	-2	3	3	97	99
-1	1	2	20	21	-4	4	2	90	89	-5	7	2	18	16	4	0	3	28	30	-1	3	3	124	129
0	1	2	254	254	-3	4	2	73	70	-4	7	2	38	37	5	0	3	109	112	0	3	3	317	301
2	1	2	151	153	-2	4	2	106	108	-2	7	2	76	80	6	0	3	16	15	1	3	3	139	141
3	1	2	137	141	-1	4	2	257	248	-1	7	2	32	31	-6	1	3	60	53	2	3	3	82	79
4	1	2	58	56	0	4	2	128	120	0	7	2	101	101	-5	1	3	60	58	3	3	3	49	43
5	1	2	78	78	1	4	2	79	68	1	7	2	15	17	-4	1	3	31	30	4	3	3	63	63
6	1	2	46	42	2	4	2	65	60	2	7	2	66	65	-3	1	3	85	78	5	3	3	64	66
-6	2	2	34	34	3	4	2	10	6	3	7	2	45	45	-2	1	3	72	73	6	3	3	41	40
-4	2	2	105	103	4	4	2	83	83	-4	8	2	24	21	-1	1	3	13	10	-5	4	3	42	40

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR N-ACETYL METHIONINE

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	4	3	19	23	-4	7	3	51	52	3	0	4	90	94	2	3	4	109	109	1	6	4	26	27
-3	4	3	104	96	-3	7	3	38	38	-6	1	4	33	29	3	3	4	17	19	2	6	4	95	95
-2	4	3	32	35	-2	7	3	69	68	-5	1	4	26	22	4	3	4	37	38	3	6	4	14	17
-1	4	3	163	164	-1	7	3	52	49	-4	1	4	67	66	5	3	4	45	46	5	6	4	13	14
0	4	3	182	173	0	7	3	96	101	-3	1	4	172	169	6	3	4	26	28	-3	7	4	30	30
1	4	3	203	191	1	7	3	24	22	-2	1	4	36	32	-6	4	4	17	14	-2	7	4	43	38
2	4	3	96	98	3	7	3	92	95	-1	1	4	195	194	-5	4	4	58	51	0	7	4	44	42
3	4	3	38	41	4	7	3	27	27	0	1	4	87	79	-4	4	4	48	52	1	7	4	75	72
4	4	3	38	41	-4	8	3	28	24	1	1	4	185	184	-3	4	4	20	20	2	7	4	18	18
5	4	3	43	42	-3	8	3	50	46	2	1	4	46	52	-2	4	4	42	45	3	7	4	14	14
6	4	3	39	39	-2	8	3	19	24	3	1	4	66	67	-1	4	4	61	56	-3	8	4	18	16
-5	5	3	24	24	-1	8	3	63	66	4	1	4	96	97	0	4	4	152	148	-2	8	4	21	21
-4	5	3	35	35	0	8	3	55	57	5	1	4	51	56	1	4	4	124	132	-1	8	4	13	12
-3	5	3	66	66	2	8	3	59	59	6	1	4	34	33	2	4	4	50	51	0	8	4	33	33
-2	5	3	106	106	3	8	3	16	17	-6	2	4	41	38	3	4	4	95	98	1	8	4	20	21
-1	5	3	94	92	4	8	3	31	31	-5	2	4	26	27	4	4	4	72	72	2	8	4	14	12
0	5	3	123	118	-2	9	3	32	32	-4	2	4	102	103	5	4	4	33	33	4	8	4	19	20
1	5	3	158	154	-1	9	3	13	16	-3	2	4	103	106	6	4	4	22	25	-1	9	4	30	31
2	5	3	32	30	0	9	3	37	40	-2	2	4	127	125	-4	5	4	32	32	0	9	4	38	39
3	5	3	90	89	1	9	3	49	54	-1	2	4	89	87	-3	5	4	23	20	2	9	4	28	27
4	5	3	70	70	3	9	3	25	25	0	2	4	237	233	-2	5	4	75	69	-1	10	4	15	17
5	5	3	37	37	0	10	3	45	47	1	2	4	114	126	-1	5	4	101	97	0	10	4	32	31
-5	6	3	49	44	1	10	3	13	15	2	2	4	93	99	0	5	4	78	72	1	10	4	19	21
-4	6	3	60	54	-6	0	4	21	22	3	2	4	35	36	1	5	4	92	94	-5	0	5	16	27
-3	6	3	35	37	-5	0	4	86	82	4	2	4	31	31	2	5	4	60	62	-2	0	5	130	133
-2	6	3	102	98	-4	0	4	12	16	5	2	4	20	22	3	5	4	98	100	-1	0	5	63	61
-1	6	3	81	83	-3	0	4	22	22	-6	3	4	30	29	4	5	4	45	48	1	0	5	135	129
0	6	3	18	20	-2	0	4	273	274	-5	3	4	47	51	5	5	4	26	29	2	0	5	22	20
1	6	3	53	59	-1	0	4	16	18	-4	3	4	112	107	-5	6	4	46	42	3	0	5	110	113
2	6	3	127	127	0	0	4	114	122	-3	3	4	78	74	-4	6	4	23	20	4	0	5	16	14
3	6	3	59	62	1	0	4	79	72	-2	3	4	172	171	-3	6	4	43	42	5	0	5	37	40
4	6	3	60	63	2	0	4	14	18	-1	3	4	168	168	-2	6	4	43	44	6	0	5	20	20
5	6	3	59	60	3	0	4	155	155	0	3	4	106	101	-1	6	4	73	69	-6	1	5	24	24
-5	7	3	26	27	4	0	4	36	36	1	3	4	120	108	0	6	4	42	38	-5	1	5	24	29

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR N-ACETYL METHIONINE

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	1	5	15	17	-4	4	5	59	49	3	7	5	34	33	-3	2	6	48	45	1	5	6	76	78
-3	1	5	90	95	-3	4	5	33	32	4	7	5	41	40	-2	2	6	32	31	2	5	6	30	31
-2	1	5	63	63	-2	4	5	123	119	-3	8	5	15	11	-1	2	6	46	50	3	5	6	29	31
-1	1	5	88	85	-1	4	5	89	90	-2	8	5	27	28	0	2	6	76	72	4	5	6	30	29
0	1	5	134	135	0	4	5	109	107	-1	8	5	27	29	1	2	6	120	116	-4	6	6	26	21
1	1	5	102	100	1	4	5	192	191	0	8	5	39	40	2	2	6	106	106	-3	6	6	41	40
2	1	5	205	208	2	4	5	86	89	1	8	5	51	54	3	2	6	69	67	-2	6	6	16	16
3	1	5	42	42	3	4	5	38	36	2	8	5	34	36	4	2	6	68	69	-1	6	6	15	14
4	1	5	62	62	4	4	5	76	81	3	8	5	38	39	5	2	6	15	13	0	6	6	91	88
5	1	5	25	26	-4	5	5	16	13	-2	9	5	18	20	-5	3	6	31	27	1	6	6	44	46
6	1	5	30	30	-3	5	5	87	85	-1	9	5	22	22	-4	3	6	81	78	2	6	6	88	88
-5	2	5	51	48	-2	5	5	95	90	0	9	5	30	29	-3	3	6	61	55	4	6	6	18	17
-4	2	5	60	55	-1	5	5	120	121	2	9	5	30	30	-2	3	6	44	43	-2	7	6	20	19
-3	2	5	42	38	0	5	5	111	110	-6	0	6	24	26	-1	3	6	35	34	-1	7	6	29	28
-2	2	5	95	100	1	5	5	96	101	-3	0	6	67	77	0	3	6	134	134	0	7	6	32	34
-1	2	5	72	71	2	5	5	91	86	-2	0	6	13	12	1	3	6	77	80	1	7	6	29	28
0	2	5	87	88	3	5	5	42	42	-1	0	6	203	207	2	3	6	69	67	2	7	6	28	30
1	2	5	145	149	4	5	5	14	18	0	0	6	99	96	3	3	6	74	80	3	7	6	13	12
2	2	5	102	103	5	5	5	44	47	1	0	6	82	84	4	3	6	18	22	-3	8	6	14	14
3	2	5	92	92	-4	6	5	48	47	2	0	6	44	41	5	3	6	13	10	-2	8	6	19	17
4	2	5	63	61	-3	6	5	27	28	3	0	6	20	19	-5	4	6	23	22	-1	8	6	28	30
6	2	5	32	33	-2	6	5	81	78	4	0	6	28	28	-4	4	6	52	49	0	8	6	20	19
-6	3	5	28	23	-1	6	5	86	84	-6	1	6	16	13	-3	4	6	26	23	1	8	6	20	20
-4	3	5	79	78	0	6	5	102	109	-5	1	6	32	35	-2	4	6	39	36	0	9	6	36	36
-3	3	5	45	45	1	6	5	92	92	-4	1	6	42	36	-1	4	6	56	53	1	9	6	27	26
-2	3	5	36	31	2	6	5	15	16	-2	1	6	44	44	0	4	6	44	45	-3	0	7	67	98
-1	3	5	185	182	3	6	5	43	44	-1	1	6	116	120	1	4	6	20	20	0	0	7	138	132
0	3	5	61	63	4	6	5	42	43	0	1	6	39	37	2	4	6	82	86	1	0	7	48	42
1	3	5	39	39	-4	7	5	20	20	1	1	6	116	109	3	4	6	27	28	2	0	7	18	19
2	3	5	76	80	-3	7	5	14	10	2	1	6	35	36	4	4	6	40	41	3	0	7	16	12
3	3	5	147	148	-2	7	5	21	20	3	1	6	92	89	-5	5	6	43	35	-2	1	7	28	27
4	3	5	36	33	-1	7	5	46	43	4	1	6	36	38	-4	5	6	35	35	-1	1	7	64	64
5	3	5	32	33	0	7	5	35	36	5	1	6	34	35	-3	5	6	43	39	0	1	7	44	43
-5	4	5	53	51	2	7	5	30	49	6	1	6	49	47	0	5	6	34	30	1	1	7	28	25

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR N-ACETYL METHIONINE

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	1	7	69	71	-4	5	7	30	29	4	0	8	17	16	1	4	8	41	41	1	3	9	35	33
2	1	7	87	86	-3	5	7	41	36	-4	1	8	15	30	2	4	8	26	25	2	3	9	36	33
3	1	7	27	21	-2	5	7	35	37	-3	1	8	12	10	3	4	8	49	49	3	3	9	13	15
4	1	7	47	47	-1	5	7	60	57	-2	1	8	38	43	-3	5	8	21	22	-3	4	9	15	19
-5	2	7	25	25	0	5	7	13	10	-1	1	8	93	93	-2	5	8	17	19	-2	4	9	34	33
-4	2	7	19	22	1	5	7	86	87	0	1	8	12	8	-1	5	8	27	27	-1	4	9	35	33
-3	2	7	45	54	2	5	7	25	23	1	1	8	65	61	1	5	8	33	33	0	4	9	13	15
-2	2	7	81	85	3	5	7	16	19	2	1	8	90	88	2	5	8	36	37	1	4	9	21	19
-1	2	7	106	104	4	5	7	48	47	3	1	8	20	22	-2	6	8	32	29	-2	5	9	16	16
0	2	7	67	63	-3	6	7	37	34	4	1	8	64	58	-1	6	8	14	11	-1	5	9	34	34
1	2	7	34	31	-2	6	7	29	28	-4	2	8	28	27	0	6	8	31	27	0	5	9	44	42
2	2	7	74	79	-1	6	7	27	28	-3	2	8	54	56	1	6	8	29	29	1	5	9	20	21
3	2	7	60	61	0	6	7	93	91	-2	2	8	15	19	2	6	8	32	33	2	5	9	28	28
-5	3	7	17	16	1	6	7	42	42	-1	2	8	27	30	-1	7	8	38	34	-1	6	9	17	17
-4	3	7	12	6	2	6	7	39	37	0	2	8	87	85	1	7	8	23	18	-2	0	10	34	34
-3	3	7	32	36	3	6	7	23	25	1	2	8	83	82	-2	0	9	12	13	-1	0	10	92	91
-2	3	7	117	116	-3	7	7	25	23	2	2	8	41	44	-1	0	9	70	67	0	0	10	23	26
-1	3	7	101	99	-2	7	7	31	31	3	2	8	51	49	0	0	9	16	17	1	0	10	15	15
0	3	7	76	77	-1	7	7	36	36	4	2	8	28	31	1	0	9	90	88	2	0	10	61	56
1	3	7	103	101	0	7	7	24	26	-4	3	8	24	25	-3	1	9	17	23	-2	1	10	35	34
2	3	7	42	45	1	7	7	45	46	-3	3	8	25	21	-2	1	9	36	38	-1	1	10	33	32
3	3	7	35	38	2	7	7	37	40	-2	3	8	19	21	0	1	9	93	89	0	1	10	75	72
4	3	7	44	43	-1	8	7	21	19	-1	3	8	48	50	1	1	9	38	34	1	1	10	28	24
-4	4	7	33	27	0	8	7	45	47	0	3	8	19	13	2	1	9	26	26	-1	2	10	39	39
-3	4	7	28	33	-4	0	8	18	41	1	3	8	96	97	-3	2	9	17	20	0	2	10	33	30
-2	4	7	28	29	-3	0	8	12	15	2	3	8	35	34	-2	2	9	21	17	1	2	10	47	45
-1	4	7	91	93	-2	0	8	91	90	3	3	8	26	24	-1	2	9	68	61	2	2	10	39	37
0	4	7	64	65	-1	0	8	19	18	4	3	8	46	44	1	2	9	20	21	-1	3	10	22	23
1	4	7	40	39	0	0	8	59	62	-3	4	8	22	23	2	2	9	22	20	0	3	10	24	23
2	4	7	74	72	1	0	8	86	85	-2	4	8	40	38	-2	3	9	44	42	1	3	10	32	31
3	4	7	68	62	2	0	8	50	46	-1	4	8	14	11	-1	3	9	29	32	0	4	10	22	21
4	4	7	14	16	3	0	8	91	92	0	4	8	51	50	0	3	9	14	12	1	4	10	37	34

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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	0	0	501	-490	2	8	0	405	-411	4	15	0	168	-169	0	4	1	1253	-1288	5	7	1	90	93
6	0	0	565	564	4	8	0	78	78	6	15	0	122	134	1	4	1	182	174	6	7	1	297	298
8	0	0	207	217	6	8	0	201	222	0	16	0	164	154	3	4	1	76	-76	8	7	1	104	-109
10	0	0	130	-123	8	8	0	200	-214	2	16	0	83	-79	4	4	1	33	25	9	7	1	43	-39
4	1	0	77	-83	10	8	0	66	-61	4	16	0	85	-85	5	4	1	80	76	10	7	1	123	117
6	1	0	121	111	2	9	0	294	-309	2	17	0	23	30	6	4	1	70	-75	1	8	1	245	236
8	1	0	419	-418	4	9	0	155	142	0	18	0	131	136	7	4	1	169	-157	2	8	1	466	454
10	1	0	134	138	6	9	0	304	320	2	18	0	144	-146	8	4	1	129	-132	4	8	1	153	-138
4	2	0	807	786	8	9	0	54	-62	4	18	0	36	39	1	5	1	194	214	5	8	1	216	-226
6	2	0	180	-172	10	9	0	48	-51	1	1	1	69	-62	2	5	1	198	-184	6	8	1	184	181
8	2	0	95	-94	0	10	0	125	124	2	1	1	40	-42	3	5	1	624	-620	7	8	1	130	140
10	2	0	91	102	2	10	0	120	-115	3	1	1	178	173	4	5	1	197	186	9	8	1	103	97
2	3	0	886	885	4	10	0	376	378	4	1	1	364	-352	5	5	1	185	178	1	9	1	420	419
4	3	0	343	-338	6	10	0	172	-166	5	1	1	289	278	6	5	1	37	51	2	9	1	125	130
6	3	0	738	-734	8	10	0	63	69	8	1	1	82	-89	7	5	1	139	126	3	9	1	553	-550
8	3	0	323	315	10	10	0	62	74	9	1	1	249	-237	8	5	1	242	235	4	9	1	169	167
10	3	0	234	-236	2	11	0	59	-60	11	1	1	188	192	9	5	1	163	-160	6	9	1	200	-205
2	4	0	1218	-1227	4	11	0	52	-49	1	2	1	189	-195	10	5	1	149	-148	7	9	1	174	167
6	4	0	59	56	6	11	0	331	324	3	2	1	551	-519	0	6	1	501	492	8	9	1	144	138
10	4	0	129	-129	8	11	0	140	-145	4	2	1	260	-253	1	6	1	616	-631	10	9	1	74	-69
2	5	0	935	-942	0	12	0	462	459	5	2	1	326	291	2	6	1	455	-452	0	10	1	313	307
4	5	0	300	279	2	12	0	43	34	6	2	1	155	-134	3	6	1	276	-259	3	10	1	199	207
8	5	0	242	-237	4	12	0	108	-104	7	2	1	152	160	4	6	1	245	230	4	10	1	191	182
10	5	0	88	84	6	12	0	117	116	9	2	1	201	-203	5	6	1	405	401	5	10	1	34	-35
0	6	0	634	-648	8	12	0	64	70	10	2	1	66	57	6	6	1	126	-121	6	10	1	89	95
2	6	0	362	-364	2	13	0	127	-125	2	3	1	782	771	7	6	1	63	-53	7	10	1	64	-53
4	6	0	35	40	4	13	0	196	192	3	3	1	140	142	8	6	1	87	90	8	10	1	67	-71
6	6	0	298	-305	6	13	0	81	-78	4	3	1	409	-392	9	6	1	127	-135	1	11	1	196	-197
8	6	0	40	-32	8	13	0	71	-76	5	3	1	199	-183	10	6	1	56	54	2	11	1	21	-19
10	6	0	136	132	0	14	0	543	547	6	3	1	79	75	11	6	1	93	101	3	11	1	329	331
2	7	0	624	611	2	14	0	377	-390	8	3	1	172	-164	1	7	1	198	187	4	11	1	223	-234
4	7	0	295	-289	4	14	0	224	232	9	3	1	25	33	2	7	1	104	-98	6	11	1	176	180
8	7	0	111	121	6	14	0	34	25	10	3	1	112	117	3	7	1	408	-406	7	11	1	69	-72
10	7	0	94	95	8	14	0	75	-79	11	3	1	41	40	4	7	1	52	50	8	11	1	71	-71

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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
0	12	1	95	-97	2	18	1	31	42	7	3	2	210	207	5	7	2	113	109	8	11	2	46	-46
1	12	1	71	-76	3	18	1	113	-111	9	3	2	114	-120	7	7	2	42	52	0	12	2	236	235
2	12	1	192	198	2	19	1	66	55	10	3	2	130	122	9	7	2	135	-131	1	12	2	50	-45
3	12	1	166	177	0	0	2	258	264	0	4	2	301	298	0	8	2	381	379	2	12	2	368	-367
4	12	1	187	-175	4	0	2	576	-557	1	4	2	344	331	2	8	2	188	-181	3	12	2	70	72
5	12	1	131	-146	8	0	2	143	-128	2	4	2	49	38	3	8	2	159	-155	4	12	2	71	68
8	12	1	57	63	6	0	2	154	146	3	4	2	87	-81	4	8	2	322	315	5	12	2	76	-73
9	12	1	128	124	7	0	2	237	-235	4	4	2	208	209	5	8	2	89	90	6	12	2	72	-72
1	13	1	105	99	9	0	2	68	60	5	4	2	132	-133	6	8	2	57	-61	9	12	2	111	-110
3	13	1	132	-133	10	0	2	62	-66	7	4	2	211	212	7	8	2	116	109	1	13	2	200	-209
4	13	1	65	-59	1	1	2	273	-281	8	4	2	40	31	8	8	2	59	-61	2	13	2	103	-96
5	13	1	55	64	2	1	2	179	177	9	4	2	32	-32	9	8	2	38	-37	3	13	2	172	173
6	13	1	79	-71	3	1	2	246	-209	11	4	2	73	-67	1	9	2	242	235	5	13	2	90	85
7	13	1	74	76	4	1	2	126	117	1	5	2	55	46	2	9	2	145	-143	6	13	2	104	119
8	13	1	51	-51	5	1	2	299	273	2	5	2	153	150	3	9	2	126	-123	7	13	2	163	-166
0	14	1	68	-74	6	1	2	159	-144	3	5	2	166	-158	4	9	2	81	-83	8	13	2	95	-93
1	14	1	122	-125	8	1	2	209	208	4	5	2	219	198	5	9	2	150	159	0	14	2	125	129
2	14	1	30	-28	9	1	2	194	-200	5	5	2	315	-307	6	9	2	52	38	1	14	2	205	-209
4	14	1	69	-79	10	1	2	76	-71	6	5	2	126	-116	7	9	2	97	-96	2	14	2	98	-98
5	14	1	99	96	11	1	2	91	94	7	5	2	148	146	8	9	2	131	-129	3	14	2	38	-43
7	14	1	63	-64	1	2	2	286	-284	8	5	2	91	83	9	9	2	90	81	4	14	2	93	-92
1	15	1	71	-79	2	2	2	1060	-1063	9	5	2	104	100	1	10	2	418	427	5	14	2	47	46
2	15	1	74	82	3	2	2	183	-150	10	5	2	62	55	2	10	2	141	-151	6	14	2	56	63
3	15	1	137	142	5	2	2	45	-44	0	6	2	667	667	3	10	2	75	-78	1	15	2	108	104
4	15	1	80	-71	6	2	2	69	-56	1	6	2	808	798	4	10	2	28	33	3	15	2	224	-221
5	15	1	50	-52	7	2	2	249	249	2	6	2	122	-118	5	10	2	130	-127	4	15	2	63	55
1	16	1	57	60	8	2	2	50	58	3	6	2	200	202	6	10	2	60	-62	7	15	2	63	58
2	16	1	126	-126	9	2	2	192	-191	4	6	2	483	478	7	10	2	72	65	0	16	2	242	248
3	16	1	62	61	1	3	2	42	-29	5	6	2	128	-118	1	11	2	88	91	1	16	2	65	-57
5	16	1	106	-114	2	3	2	594	600	7	6	2	67	-67	2	11	2	230	-232	2	16	2	138	-135
6	16	1	37	33	3	3	2	240	-255	8	6	2	53	-61	3	11	2	25	-25	4	16	2	65	66
1	17	1	33	-41	4	3	2	119	-113	9	6	2	50	46	4	11	2	81	74	1	17	2	24	-7
4	17	1	60	-66	5	3	2	318	-318	1	7	2	149	-173	6	11	2	42	-32	2	17	2	82	76
1	18	1	119	-119	6	3	2	103	107	3	7	2	230	233	7	11	2	57	-52	4	17	2	30	-29

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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	17	2	51	-55	1	4	3	893	-905	9	7	3	140	-144	7	11	3	90	88	3	17	3	96	98
1	18	2	35	35	2	4	3	91	97	0	8	3	142	149	8	11	3	79	71	4	17	3	82	-81
3	18	2	84	66	3	4	3	111	-94	1	8	3	143	-140	9	11	3	41	-45	5	17	3	48	-48
4	18	2	42	41	4	4	3	646	607	2	8	3	228	-238	0	12	3	58	58	1	18	3	91	-77
1	1	3	331	-343	5	4	3	354	343	4	8	3	202	-210	1	12	3	93	93	3	18	3	149	139
2	1	3	534	529	6	4	3	96	-89	5	8	3	44	49	2	12	3	75	83	1	19	3	40	35
3	1	3	177	-170	7	4	3	226	230	6	8	3	82	70	3	12	3	39	-63	2	19	3	73	71
4	1	3	303	-289	8	4	3	104	-110	7	8	3	87	-79	4	12	3	193	-189	0	0	4	1291	-1300
5	1	3	64	-60	9	4	3	147	-156	8	8	3	51	-33	5	12	3	62	61	1	0	4	350	-355
6	1	3	233	226	11	4	3	26	27	9	8	3	142	-147	7	12	3	77	-74	2	0	4	531	537
7	1	3	96	-109	1	5	3	326	326	1	9	3	179	-181	8	12	3	113	126	3	0	4	30	-18
9	1	3	28	30	2	5	3	106	-100	2	9	3	79	76	1	13	3	175	-180	4	0	4	707	699
10	1	3	129	123	3	5	3	168	154	3	9	3	276	286	2	13	3	170	166	5	0	4	252	253
11	1	3	53	41	4	5	3	289	-286	4	9	3	123	-124	3	13	3	174	176	6	0	4	256	-259
0	2	3	743	750	5	5	3	129	-123	5	9	3	100	-104	4	13	3	55	-58	7	0	4	232	226
2	2	3	114	113	6	5	3	233	229	6	9	3	57	61	5	13	3	91	-94	9	0	4	170	-176
3	2	3	275	260	7	5	3	118	116	8	9	3	67	-72	6	13	3	67	70	10	0	4	43	40
4	2	3	84	81	8	5	3	92	-91	9	9	3	110	-107	7	13	3	44	-40	11	0	4	125	132
6	2	3	99	99	9	5	3	29	21	0	10	3	131	-134	2	14	3	75	78	1	1	4	246	250
7	2	3	64	-60	10	5	3	74	-75	1	10	3	48	42	3	14	3	149	146	2	1	4	224	237
8	2	3	64	-55	0	6	3	257	-243	2	10	3	30	-34	5	14	3	134	-138	3	1	4	84	70
9	2	3	99	93	1	6	3	139	151	3	10	3	64	-69	7	14	3	33	33	4	1	4	308	-299
10	2	3	64	68	2	6	3	157	147	4	10	3	187	-182	2	15	3	169	173	6	1	4	68	-78
1	3	3	92	-89	3	6	3	164	161	5	10	3	157	-153	3	15	3	84	-77	7	1	4	32	44
2	3	3	802	801	5	6	3	133	-136	6	10	3	141	138	4	15	3	86	-85	8	1	4	208	218
3	3	3	103	-108	6	6	3	64	-75	7	10	3	23	7	5	15	3	144	144	10	1	4	48	-39
4	3	3	395	-388	9	6	3	130	122	8	10	3	67	-67	7	15	3	32	-42	0	2	4	1335	1377
5	3	3	234	228	10	6	3	42	20	9	10	3	82	97	0	16	3	77	72	1	2	4	196	199
6	3	3	27	23	1	7	3	425	417	1	11	3	162	169	1	16	3	95	-104	2	2	4	234	-244
7	3	3	60	61	2	7	3	368	-352	2	11	3	123	126	3	16	3	87	-88	3	2	4	40	-43
8	3	3	31	8	3	7	3	217	-207	3	11	3	368	-374	5	16	3	197	187	4	2	4	542	-524
9	3	3	210	-207	4	7	3	32	28	4	11	3	198	-206	6	16	3	116	100	6	2	4	290	271
11	3	3	97	99	7	7	3	279	278	5	11	3	33	-36	1	17	3	32	-6	7	2	4	145	137
0	4	3	120	-110	8	7	3	66	-79	6	11	3	148	166	2	17	3	24	20	8	2	4	147	152

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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
10	2	4	165	-173	3	6	4	55	56	6	10	4	91	96	1	17	4	96	87	3	4	5	207	-222
11	2	4	70	-56	4	6	4	113	109	7	10	4	152	152	3	17	4	129	-115	4	4	5	31	-43
1	3	4	686	683	5	6	4	349	-356	9	10	4	51	-47	1	18	4	81	68	5	4	5	37	40
2	3	4	371	-365	6	6	4	49	36	1	11	4	142	-143	2	18	4	44	-42	6	4	5	31	-61
3	3	4	295	-293	8	6	4	51	-53	2	11	4	108	-112	3	18	4	68	-61	9	4	5	46	-50
4	3	4	79	69	10	6	4	106	-110	4	11	4	164	158	1	1	5	220	217	10	4	5	66	60
5	3	4	148	145	1	7	4	72	77	5	11	4	84	86	2	1	5	557	551	1	5	5	208	-202
6	3	4	116	120	2	7	4	462	-474	7	11	4	106	-110	3	1	5	296	295	2	5	5	206	208
7	3	4	188	191	3	7	4	277	-278	9	11	4	51	50	4	1	5	291	-283	4	5	5	200	-203
8	3	4	341	-357	4	7	4	102	102	1	12	4	48	44	5	1	5	44	-47	5	5	5	271	-284
9	3	4	45	-47	6	7	4	246	257	3	12	4	45	-46	6	1	5	97	-100	6	5	5	260	269
10	3	4	131	143	8	7	4	116	-119	4	12	4	164	169	7	1	5	59	65	7	5	5	72	72
0	4	4	280	-283	9	7	4	36	32	5	12	4	79	80	9	1	5	46	48	8	5	5	207	-216
1	4	4	316	304	1	8	4	264	258	7	12	4	61	60	10	1	5	44	49	9	5	5	66	-64
2	4	4	80	-73	2	8	4	125	-120	8	12	4	33	-37	0	2	5	781	-791	10	5	5	104	119
3	4	4	52	-43	4	8	4	139	163	2	13	4	60	-63	1	2	5	251	-256	0	6	5	911	-907
4	4	4	223	223	5	8	4	165	-166	3	13	4	77	74	2	2	5	126	123	1	6	5	130	134
5	4	4	197	-198	6	8	4	252	-264	4	13	4	54	-53	3	2	5	135	118	2	6	5	249	253
6	4	4	244	-252	8	8	4	103	116	5	13	4	29	26	4	2	5	198	203	3	6	5	148	143
7	4	4	175	172	10	8	4	109	110	6	13	4	68	71	5	2	5	172	-163	4	6	5	44	45
8	4	4	25	-26	1	9	4	200	-198	8	13	4	78	65	7	2	5	141	-139	5	6	5	309	-293
9	4	4	164	-171	2	9	4	116	109	0	14	4	56	46	8	2	5	134	-137	6	6	5	32	27
10	4	4	111	108	3	9	4	47	-50	1	14	4	123	-127	9	2	5	33	30	7	6	5	133	131
1	5	4	127	135	4	9	4	102	99	2	14	4	86	91	2	3	5	36	43	8	6	5	71	-75
2	5	4	784	777	6	9	4	127	-132	3	14	4	52	50	3	3	5	41	-39	1	7	5	214	-220
3	5	4	162	158	7	9	4	44	-46	4	14	4	96	-100	4	3	5	237	-248	2	7	5	62	63
4	5	4	379	-362	8	9	4	78	87	5	14	4	39	46	5	3	5	96	78	3	7	5	29	42
5	5	4	89	-92	9	9	4	76	-75	6	14	4	57	-54	6	3	5	116	114	4	7	5	62	-59
6	5	4	204	-209	0	10	4	317	313	7	14	4	78	76	7	3	5	35	38	5	7	5	238	-249
7	5	4	86	87	1	10	4	82	86	1	15	4	63	68	8	3	5	98	97	6	7	5	125	120
8	5	4	109	116	2	10	4	165	-164	4	15	4	46	47	9	3	5	144	-147	7	7	5	134	130
0	6	4	232	230	3	10	4	144	139	9	15	4	38	48	10	3	5	85	-81	10	7	5	27	-22
1	6	4	186	183	4	10	4	55	52	6	15	4	83	-76	1	4	5	277	-265	0	8	5	220	223
2	6	4	482	-484	5	10	4	230	-226	1	16	4	31	-45	2	4	5	39	-37	1	8	5	106	-103

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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	8	5	166	-168	6	12	5	53	56	2	1	6	21	-38	5	5	6	124	124	0	10	6	99	95
4	8	5	66	58	2	13	5	92	93	3	1	6	210	-217	6	5	6	168	170	1	10	6	37	-40
5	8	5	232	230	3	13	5	147	146	4	1	6	23	-18	8	5	6	56	-59	2	10	6	102	-98
6	8	5	107	100	4	13	5	121	-128	5	1	6	227	-225	9	5	6	41	-34	4	10	6	58	66
7	8	5	123	-120	5	13	5	50	52	6	1	6	134	162	0	6	6	108	-113	8	10	6	35	15
0	9	5	55	-49	6	13	5	38	39	7	1	6	67	63	1	6	6	36	-39	1	11	6	90	-97
9	8	5	40	47	7	13	5	107	-109	8	1	6	213	-225	2	6	6	109	115	2	11	6	34	36
1	9	5	163	-172	1	14	5	140	151	9	1	6	83	88	3	6	6	116	115	3	11	6	210	212
2	9	5	132	-135	4	14	5	48	-47	10	1	6	136	138	4	6	6	45	-44	4	11	6	92	-88
3	9	5	234	237	5	14	5	79	-71	0	2	6	389	-387	5	6	6	107	-109	6	11	6	64	56
4	9	5	187	-204	6	14	5	42	27	2	2	6	333	319	6	6	6	86	-101	8	11	6	32	-12
5	9	5	44	-40	7	14	5	26	24	3	2	6	60	61	7	6	6	266	261	0	12	6	113	-122
6	9	5	376	390	2	15	5	51	57	5	2	6	199	-203	8	6	6	102	109	1	12	6	102	-106
8	9	5	154	-167	4	15	5	54	-53	7	2	6	54	54	9	6	6	26	-12	2	12	6	100	111
9	9	5	44	-39	5	15	5	74	-73	9	2	6	124	-119	1	7	6	234	236	4	12	6	61	-55
0	10	5	207	207	1	16	5	252	-252	10	2	6	47	-51	2	7	6	49	50	5	12	6	101	-101
1	10	5	65	65	3	16	5	53	59	1	3	6	84	-80	3	7	6	77	-73	7	12	6	72	75
2	10	5	68	67	4	16	5	41	-49	2	3	6	95	-91	5	7	6	60	-57	1	13	6	65	62
4	10	5	215	-216	2	17	5	122	121	3	3	6	388	-394	6	7	6	38	-43	2	13	6	57	53
5	10	5	43	42	4	17	5	38	-31	4	3	6	154	154	7	7	6	72	77	3	13	6	132	-142
6	10	5	97	91	0	18	5	104	-90	6	3	6	114	-124	9	7	6	88	84	5	13	6	30	-29
7	10	5	120	112	1	18	5	77	74	7	3	6	48	-48	0	8	6	65	-61	6	13	6	89	-83
8	10	5	73	86	2	18	5	42	20	8	3	6	84	92	1	8	6	183	175	1	14	6	64	62
1	11	5	235	233	0	0	6	253	259	9	3	6	75	75	3	8	6	407	415	2	14	6	53	-48
2	11	5	204	198	1	0	6	82	-84	10	3	6	48	-51	5	8	6	325	-331	3	14	6	77	-73
3	11	5	109	-109	2	0	6	119	-131	0	4	6	338	329	6	8	6	37	-37	4	14	6	136	131
4	11	5	82	-77	3	0	6	433	-444	2	4	6	221	-210	7	8	6	96	89	6	14	6	113	-110
5	11	5	30	-34	4	0	6	125	131	3	4	6	145	137	8	8	6	59	61	1	15	6	92	-100
6	11	5	73	-67	5	0	6	91	83	4	4	6	60	57	9	8	6	89	84	2	15	6	27	-21
7	11	5	223	221	6	0	6	208	-210	5	4	6	81	-81	2	9	6	49	-48	3	15	6	27	16
0	12	5	221	225	7	0	6	147	154	8	4	6	25	2	3	9	6	193	199	5	15	6	34	26
2	12	5	129	-130	8	0	6	138	150	10	4	6	41	-30	4	9	6	78	81	0	16	6	119	-118
3	12	5	42	31	9	0	6	188	-198	3	5	6	281	-283	5	9	6	102	97	2	16	6	78	78
5	12	5	29	35	1	1	6	87	88	4	5	6	132	-133	6	9	6	81	-81	3	16	6	40	-40

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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	17	6	66	-68	2	5	7	43	45	7	9	7	36	45	0	0	8	103	100	3	4	8	164	160
3	17	6	123	-107	3	6	7	126	124	0	10	7	366	376	2	0	8	35	35	4	4	8	180	-178
1	1	7	203	-207	4	6	7	24	33	1	10	7	144	-138	3	0	8	211	225	5	4	8	152	-150
2	1	7	293	283	5	6	7	114	111	2	10	7	160	-164	4	0	8	360	-373	6	4	8	142	138
4	1	7	137	-129	6	6	7	60	-61	5	10	7	81	85	5	0	8	168	-179	1	5	8	264	266
5	1	7	172	-183	8	6	7	44	40	7	10	7	121	-130	6	0	8	92	84	2	5	8	463	-474
6	1	7	122	-125	0	6	7	169	172	8	10	7	53	67	7	0	8	197	186	3	5	8	284	-281
7	1	7	110	115	2	6	7	216	-208	1	11	7	217	-213	8	0	8	86	86	4	5	8	268	274
8	1	7	100	97	3	6	7	118	-128	3	11	7	158	162	1	1	8	109	109	5	5	8	88	86
9	1	7	67	-76	4	6	7	155	151	4	11	7	44	-40	2	1	8	240	-252	6	5	8	87	89
0	2	7	364	-364	5	6	7	161	159	6	11	7	85	85	3	1	8	62	-64	8	5	8	29	-36
3	2	7	85	-88	8	6	7	79	-86	7	11	7	71	-65	4	1	8	321	333	0	6	8	34	-43
4	2	7	177	184	9	6	7	75	-65	0	12	7	33	30	5	1	8	86	87	1	6	8	29	-31
5	2	7	172	174	1	7	7	120	-126	4	12	7	45	-47	6	1	8	110	-109	2	6	8	186	190
6	2	7	130	-125	2	7	7	122	126	5	12	7	124	-117	7	1	8	27	19	3	6	8	81	86
7	2	7	61	67	3	7	7	440	453	6	12	7	79	73	0	2	8	428	-426	5	6	8	131	-144
9	2	7	34	16	4	7	7	280	-283	7	12	7	98	95	1	2	8	169	-165	6	6	8	97	-104
3	3	7	133	134	5	7	7	72	-72	1	13	7	28	22	2	2	8	65	-74	8	6	8	111	122
4	3	7	108	-107	6	7	7	362	354	2	13	7	70	70	3	2	8	30	22	1	7	8	88	-82
5	3	7	238	-248	7	7	7	76	-78	3	13	7	110	-115	4	2	8	477	486	2	7	8	282	278
6	3	7	150	149	8	7	7	184	-188	4	13	7	54	-58	5	2	8	89	86	4	7	8	89	-94
7	3	7	102	106	9	7	7	34	43	5	13	7	93	-89	6	2	8	344	-342	6	7	8	145	-141
8	3	7	118	-125	0	8	7	207	-208	6	13	7	63	-55	7	2	8	94	86	7	7	8	32	-31
9	3	7	42	38	1	8	7	49	-45	0	14	7	125	120	8	2	8	46	-28	8	7	8	80	84
0	4	7	346	-345	2	8	7	205	211	1	14	7	101	-93	9	2	8	90	-91	0	8	8	53	50
1	4	7	357	371	3	8	7	26	18	2	14	7	58	-49	1	3	8	74	82	2	8	8	31	41
2	4	7	218	215	4	8	7	74	-76	4	14	7	83	-87	2	3	8	171	171	3	8	8	101	99
3	4	7	266	263	5	8	7	151	-150	5	14	7	70	71	3	3	8	189	193	4	8	8	71	-80
4	4	7	181	-183	7	8	7	61	54	2	15	7	60	48	4	3	8	130	-128	5	8	8	229	-230
5	4	7	192	-190	1	9	7	109	114	3	15	7	102	92	5	3	8	43	-49	6	8	8	103	99
6	4	7	62	63	2	9	7	67	-71	0	16	7	108	-107	6	3	8	45	52	7	8	8	74	78
8	4	7	41	-33	3	9	7	126	-129	1	16	7	135	135	8	3	8	106	103	1	9	8	86	-92
9	4	7	119	120	5	9	7	70	-62	2	16	7	59	50	1	4	8	210	-207	4	9	8	84	-86
1	5	7	49	39	6	9	7	46	41	3	16	7	80	66	2	4	8	156	154	5	9	8	64	-66

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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
6	9	8	105	105	5	1	9	72	-79	4	6	9	127	-128	0	14	9	52	59	2	6	10	76	-66
7	9	8	25	-17	6	1	9	110	109	5	6	9	53	56	1	14	9	87	-89	3	6	10	168	169
0	10	8	252	-249	7	1	9	127	130	6	6	9	99	96	2	14	9	26	25	4	6	10	108	-105
1	10	8	135	-145	0	2	9	114	116	7	6	9	78	-76	0	0	10	101	-87	5	6	10	77	-80
2	10	8	161	165	1	2	9	165	159	1	7	9	101	-108	1	0	10	153	-142	6	6	10	50	47
3	10	8	92	99	2	2	9	126	126	2	7	9	105	-110	2	0	10	60	45	7	6	10	31	-25
4	10	8	71	-65	3	2	9	66	-64	3	7	9	74	77	3	0	10	210	205	9	7	10	93	90
6	10	8	65	-69	4	2	9	40	-52	6	7	9	29	-42	4	0	10	255	257	6	7	10	60	57
1	11	8	31	-27	5	2	9	81	-81	7	7	9	158	-159	5	0	10	190	-188	1	8	10	83	-81
2	11	8	116	114	8	2	9	72	74	1	8	9	120	122	1	1	10	132	-130	3	8	10	46	-53
4	11	8	91	-84	1	3	9	37	-30	2	8	9	76	-78	2	1	10	48	52	4	8	10	118	-118
5	11	8	44	26	2	3	9	68	60	6	8	9	113	-112	3	1	10	117	121	5	8	10	96	90
6	11	8	56	-66	3	3	9	107	108	7	8	9	112	103	4	1	10	123	-125	1	9	10	48	52
0	12	8	94	92	4	3	9	100	108	1	9	9	35	-28	0	2	10	34	-32	2	9	10	140	142
1	12	8	39	-31	5	3	9	134	-137	2	9	9	98	91	1	2	10	260	-261	4	9	10	77	-68
2	12	8	110	-108	6	3	9	109	-114	3	9	9	68	61	2	2	10	111	121	1	10	10	56	-55
3	12	8	89	89	0	4	9	43	50	4	9	9	40	-42	3	2	10	103	107	2	10	10	128	120
4	12	8	44	38	1	4	9	228	243	5	9	9	46	-50	5	2	10	63	-71	3	10	10	110	-112
6	12	8	105	-102	2	4	9	81	-85	0	10	9	180	-181	7	2	10	30	22	1	11	10	64	68
2	13	8	112	117	3	4	9	21	-8	1	10	9	135	-138	2	3	10	136	-136	2	11	10	127	125
4	13	8	75	-67	4	4	9	270	276	2	10	9	54	56	3	3	10	48	-53	3	11	10	100	-90
5	13	8	83	78	5	4	9	146	-142	3	10	9	70	63	5	3	10	58	-50	0	12	10	104	-99
0	14	8	210	-205	7	4	9	43	-33	4	10	9	82	-71	0	4	10	225	-219	1	12	10	78	-76
1	14	8	33	-57	8	4	9	67	-70	5	10	9	65	54	1	4	10	133	-131	2	12	10	98	96
2	14	8	48	82	1	5	9	162	-159	6	10	9	68	64	2	4	10	71	64	3	12	10	71	69
3	14	8	97	91	2	5	9	22	-19	1	11	9	64	-64	3	4	10	61	63	1	13	10	61	-56
4	14	8	75	73	3	5	9	108	104	2	11	9	146	-144	6	4	10	149	-159	1	1	11	63	57
1	15	8	39	37	4	5	9	101	-102	3	11	9	158	155	7	4	10	54	49	2	1	11	84	-83
2	15	8	79	-73	5	5	9	64	-65	4	11	9	66	68	2	5	10	76	-81	3	1	11	99	-98
0	16	8	98	86	6	5	9	82	79	0	12	9	63	68	3	5	10	40	44	0	2	11	38	71
1	1	9	86	89	8	5	9	58	-47	3	12	9	51	-41	4	5	10	57	57	1	2	11	134	131
2	1	9	71	77	1	6	9	49	-55	4	12	9	80	-74	5	5	10	121	-122	2	2	11	119	-121
3	1	9	58	66	2	6	9	43	45	1	13	9	78	82	6	5	10	34	-40	3	2	11	123	121
4	1	9	74	-75	3	6	9	34	-22	2	13	9	78	65	7	5	10	64	70	4	2	11	68	-62

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR dl Glutamic Acid

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	11	166	-167	5	5	11	48	-49	4	9	11	43	42	2	2	12	65	59	0	6	12	133	130
1	3	11	52	-51	6	5	11	34	40	0	10	11	136	-128	3	2	12	166	160	1	6	12	100	-99
2	3	11	61	58	0	6	11	30	-43	1	10	11	97	87	4	2	12	197	-177	1	7	12	48	45
3	3	11	51	48	1	6	11	60	66	2	10	11	130	119	5	2	12	171	-171	2	7	12	138	-147
4	3	11	23	-29	3	6	11	115	111	3	10	11	102	-96	2	3	12	145	-132	0	8	12	88	91
5	3	11	39	29	5	6	11	142	-137	0	0	12	327	-337	4	3	12	120	109	1	8	12	204	-194
0	4	11	22	19	1	7	11	24	35	1	0	12	190	-200	0	4	12	63	-80	2	1	13	75	-78
1	4	11	60	-65	3	7	11	52	-44	4	0	12	171	161	1	4	12	95	-99	3	1	13	78	-72
2	4	11	116	-113	4	7	11	93	93	5	0	12	129	112	2	4	12	48	52	0	2	13	80	81
3	4	11	44	-34	0	8	11	90	86	2	1	12	127	120	4	4	12	125	118	3	2	13	28	14
5	4	11	83	74	3	8	11	46	-37	3	1	12	68	63	1	5	12	116	-111	0	4	13	30	43
1	5	11	46	-38	1	9	11	143	-140	4	1	12	167	-162	2	5	12	147	141	1	4	13	90	-85
2	5	11	27	-39	2	9	11	27	-38	0	2	12	46	-42	3	5	12	169	165	2	4	13	129	119
3	5	11	131	124	3	9	11	177	174	1	2	12	43	-33	4	5	12	94	-86					

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
2	0	0	449	462
4	0	0	88	86
6	0	0	303	303
8	0	0	51	53
10	0	0	129	134
1	1	0	639	657
2	1	0	668	706
3	1	0	57	59
4	1	0	410	396
5	1	0	68	66
6	1	0	116	113
7	1	0	210	212
9	1	0	109	113
11	1	0	88	79
12	1	0	74	72
0	2	0	787	807
1	2	0	394	412
2	2	0	508	531
3	2	0	469	452
4	2	0	131	127
5	2	0	377	371
6	2	0	103	98
7	2	0	191	189
9	2	0	34	34
10	2	0	233	244
11	2	0	60	57
1	3	0	278	280
2	3	0	310	290
3	3	0	365	349
4	3	0	358	366
5	3	0	215	214
6	3	0	117	114
7	3	0	87	86
9	3	0	177	178
10	3	0	83	88
0	4	0	475	483
2	4	0	81	80
3	4	0	199	198
4	4	0	147	146
8	4	0	107	108
9	4	0	57	60
5	8	0	180	177
1	9	0	89	87
2	9	0	97	97
4	9	0	132	130
5	9	0	73	73
6	9	0	114	110
7	9	0	183	178
8	9	0	54	54
9	9	0	55	49
10	9	0	74	70
0	10	0	53	55
2	10	0	41	44
4	10	0	91	88
5	10	0	89	88
7	10	0	86	79
8	10	0	46	50
1	11	0	64	49
2	11	0	56	53
3	11	0	114	129
4	11	0	82	73
0	12	0	77	79
4	12	0	231	234
1	13	0	67	74
3	13	0	56	50
0	14	0	43	33
1	14	0	63	65
1	0	1	779	872
3	0	1	333	322
2	0	1	78	72
4	0	1	310	306
6	0	1	96	95
8	0	1	308	295
9	0	1	166	168
10	0	1	173	179
11	0	1	91	86
0	1	1	538	562
2	1	1	734	761
3	1	1	208	216
4	1	1	60	61
5	1	1	618	601
6	1	1	156	145
7	1	1	143	133
8	1	1	99	98
10	1	1	156	153
11	1	1	57	70
0	2	1	360	406
1	2	1	178	180
3	2	1	257	256
5	2	1	332	328
6	2	1	164	170
9	2	1	333	332
10	2	1	270	269
11	2	1	231	230
0	3	1	100	103
2	3	1	167	177
4	3	1	93	91
1	4	1	57	56
2	4	1	264	283
3	4	1	584	607
4	4	1	221	217
5	4	1	333	322
4	5	1	80	73
6	5	1	220	200
7	5	1	112	108
8	5	1	130	127
9	5	1	72	73
0	6	1	43	42
1	6	1	237	225
2	6	1	496	488
3	6	1	250	254
4	6	1	410	388
5	6	1	298	300
6	6	1	43	43
7	6	1	140	141
8	6	1	94	92
9	6	1	103	113
10	6	1	149	152
11	6	1	81	59
1	8	1	178	180
3	8	1	287	286
4	8	1	80	47
6	8	1	85	87
7	8	1	92	96
8	8	1	60	61
9	8	1	139	135
10	8	1	70	67
11	8	1	82	92
12	8	1	61	58
0	8	1	96	102
1	6	1	72	71
2	6	1	47	50
1	7	1	49	50
3	7	1	433	423
4	6	1	106	106
5	6	1	244	247

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
7	6	1	232	235
8	6	1	68	69
9	6	1	176	182
10	6	1	80	81
11	6	1	74	73
12	6	1	50	53
0	7	1	203	204
2	7	1	207	202
3	7	1	122	121
4	7	1	75	76
5	7	1	48	52
6	7	1	129	136
7	7	1	158	165
8	7	1	80	86
9	7	1	70	73
10	7	1	61	63
11	7	1	88	80
0	8	1	104	110
1	8	1	107	105
2	8	1	149	146
3	8	1	136	131
4	8	1	77	75
5	8	1	141	149
6	8	1	117	121
7	8	1	106	110
8	8	1	127	118
9	8	1	47	45
10	8	1	43	43
0	9	1	60	58
1	9	1	99	91
2	9	1	152	157
3	9	1	122	121
4	9	1	60	63
5	9	1	91	91
6	9	1	73	78
7	9	1	96	101
10	9	1	71	65
1	10	1	117	120
2	10	1	140	142
3	10	1	67	64
4	10	1	83	78
5	10	1	103	102
6	10	1	43	53
8	10	1	69	67
0	11	1	101	100
1	11	1	165	173
2	11	1	180	176
5	11	1	55	52
6	11	1	73	74
1	12	1	58	57
3	12	1	82	91
4	12	1	62	68
5	12	1	117	111
0	13	1	67	78
1	13	1	83	78
3	13	1	77	75
0	0	2	261	263
1	0	2	109	112
2	0	2	247	256
3	0	2	226	218
4	0	2	137	141
5	0	2	190	186
6	0	2	446	425
7	0	2	304	309
8	0	2	87	93
10	0	2	290	297
0	1	2	671	669
1	1	2	547	565
2	1	2	640	653
3	1	2	111	108
4	1	2	218	214
5	1	2	245	251
6	1	2	197	195
7	1	2	54	54
8	1	2	137	139
9	1	2	41	48
10	1	2	111	115
11	1	2	93	98
0	2	2	1361	1716
1	2	2	919	932
3	2	2	151	151
4	2	2	150	149
5	2	2	54	56
6	2	2	207	194
7	2	2	75	77
8	2	2	128	127
9	2	2	183	187
10	2	2	85	88
11	2	2	41	55
0	3	2	191	205
1	3	2	480	471
2	3	2	256	267
3	3	2	55	52
4	3	2	370	361
5	3	2	213	199
6	3	2	169	162
7	3	2	185	185
8	3	2	72	83
9	3	2	189	184
12	3	2	37	37
0	4	2	415	429
1	4	2	282	289
2	4	2	288	298
4	4	2	194	184
4	4	2	41	40
5	4	2	247	231
6	4	2	120	107
7	4	2	109	107
8	4	2	97	96
9	4	2	58	57
10	4	2	176	177
0	5	2	69	70
1	5	2	163	163
2	5	2	295	280
3	5	2	272	260
4	5	2	236	226
5	5	2	114	109
7	5	2	68	70
8	5	2	160	163
10	5	2	103	104
11	5	2	48	47
12	5	2	49	52
0	6	2	148	154
1	6	2	143	147
2	6	2	96	94
3	6	2	134	129
4	6	2	113	115
5	6	2	122	118
6	6	2	86	84
7	6	2	114	119
8	6	2	84	87
9	6	2	122	118
10	6	2	79	78
11	6	2	88	89
4	7	2	157	164
5	7	2	96	92
6	7	2	187	188
7	7	2	133	134
8	7	2	181	191
10	7	2	47	38
0	8	2	166	158
2	8	2	42	43
3	8	2	120	116
4	8	2	69	68
5	8	2	132	133
6	8	2	96	97
7	8	2	150	154
8	8	2	122	128
9	8	2	51	61
10	8	2	52	44
12	8	2	72	76
0	9	2	125	118
1	9	2	198	194
2	9	2	83	87
3	9	2	214	210
4	9	2	74	70
5	9	2	124	120
6	9	2	63	74
8	9	2	88	93
0	10	2	86	86
1	10	2	115	117
2	10	2	126	132
3	10	2	85	86
4	10	2	73	69
8	10	2	60	51
7	10	2	97	106
8	10	2	37	38

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
2	11	2	114	111
3	11	2	80	85
4	11	2	90	93
7	11	2	78	66
8	11	2	97	48
0	12	2	50	47
1	12	2	38	34
2	12	2	120	113
3	12	2	35	41
4	12	2	70	63
6	12	2	77	70
1	0	3	152	144
2	0	3	623	633
3	0	3	346	349
4	0	3	323	324
5	0	3	332	322
6	0	3	39	40
7	0	3	64	64
8	0	3	63	60
9	0	3	195	199
10	0	3	205	199
12	0	3	87	86
0	1	3	736	732
1	1	3	909	886
2	1	3	395	398
3	1	3	154	156
4	1	3	114	114
5	1	3	108	102
6	1	3	203	196
7	1	3	67	68
8	1	3	39	41
9	1	3	120	123
10	1	3	66	73
11	1	3	43	37
12	1	3	34	34
0	2	3	177	171
1	2	3	810	813
2	2	3	96	92
3	2	3	253	248
4	2	3	207	206
5	2	3	120	112
6	2	3	140	135
7	2	3	210	207
8	2	3	127	130
10	2	3	43	40
11	2	3	125	131
12	2	3	60	51
0	3	3	544	542
1	3	3	108	114
2	3	3	233	235
3	3	3	67	68
4	3	3	256	246
5	3	3	114	98
6	3	3	175	170
7	3	3	92	90
8	3	3	119	118
9	3	3	147	152
12	3	3	38	40
0	4	3	388	377
1	4	3	340	332
2	4	3	265	263
3	4	3	318	307
4	4	3	361	350
5	4	3	79	77
6	4	3	153	150
7	4	3	179	173
8	4	3	235	242
9	4	3	163	165
10	4	3	104	105
0	5	3	42	47
1	5	3	497	468
3	5	3	399	388
4	5	3	43	45
5	5	3	109	108
6	5	3	247	240
7	5	3	116	119
8	5	3	63	62
9	5	3	90	92
0	6	3	112	111
1	6	3	77	74
2	6	3	127	120
3	6	3	134	148
4	6	3	76	80
5	6	3	148	153
6	6	3	47	45
7	6	3	200	204
8	6	3	108	108
11	6	3	55	50
0	7	3	104	95
1	7	3	98	102
2	7	3	211	201
3	7	3	47	48
4	7	3	193	187
5	7	3	161	164
6	7	3	204	216
7	7	3	52	58
8	7	3	123	124
9	7	3	54	50
10	7	3	73	64
11	7	3	80	77
0	8	3	168	165
1	0	3	52	56
2	0	3	177	180
3	0	3	59	62
4	0	3	182	186
5	0	3	161	166
6	0	3	134	136
7	0	3	95	88
8	0	3	129	129
1	9	3	104	112
2	9	3	275	269
4	9	3	86	81
5	9	3	79	84
6	9	3	91	90
7	9	3	92	49
1	10	3	120	124
2	10	3	129	127
3	10	3	72	75
4	10	3	39	42
5	10	3	59	59
7	10	3	77	80
8	10	3	58	48
0	11	3	109	112
1	11	3	35	41
2	11	3	63	61
3	11	3	89	95
4	11	3	57	71
5	11	3	78	85
0	12	3	115	113
1	12	3	73	71
2	12	3	39	38
3	12	3	44	41
4	12	3	74	73
5	12	3	61	58
6	12	3	79	78
0	0	4	821	809
1	0	4	838	844
2	0	4	250	245
3	0	4	87	82
5	0	4	71	76
7	0	4	38	38
8	0	4	166	172
9	0	4	85	83
10	0	4	65	68
11	0	4	80	80
1	1	4	132	126
2	1	4	46	48
3	1	4	111	104
4	1	4	122	122
5	1	4	144	140
6	1	4	138	135
7	1	4	94	94
8	1	4	80	82
9	1	4	170	169
12	1	4	64	60
1	2	4	401	399
2	2	4	288	301
3	2	4	247	239
4	2	4	107	106
5	2	4	347	336
6	2	4	194	189
7	2	4	108	113
8	2	4	177	173
9	2	4	137	138
10	2	4	127	120
12	2	4	42	38
3	3	4	344	351
2	3	4	242	250
3	3	4	294	285

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
4	3	4	275	268
5	3	4	166	169
6	3	4	76	77
7	3	4	203	205
8	3	4	130	129
9	3	4	136	141
11	3	4	62	57
0	4	4	420	419
1	4	4	568	573
2	4	4	284	276
3	4	4	195	186
4	4	4	89	90
5	4	4	80	78
6	4	4	180	177
7	4	4	100	102
8	4	4	151	160
9	4	4	54	56
10	4	4	37	24
11	4	4	53	53
0	5	4	484	472
1	5	4	219	203
2	5	4	82	81
3	5	4	105	101
4	5	4	134	139
5	5	4	177	164
6	5	4	177	180
7	5	4	184	181
8	5	4	132	133
9	5	4	195	201
0	6	4	117	119
1	6	4	254	254
2	6	4	64	68
4	6	4	35	39
5	6	4	123	126
6	6	4	75	75
7	6	4	131	135
8	6	4	117	121
1	7	4	98	95
2	7	4	91	98
3	7	4	168	173
4	7	4	132	135
5	7	4	135	140
6	7	4	81	81
7	7	4	92	86
10	7	4	57	52
1	8	4	94	103
2	8	4	138	136
3	8	4	64	67
4	8	4	90	89
5	8	4	68	66
7	8	4	67	70
8	8	4	51	50
9	8	4	32	49
0	9	4	140	139
1	9	4	91	96
2	9	4	136	143
4	9	4	54	61
5	9	4	158	172
6	9	4	81	85
7	9	4	49	45
8	9	4	58	59
9	9	4	61	50
0	10	4	85	90
1	10	4	116	117
2	10	4	105	107
3	10	4	51	58
6	10	4	83	86
7	10	4	83	74
2	11	4	86	81
3	11	4	93	93
4	11	4	57	59
5	11	4	78	82
6	11	4	58	58
7	11	4	43	45
8	11	4	134	136
9	11	4	109	108
0	12	4	247	240
1	12	4	116	119
2	12	4	63	62
4	12	4	90	92
5	12	4	112	111
7	12	4	127	120
8	12	4	76	80
9	12	4	148	153
10	12	4	72	75
11	12	4	47	45
0	13	4	112	105
1	13	4	211	194
2	13	4	436	431
3	13	4	111	115
4	13	4	39	37
5	13	4	175	166
6	13	4	64	66
0	14	4	588	579
1	14	4	174	177
2	14	4	73	75
3	14	4	39	39
4	14	4	184	178
5	14	4	39	44
6	14	4	129	131
7	14	4	162	166
8	14	4	72	69
9	14	4	92	98
0	15	4	231	228
1	15	4	489	490
2	15	4	259	264
3	15	4	74	76
5	15	4	135	163
6	15	4	88	84
7	15	4	90	92
8	2	5	213	210
9	2	5	120	118
10	2	5	70	69
11	2	5	74	73
12	2	5	53	51
0	3	5	341	330
1	3	5	375	362
2	3	5	176	177
3	3	5	265	265
4	3	5	234	221
5	3	5	83	79
6	3	5	243	239
7	3	5	69	69
9	3	5	139	142
10	3	5	66	70
11	3	5	69	71
0	4	5	394	396
1	4	5	175	170
2	4	5	249	246
3	4	5	145	141
4	4	5	103	101
5	4	5	142	132
6	4	5	132	131
8	4	5	118	118
9	4	5	73	72
1	5	5	47	44
2	5	5	192	202
4	5	5	44	44
5	5	5	67	61
6	5	5	102	106
7	5	5	40	31
9	5	5	65	68
10	5	5	40	49
11	5	5	47	47
0	6	5	214	211
1	6	5	106	107
2	6	5	49	44
3	6	5	50	46
4	6	5	111	106
6	6	5	98	93
7	6	5	99	101
8	6	5	56	55
1	7	5	167	164
2	7	5	175	173
3	7	5	74	74
5	7	5	37	37
6	7	5	116	109
7	7	5	81	77
8	7	5	104	110
1	8	5	80	77
2	8	5	42	70
3	8	5	117	112
4	8	5	61	55
5	8	5	117	118
6	8	5	195	196
7	8	5	62	58
0	9	5	134	125
1	9	5	47	44
4	9	5	192	202
7	9	5	44	44
8	9	5	67	61
2	10	5	62	57
3	10	5	102	106
4	10	5	105	106
7	10	5	40	31
1	11	5	65	68

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
2	11	5	93	95
0	12	5	144	143
1	12	5	50	47
2	12	5	96	92
3	12	5	74	76
0	0	6	76	76
1	0	6	98	101
3	0	6	230	216
4	0	6	168	164
5	0	6	149	143
7	0	6	63	68
8	0	6	134	136
0	1	6	168	167
1	1	6	220	218
3	1	6	272	269
4	1	6	87	91
5	1	6	160	159
6	1	6	114	111
7	1	6	62	60
8	1	6	86	87
9	1	6	90	85
12	1	6	68	57
0	2	6	247	247
1	2	6	382	379
2	2	6	224	219
3	2	6	97	92
4	2	6	170	171
5	2	6	31	36
7	2	6	193	185
8	2	6	64	60
9	2	6	175	174
0	3	6	294	306
1	3	6	174	179
2	3	6	142	155
3	3	6	203	194
5	3	6	144	143
6	3	6	68	69
7	3	6	205	206
8	3	6	226	231
9	3	6	160	167
10	3	6	260	269
0	4	6	43	41
1	4	6	240	233
2	4	6	273	272
3	4	6	82	84
4	4	6	190	203
5	4	6	218	230
6	4	6	158	161
7	4	6	50	53
8	4	6	212	213
9	4	6	66	70
10	4	6	54	52
11	4	6	58	57
0	5	6	99	102
1	5	6	312	298
2	5	6	114	108
3	5	6	206	206
4	5	6	131	134
5	5	6	217	216
6	5	6	40	39
7	5	6	206	204
8	5	6	74	67
9	5	6	78	71
11	5	6	53	43
0	6	6	125	124
1	6	6	152	157
2	6	6	95	93
3	6	6	143	140
4	6	6	187	196
5	6	6	90	95
6	6	6	69	68
7	6	6	101	109
0	7	6	80	80
1	7	6	176	184
2	7	6	97	49
3	7	6	137	144
4	7	6	46	42
5	7	6	52	51
6	7	6	98	95
7	7	6	45	38
8	7	6	71	60
0	8	6	42	38
1	8	6	136	143
2	8	6	150	144
3	8	6	46	35
4	8	6	108	110
5	8	6	104	102
6	8	6	75	75
7	8	6	106	92
9	8	6	83	78
1	9	6	60	61
3	9	6	89	91
4	9	6	93	101
5	9	6	85	89
1	10	6	107	117
2	10	6	54	57
4	10	6	68	62
5	10	6	120	126
7	10	6	41	36
1	11	6	57	72
2	11	6	46	36
3	11	6	98	96
4	11	6	76	66
5	11	6	49	51
0	12	6	46	37
2	0	7	246	239
3	0	7	302	299
4	0	7	240	243
5	0	7	98	96
6	0	7	116	119
7	0	7	212	208
8	0	7	170	167
9	0	7	86	84
11	0	7	72	72
0	1	7	235	233
1	1	7	167	171
2	1	7	287	283
3	1	7	43	38
4	1	7	135	125
5	1	7	98	99
6	1	7	57	58
7	1	7	118	121
8	1	7	39	38
9	1	7	77	87
10	1	7	62	62
11	1	7	74	80
0	2	7	135	113
1	2	7	207	196
2	2	7	115	116
3	2	7	79	80
4	2	7	79	78
7	2	7	93	86
8	2	7	91	91
9	2	7	44	44
0	3	7	118	119
1	3	7	390	385
2	3	7	100	97
3	3	7	88	87
4	3	7	94	100
5	3	7	129	124
6	3	7	142	148
7	3	7	91	91
8	3	7	49	47
11	3	7	46	44
0	4	7	97	93
1	4	7	156	145
2	4	7	242	232
3	4	7	116	117
4	4	7	86	92
5	4	7	176	180
6	4	7	88	86
7	4	7	163	165
8	4	7	72	70
0	5	7	290	277
4	5	7	183	190
5	5	7	95	94
6	5	7	121	120
7	5	7	76	73
8	5	7	91	99
9	5	7	69	70
0	6	7	251	261
1	6	7	77	85
3	6	7	191	188
4	6	7	190	193
5	6	7	181	182
6	6	7	136	135
7	6	7	71	69
8	6	7	79	82

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
6	7	9	75	71
1	8	9	70	73
3	8	9	53	50
4	8	9	67	61
0	9	9	95	89
1	9	9	45	50
3	9	9	62	56
5	9	9	50	63
0	10	9	40	37
1	10	9	49	50
4	10	9	75	71
0	11	9	62	58
0	0	10	110	107
3	0	10	87	89
4	0	10	128	131
5	0	10	135	144
6	0	10	97	98
7	0	10	136	136
8	0	10	49	46
0	1	10	161	169
1	1	10	189	178
2	1	10	83	83
3	1	10	175	174
6	1	10	76	82
7	1	10	143	142
8	1	10	92	96
0	2	10	291	288
1	2	10	94	99
2	2	10	189	189
4	2	10	60	59
6	2	10	149	147
8	2	10	56	61
0	3	10	54	59
1	3	10	104	105
2	3	10	132	132
3	3	10	71	65
4	3	10	75	76
0	4	10	179	189
1	4	10	83	81
2	4	10	72	81
3	4	10	92	90
4	4	10	87	95
5	4	10	78	77
6	4	10	66	68
7	4	10	61	56
8	4	10	59	49
1	5	10	86	84
2	5	10	72	67
3	5	10	67	65
4	5	10	67	67
7	5	10	51	59
8	5	10	68	68
0	6	10	66	61
1	6	10	84	87
2	6	10	66	57
3	6	10	116	127
4	6	10	47	50
5	6	10	47	47
6	6	10	86	90
1	7	10	37	33
2	7	10	83	82
3	7	10	70	70
4	7	10	60	58
5	7	10	80	84
0	8	10	85	80
1	8	10	74	78
5	8	10	46	44
1	9	10	40	26
2	9	10	51	51
4	9	10	75	65
0	10	10	56	46
1	10	10	43	27
5	0	11	125	130
6	0	11	134	134
8	0	11	53	55
0	1	11	87	87
1	1	11	74	79
2	1	11	73	68
3	1	11	88	92
4	1	11	115	119
6	1	11	100	99
7	1	11	67	65
8	1	11	63	58
0	2	11	125	132
1	2	11	92	94
2	2	11	55	56
3	2	11	44	40
4	2	11	73	77
5	2	11	86	82
6	2	11	117	113
7	2	11	43	53
8	2	11	42	45
3	3	11	62	64
4	3	11	93	93
5	3	11	68	62
6	3	11	93	93
7	3	11	73	73
8	3	11	83	79
2	4	11	60	65
3	4	11	48	46
4	4	11	84	85
6	4	11	40	40
0	5	11	58	58
1	5	11	81	72
2	5	11	152	154
3	5	11	86	87
4	5	11	38	36
5	5	11	70	68
6	5	11	89	80
7	5	11	46	54
0	6	11	36	33
1	6	11	79	89
2	6	11	92	92
3	6	11	58	59
4	6	11	94	95
5	6	11	74	81
6	6	11	46	57
0	7	11	45	39
1	7	11	42	54
2	7	11	61	65
1	8	11	76	70
0	9	11	78	76
0	0	12	180	185
1	0	12	89	88
2	0	12	46	38
3	0	12	92	89
4	0	12	102	103
6	0	12	129	134
7	0	12	86	83
1	1	12	114	112
2	1	12	92	89
3	1	12	56	60
7	1	12	65	62
0	2	12	40	46
2	2	12	48	51
2	2	12	87	89
3	2	12	42	46
4	2	12	143	147
5	2	12	49	49
0	3	12	99	97
1	3	12	84	89
2	3	12	183	189
3	3	12	72	71
1	5	12	36	38
2	5	12	93	97
3	5	12	48	41
4	5	12	92	86
5	5	12	44	49
0	6	12	108	109
4	6	12	37	39
1	7	12	78	86
2	7	12	59	60
3	0	13	70	70
5	0	13	42	46
0	1	13	114	114
3	1	13	92	95
4	1	13	101	95
4	2	13	76	67
5	2	13	68	68
1	3	13	89	89
3	3	13	69	72
2	4	13	51	36
3	4	13	80	79
4	4	13	91	88
0	5	13	38	38
1	5	13	69	64
2	5	13	71	65
0	6	13	35	42
0	0	14	111	112

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
9	6	7	37	44
0	7	7	239	250
1	7	7	82	75
2	7	7	149	152
3	7	7	40	43
4	7	7	185	193
5	7	7	73	68
7	7	7	86	88
8	7	7	96	95
0	8	7	104	104
1	8	7	119	116
2	8	7	129	130
4	8	7	57	50
6	8	7	50	52
1	9	7	95	89
2	9	7	81	75
3	9	7	95	105
5	9	7	68	69
7	9	7	40	44
3	10	7	94	86
6	10	7	70	73
2	11	7	41	34
3	11	7	65	63
1	0	8	64	64
2	0	8	249	262
3	0	8	47	44
5	0	8	138	130
7	0	8	73	72
8	0	8	40	37
9	0	8	49	46
11	0	8	108	108
1	1	8	61	60
2	1	8	93	92
3	1	8	165	159
4	1	8	68	70
5	1	8	66	66
6	1	8	117	121
7	1	8	201	200
8	1	8	178	176
9	1	8	120	122
10	1	8	104	110
0	2	8	62	58
1	2	8	200	204
2	2	8	138	150
3	2	8	99	99
4	2	8	117	112
5	2	8	93	82
7	2	8	172	169
8	2	8	86	90
9	2	8	93	97
10	2	8	73	73
0	3	8	256	261
1	3	8	131	137
2	3	8	117	120
4	3	8	67	68
5	3	8	80	84
6	3	8	84	86
8	3	8	96	95
0	4	8	59	58
1	4	8	99	99
2	4	8	148	152
3	4	8	92	95
4	4	8	65	65
5	4	8	99	104
6	4	8	130	128
7	4	8	68	66
10	4	8	44	46
0	5	8	166	161
1	5	8	104	115
2	5	8	204	201
3	5	8	187	181
4	5	8	193	197
5	5	8	89	91
7	5	8	110	114
8	5	8	87	77
9	5	8	87	82
0	6	8	235	235
1	6	8	186	194
2	6	8	96	97
3	6	8	90	92
4	6	8	184	191
5	6	8	90	86
6	6	8	79	74
7	6	8	121	119
8	6	8	41	44
0	7	8	75	75
1	7	8	196	195
2	7	8	66	73
3	7	8	233	237
4	7	8	86	95
5	7	8	66	60
7	7	8	75	69
8	7	8	49	38
0	8	8	89	94
1	8	8	105	108
2	8	8	112	114
5	8	8	51	59
6	8	8	76	74
8	8	8	42	42
1	9	8	53	48
5	9	8	40	32
7	9	8	67	57
1	10	8	37	33
2	10	8	63	74
3	10	8	63	49
2	11	8	47	43
3	11	8	62	51
1	0	9	69	67
2	0	9	132	133
3	0	9	136	133
4	0	9	53	54
5	0	9	61	56
6	0	9	85	81
7	0	9	100	89
8	0	9	198	187
0	1	9	147	143
1	1	9	102	106
2	1	9	95	94
3	1	9	87	92
4	1	9	84	89
5	1	9	83	81
6	1	9	147	149
8	1	9	100	98
9	1	9	94	92
10	1	9	48	41
0	2	9	39	41
1	2	9	151	147
2	2	9	81	81
3	2	9	253	257
4	2	9	39	35
5	2	9	90	88
6	2	9	66	59
7	2	9	87	91
8	2	9	120	123
10	2	9	52	57
1	3	9	212	224
2	3	9	93	90
3	3	9	163	166
4	3	9	130	124
5	3	9	85	79
9	3	9	77	76
0	4	9	179	179
1	4	9	219	223
2	4	9	74	75
3	4	9	213	217
4	4	9	69	69
5	4	9	124	119
6	4	9	82	77
8	4	9	100	92
9	4	9	78	71
0	5	9	178	176
1	5	9	131	129
2	5	9	188	184
3	5	9	89	87
4	5	9	120	124
5	5	9	113	126
6	5	9	97	94
7	5	9	85	84
8	5	9	98	93
0	6	9	88	81
1	6	9	99	65
2	6	9	77	80
3	6	9	97	90
4	6	9	82	78
5	6	9	94	94
6	6	9	98	97
0	7	9	187	179
1	7	9	97	101
2	7	9	73	77
4	7	9	91	101

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine

H	K	L	10FO	10FC
0	1	14	44	37
1	1	14	62	69
2	1	14	64	65
1	2	14	48	53
2	2	14	112	111

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	707	705	3	6	0	222	223	5	12	0	155	166	6	19	0	71	66	1	33	0	114	111
4	0	0	65	66	4	6	0	159	156	6	12	0	216	219	0	20	0	375	384	1	0	1	117	133
6	0	0	232	222	5	6	0	80	81	8	12	0	73	57	1	20	0	57	66	2	0	1	848	845
8	0	0	95	75	1	7	0	947	833	1	13	0	55	62	2	20	0	137	129	4	0	1	156	150
1	1	0	176	204	2	7	0	77	86	3	13	0	214	210	3	20	0	80	85	5	0	1	98	83
2	1	0	294	244	3	7	0	336	329	5	13	0	104	103	5	20	0	90	87	6	0	1	91	100
3	1	0	189	182	4	7	0	169	166	6	13	0	125	125	6	20	0	82	72	8	0	1	81	98
4	1	0	313	307	5	7	0	82	92	7	13	0	92	104	2	21	0	172	184	2	1	1	630	616
5	1	0	136	128	6	7	0	76	81	1	14	0	361	334	3	21	0	109	118	3	1	1	359	360
1	2	0	1165	1207	0	8	0	851	873	2	14	0	241	247	4	21	0	143	145	4	1	1	261	262
2	2	0	236	245	1	8	0	1265	1275	3	14	0	368	359	5	21	0	147	134	5	1	1	153	167
3	2	0	40	28	2	8	0	340	336	5	14	0	274	273	0	22	0	81	68	6	1	1	119	118
7	2	0	74	58	3	8	0	58	52	6	14	0	65	65	4	22	0	76	72	7	1	1	86	98
1	3	0	93	45	4	8	0	265	276	1	15	0	443	434	1	23	0	189	183	1	2	1	597	605
2	3	0	1034	1053	5	8	0	97	104	2	15	0	116	114	3	23	0	145	138	2	2	1	343	336
3	3	0	183	187	2	9	0	40	42	3	15	0	202	186	4	23	0	91	86	3	2	1	228	228
4	3	0	107	108	3	9	0	445	446	4	15	0	116	123	0	24	0	94	95	4	2	1	117	112
6	3	0	73	62	4	9	0	284	267	6	15	0	109	110	1	24	0	153	143	5	2	1	164	171
7	3	0	80	71	5	9	0	67	54	1	16	0	167	169	2	24	0	96	97	6	2	1	118	120
1	4	0	438	430	0	10	0	768	752	3	16	0	102	103	3	24	0	144	134	7	2	1	97	105
2	4	0	841	828	1	10	0	150	139	4	16	0	90	104	2	25	0	140	135	0	3	1	355	352
3	4	0	415	399	2	10	0	237	218	6	16	0	120	124	4	25	0	80	75	1	3	1	527	530
4	4	0	381	379	3	10	0	120	112	1	17	0	93	106	5	25	0	65	63	2	3	1	377	372
5	4	0	137	133	4	10	0	122	125	2	17	0	220	223	6	25	0	88	83	3	3	1	173	161
7	4	0	123	115	1	11	0	265	252	4	17	0	91	93	0	26	0	98	89	4	3	1	79	71
1	5	0	1268	1292	3	11	0	811	790	5	17	0	148	130	3	26	0	91	82	5	3	1	212	229
2	5	0	483	490	4	11	0	77	76	0	18	0	120	125	5	26	0	79	64	6	3	1	90	92
3	5	0	179	160	5	11	0	56	57	1	18	0	126	110	2	27	0	119	116	0	4	1	930	921
4	5	0	517	504	6	11	0	59	60	3	18	0	57	53	3	27	0	169	180	1	4	1	678	700
5	5	0	116	114	0	12	0	272	262	5	18	0	174	174	0	28	0	104	99	2	4	1	414	414
6	5	0	92	87	1	12	0	246	241	1	19	0	130	134	2	28	0	104	115	4	4	1	139	126
0	6	0	36	51	2	12	0	639	628	2	19	0	119	103	2	29	0	72	88	5	4	1	149	147
1	6	0	273	286	3	12	0	201	195	3	19	0	69	65	3	29	0	77	80	7	4	1	85	60
2	6	0	293	297	4	12	0	75	56	4	19	0	246	259	2	31	0	93	80	8	4	1	80	90

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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
0	5	1	194	169	3	10	1	250	227	0	16	1	52	46	1	22	1	222	242	1	33	1	67	61
1	5	1	1264	1320	4	10	1	152	142	1	16	1	221	217	2	22	1	100	92	1	35	1	78	70
2	5	1	303	292	5	10	1	112	115	2	16	1	94	103	3	22	1	134	141	2	35	1	100	109
4	5	1	139	146	7	10	1	83	84	3	16	1	132	133	4	22	1	160	168	0	36	1	109	94
5	5	1	191	180	8	10	1	70	44	4	16	1	100	82	0	23	1	98	117	1	36	1	83	63
6	5	1	96	106	0	11	1	574	577	5	16	1	148	149	1	23	1	120	99	1	0	2	251	255
0	6	1	302	314	1	11	1	411	409	6	16	1	78	86	2	23	1	125	126	2	0	2	234	212
1	6	1	784	780	2	11	1	260	256	0	17	1	174	162	4	23	1	172	180	4	0	2	199	201
2	6	1	438	429	3	11	1	71	81	1	17	1	157	177	6	23	1	80	97	5	0	2	114	116
3	6	1	554	552	4	11	1	189	182	2	17	1	153	156	0	24	1	113	124	6	0	2	317	344
4	6	1	249	250	5	11	1	72	75	3	17	1	134	137	1	24	1	102	106	7	0	2	105	109
5	6	1	118	124	6	11	1	146	140	5	17	1	100	101	2	24	1	191	204	8	0	2	69	84
7	6	1	160	158	0	12	1	237	203	0	18	1	99	94	3	24	1	236	236	1	1	2	99	118
0	7	1	426	416	1	12	1	458	452	2	18	1	86	113	6	24	1	94	81	2	1	2	255	246
1	7	1	288	285	2	12	1	218	229	3	18	1	183	194	0	25	1	184	173	3	1	2	273	271
2	7	1	615	614	3	12	1	300	287	5	18	1	79	86	3	25	1	110	112	4	1	2	227	228
3	7	1	201	201	5	12	1	99	100	6	18	1	63	73	0	26	1	102	76	5	1	2	232	234
4	7	1	213	218	6	12	1	130	129	0	19	1	127	129	1	26	1	181	187	6	1	2	86	89
5	7	1	91	90	0	13	1	602	592	1	19	1	184	190	3	26	1	90	87	8	1	2	95	99
1	8	1	84	88	1	13	1	103	97	2	19	1	358	364	5	26	1	88	92	1	2	2	367	375
2	8	1	241	233	2	13	1	358	343	3	19	1	105	105	1	27	1	77	45	2	2	2	365	351
3	8	1	139	139	4	13	1	150	154	4	19	1	165	175	2	27	1	158	162	3	2	2	317	317
5	8	1	53	37	5	13	1	91	92	6	19	1	72	80	1	28	1	98	114	4	2	2	233	232
6	8	1	137	164	6	13	1	127	123	0	20	1	160	158	2	28	1	74	101	5	2	2	75	73
7	8	1	78	87	0	14	1	296	275	1	20	1	192	180	3	28	1	79	81	7	2	2	162	156
0	9	1	81	69	1	14	1	181	166	2	20	1	78	62	5	28	1	80	56	8	2	2	94	106
1	9	1	88	88	2	14	1	181	172	3	20	1	165	172	2	29	1	134	140	0	3	2	516	523
2	9	1	242	239	3	14	1	373	377	4	20	1	192	193	3	29	1	68	61	1	3	2	485	498
3	9	1	266	262	4	14	1	185	190	5	20	1	67	61	0	30	1	81	85	2	3	2	158	154
4	9	1	71	77	5	14	1	148	156	0	21	1	70	48	2	30	1	78	109	3	3	2	214	219
5	9	1	53	34	0	15	1	86	84	1	21	1	158	162	0	32	1	67	58	4	3	2	82	89
0	10	1	213	209	1	15	1	405	408	2	21	1	116	119	1	32	1	95	76	5	3	2	154	147
1	10	1	299	299	2	15	1	144	143	3	21	1	70	64	2	32	1	70	75	8	3	2	86	91
2	10	1	192	188	4	15	1	97	101	5	21	1	67	52	0	33	1	83	67	0	4	2	112	68

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	4	2	319	335	7	8	2	106	114	4	13	2	56	67	1	19	2	136	165	0	27	2	78	71
2	4	2	416	420	0	9	2	70	75	5	13	2	111	101	2	19	2	181	163	2	27	2	136	148
3	4	2	192	173	1	9	2	120	107	6	13	2	129	123	3	19	2	123	128	0	28	2	180	185
4	4	2	148	144	2	9	2	427	415	7	13	2	84	90	4	19	2	186	201	1	28	2	89	92
5	4	2	132	128	3	9	2	249	238	0	14	2	200	194	5	19	2	113	116	0	29	2	184	171
6	4	2	188	184	4	9	2	139	138	1	14	2	322	309	0	20	2	150	109	1	29	2	79	89
7	4	2	85	84	5	9	2	130	143	2	14	2	282	277	1	20	2	85	83	2	29	2	96	105
0	5	2	304	297	6	9	2	124	118	3	14	2	127	123	3	20	2	159	178	3	29	2	120	111
1	5	2	423	396	0	10	2	107	101	4	14	2	226	239	4	20	2	130	130	1	30	2	68	70
2	5	2	510	521	1	10	2	148	152	5	14	2	248	260	7	20	2	76	65	1	32	2	83	78
3	5	2	117	102	2	10	2	268	270	0	15	2	87	70	0	21	2	182	174	0	33	2	61	52
4	5	2	97	105	3	10	2	355	357	1	15	2	270	268	1	21	2	85	100	2	33	2	72	67
5	5	2	189	195	4	10	2	222	230	2	15	2	357	383	3	21	2	105	128	1	34	2	114	109
6	5	2	103	113	5	10	2	83	62	3	15	2	139	135	0	22	2	69	17	1	0	3	337	334
0	6	2	434	434	6	10	2	108	125	4	15	2	70	72	1	22	2	79	67	2	0	3	74	60
1	6	2	638	634	7	10	2	72	63	5	15	2	163	176	2	22	2	78	71	3	0	3	207	209
2	6	2	316	306	0	11	2	337	323	6	15	2	90	85	3	22	2	91	101	4	0	3	149	150
3	6	2	434	415	1	11	2	106	119	7	15	2	67	58	4	22	2	130	117	5	0	3	70	76
4	6	2	95	93	2	11	2	419	412	0	16	2	149	149	6	22	2	85	102	6	0	3	109	100
5	6	2	142	152	3	11	2	372	363	1	16	2	98	86	0	23	2	245	247	8	0	3	74	48
0	7	2	243	243	4	11	2	216	219	2	16	2	109	108	1	23	2	77	80	1	1	3	403	398
1	7	2	453	447	5	11	2	112	113	3	16	2	180	171	2	23	2	61	56	2	1	3	530	505
2	7	2	385	385	6	11	2	101	114	4	16	2	136	140	5	23	2	88	99	3	1	3	189	176
3	7	2	187	175	0	12	2	334	327	0	17	2	185	191	0	24	2	96	92	4	1	3	60	58
4	7	2	137	144	1	12	2	118	113	1	17	2	283	290	1	24	2	99	105	5	1	3	79	86
5	7	2	123	121	2	12	2	320	328	2	17	2	178	179	2	24	2	87	89	6	1	3	187	188
6	7	2	153	149	3	12	2	257	264	3	17	2	103	96	4	24	2	84	85	7	1	3	115	113
7	7	2	126	128	4	12	2	109	113	6	17	2	121	124	1	25	2	193	203	1	2	3	393	386
0	8	2	178	146	5	12	2	91	88	7	17	2	108	105	3	25	2	71	87	2	2	3	316	307
1	8	2	282	298	6	12	2	62	61	0	18	2	249	240	4	25	2	126	112	3	2	3	160	154
2	8	2	328	321	8	12	2	78	81	3	18	2	200	198	5	25	2	71	78	4	2	3	116	128
3	8	2	273	259	0	13	2	88	88	4	18	2	217	229	0	26	2	98	117	5	2	3	70	77
4	8	2	31	47	1	13	2	300	296	5	18	2	118	113	1	26	2	135	108	6	2	3	119	119
6	8	2	69	59	3	13	2	265	263	0	19	2	293	278	2	26	2	157	153	0	3	3	486	480

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	3	3	170	168	5	7	3	104	121	4	12	3	188	189	4	18	3	101	112	1	26	3	218	211
2	3	3	298	281	0	8	3	194	195	5	12	3	225	232	5	18	3	92	102	3	26	3	80	69
3	3	3	377	356	1	8	3	98	77	6	12	3	74	89	0	19	3	108	105	0	27	3	102	80
4	3	3	148	143	2	8	3	358	338	7	12	3	78	84	1	19	3	71	85	1	27	3	129	117
5	3	3	117	119	3	8	3	105	97	1	13	3	130	127	2	19	3	66	75	2	27	3	78	87
6	3	3	66	75	4	8	3	166	181	2	13	3	129	129	3	19	3	167	167	0	28	3	65	48
0	4	3	78	63	5	8	3	200	195	3	13	3	118	115	4	19	3	122	126	1	28	3	132	111
1	4	3	402	387	6	8	3	137	142	4	13	3	148	156	5	19	3	132	138	2	28	3	70	76
2	4	3	115	121	7	8	3	103	94	0	14	3	149	133	0	20	3	108	116	0	29	3	98	104
3	4	3	190	184	0	9	3	216	199	1	14	3	102	93	2	20	3	63	48	1	29	3	69	58
5	4	3	73	87	1	9	3	127	114	2	14	3	60	58	3	20	3	163	164	0	30	3	179	188
6	4	3	89	64	2	9	3	217	228	3	14	3	79	74	4	20	3	109	115	2	30	3	68	60
7	4	3	68	50	3	9	3	91	82	4	14	3	152	153	0	21	3	178	192	4	30	3	84	80
0	5	3	477	475	4	9	3	74	83	5	14	3	125	115	1	21	3	151	145	0	31	3	126	118
1	5	3	188	183	5	9	3	138	146	6	14	3	81	85	2	21	3	171	204	1	0	4	236	248
2	5	3	67	75	6	9	3	96	85	1	15	3	66	72	3	21	3	75	72	2	0	4	48	48
3	5	3	103	91	0	10	3	240	234	2	15	3	92	77	4	21	3	111	113	3	0	4	185	179
4	5	3	183	169	1	10	3	228	223	3	15	3	159	165	6	21	3	63	44	4	0	4	109	128
5	5	3	83	77	3	10	3	173	170	4	15	3	144	141	0	22	3	99	93	5	0	4	122	131
6	5	3	93	107	4	10	3	284	288	5	15	3	105	126	1	22	3	134	132	1	1	4	197	198
7	5	3	88	76	5	10	3	121	128	2	16	3	91	97	2	22	3	84	103	3	1	4	97	102
0	6	3	49	28	6	10	3	75	61	3	16	3	171	164	3	22	3	148	162	4	1	4	86	97
1	6	3	498	497	7	10	3	73	65	4	16	3	66	70	4	22	3	139	123	5	1	4	62	59
2	6	3	206	197	0	11	3	380	373	5	16	3	140	136	0	23	3	138	139	1	2	4	202	192
3	6	3	83	85	1	11	3	53	54	7	16	3	79	78	1	23	3	99	81	2	2	4	349	340
4	6	3	186	174	2	11	3	301	281	1	17	3	178	191	3	23	3	187	184	3	2	4	247	253
5	6	3	101	102	3	11	3	141	144	2	17	3	68	55	4	23	3	92	95	5	2	4	181	167
6	6	3	102	99	4	11	3	92	78	3	17	3	110	112	1	24	3	78	75	6	2	4	108	108
7	6	3	132	129	5	11	3	131	142	4	17	3	183	189	2	24	3	100	103	0	3	4	96	97
0	7	3	210	212	6	11	3	82	94	6	17	3	67	67	3	24	3	144	151	1	3	4	69	68
1	7	3	54	61	0	12	3	207	193	7	17	3	126	115	0	25	3	78	66	2	3	4	228	213
2	7	3	233	225	1	12	3	122	115	0	18	3	60	69	1	25	3	125	140	3	3	4	99	100
3	7	3	175	181	2	12	3	144	162	2	18	3	172	178	2	25	3	93	114	4	3	4	213	207
4	7	3	160	170	3	12	3	62	68	3	18	3	61	57	0	26	3	211	210	0	4	4	129	118

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	4	4	581	568	5	9	4	216	214	5	16	4	66	65	0	28	4	101	91	5	5	5	115	121
2	4	4	157	147	7	9	4	101	95	0	17	4	206	198	1	29	4	88	68	6	5	5	69	53
3	4	4	214	213	0	10	4	198	192	1	17	4	243	263	0	31	4	83	73	6	5	5	81	77
4	4	4	305	296	1	10	4	172	174	2	17	4	89	93	1	32	4	74	59	0	6	5	175	202
5	4	4	88	88	2	10	4	93	88	3	17	4	94	90	1	33	4	87	83	1	6	5	175	180
0	5	4	137	121	3	10	4	97	95	4	17	4	103	119	1	0	5	188	186	2	6	5	106	113
1	5	4	180	178	4	10	4	167	169	5	17	4	132	134	2	0	5	102	99	3	6	5	200	199
2	5	4	173	167	0	11	4	53	49	0	18	4	218	211	3	0	5	250	246	4	6	5	87	87
3	5	4	89	93	1	11	4	93	97	2	18	4	167	171	4	0	5	114	113	5	6	5	129	128
4	5	4	61	56	2	11	4	186	175	3	18	4	114	129	5	0	5	126	138	0	7	5	173	184
5	5	4	71	73	3	11	4	74	75	6	18	4	85	77	1	1	5	224	233	1	7	5	110	113
6	5	4	71	65	6	11	4	87	90	0	19	4	87	67	2	1	5	203	191	2	7	5	179	185
1	6	4	156	155	0	12	4	61	74	1	19	4	90	78	3	1	5	87	84	3	7	5	107	100
2	6	4	207	197	1	12	4	236	225	2	19	4	70	78	4	1	5	112	99	4	7	5	111	117
4	6	4	105	114	2	12	4	295	307	3	19	4	87	101	5	1	5	104	97	5	7	5	158	151
5	6	4	165	165	3	12	4	226	221	4	19	4	149	168	1	2	5	95	91	6	7	5	86	102
0	7	4	50	50	4	12	4	138	144	2	20	4	144	155	2	2	5	69	63	7	7	5	75	82
1	7	4	120	127	5	12	4	95	88	5	20	4	91	90	4	2	5	85	77	0	8	5	204	210
2	7	4	59	56	1	13	4	132	137	0	21	4	65	56	5	2	5	76	78	1	8	5	66	79
3	7	4	122	120	3	13	4	140	144	1	21	4	134	136	6	2	5	64	56	5	8	5	78	87
4	7	4	134	134	4	13	4	110	113	2	21	4	161	166	0	3	5	58	48	6	8	5	73	73
5	7	4	80	91	5	13	4	140	146	3	21	4	90	97	1	3	5	219	236	0	9	5	56	58
6	7	4	111	110	0	14	4	60	66	2	22	4	233	245	4	3	5	144	150	1	9	5	92	99
0	8	4	226	244	2	14	4	127	137	3	22	4	73	64	5	3	5	103	112	2	9	5	145	137
2	8	4	157	164	3	14	4	256	268	0	23	4	229	228	7	3	5	92	86	3	9	5	93	94
3	8	4	71	72	4	14	4	142	136	1	23	4	91	87	1	4	5	103	103	0	10	5	94	104
4	8	4	70	77	1	15	4	180	181	5	23	4	91	73	2	4	5	56	60	1	10	5	167	173
5	8	4	126	126	4	15	4	134	136	0	24	4	163	162	3	4	5	102	102	2	10	5	230	243
6	8	4	73	74	5	15	4	93	109	1	24	4	212	204	4	4	5	71	80	3	10	5	92	88
0	9	4	272	273	6	15	4	83	67	0	25	4	132	119	5	4	5	163	162	4	10	5	84	83
1	9	4	156	162	1	16	4	161	175	1	25	4	227	212	0	5	5	150	167	5	10	5	64	49
2	9	4	122	127	2	16	4	153	162	5	26	4	72	73	1	5	5	44	32	0	11	5	98	91
3	9	4	124	132	3	16	4	200	208	0	27	4	112	110	2	5	5	65	75	2	11	5	96	93
4	9	4	116	114	4	16	4	84	86	2	27	4	90	86	4	5	5	96	102	4	11	5	163	169

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	12	5	107	123	4	19	5	80	80	2	4	6	170	171	1	13	6	158	162	1	1	7	214	236
2	12	5	67	69	2	20	5	218	234	4	4	6	118	116	2	13	6	80	68	2	1	7	167	161
3	12	5	98	109	0	21	5	114	126	0	5	6	97	79	3	13	6	72	88	2	2	7	103	101
4	12	5	120	133	1	21	5	125	130	1	5	6	193	191	6	13	6	76	40	4	2	7	70	74
5	12	5	142	143	2	21	5	127	126	2	5	6	121	114	1	14	6	106	108	0	3	7	76	92
6	12	5	74	76	3	21	5	111	115	3	5	6	163	166	2	14	6	98	102	1	3	7	63	78
1	13	5	90	90	3	22	5	68	76	0	6	6	97	106	4	14	6	80	83	2	3	7	69	55
3	13	5	86	96	3	23	5	75	73	1	6	6	45	31	1	15	6	86	108	1	4	7	119	119
4	13	5	69	40	0	24	5	96	95	2	6	6	104	101	2	15	6	81	95	1	5	7	165	180
0	14	5	185	192	1	24	5	95	87	3	6	6	221	210	4	15	6	64	53	3	5	7	162	148
1	14	5	157	164	2	24	5	82	104	5	6	6	92	92	2	16	6	129	126	0	6	7	195	203
2	14	5	155	147	1	25	5	44	57	1	7	6	95	93	3	16	6	72	69	2	6	7	86	88
3	14	5	77	76	0	26	5	147	128	2	7	6	87	90	4	16	6	89	92	3	6	7	164	155
4	14	5	68	83	3	26	5	122	121	3	7	6	124	145	0	17	6	62	67	4	6	7	76	55
0	15	5	96	94	0	29	5	71	36	4	7	6	133	132	3	17	6	78	68	0	7	7	181	180
1	15	5	104	105	1	0	6	115	115	0	8	6	65	83	0	18	6	71	59	1	7	7	91	98
2	15	5	105	111	2	0	6	178	195	2	8	6	59	60	2	18	6	69	45	2	7	7	107	102
3	15	5	130	130	3	0	6	92	90	1	9	6	60	60	3	18	6	99	88	3	7	7	101	90
4	15	5	60	58	4	0	6	97	95	3	9	6	143	155	4	18	6	82	84	0	8	7	106	115
1	16	5	122	121	5	0	6	137	128	4	9	6	107	105	0	19	6	105	89	1	8	7	91	85
2	16	5	171	163	1	1	6	132	147	0	10	6	112	114	1	19	6	99	115	2	8	7	69	70
5	16	5	97	106	3	1	6	94	94	1	10	6	93	91	2	19	6	91	98	3	8	7	77	67
0	17	5	78	70	4	1	6	186	167	3	10	6	71	73	3	19	6	61	36	4	8	7	76	62
1	17	5	217	226	6	1	6	67	58	5	10	6	74	93	0	20	6	123	122	5	8	7	70	50
2	17	5	124	121	1	2	6	193	193	1	11	6	34	31	2	21	6	114	106	0	9	7	74	70
3	17	5	113	127	2	2	6	96	91	2	11	6	192	192	0	23	6	103	113	1	9	7	100	109
6	17	5	73	43	3	2	6	173	167	4	11	6	87	83	1	23	6	90	88	2	9	7	91	98
0	18	5	122	124	4	2	6	85	97	5	11	6	92	98	2	23	6	87	74	5	9	7	68	52
1	18	5	156	162	6	2	6	86	77	6	11	6	66	35	0	24	6	80	71	0	10	7	67	57
2	18	5	66	69	1	3	6	184	198	1	12	6	74	85	1	25	6	101	71	1	10	7	67	66
5	18	5	75	91	2	3	6	57	58	2	12	6	154	164	3	25	6	74	35	2	10	7	106	98
6	18	5	86	80	3	3	6	73	66	3	12	6	134	133	1	26	6	75	64	4	10	7	109	90
0	19	5	192	203	0	4	6	124	141	4	12	6	77	79	1	0	7	123	131	0	11	7	96	92
2	19	5	84	93	1	4	6	173	183	0	13	6	74	91	2	0	7	132	127	1	11	7	89	84

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR STRYCHNINE NITRATE

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	11	7	77	70	3	15	7	93	78	1	21	7	72	53	3	5	8	119	116	3	13	8	66	53
4	11	7	102	88	0	16	7	123	112	2	0	8	63	55	1	6	8	61	77	1	13	8	118	129
0	12	7	92	88	1	16	7	71	62	4	0	8	95	91	3	6	8	88	87	2	13	8	65	61
1	12	7	131	128	0	17	7	68	71	1	1	8	78	76	1	7	8	58	52	1	14	8	63	70
0	13	7	99	106	1	17	7	59	61	2	1	8	88	97	2	7	8	96	100	2	14	8	82	67
2	13	7	59	65	4	17	7	70	70	4	1	8	74	59	0	8	8	62	78	3	14	8	65	50
3	13	7	89	94	2	18	7	73	88	1	2	8	85	96	2	9	8	65	59	0	15	8	69	60
4	13	7	95	90	1	19	7	71	79	2	2	8	60	51	3	9	8	72	66	2	15	8	59	64
0	14	7	99	108	3	19	7	73	73	3	2	8	65	60	4	9	8	63	51	1	16	8	49	52
1	14	7	98	75	2	20	7	60	22	4	2	8	82	70	2	10	8	75	90	3	16	8	71	68
2	14	7	82	75	0	21	7	63	49	1	3	8	48	19	2	12	8	78	80	2	18	8	72	77
2	15	7	62	70																				

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

H	K	L	10FO	10FC
4	0	0	656	681
6	0	0	68	73
8	0	0	50	53
1	1	0	848	840
2	1	0	461	480
3	1	0	99	96
4	1	0	655	652
5	1	0	78	68
7	1	0	92	85
8	1	0	51	48
1	2	0	918	931
2	2	0	730	741
3	2	0	188	184
4	2	0	211	205
5	2	0	67	74
6	2	0	154	156
7	2	0	165	161
9	2	0	143	145
1	3	0	74	74
2	3	0	295	284
3	3	0	159	156
4	3	0	78	77
5	3	0	95	95
6	3	0	183	184
7	3	0	80	92
0	4	0	101	97
1	4	0	270	269
2	4	0	371	392
3	4	0	243	244
4	4	0	36	37
6	4	0	118	117
1	5	0	889	871
3	5	0	241	247
4	5	0	69	72
6	5	0	95	89
7	5	0	85	83
8	5	0	63	72
0	6	0	767	706
1	6	0	334	331
2	6	0	419	410
5	6	0	72	64
6	6	0	70	61
1	7	0	170	161
2	7	0	168	176
3	7	0	226	220
4	7	0	94	89
5	7	0	98	96
1	1	1	991	1003
2	1	1	1012	1033
3	1	1	226	235
4	1	1	300	301
5	1	1	429	423
6	1	1	103	107
7	1	1	138	151
0	2	1	512	514
1	2	1	204	207
2	2	1	906	912
3	2	1	382	381
4	2	1	563	571
5	2	1	180	182
6	2	1	169	179
7	2	1	204	212
8	2	1	58	72
9	2	1	94	92
0	3	1	431	433
1	3	1	117	119
2	3	1	195	207
3	3	1	326	319
4	3	1	324	317
5	7	1	148	155
6	7	1	66	66
0	8	1	126	124
1	8	1	73	73
2	8	1	162	156
3	8	1	142	147
4	8	1	66	78
5	8	1	90	100
7	8	1	103	103
0	9	1	88	91
1	9	1	109	112
2	9	1	194	192
3	9	1	177	169
4	9	1	138	128
6	9	1	79	82
0	10	1	187	195
1	10	1	126	130
2	10	1	111	110
3	10	1	110	111
4	10	1	83	83
5	10	1	75	76
0	11	1	117	130
2	11	1	203	208
3	11	1	88	87
4	11	1	108	105
5	11	1	100	91
0	12	1	237	256
2	12	1	189	198
3	12	1	47	36
0	13	1	102	97
2	13	1	87	93
4	13	1	110	99
3	0	2	58	62
4	0	2	243	226

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

H	K	L	10FO	10FC
5	0	2	338	337
6	0	2	172	185
8	0	2	89	99
0	1	2	306	340
1	1	2	302	302
2	1	2	1231	1250
3	1	2	457	449
4	1	2	436	436
7	1	2	56	61
1	2	2	189	212
2	2	2	239	252
3	2	2	387	389
4	2	2	460	450
5	2	2	89	87
6	2	2	113	116
7	2	2	106	101
9	2	2	89	94
0	3	2	1278	1386
1	3	2	473	462
2	3	2	110	118
3	3	2	114	110
4	3	2	214	220
6	3	2	111	103
7	3	2	61	57
8	3	2	57	57
0	4	2	505	498
1	4	2	501	493
2	4	2	235	225
3	4	2	195	195
4	4	2	211	205
5	4	2	114	114
7	4	2	87	92
0	5	2	321	292
1	5	2	288	280
2	5	2	267	263
3	5	2	263	257
4	5	2	124	126
5	5	2	155	151
6	5	2	135	131
7	5	2	112	120
8	5	2	62	66
0	6	2	504	477
1	6	2	235	240
2	6	2	303	289
3	6	2	108	107
4	6	2	82	81
5	6	2	159	159
6	6	2	85	85
7	6	2	96	93
0	7	2	200	185
1	7	2	145	138
2	7	2	79	69
3	7	2	153	148
4	7	2	52	34
5	7	2	117	111
6	7	2	61	67
0	8	2	219	216
1	8	2	276	273
2	8	2	242	245
3	8	2	136	124
4	8	2	117	121
5	8	2	67	62
7	8	2	70	58
0	9	2	66	57
1	9	2	152	164
2	9	2	119	120
3	9	2	240	238
4	9	2	42	33
5	9	2	190	190
7	9	2	65	56
0	10	2	120	127
1	10	2	49	55
2	10	2	104	104
3	10	2	93	94
4	10	2	86	84
5	11	2	74	78
6	11	2	56	49
0	12	2	67	77
2	12	2	79	70
3	12	2	112	109
5	12	2	73	66
1	13	2	57	57
1	14	2	66	60
2	0	3	610	625
3	0	3	405	390
4	0	3	41	42
5	0	3	68	64
6	0	3	116	121
8	0	3	86	88
1	1	3	965	979
2	1	3	425	431
3	1	3	189	184
4	1	3	259	264
5	1	3	170	170
6	1	3	153	159
7	1	3	227	232
8	1	3	81	70
0	2	3	463	504
1	2	3	494	491
2	2	3	126	128
3	2	3	208	208
4	2	3	222	209
5	2	3	175	169
6	2	3	216	222
7	2	3	149	136
0	3	3	1149	1161
1	3	3	594	606
2	3	3	197	196
3	3	3	318	321
4	3	3	326	302
5	3	3	156	151
6	3	3	133	129
7	3	3	142	137
8	3	3	187	186
0	4	3	203	206
1	4	3	180	190
2	4	3	137	142
3	4	3	137	134
4	4	3	92	94
5	4	3	108	107
6	4	3	211	299
8	4	3	110	107
0	5	3	356	365
1	5	3	218	213
2	5	3	230	211
3	5	3	72	81
4	5	3	178	181
6	5	3	86	83
0	6	3	488	489
1	6	3	287	291
2	6	3	84	81
3	6	3	70	67
4	6	3	62	62
5	6	3	78	64
6	6	3	78	65
0	7	3	93	71
0	7	3	243	244
1	7	3	70	70
2	7	3	73	67
3	7	3	190	188
4	7	3	135	138
5	7	3	129	133
7	7	3	67	60
0	8	3	164	154
1	8	3	113	108
2	8	3	303	316
3	8	3	192	156
4	8	3	207	208
6	8	3	69	75
7	8	3	72	72
0	9	3	180	184
1	9	3	150	146
2	9	3	94	87
3	9	3	102	99
4	9	3	70	58
5	9	3	63	64
0	10	3	138	136
1	10	3	59	49
2	10	3	121	125
4	10	3	139	134
6	10	3	86	77
0	11	3	74	82
1	11	3	145	138
2	11	3	60	73
3	11	3	96	96
4	11	3	92	99
5	11	3	60	62

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

H	K	L	10FO	10FC
0	12	3	71	73
3	12	3	96	101
0	13	3	73	84
1	13	3	69	58
2	13	3	112	100
4	13	3	70	72
0	0	4	256	265
1	0	4	462	459
2	0	4	622	620
3	0	4	537	533
4	0	4	98	100
5	0	4	328	331
6	0	4	104	101
7	0	4	247	263
0	1	4	520	520
1	1	4	386	378
2	1	4	818	814
3	1	4	294	262
4	1	4	133	142
5	1	4	165	166
6	1	4	142	135
7	1	4	152	151
0	2	4	464	463
1	2	4	165	157
2	2	4	940	973
3	2	4	221	224
4	2	4	267	265
5	2	4	100	100
6	2	4	87	80
0	3	4	442	419
1	3	4	177	167
2	3	4	512	503
3	3	4	201	190
4	3	4	169	166
5	3	4	68	62
6	3	4	88	69
7	3	4	58	61
0	4	4	210	232
1	4	4	421	423
2	4	4	80	82
3	4	4	136	139
4	4	4	57	54
5	4	4	140	149
6	4	4	170	159
7	4	4	474	476
2	5	4	350	353
3	5	4	303	298
4	5	4	127	126
5	5	4	93	97
7	5	4	127	136
8	5	4	53	44
0	6	4	406	396
1	6	4	138	149
2	6	4	211	207
3	6	4	51	61
4	6	4	62	47
5	6	4	153	158
6	6	4	86	77
0	7	4	56	60
1	7	4	169	153
2	7	4	119	125
3	7	4	183	190
4	7	4	109	115
5	7	4	131	133
7	7	4	279	272
0	8	4	190	189
2	8	4	157	166
3	8	4	.109	103
4	8	4	154	144
5	8	4	58	38
6	8	4	96	98
0	9	4	132	132
1	9	4	197	185
2	9	4	141	150
3	9	4	262	269
4	9	4	154	144
5	9	4	162	161
7	9	4	49	46
0	10	4	100	94
1	10	4	264	251
2	10	4	166	174
3	10	4	152	155
0	11	4	68	67
1	11	4	140	142
2	11	4	49	40
3	11	4	84	90
4	11	4	90	88
0	12	4	87	93
2	12	4	86	86
4	12	4	107	96
0	13	4	85	90
2	13	4	77	77
0	14	4	58	60
1	0	5	168	158
2	0	5	524	513
3	0	5	339	340
4	0	5	236	242
6	0	5	89	85
7	0	5	51	48
0	1	5	602	604
1	1	5	143	144
2	1	5	318	317
3	1	5	342	345
5	1	5	53	48
6	1	5	228	232
0	2	5	252	249
1	2	5	224	211
2	2	5	232	223
4	2	5	227	232
6	2	5	156	163
7	2	5	44	40
0	3	5	908	925
1	3	5	331	331
3	3	5	192	198
4	3	5	302	300
5	3	5	174	166
6	3	5	181	186
0	4	5	192	187
1	4	5	938	934
2	4	5	303	289
3	4	5	350	348
4	4	5	277	276
5	4	5	174	177
0	5	5	490	487
1	5	5	168	166
2	5	5	131	136
3	5	5	155	149
4	5	5	184	185
5	5	5	101	101
0	6	5	107	104
1	6	5	437	430
2	6	5	83	84
3	6	5	231	233
4	6	5	63	66
5	6	5	90	101
6	6	5	120	117
0	7	5	80	76
1	7	5	243	238
2	7	5	137	133
3	7	5	224	220
4	7	5	69	61
5	7	5	130	136
6	7	5	46	50
7	7	5	64	66
8	7	5	116	113
0	8	5	92	89
1	8	5	55	58
3	8	5	83	78
4	8	5	167	165
6	8	5	82	95
1	9	5	116	125
2	9	5	189	191
3	9	5	130	125
5	9	5	100	102
6	9	5	65	64
0	10	5	229	225
1	10	5	194	197
2	10	5	230	227
3	10	5	84	79
4	10	5	73	73
6	10	5	35	30
0	11	5	63	69
1	11	5	90	96
2	11	5	96	98
3	11	5	120	123
4	11	5	95	100
6	11	5	48	48
0	12	5	73	75
4	12	5	67	56
1	13	5	51	40

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

H	K	L	10FO	10FC
0	0	6	704	714
1	0	6	302	309
2	0	6	127	127
4	0	6	443	436
5	0	6	154	162
6	0	6	163	163
7	0	6	101	103
8	0	6	118	118
0	1	6	142	133
1	1	6	407	406
2	1	6	162	163
3	1	6	89	86
4	1	6	305	309
5	1	6	136	138
6	1	6	122	121
0	2	6	612	605
1	2	6	155	160
2	2	6	364	363
3	2	6	162	164
4	2	6	72	74
5	2	6	61	60
0	3	6	269	298
1	3	6	422	407
2	3	6	219	215
3	3	6	146	143
4	3	6	133	128
5	3	6	108	107
6	3	6	57	64
0	4	6	169	188
1	4	6	62	54
2	4	6	234	234
3	4	6	101	100
4	4	6	131	131
5	4	6	92	92
6	4	6	74	68
7	4	6	65	64
8	4	6	68	75
0	5	6	466	452
1	5	6	254	253
2	5	6	203	204
3	5	6	99	107
4	5	6	178	180
5	5	6	91	91
6	5	6	78	78
7	5	6	77	77
8	5	6	63	53
0	6	6	75	78
1	6	6	69	76
2	6	6	145	143
3	6	6	169	168
4	6	6	182	181
5	6	6	61	53
6	6	6	110	105
7	6	6	68	67
8	6	6	83	84
1	8	6	60	64
2	8	6	94	89
4	8	6	91	91
5	8	6	135	134
7	8	6	49	42
0	9	6	59	59
1	9	6	206	211
2	9	6	194	203
3	9	6	118	122
4	9	6	112	119
6	9	6	72	68
7	9	6	85	63
0	10	6	77	70
1	10	6	105	98
2	10	6	126	136
3	10	6	139	143
4	10	6	120	122
5	10	6	49	54
6	10	6	124	126
0	11	6	75	69
1	12	6	58	35
2	13	6	47	53
3	13	6	91	59
1	0	7	187	203
2	0	7	64	69
3	0	7	398	393
4	0	7	60	60
5	0	7	110	106
6	0	7	49	55
0	1	7	84	82
1	1	7	604	595
2	1	7	355	349
3	1	7	249	243
4	1	7	133	139
5	1	7	161	169
6	1	7	102	107
7	1	7	114	111
0	2	7	382	367
1	2	7	106	103
2	2	7	81	78
3	2	7	381	364
4	2	7	71	69
5	2	7	104	93
6	2	7	55	54
7	2	7	52	46
0	3	7	372	392
1	3	7	368	368
2	3	7	344	338
3	3	7	340	333
4	3	7	114	118
5	3	7	114	119
6	3	7	79	69
0	4	7	177	188
1	4	7	281	284
2	4	7	137	137
3	4	7	363	354
4	4	7	38	40
5	4	7	188	188
6	4	7	101	101
7	4	7	93	89
0	5	7	70	62
1	5	7	213	229
2	5	7	210	196
3	5	7	210	201
4	5	7	213	209
5	5	7	119	112
6	5	7	146	143
7	5	7	141	135
0	6	7	161	158
1	6	7	344	326
2	6	7	95	94
3	6	7	178	179
4	6	7	118	120
6	6	7	142	149
8	6	7	69	67
0	7	7	38	31
1	7	7	173	176
2	7	7	63	58
3	7	7	94	98
4	7	7	272	272
5	7	7	65	60
6	7	7	125	121
0	8	7	87	87
1	8	7	127	129
2	8	7	95	90
3	8	7	136	136
4	8	7	115	109
5	8	7	101	98
0	9	7	80	78
1	9	7	73	72
2	9	7	134	140
4	9	7	62	57
0	10	7	91	91
1	10	7	254	263
3	10	7	169	174
4	10	7	93	94
5	10	7	73	76
0	11	7	126	122
1	11	7	127	129
2	11	7	64	73
0	12	7	81	76
2	12	7	88	94
1	13	7	31	30
0	0	8	41	38
1	0	8	90	22
2	0	8	143	134
3	0	8	113	120

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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	0	8	192	207	4	5	8	185	184	0	11	8	57	61	1	3	9	294	284	5	8	9	117	110
6	0	8	206	207	8	5	8	66	61	1	11	8	165	169	2	3	9	462	437	6	8	9	131	130
7	0	8	109	117	6	6	8	164	161	2	11	8	126	121	3	3	9	239	233	1	9	9	316	308
8	0	8	54	59	0	6	8	119	116	3	11	8	85	90	4	3	9	226	220	2	9	9	96	92
1	1	8	360	356	1	6	8	232	228	4	11	8	90	93	6	3	9	164	158	3	9	9	183	126
2	1	8	92	96	2	6	8	86	82	1	12	8	136	133	0	4	9	461	449	4	9	9	208	207
4	1	8	146	155	3	6	8	169	169	2	12	8	59	49	1	4	9	281	275	5	9	9	81	83
5	1	8	65	71	4	6	8	83	87	3	12	8	100	97	2	4	9	291	284	0	10	9	181	184
6	1	8	88	99	5	6	8	113	117	4	12	8	64	57	3	4	9	63	63	1	10	9	131	126
8	1	8	70	73	6	6	8	151	158	2	13	8	74	76	4	4	9	138	136	2	10	9	183	192
0	2	8	214	205	8	6	8	109	103	3	13	8	80	71	5	4	9	77	75	3	10	9	119	126
1	2	8	216	213	1	7	8	218	217	1	0	9	229	241	1	5	9	225	208	4	11	9	73	66
2	2	8	109	110	2	7	8	63	62	2	0	9	268	284	3	5	9	187	188	5	11	9	120	122
3	2	8	315	307	3	7	8	91	101	3	0	9	181	187	4	5	9	101	93	0	12	9	57	60
4	2	8	120	120	5	7	8	67	65	4	0	9	107	105	7	5	9	85	88	1	12	9	116	112
5	2	8	97	100	6	7	8	112	104	5	0	9	88	92	8	5	9	91	92	2	12	9	64	58
6	2	8	49	52	7	7	8	62	61	8	0	9	59	73	0	6	9	196	171	3	12	9	60	62
0	3	8	188	197	0	8	8	232	233	0	1	9	422	418	1	6	9	372	366	5	13	9	84	87
1	3	8	653	627	1	8	8	90	93	1	1	9	125	124	2	6	9	136	141	0	0	10	193	203
2	3	8	304	308	2	8	8	189	178	2	1	9	169	168	3	6	9	109	110	0	0	10	193	203
3	3	8	321	335	4	8	8	265	269	3	1	9	227	242	4	6	9	173	166	1	0	10	146	149
4	3	8	51	59	5	8	8	82	71	4	1	9	299	296	6	6	9	104	104	2	0	10	348	316
5	3	8	146	148	6	8	8	92	101	5	1	9	155	160	7	6	9	82	80	4	0	10	62	61
0	4	8	165	160	0	9	8	220	227	6	1	9	212	214	1	7	9	310	321	0	1	10	240	230
1	4	8	260	272	1	9	8	198	199	7	1	9	114	114	2	7	9	110	99	1	1	10	383	366
2	4	8	206	209	2	9	8	99	97	8	1	9	82	71	3	7	9	117	122	2	1	10	423	419
3	4	8	251	247	3	9	8	227	232	0	2	9	51	61	4	7	9	69	81	3	1	10	173	177
4	4	8	194	185	5	9	8	64	59	1	2	9	201	249	5	7	9	140	143	4	1	10	35	44
5	4	8	148	140	0	10	8	133	130	2	2	9	210	204	6	7	9	95	89	5	1	10	198	200
6	4	8	83	88	1	10	8	97	94	3	2	9	417	425	0	8	9	92	90	6	1	10	66	66
0	5	8	42	36	2	10	8	83	80	4	2	9	140	132	1	8	9	139	134	7	1	10	82	70
1	5	8	33	34	3	10	8	79	80	5	2	9	104	90	2	8	9	386	387	8	1	10	81	84
2	5	8	416	428	4	10	8	83	76	6	2	9	160	158	3	8	9	192	191	0	2	10	260	260
3	5	8	91	92	5	10	8	103	101	0	3	9	446	442	4	8	9	140	146	1	2	10	297	288

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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	3	12	332	326	3	8	12	167	170	3	2	13	200	202	3	7	13	64	72	4	1	14	208	206
2	3	12	383	380	4	8	12	74	71	4	2	13	198	198	4	7	13	63	67	5	1	14	63	74
3	3	12	274	273	5	8	12	97	93	5	2	13	255	254	5	7	13	75	67	6	1	14	92	103
4	3	12	300	292	1	9	12	233	232	6	2	13	166	172	6	7	13	94	93	0	2	14	231	228
5	3	12	82	82	2	9	12	242	238	0	3	13	165	162	7	7	13	62	59	1	2	14	130	144
6	3	12	147	147	4	9	12	195	191	1	3	13	268	267	0	8	13	100	98	2	2	14	121	117
7	3	12	71	66	5	9	12	97	91	3	3	13	128	132	1	8	13	280	277	3	2	14	167	171
0	4	12	100	92	0	10	12	166	169	4	3	13	102	100	2	8	13	115	110	4	2	14	209	220
1	4	12	196	186	1	10	12	140	137	5	3	13	203	203	3	8	13	152	145	5	2	14	202	201
2	4	12	166	157	2	10	12	176	183	8	3	13	81	70	4	8	13	60	30	6	2	14	82	85
3	4	12	248	239	3	10	12	150	160	0	4	13	50	36	5	8	13	125	128	7	2	14	79	76
5	4	12	89	100	4	10	12	93	89	1	4	13	158	154	6	8	13	58	59	8	2	14	65	50
6	4	12	108	109	5	10	12	86	81	2	4	13	103	107	0	9	13	80	75	0	3	14	117	119
0	5	12	88	83	0	11	12	118	120	3	4	13	51	50	1	9	13	47	53	1	3	14	94	88
1	5	12	211	215	2	11	12	106	109	4	4	13	90	91	2	9	13	231	248	2	3	14	84	85
2	5	12	134	134	1	12	12	110	113	6	4	13	97	99	3	9	13	85	76	4	3	14	201	203
4	5	12	93	78	2	12	12	82	90	7	4	13	88	87	4	9	13	143	141	5	3	14	98	106
5	5	12	83	58	1	0	13	111	116	0	5	13	163	158	6	9	13	114	110	0	4	14	191	182
6	5	12	115	113	2	0	13	257	261	1	5	13	82	75	3	10	13	70	75	1	4	14	281	263
0	6	12	74	75	3	0	13	103	104	2	5	13	102	101	4	10	13	62	61	2	4	14	69	66
1	6	12	276	275	4	0	13	122	131	3	5	13	93	95	0	11	13	94	103	3	4	14	62	54
2	6	12	258	270	5	0	13	48	53	4	5	13	48	44	2	11	13	86	89	4	4	14	121	112
4	6	12	124	131	6	0	13	89	91	5	5	13	125	130	3	11	13	70	68	6	4	14	64	56
0	7	12	369	355	0	1	13	239	251	6	5	13	92	92	3	12	13	57	46	7	4	14	61	63
1	7	12	182	183	1	1	13	434	438	7	5	13	57	59	0	0	14	200	187	1	5	14	123	118
2	7	12	310	308	2	1	13	312	312	0	6	13	175	180	1	0	14	66	71	2	5	14	169	171
3	7	12	155	158	3	1	13	48	50	1	6	13	147	142	2	0	14	241	235	3	5	14	112	109
4	7	12	182	183	4	1	13	101	89	2	6	13	75	91	3	0	14	40	33	4	5	14	100	99
5	7	12	70	76	5	1	13	133	131	3	6	13	104	115	4	0	14	70	66	6	5	14	88	80
6	7	12	104	104	6	1	13	63	54	5	6	13	80	79	5	0	14	174	170	1	6	14	119	121
7	7	12	76	78	7	1	13	77	79	7	6	13	57	53	0	1	14	62	148	2	6	14	58	59
0	8	12	140	142	0	2	13	106	99	0	7	13	43	56	1	1	14	100	110	3	6	14	144	145
1	8	12	56	53	1	2	13	230	229	1	7	13	151	157	2	1	14	174	157	5	6	14	89	89
2	8	12	157	157	2	2	13	178	181	2	7	13	89	96	3	1	14	90	90	6	6	14	64	74

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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	2	10	600	580	7	6	10	108	104	4	0	11	217	211	1	5	11	133	134	2	10	11	74	84
3	2	10	104	101	0	7	10	122	127	5	0	11	191	197	2	5	11	137	133	3	10	11	81	82
4	2	10	50	52	1	7	10	112	112	6	0	11	103	107	3	5	11	195	181	4	10	11	99	103
5	2	10	142	147	2	7	10	39	40	0	1	11	426	408	4	5	11	94	47	5	10	11	81	87
7	2	10	62	80	3	7	10	67	69	1	1	11	143	135	5	5	11	177	168	0	11	11	139	133
0	3	10	413	397	4	7	10	137	141	2	1	11	289	287	7	5	11	73	66	1	11	11	104	105
1	3	10	542	510	6	7	10	179	176	3	1	11	92	95	0	6	11	261	272	3	11	11	131	117
2	3	10	111	111	7	7	10	70	78	4	1	11	285	278	1	6	11	62	67	5	11	11	105	95
3	3	10	225	231	0	8	10	269	270	5	1	11	223	230	2	6	11	148	151	0	12	11	104	111
4	3	10	159	153	1	8	10	70	74	6	1	11	251	256	4	6	11	133	130	1	12	11	81	80
5	3	10	73	84	2	8	10	74	68	7	1	11	49	57	5	6	11	49	43	2	12	11	77	79
6	3	10	85	82	3	8	10	97	97	8	1	11	111	110	6	6	11	167	168	3	12	11	80	79
7	3	10	43	41	4	8	10	94	83	0	2	11	295	271	0	7	11	142	139	0	0	12	852	801
0	4	10	362	336	6	8	10	109	114	1	2	11	299	299	1	7	11	197	190	2	0	12	59	58
1	4	10	226	233	7	8	10	77	75	2	2	11	455	434	2	7	11	186	190	3	0	12	61	62
2	4	10	219	220	0	9	10	124	123	3	2	11	125	119	3	7	11	233	235	5	0	12	112	111
3	4	10	218	203	1	9	10	367	369	4	2	11	429	431	4	7	11	128	124	6	0	12	65	52
4	4	10	104	101	2	9	10	174	174	5	2	11	102	107	5	7	11	215	210	7	0	12	47	45
5	4	10	196	201	3	9	10	119	124	6	2	11	166	168	6	7	11	93	82	8	0	12	74	86
0	5	10	133	136	4	9	10	91	93	0	3	11	255	250	7	7	11	77	87	1	1	12	878	868
1	5	10	170	151	0	10	10	298	292	1	3	11	39	43	0	8	11	229	228	2	1	12	327	318
2	5	10	94	94	1	10	10	155	151	2	3	11	334	311	1	8	11	365	357	3	1	12	285	287
3	5	10	110	111	2	10	10	129	127	3	3	11	166	168	2	8	11	251	252	4	1	12	202	200
4	5	10	104	105	3	10	10	142	144	4	3	11	327	321	3	8	11	335	346	5	1	12	85	93
5	5	10	84	93	5	10	10	98	98	5	3	11	80	79	4	8	11	261	270	6	1	12	126	126
6	5	10	89	88	0	11	10	112	115	6	3	11	46	36	5	8	11	94	92	7	1	12	56	57
8	5	10	76	74	1	11	10	70	66	7	3	11	60	56	6	8	11	92	84	0	2	12	368	353
0	6	10	129	129	3	11	10	66	62	1	4	11	256	244	0	9	11	238	233	1	2	12	148	152
1	6	10	90	90	4	11	10	123	132	2	4	11	79	83	1	9	11	137	139	2	2	12	194	187
2	6	10	84	92	0	12	10	76	77	3	4	11	176	174	2	9	11	164	166	3	2	12	106	104
3	6	10	106	102	4	12	10	90	84	4	4	11	122	122	3	9	11	120	119	4	2	12	162	162
4	6	10	200	194	1	0	11	194	172	5	4	11	104	102	4	9	11	195	189	6	2	12	188	190
5	6	10	137	133	2	0	11	440	430	6	4	11	58	63	6	9	11	92	82	7	2	12	56	47
6	6	10	136	144	3	0	11	131	127	0	5	11	117	113	0	10	11	162	162	0	3	12	326	308

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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
0	7	14	92	92	0	2	15	84	97	5	7	15	75	80	0	4	16	275	264	3	0	17	131	128
1	7	14	106	105	1	2	15	350	352	6	7	15	85	70	1	4	16	86	98	4	0	17	136	137
2	7	14	63	58	2	2	15	112	112	1	8	15	185	177	2	4	16	212	209	6	0	17	87	89
3	7	14	115	109	3	2	15	283	268	2	8	15	107	107	3	4	16	103	102	7	0	17	41	49
4	7	14	82	79	4	2	15	86	82	3	8	15	152	154	4	4	16	102	99	0	1	17	249	255
6	7	14	78	56	5	2	15	202	204	1	9	15	226	220	5	4	16	185	191	1	1	17	91	90
0	8	14	171	166	6	2	15	56	51	2	9	15	85	87	0	5	16	39	48	2	1	17	123	113
1	8	14	79	94	0	3	15	178	175	3	9	15	193	199	1	5	16	100	105	3	1	17	196	211
2	8	14	181	182	1	3	15	134	129	5	9	15	72	76	2	5	16	104	104	4	1	17	48	62
3	8	14	183	179	2	3	15	57	53	1	11	15	85	79	3	5	16	76	66	7	1	17	52	35
4	8	14	67	60	3	3	15	180	178	2	11	15	46	60	4	5	16	64	66	2	2	17	188	188
0	9	14	126	132	4	3	15	132	131	1	0	16	180	163	6	5	16	55	48	3	2	17	137	140
1	9	14	172	174	5	3	15	147	146	2	0	16	38	49	0	6	16	192	198	4	2	17	60	70
2	9	14	101	102	6	3	15	108	110	3	0	16	220	219	1	6	16	220	210	6	2	17	107	107
3	9	14	99	99	0	4	15	200	188	4	0	16	59	69	2	6	16	92	96	0	3	17	134	133
4	9	14	112	107	1	4	15	152	155	0	1	16	93	102	3	6	16	172	168	1	3	17	56	52
0	10	14	79	84	2	4	15	152	159	1	1	16	85	77	4	6	16	104	102	2	3	17	88	92
1	10	14	47	41	3	4	15	139	140	2	1	16	165	159	0	7	16	142	130	3	3	17	161	161
2	10	14	131	124	4	4	15	77	88	3	1	16	119	123	1	7	16	182	189	4	3	17	104	94
3	10	14	121	118	5	4	15	82	89	0	2	16	46	40	2	7	16	44	54	5	3	17	99	104
1	11	14	58	66	6	4	15	87	80	1	2	16	69	68	3	7	16	183	182	7	3	17	75	66
3	11	14	54	57	0	5	15	291	291	2	2	16	278	261	4	7	16	66	57	0	4	17	77	73
1	0	15	320	313	1	5	15	243	245	3	2	16	253	252	5	7	16	138	136	1	4	17	257	255
2	0	15	53	54	2	5	15	104	115	5	2	16	178	182	0	8	16	82	87	3	4	17	121	119
3	0	15	125	123	3	5	15	114	111	6	2	16	75	82	1	8	16	154	161	0	5	17	87	86
5	0	15	70	71	5	5	15	78	70	7	2	16	64	66	2	8	16	140	147	1	5	17	41	46
6	0	15	80	74	1	6	15	132	128	0	3	16	62	68	3	8	16	59	64	2	5	17	124	129
0	1	15	329	304	2	6	15	168	176	1	3	16	159	155	4	8	16	82	77	3	5	17	144	148
1	1	15	469	465	3	6	15	132	142	2	3	16	70	77	1	9	16	66	73	4	5	17	82	83
2	1	15	191	180	0	7	15	121	118	3	3	16	179	180	1	10	16	48	46	5	5	17	73	73
3	1	15	89	85	1	7	15	271	270	4	3	16	79	77	1	11	16	114	109	1	6	17	107	99
4	1	15	112	113	2	7	15	161	164	5	3	16	95	92	2	11	16	91	83	2	6	17	102	104
5	1	15	90	102	3	7	15	144	154	6	3	16	72	75	1	12	16	127	110	3	6	17	124	123
6	1	15	66	61	4	7	15	99	84	7	3	16	91	89	1	0	17	289	280	4	6	17	124	119

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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
0	7	17	230	232	0	3	18	167	150	2	10	18	77	67	6	6	19	92	98	3	3	20	201	206
2	7	17	144	142	1	3	18	203	204	1	0	19	61	70	0	7	19	115	117	4	3	20	176	183
3	7	17	64	59	2	3	18	90	91	2	0	19	58	54	2	7	19	87	78	0	4	20	100	106
5	7	17	78	68	3	3	18	133	130	3	0	19	116	114	3	7	19	91	81	1	4	20	118	121
1	8	17	88	66	4	3	18	88	85	5	0	19	240	235	0	8	19	117	123	2	4	20	83	95
2	8	17	122	121	5	3	18	80	81	6	0	19	119	121	1	8	19	99	96	3	4	20	81	78
3	8	17	54	44	6	3	18	90	96	7	0	19	103	106	2	8	19	79	80	0	5	20	357	359
4	8	17	131	133	7	3	18	109	107	1	1	19	132	126	3	8	19	48	55	1	5	20	178	188
5	8	17	45	41	0	4	18	90	95	3	1	19	58	49	3	9	19	104	96	2	5	20	224	228
1	9	17	64	57	1	4	18	49	96	4	1	19	168	171	0	10	19	137	137	3	5	20	170	170
2	9	17	129	124	2	4	18	115	120	5	1	19	62	62	1	10	19	84	77	0	6	20	67	79
3	9	17	90	90	3	4	18	164	161	6	1	19	104	97	0	11	19	111	110	2	6	20	89	99
4	9	17	109	105	4	4	18	55	49	2	2	19	219	232	1	11	19	52	46	5	6	20	52	62
0	10	17	111	103	5	4	18	72	73	3	2	19	167	163	0	0	20	300	302	1	7	20	92	95
1	10	17	101	99	0	5	18	273	281	4	2	19	148	149	1	0	20	466	490	3	7	20	101	113
2	10	17	56	52	1	5	18	103	99	6	2	19	71	69	2	0	20	237	220	4	7	20	109	103
0	11	17	70	65	2	5	18	206	206	0	3	19	71	79	3	0	20	81	75	0	8	20	89	77
2	11	17	84	64	3	5	18	75	75	1	3	19	61	53	5	0	20	143	139	1	8	20	121	131
1	0	18	115	108	1	6	18	237	227	2	3	19	149	147	6	0	20	85	83	2	8	20	64	70
2	0	18	77	75	2	6	18	90	82	3	3	19	104	107	0	1	20	165	165	3	8	20	113	115
3	0	18	70	78	3	6	18	67	67	5	3	19	166	169	1	1	20	313	317	4	8	20	61	60
4	0	18	180	172	4	6	18	39	48	0	4	19	71	82	2	1	20	208	216	0	9	20	82	92
6	0	18	91	83	0	7	18	57	46	1	4	19	298	306	3	1	20	75	80	1	9	20	79	76
1	1	18	75	78	1	7	18	168	175	2	4	19	267	269	5	1	20	46	47	1	10	20	93	90
3	1	18	196	210	2	7	18	169	169	3	4	19	156	142	0	2	20	524	531	2	10	20	64	59
4	1	18	77	72	3	7	18	132	130	4	4	19	137	144	1	2	20	48	48	1	0	21	81	79
5	1	18	79	75	4	7	18	156	160	6	4	19	95	79	2	2	20	387	379	2	0	21	83	77
7	1	18	102	94	5	7	18	95	90	0	5	19	219	222	3	2	20	86	90	3	0	21	226	223
0	2	18	173	170	4	8	18	50	50	2	5	19	92	99	4	2	20	187	183	5	0	21	130	124
1	2	18	126	130	5	8	18	96	91	3	5	19	157	150	5	2	20	58	64	6	0	21	94	93
2	2	18	143	148	0	9	18	72	80	4	5	19	129	125	6	2	20	39	73	1	1	21	96	89
4	2	18	79	70	1	9	18	69	74	5	5	19	72	68	0	3	20	261	272	2	1	21	195	203
5	2	18	113	107	2	9	18	59	58	0	6	19	101	103	1	3	20	95	98	3	1	21	172	176
7	2	18	90	87	1	10	18	66	65	1	6	19	304	307	2	3	20	200	197	4	1	21	73	73

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Brucine Free Base Ethanol/water solvate

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	1	21	104	97	2	7	21	48	54	3	7	22	99	87	3	8	23	71	59	1	0	26	84	89
6	1	21	62	57	4	8	21	75	74	1	8	22	96	100	0	0	24	344	361	4	0	26	47	37
1	2	21	233	225	3	9	21	88	89	1	0	23	88	84	2	0	24	240	248	0	2	26	72	69
2	2	21	116	127	0	0	22	132	127	3	0	23	81	74	3	0	24	84	91	1	2	26	45	43
3	2	21	281	277	1	0	22	88	81	0	1	23	264	277	4	0	24	108	109	2	2	26	65	52
4	2	21	152	161	2	0	22	105	104	1	1	23	73	76	0	1	24	87	98	0	3	26	102	114
5	2	21	140	140	3	0	22	219	230	2	1	23	188	192	1	1	24	88	85	0	4	26	46	57
6	2	21	90	97	0	1	22	288	301	4	1	23	75	82	2	1	24	80	90	3	4	26	51	44
1	3	21	131	134	1	1	22	248	247	5	1	23	114	115	4	1	24	81	85	2	0	27	75	67
2	3	21	161	157	2	1	22	243	242	0	2	23	133	135	5	1	24	58	55	0	1	27	58	40
4	3	21	147	148	4	1	22	232	224	1	2	23	162	149	2	2	24	85	88	1	1	27	125	117
5	3	21	104	105	0	2	22	137	150	2	2	23	177	179	3	2	24	59	51	1	2	27	97	86
6	3	21	82	78	1	2	22	84	81	3	2	23	80	78	5	2	24	75	64	3	2	27	74	69
0	4	21	80	66	2	2	22	159	152	5	2	23	95	76	2	3	24	49	62	0	3	27	72	75
1	4	21	178	175	3	2	22	149	157	0	3	23	106	126	3	3	24	122	120	2	3	27	81	77
2	4	21	123	129	4	2	22	57	63	2	3	23	174	168	4	3	24	97	102	3	3	27	46	41
3	4	21	162	171	2	3	22	303	306	3	3	23	151	146	4	4	24	65	60	2	4	27	81	60
4	4	21	76	71	3	3	22	103	95	4	3	23	103	110	0	5	24	50	50	2	5	27	68	62
6	4	21	62	72	4	3	22	150	146	5	3	23	68	60	0	7	24	106	104	0	0	28	73	69
0	5	21	144	149	5	3	22	92	89	3	4	23	82	84	1	8	24	57	57	1	0	28	191	152
1	5	21	99	91	0	4	22	153	158	4	4	23	108	100	1	0	25	126	125	3	0	28	59	64
2	5	21	158	169	1	4	22	91	86	0	5	23	116	114	3	0	25	79	73	2	1	28	97	49
3	5	21	95	90	2	4	22	103	111	1	5	23	85	70	1	1	25	69	87	3	1	28	57	56
4	5	21	61	62	3	4	22	69	73	2	5	23	69	72	2	1	25	48	54	1	2	28	67	73
0	6	21	201	196	1	5	22	153	163	4	5	23	91	91	0	2	25	114	115	1	3	28	55	36
1	6	21	139	140	2	5	22	53	58	0	6	23	120	121	2	2	25	62	57	2	3	28	66	64
3	6	21	82	76	4	5	22	47	40	1	6	23	53	69	4	2	25	76	68	0	1	29	148	148
4	6	21	104	105	5	5	22	70	58	3	6	23	81	88	1	4	25	49	44	1	1	29	57	52
5	6	21	73	76	2	6	22	86	85	4	6	23	66	67	4	4	25	62	61	2	1	29	87	83
0	7	21	115	110	4	6	22	59	43	0	7	23	54	50	3	5	25	49	21	1	3	29	59	60
1	7	21	79	81	2	7	22	109	110	2	8	23	56	61	3	6	25	51	49					

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine Fixed water

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	87	89	2	3	0	204	159	6	6	0	64	62	1	1	1	537	484	-13	3	1	71	72
3	0	0	708	643	3	3	0	89	76	7	6	0	43	52	2	1	1	267	297	-10	3	1	111	118
4	0	0	298	311	4	3	0	296	278	9	6	0	57	47	3	1	1	402	418	-9	3	1	87	78
5	0	0	139	139	5	3	0	76	101	1	7	0	72	68	5	1	1	425	431	-7	3	1	326	270
7	0	0	304	334	6	3	0	139	123	2	7	0	72	68	5	1	1	203	188	-6	3	1	266	287
8	0	0	92	92	7	3	0	144	146	1	8	0	52	42	6	1	1	299	297	-5	3	1	187	206
9	0	0	130	113	8	3	0	64	70	-11	0	1	65	49	7	1	1	61	73	-4	3	1	62	70
10	0	0	147	143	9	3	0	147	132	-10	0	1	165	165	8	1	1	198	215	-3	3	1	185	203
12	0	0	72	69	10	3	0	67	73	-8	0	1	336	342	9	1	1	43	41	-2	3	1	72	73
1	1	0	889	780	11	3	0	51	54	-6	0	1	227	216	10	1	1	83	91	-1	3	1	199	213
2	1	0	257	318	0	4	0	598	575	-5	0	1	100	98	11	1	1	77	80	0	3	1	320	329
3	1	0	439	427	1	4	0	288	251	-4	0	1	222	189	-12	2	1	90	89	1	3	1	208	214
4	1	0	184	183	2	4	0	39	30	-3	0	1	205	273	-11	2	1	74	84	2	3	1	226	228
5	1	0	105	105	3	4	0	375	395	-2	0	1	561	474	-9	2	1	87	98	3	3	1	101	100
6	1	0	170	174	4	4	0	257	288	2	0	1	65	71	-8	2	1	106	107	4	3	1	93	110
7	1	0	206	198	5	4	0	109	98	3	0	1	65	59	-7	2	1	95	104	5	3	1	89	101
8	1	0	169	181	6	4	0	127	149	4	0	1	111	109	-6	2	1	268	281	6	3	1	103	96
9	1	0	136	117	7	4	0	122	123	5	0	1	173	153	-5	2	1	58	62	8	3	1	65	64
10	1	0	78	84	8	4	0	61	50	7	0	1	328	309	-4	2	1	437	379	9	3	1	105	99
11	1	0	68	62	10	4	0	54	54	8	0	1	163	163	-3	2	1	352	300	-10	4	1	55	56
13	1	0	61	58	11	4	0	47	65	11	0	1	67	60	-2	2	1	584	571	-9	4	1	67	76
0	2	0	1945	1896	13	4	0	51	39	12	0	1	101	111	-1	2	1	908	951	-8	4	1	116	135
1	2	0	300	280	1	5	0	155	168	-11	1	1	60	45	0	2	1	991	688	-7	4	1	73	66
2	2	0	823	843	2	5	0	124	117	-10	1	1	129	116	1	2	1	462	471	-6	4	1	209	203
3	2	0	748	687	3	5	0	53	40	-9	1	1	77	80	2	2	1	410	391	-5	4	1	100	101
4	2	0	632	597	4	5	0	82	90	-8	1	1	167	152	3	2	1	291	282	-4	4	1	200	206
5	2	0	231	191	5	5	0	49	54	-7	1	1	231	232	4	2	1	224	189	-3	4	1	160	173
7	2	0	213	202	8	5	0	64	70	-6	1	1	65	72	5	2	1	78	83	-2	4	1	192	192
9	2	0	75	70	9	5	0	69	61	-5	1	1	102	96	6	2	1	144	159	-1	4	1	367	371
10	2	0	67	71	0	6	0	93	84	-4	1	1	484	467	7	2	1	212	196	0	4	1	168	180
11	2	0	66	59	1	6	0	126	133	-3	1	1	677	669	9	2	1	79	71	1	4	1	174	202
12	2	0	61	53	2	6	0	85	88	-2	1	1	750	893	10	2	1	67	54	2	4	1	91	109
13	2	0	70	62	3	6	0	115	113	-1	1	1	490	632	12	2	1	98	104	3	4	1	204	212
1	3	0	101	110	5	6	0	47	39	0	1	1	572	676	13	2	1	76	71	4	4	1	149	136

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine Fixed water

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	4	1	94	104	-1	8	1	46	47	0	1	2	564	536	8	2	2	68	72	-1	4	2	136	122
6	4	1	114	122	-16	0	2	67	58	1	1	2	277	251	10	2	2	89	85	0	4	2	170	171
9	4	1	46	71	-12	0	2	57	40	2	1	2	474	374	11	2	2	101	100	1	4	2	85	79
12	4	1	49	48	-10	0	2	78	86	3	1	2	88	89	12	2	2	71	80	2	4	2	151	139
-9	5	1	143	148	-9	0	2	137	150	4	1	2	343	278	-13	3	2	76	74	3	4	2	148	129
-8	5	1	111	116	-8	0	2	170	163	5	1	2	219	227	-12	3	2	66	71	5	4	2	72	66
-7	5	1	111	97	-7	0	2	217	195	6	1	2	198	152	-11	3	2	71	80	6	4	2	99	104
-5	5	1	93	77	-6	0	2	123	100	7	1	2	191	175	-10	3	2	49	56	7	4	2	52	42
-3	5	1	137	128	-5	0	2	126	139	8	1	2	111	118	-9	3	2	132	147	9	4	2	52	58
-2	5	1	71	75	-4	0	2	516	461	9	1	2	129	134	-8	3	2	196	196	12	4	2	68	73
-1	5	1	46	42	-3	0	2	64	77	10	1	2	67	66	-7	3	2	137	128	-9	5	2	165	178
0	5	1	150	136	-2	0	2	293	330	11	1	2	51	53	-6	3	2	351	352	-8	5	2	181	163
1	5	1	99	94	-1	0	2	486	373	12	1	2	64	64	-5	3	2	187	206	-7	5	2	44	53
2	5	1	54	78	1	0	2	68	54	-14	2	2	68	54	-4	3	2	191	188	-6	5	2	77	87
3	5	1	79	82	2	0	2	632	617	-13	2	2	70	69	-3	3	2	309	289	-5	5	2	99	103
6	5	1	105	104	3	0	2	287	233	-11	2	2	70	55	-2	3	2	178	183	0	5	2	49	55
7	5	1	52	57	5	0	2	65	41	-10	2	2	76	84	-1	3	2	97	109	1	5	2	108	117
12	5	1	70	27	6	0	2	111	131	-9	2	2	166	169	0	3	2	231	230	1	5	2	73	80
-8	6	1	60	76	8	0	2	97	117	-8	2	2	225	218	1	3	2	95	108	2	5	2	67	78
-7	6	1	72	86	9	0	2	99	78	-7	2	2	118	102	2	3	2	150	174	3	5	2	119	136
-6	6	1	83	66	10	0	2	111	92	-6	2	2	274	279	3	3	2	184	141	5	5	2	91	90
-4	6	1	63	59	11	0	2	133	120	-5	2	2	98	87	4	3	2	221	183	9	5	2	91	90
-3	6	1	80	66	-13	1	2	111	96	-4	2	2	222	248	5	3	2	50	58	10	5	2	94	66
-2	6	1	98	94	-11	1	2	81	90	-3	2	2	456	448	8	3	2	59	61	-9	6	2	58	38
-1	6	1	136	172	-10	1	2	129	126	-2	2	2	676	663	9	3	2	59	61	-8	6	2	66	82
0	6	1	70	59	-9	1	2	149	159	-1	2	2	104	110	-11	4	2	45	31	-3	6	2	102	108
1	6	1	107	118	-8	1	2	139	111	0	2	2	741	662	-10	4	2	56	54	-2	6	2	78	83
2	6	1	52	52	-7	1	2	232	218	1	2	2	156	103	-9	4	2	126	109	-1	6	2	77	65
5	6	1	96	90	-6	1	2	437	434	2	2	2	317	262	-8	4	2	121	127	0	6	2	82	81
6	6	1	64	66	-5	1	2	208	249	3	2	2	142	161	-7	4	2	57	60	2	6	2	77	65
-2	7	1	79	81	-4	1	2	611	569	4	2	2	218	271	-6	4	2	112	105	4	6	2	82	69
1	7	1	62	55	-3	1	2	437	441	5	2	2	133	105	-5	4	2	152	149	5	6	2	77	63
2	7	1	65	50	-2	1	2	454	499	6	2	2	275	269	-3	4	2	159	152	-3	7	2	57	60
7	7	1	58	40	-1	1	2	450	292	7	2	2	96	109	-2	4	2	205	214	1	7	2	80	79

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine Fixed water

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	7	2	68	54	0	1	3	430	429	13	2	3	67	46	5	4	3	62	58	3	0	4	90	93
-4	8	2	58	34	1	1	3	321	339	-9	3	3	53	30	-11	5	3	58	47	5	0	4	194	181
-15	0	3	94	113	2	1	3	402	291	-8	3	3	200	234	-9	5	3	70	78	6	0	4	104	94
-14	0	3	112	113	3	1	3	219	224	-7	3	3	47	47	-8	5	3	99	92	7	0	4	133	142
-12	0	3	118	115	4	1	3	265	256	-6	3	3	181	172	-7	5	3	53	57	10	0	4	74	86
-11	0	3	94	81	5	1	3	120	124	-5	3	3	186	214	-6	5	3	61	64	-13	1	4	79	63
-9	0	3	88	89	6	1	3	246	219	-4	3	3	221	188	-5	5	3	50	52	-12	1	4	66	70
-8	0	3	117	109	7	1	3	151	172	-3	3	3	111	122	-4	5	3	106	117	-10	1	4	68	68
-7	0	3	37	72	8	1	3	69	57	-2	3	3	215	190	-3	5	3	56	60	-9	1	4	92	97
-6	0	3	103	74	10	1	3	120	112	-1	3	3	478	448	-2	5	3	57	33	-8	1	4	181	191
-5	0	3	192	210	11	1	3	84	57	0	3	3	92	107	-1	5	3	97	118	-7	1	4	77	79
-4	0	3	442	330	-15	2	3	77	79	1	3	3	190	168	0	5	3	96	88	-6	1	4	162	158
-3	0	3	382	370	-14	2	3	106	101	2	3	3	134	110	2	5	3	57	63	-5	1	4	174	144
-2	0	3	465	554	-12	2	3	44	42	3	3	3	109	117	3	5	3	52	45	-4	1	4	103	126
-1	0	3	902	114	-11	2	3	78	71	4	3	3	127	122	4	5	3	90	83	-3	1	4	266	276
1	0	3	97	64	-9	2	3	133	151	5	3	3	89	110	5	5	3	89	100	-2	1	4	311	328
3	0	3	57	39	-8	2	3	129	149	6	3	3	52	66	-3	6	3	71	75	-1	1	4	578	563
4	0	3	172	165	-7	2	3	74	86	8	3	3	62	61	2	6	3	75	79	0	1	4	186	179
5	0	3	80	87	-6	2	3	97	103	9	3	3	56	39	3	6	3	97	114	1	1	4	302	295
7	0	3	107	94	-5	2	3	234	236	10	3	3	91	93	2	7	3	69	72	2	1	4	74	93
8	0	3	149	138	-4	2	3	186	138	-14	4	3	61	64	3	7	3	68	80	3	1	4	199	196
10	0	3	73	77	-3	2	3	176	213	-13	4	3	51	42	-13	0	4	64	68	4	1	4	144	148
13	0	3	93	91	-2	2	3	113	99	-11	4	3	79	77	-14	0	4	96	71	5	1	4	67	55
-13	1	3	82	78	-1	2	3	121	130	-10	4	3	99	106	-11	0	4	49	40	6	1	4	89	86
-10	1	3	129	138	0	2	3	84	74	-9	4	3	73	78	-10	0	4	138	138	7	1	4	66	66
-9	1	3	188	144	1	2	3	135	153	-8	4	3	133	134	-8	0	4	49	43	8	1	4	55	62
-8	1	3	102	110	2	2	3	176	138	-7	4	3	82	71	-6	0	4	91	90	9	1	4	55	62
-7	1	3	30	63	3	2	3	204	200	-6	4	3	157	156	-4	0	4	431	493	10	1	4	78	98
-6	1	3	327	368	4	2	3	237	218	-1	4	3	169	164	-3	0	4	133	99	11	1	4	88	86
-5	1	3	239	234	5	2	3	163	173	0	4	3	104	125	-2	0	4	505	545	-14	2	4	78	77
-4	1	3	149	126	6	2	3	77	72	1	4	3	162	197	-1	0	4	90	76	-11	2	4	53	86
-3	1	3	220	170	7	2	3	102	111	2	4	3	40	54	0	0	4	763	694	-8	2	4	88	70
-2	1	3	446	399	8	2	3	62	46	3	4	3	107	101	1	0	4	358	282	-7	2	4	108	109
-1	1	3	628	680	10	2	3	73	72	4	4	3	91	56	2	0	4	385	355	-6	2	4	149	153

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine Fixed water

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	4	127	107	-8	4	4	49	56	-1	0	5	391	305	-8	2	5	85	73	-5	4	5	97	90
-4	2	4	344	362	-7	4	4	80	74	0	0	5	680	547	-7	2	5	118	106	-4	4	5	67	66
-3	2	4	222	252	-6	4	4	82	89	1	0	5	128	104	-6	2	5	89	80	-3	4	5	194	198
-1	2	4	149	148	-5	4	4	77	74	2	0	5	305	291	-5	2	5	301	305	-1	4	5	148	147
0	2	4	166	216	-4	4	4	203	189	4	0	5	107	110	-4	2	5	306	277	0	4	5	168	169
2	2	4	118	109	-3	4	4	84	79	6	0	5	53	60	-3	2	5	261	249	2	4	5	116	113
3	2	4	93	99	-2	4	4	173	170	10	0	5	89	93	-2	2	5	224	232	3	4	5	91	105
4	2	4	173	151	-1	4	4	53	49	-10	1	5	89	87	0	2	5	222	219	4	4	5	98	99
6	2	4	82	78	2	4	4	43	52	-9	1	5	129	132	1	2	5	55	49	-9	5	5	99	78
9	2	4	81	72	4	4	4	59	56	-8	1	5	128	149	2	2	5	134	148	-8	5	5	118	124
10	2	4	55	54	5	4	4	72	72	-7	1	5	43	52	3	2	5	184	173	-6	5	5	64	49
-12	3	4	49	68	-11	5	4	46	40	-6	1	5	202	152	6	2	5	70	72	-5	5	5	74	63
-9	3	4	63	58	-8	5	4	52	49	-5	1	5	108	106	8	2	5	46	49	2	5	5	99	97
-8	3	4	97	69	-5	5	4	131	139	-4	1	5	129	118	-8	3	5	58	55	4	5	5	52	60
-7	3	4	39	47	-4	5	4	96	96	-3	1	5	45	41	-7	3	5	80	88	5	5	5	113	107
-6	3	4	89	88	-1	5	4	116	116	-2	1	5	149	188	-6	3	5	181	171	-8	6	5	81	74
-5	3	4	174	185	0	5	4	88	109	-1	1	5	321	311	-5	3	5	143	150	-7	6	5	68	96
-4	3	4	189	179	-8	6	4	55	60	0	1	5	376	341	-4	3	5	108	106	-6	6	5	70	71
-3	3	4	190	184	0	6	4	96	90	1	1	5	224	240	-3	3	5	118	125	-1	6	5	86	89
-2	3	4	67	59	1	6	4	64	64	2	1	5	319	340	-2	3	5	100	99	1	6	5	82	88
-1	3	4	336	363	-5	7	4	64	64	3	1	5	140	127	0	3	5	64	72	2	6	5	60	81
0	3	4	59	56	-4	7	4	79	82	4	1	5	177	138	1	3	5	116	98	-5	7	5	99	84
1	3	4	153	144	5	7	4	57	61	5	1	5	134	111	2	3	5	161	158	-4	7	5	64	56
2	3	4	92	88	-16	0	5	54	54	6	1	5	98	91	3	3	5	124	130	-14	0	6	138	139
3	3	4	177	173	-14	0	5	84	76	8	1	5	82	70	4	3	5	82	85	-11	0	6	184	202
4	3	4	60	60	-10	0	5	70	58	9	1	5	89	74	6	3	5	67	73	-9	0	6	133	138
5	3	4	92	84	-9	0	5	78	73	11	1	5	89	91	7	3	5	93	83	-8	0	6	172	209
6	3	4	73	83	-8	0	5	83	77	-14	2	5	89	88	8	3	5	66	68	-7	0	6	370	448
7	3	4	69	68	-7	0	5	141	144	-13	2	5	80	72	10	3	5	73	74	-6	0	6	221	223
11	3	4	55	53	-6	0	5	83	33	-12	2	5	63	69	11	3	5	50	50	-5	0	6	160	138
-14	4	4	55	53	-4	0	5	55	67	-11	2	5	58	47	-11	4	5	47	42	-4	0	6	399	376
-11	4	4	75	74	-3	0	5	199	209	-10	2	5	67	62	-8	4	5	106	111	-3	0	6	289	199
-10	4	4	91	47	-2	0	5	228	249	-9	2	5	95	92	-7	4	5	78	83	-2	0	6	488	388

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine fixed water PAGE 5

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	0	6	383	392	-3	2	6	44	46	-5	4	6	66	52	1	0	7	256	266	2	2	7	43	38
1	0	6	417	374	-2	2	6	349	331	-3	4	6	44	38	2	0	7	117	110	3	2	7	86	93
2	0	6	143	152	-1	2	6	254	289	-2	4	6	59	56	4	0	7	69	74	4	2	7	62	53
4	0	6	183	173	0	2	6	189	178	-1	4	6	162	160	6	0	7	48	55	5	2	7	69	74
5	0	6	120	106	1	2	6	400	393	1	4	6	135	142	8	0	7	92	111	8	2	7	100	120
8	0	6	141	115	2	2	6	196	202	2	4	6	121	99	9	0	7	72	69	9	2	7	85	84
11	0	6	57	59	3	2	6	55	51	4	4	6	89	84	-17	1	7	53	59	-14	3	7	93	97
-12	1	6	68	79	4	2	6	194	188	5	4	6	63	62	-14	1	7	111	128	-12	3	7	80	98
-10	1	6	96	111	5	2	6	178	176	6	4	6	61	66	-11	1	7	165	148	-11	3	7	63	67
-9	1	6	68	80	6	2	6	84	82	8	4	6	53	59	-10	1	7	80	72	-10	3	7	50	56
-8	1	6	300	247	7	2	6	69	75	-10	5	6	71	75	-9	1	7	114	124	-9	3	7	111	120
-7	1	6	48	39	8	2	6	88	87	-9	5	6	65	65	-8	1	7	70	70	-8	3	7	96	78
-6	1	6	281	312	-12	3	6	86	78	-5	5	6	71	72	-6	1	7	279	225	-6	3	7	150	137
-5	1	6	273	321	-8	3	6	109	123	-4	5	6	69	68	-5	1	7	257	229	-5	3	7	62	63
-4	1	6	362	410	-7	3	6	84	85	-3	5	6	76	74	-4	1	7	128	108	-4	3	7	127	118
-3	1	6	130	122	-6	3	6	134	141	-1	5	6	59	82	-3	1	7	219	240	-3	3	7	45	64
-2	1	6	324	334	-5	3	6	105	90	0	5	6	55	44	-2	1	7	121	92	-2	3	7	63	57
-1	1	6	495	469	-4	3	6	114	113	2	5	6	54	46	0	1	7	128	83	-1	3	7	108	99
0	1	6	173	94	-3	3	6	78	69	5	5	6	60	55	1	1	7	116	107	0	3	7	67	73
1	1	6	78	97	-2	3	6	49	62	7	5	6	65	52	2	1	7	81	56	1	3	7	73	64
3	1	6	89	63	-1	3	6	179	165	-1	6	6	66	75	3	1	7	99	97	3	3	7	90	94
4	1	6	72	74	0	3	6	95	111	6	6	6	54	41	4	1	7	47	35	4	3	7	87	91
5	1	6	68	72	1	3	6	118	114	-14	0	7	94	87	5	1	7	96	93	5	3	7	74	71
7	1	6	119	113	2	3	6	124	102	-12	0	7	59	63	7	1	7	126	134	7	3	7	91	95
8	1	6	93	97	3	3	6	116	113	-10	0	7	54	42	8	1	7	76	59	8	3	7	101	96
9	1	6	74	83	8	3	6	98	112	-9	0	7	139	133	-11	2	7	113	106	-9	4	7	48	46
10	1	6	71	54	9	3	6	83	93	-7	0	7	161	132	-8	2	7	48	61	-8	4	7	97	85
-14	2	6	139	146	-14	4	6	64	64	-6	0	7	57	30	-7	2	7	104	109	-7	4	7	87	78
-13	2	6	61	69	-11	4	6	117	116	-5	0	7	114	104	-6	2	7	124	103	-6	4	7	71	80
-11	2	6	143	150	-10	4	6	75	80	-4	0	7	483	448	-5	2	7	226	206	-5	4	7	56	50
-9	2	6	58	64	-9	4	6	76	85	-3	0	7	40	13	-3	2	7	78	75	-2	4	7	119	104
-7	2	6	204	177	-8	4	6	95	109	-2	0	7	402	421	-1	2	7	262	254	-1	4	7	148	139
-5	2	6	112	94	-7	4	6	88	74	-1	0	7	382	355	0	2	7	53	59	0	4	7	89	103
-4	2	6	197	196	-6	4	6	52	43	0	0	7	152	150	1	2	7	141	135	1	4	7	43	23

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine fixed water PAGE 6

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	4	7	94	98	-4	1	8	110	117	-5	3	8	93	96	0	0	9	95	116	-8	3	9	65	53
5	4	7	78	82	-3	1	8	335	302	-4	3	8	73	78	1	0	9	87	74	-6	3	9	91	119
6	4	7	56	52	-2	1	8	152	113	-3	3	8	155	140	6	0	9	100	116	-5	3	9	97	103
-9	5	7	86	78	-1	1	8	48	48	-2	3	8	89	81	-9	1	9	53	53	-3	3	9	55	54
-5	5	7	30	29	0	1	8	43	30	-1	3	8	139	123	-6	1	9	181	170	1	3	9	110	109
-3	5	7	92	85	1	1	8	87	99	1	3	8	74	73	-5	1	9	68	72	2	3	9	54	42
0	5	7	51	37	2	1	8	117	109	2	3	8	51	60	-4	1	9	126	130	3	3	9	73	60
3	5	7	53	41	3	1	8	46	37	4	3	8	147	131	-3	1	9	94	98	4	3	9	67	68
1	6	7	91	65	4	1	8	126	109	6	3	8	55	74	0	1	9	54	63	5	3	9	82	63
3	6	7	58	43	6	1	8	146	148	7	3	8	101	93	1	1	9	45	39	-14	4	9	52	48
-4	7	7	51	50	7	1	8	116	112	-12	4	8	66	88	2	1	9	77	77	-13	4	9	65	82
-12	0	8	110	101	8	1	8	63	50	-10	4	8	80	77	3	1	9	52	54	-10	4	9	62	68
-11	0	8	79	72	-11	2	8	69	75	-9	4	8	66	70	4	1	9	78	84	-9	4	9	82	75
-10	0	8	89	89	-10	2	8	76	77	-8	4	8	67	66	5	1	9	127	138	-6	4	9	48	47
-9	0	8	55	42	-8	2	8	92	102	-2	4	8	106	118	6	1	9	61	73	-4	4	9	65	54
-7	0	8	128	128	-7	2	8	122	101	-1	4	8	68	67	-13	2	9	69	77	-1	4	9	57	75
-6	0	8	125	113	-5	2	8	76	83	4	4	8	57	53	-12	2	9	109	98	-6	5	9	48	67
-5	0	8	297	325	-3	2	8	54	53	5	4	8	69	51	-9	2	9	67	62	-5	5	9	56	69
-4	0	8	66	83	-2	2	8	113	124	-11	5	8	49	66	-8	2	9	70	63	-5	6	9	56	38
-3	0	8	157	164	-1	2	8	124	115	-9	5	8	74	83	-7	2	9	130	126	-9	0	10	94	70
-2	0	8	275	242	0	2	8	67	72	-6	5	8	55	50	-6	2	9	122	113	-8	0	10	91	87
0	0	8	97	82	1	2	8	112	111	-1	5	8	70	68	-5	2	9	102	103	-6	0	10	164	125
7	0	8	98	100	3	2	8	112	128	0	5	8	79	86	-4	2	9	41	33	-5	0	10	128	127
8	0	8	74	71	4	2	8	57	65	4	5	8	60	71	-2	2	9	160	170	-4	0	10	130	142
-17	1	8	61	63	5	2	8	67	67	-4	7	8	59	56	-1	2	9	58	48	0	0	10	65	72
-14	1	8	103	91	7	2	8	94	102	-14	0	9	77	79	0	2	9	88	104	3	0	10	101	96
-13	1	8	102	103	8	2	8	79	72	-13	0	9	102	113	1	2	9	84	84	-13	1	10	50	64
-11	1	8	83	77	-14	3	8	77	75	-12	0	9	115	104	2	2	9	85	95	-9	1	10	76	74
-10	1	8	96	84	-13	3	8	63	53	-8	0	9	62	69	3	2	9	45	29	-7	1	10	107	118
-9	1	8	75	78	-12	3	8	85	98	-6	0	9	61	57	4	2	9	45	62	-6	1	10	77	89
-8	1	8	100	114	-11	3	8	89	92	-5	0	9	63	58	6	2	9	81	94	-5	1	10	68	82
-7	1	8	56	49	-10	3	8	69	71	-4	0	9	185	189	-13	3	9	50	61	-4	1	10	49	51
-6	1	8	121	119	-9	3	8	129	124	-3	0	9	63	68	-11	3	9	49	51	-3	1	10	96	84
-5	1	8	88	89	-6	3	8	61	53	-1	0	9	191	161	-9	3	9	49	62	-2	1	10	50	57

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Strychnine Tyrosine fixed water PAGE 7

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	1	10	147	146	-7	4	10	68	72	-2	2	11	133	128	-9	1	12	153	132	-11	1	13	90	99
0	1	10	121	108	-6	4	10	86	80	-1	2	11	66	61	-8	1	12	78	65	-10	1	13	71	81
3	1	10	53	55	-5	4	10	73	88	-9	3	11	60	54	-7	1	12	148	136	-9	1	13	77	69
4	1	10	135	133	-4	4	10	54	68	-8	3	11	90	89	-4	1	12	63	57	-7	1	13	91	97
5	1	10	74	83	-2	4	10	58	60	-7	3	11	92	87	-3	1	12	100	115	-4	1	13	86	86
-12	2	10	62	68	-1	4	10	77	81	-6	3	11	78	86	-2	1	12	60	66	-3	1	13	68	60
-10	2	10	98	106	0	4	10	55	68	-4	3	11	86	93	-1	1	12	71	62	0	1	13	67	79
-8	2	10	62	61	-7	5	10	61	53	-3	3	11	60	47	-10	2	12	92	83	-8	2	13	92	90
-7	2	10	95	86	-6	5	10	60	78	-2	3	11	111	109	-9	2	12	65	51	-6	2	13	81	96
-6	2	10	128	127	-3	5	10	59	53	-1	3	11	76	70	-6	2	12	130	119	-5	2	13	83	79
-5	2	10	87	91	-13	0	11	80	101	3	3	11	75	65	-4	2	12	71	69	-3	2	13	76	67
-4	2	10	62	47	-8	0	11	68	54	-6	4	11	93	79	-11	3	12	70	68	1	2	13	45	29
-2	2	10	73	78	-7	0	11	86	92	-5	4	11	74	79	-8	3	12	77	62	-10	3	13	59	66
-1	2	10	123	109	-5	0	11	115	122	-4	4	11	51	43	-7	3	12	101	95	-3	3	13	54	67
0	2	10	84	80	-4	0	11	166	191	-3	4	11	65	86	-4	3	12	103	105	-5	4	13	62	61
1	2	10	79	78	-2	0	11	67	60	-1	4	11	86	85	-3	3	12	78	78	-10	0	14	68	72
2	2	10	79	81	3	0	11	105	116	1	4	11	48	32	-2	3	12	79	83	-6	0	14	77	88
4	2	10	59	50	-8	1	11	91	78	-7	5	11	53	32	-6	4	12	83	87	-10	1	14	95	78
-13	3	10	55	51	-7	1	11	88	99	-2	5	11	52	52	-1	4	12	60	53	-6	1	14	93	74
-12	3	10	72	67	-6	1	11	76	79	-14	0	12	82	93	-11	0	13	63	48	-4	1	14	47	44
-9	3	10	71	55	-5	1	11	49	43	-12	0	12	71	81	-10	0	13	66	65	-1	1	14	53	49
-7	3	10	77	75	-3	1	11	67	57	-9	0	12	70	60	-8	0	13	76	66	-10	2	14	62	57
-6	3	10	123	121	-2	1	11	56	50	-6	0	12	52	78	-6	0	13	142	139	-8	2	14	49	28
-5	3	10	71	56	-1	1	11	53	41	-3	0	12	77	83	-5	0	13	57	52	-7	2	14	57	46
-3	3	10	84	75	3	1	11	74	59	-4	0	12	65	64	-3	0	13	68	79	-6	2	14	53	56
-1	3	10	64	76	-10	2	11	46	55	-3	0	12	62	63	-2	0	13	81	72	-4	2	14	54	52
0	3	10	121	119	-8	2	11	79	83	-2	0	12	53	43	-1	0	13	53	41	-12	3	14	54	27
1	3	10	92	70	-7	2	11	119	108	-13	1	12	54	58	-13	1	13	53	47	-4	1	15	46	40
2	3	10	53	49	-5	2	11	56	50															