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Conformational analysis of internal rotation

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CONFORMATIONAL ANALYSIS

OF

INTERNAL ROTATION

by

LOUIS DENNER

A thesis submitted to the Faculty of Science, University of the Witwatersrand, Johannesburg, in fulfilment of the requirements for the degree of Doctor of Philosophy.

February, 1967.

Declaration

I declare that this thesis is my own, unaided work. It is being submitted for the degree of Doctor of Philosophy at the University of the Witwatersrand, Johannesburg. It has not been submitted for any degree or examination at any other university.



Louis Denner

ABSTRACT

The concept of internal rotation is fundamental in the analysis of conformational preferences in molecular structure. Restricted internal rotation is invoked to explain the existence of rotational isomers, barrier heights and, sometimes, novel stereoisomers, which again leads to different chemical reactivities. The particular pharmacological activity of certain compounds have been linked to specific conformational preferences.

This thesis describes a conformational study of internal rotation in classes of compounds such as: amides, peri-substituted naphthalenes, fulgides, as well as a variety of exploratory studies. The techniques of single crystal X-ray crystallography, complemented by the theoretical method of molecular mechanics were used as tools in this investigation. The single crystal structure analyses of 24 organic compounds were performed. The two-dimensional mapping of steric energy of 10 compounds were studied to elucidate rotational isomerism pathways and barrier heights to interconversion. The conformations predicted for compounds are compared with analogous systems in the literature. For the first time, the structures of the rotameric forms of o-methylformanilide were studied crystallographically. A point worth mentioning is the isolation of rotamers of E,E-bis(p-methoxybenzylidene)-succinic anhydride. This compound crystallises with different orientations for the p-methoxy groups.

Finally, this work contributes to the understanding of conformational isomerism, and it shows that X-ray crystallography, combined with theoretical methods, is a very powerful tool for conformational analysis.

ACKNOWLEDGEMENTS

I am most grateful to my supervisor, Professor Jan Boeyens, who created and stimulated a perfect research environment for me. I wish to thank him for the funds he provided me to attend many conferences to present my work, including two international meetings, namely:

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To Leticia with love and thanks

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CHAPTER 1

INTRODUCTION

One of the most intriguing and important concepts in the whole of chemistry must be that of molecular structure. The properties of matter are linked to it, and explanations and discussions of reactivity are based on this concept. Hence, the understanding and development of structural models are of prime importance.

Both molecular shape and conformation are defined by a set of three different geometric parameters, namely, bond lengths, bond angles and torsion or dihedral angles. The rules of conformational analysis are based on a rigid structural model, defined by standard bond lengths and angles, and torsion angles that favour staggered arrangements. Structural models of this type allow the discussion of reactivity without experimental structural analysis of a whole series of compounds. However, this simple rigid model reaches its limits when used to explain steric effects and restricted rotation in complicated structures. If a part of a molecule is, for example, distorted by non-bonded interactions, the strain is eventually dispersed throughout the entire molecule. However, relatively large energies are needed to bring about significant changes, particularly in bond lengths and to a lesser extent in bond angles. Torsional mobility about single and partial double bonds is energetically less demanding. Since torsion angles determine molecular conformation, it is clear that for molecules which possess torsional flexibility, various molecular conformations are possible.

The study of steric influence on conformation is of growing importance, not only in organic chemistry. Studies of the relationship between structure and activity

of drugs are related to it. As another example, special material properties of polymers resulting from preferred conformations in a polymer chain should be mentioned. To understand this type of relationship between conformation and function, a flexible structural model which allows variation of the structural parameters, and some knowledge of the dynamic behaviour of organic molecules are essential. In modern stereochemistry not only static, but also dynamic behaviour of molecules are important.

In practice, three different methods can be applied to determine the conformation of organic molecules. These are: X-ray crystallography for the solid state, NMR spectroscopy for the liquid and solution state and computational techniques for an isolated molecule. The enormous contribution that crystallography has made to the study of molecular conformation is generally appreciated. It is still the most powerful method of determining relative atomic positions in a molecule. NMR spectroscopy, especially in techniques like dynamic nuclear magnetic resonance spectroscopy and nuclear Overhauser enhancement experiments, which demonstrate spatial relationships between chemical entities, is of similar importance for dispersed molecules. Theoretical calculations, on the other hand, have been widely applied to interpret and organise results and to resolve chemical problems. The most important future role of theoretical calculations is probably to make reliable predictions before any costly experimental work begins. Unfortunately, there is currently no single method which is adequate for all problems.

The idea of calculating by some means the geometry and energy of a given molecule has long intrigued chemists. The procedure known as molecular mechanics has been used widely to determine strain energies, conformations and enthalpies of various compounds. Like many other chemical concepts, the concept of steric strain is only semi-

quantitative and lacks precise definition. Molecules are considered strained if they contain internal coordinates which deviate from values regarded as "normal" or "strain free", which in their turn again can be considered as equilibrium situations. Although early computational studies in structural chemistry have for the most part been concerned with equilibrium structures (minima on the potential energy surface), many studies are now appearing that deal with movements across saddle points on the energy surface, simulating reaction pathways. This is often accomplished by rotating certain torsional degrees of freedom for a specific molecule. This method of stereodynamics represents an important application of molecular mechanics calculation. The rotation about a chemical bond of one part of a molecule with respect to another, is usually accompanied by a change in potential energy. In some cases internal rotation of this kind gives rise to the phenomenon of rotational isomerism, where the molecules exist as an equilibrium mixture of two or more conformers or rotamers. One of the classic examples of a molecule exhibiting rotational isomerism is 1,2-dichloroethane, where the variation of potential energy with azimuthal angle is as shown in Figure 1.

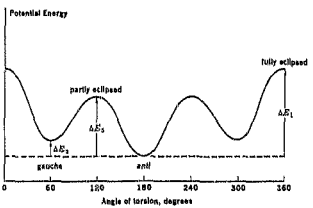
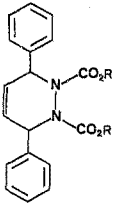


Fig.1. Potential energy curve for 1,2-dichloroethane.

The minima in this potential energy curve correspond to the stable staggered conformations of the molecule which are the rotamers. The difference in energy between the minima is the enthalphy difference between the isomers, and the difference between the neighbouring maxima and minima is the potential barrier hindering the rotation.

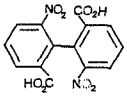
Rotational isomerism of this kind is now basic in theories covering large areas of chemistry, including the conformational analysis of acyclic structures and the physical properties of polymers. The phenomenon also has considerable importance in protein chemistry, where the coiling and uncoiling of polypeptide chains play an important role in biological systems. Hence, provided the barrier to rotation about a certain bond in a molecule is sufficiently high, separation of rotational isomers should be possible. Intramolecular movements with activation energies of 3-16 kcal/mol (1 kcal = 4.184 kJ) are so fast that the resulting isomers cannot be isolated at room temperature. In general, resolution of isomers separated by an energy barrier of ca. 17 kcal/mol or greater is physically possible. For example, at least three hindered rate processes occur in compounds like 1 (Ereliere and Lehn, 1965):



1

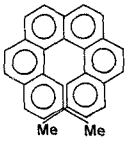
Two of these processes are *hindered rotations* about the N-CO_2R bonds and the third is a ring inversion process. These authors did not attempt to resolve any of these conformational isomers.

A direct and interesting consequence of restricted internal rotation is chirality. Many compounds have no *asymmetric carbon* atom yet they have a chiral structure. The most common examples of chiral molecules without asymmetric carbon atoms are ortho-substituted biphenyl derivatives, like 2:



2

Since the two rings cannot lie in a common plane *there is* no plane of symmetry, hence the enantiomers are optically active. Enantiomers may even result from a reversal of helicity in compounds like 3:



3

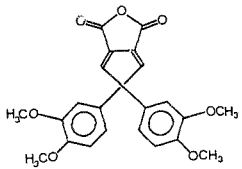
The factors which govern the activation energy for rotation about chemical bonds are manifold, and of prime importance when it comes to understanding molecular conformation and attempting to isolate possible rotamers. No attempt was made to analyse all aspects of conformational stereodynamics in the present work.

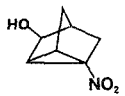
The aims of this project on conformational analysis of internal rotation are as outlined below:

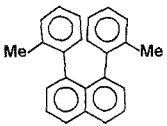
(i) to gain a better understanding of molecular structure and conformation

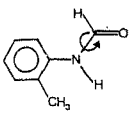
(ii) to master some of the methods used in modern conformational analysis

(iii) to examine, both theoretically and experimentally, conformational isomerism in model systems such as these listed below:









(iv) to perform stereodynamical molecular mechanics calculations on a subset of such molecules, and

(v) to discuss the stereo-isomerism and describe some of the novel stereochemistry which result from restricted internal rotation.

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CHAPTER 2

HISTORICAL REVIEW OF INTERNAL ROTATION

2.1 Introduction

Atoms in molecules are held together by electrostatic interactions between nuclei and electrons. Chemical bonds are a manifestation of these interactions. In a compound in which two carbon atoms are joined by a single bond, and rotation occurs, the substituent attached to the carbon atoms can assume an infinite number of positions relative to each other. The various conformations, corresponding to the relative disposition of the substituents during rotation around the carbon-carbon link, are not all of the same energy. Depending upon the nature of the substituents, one or more of the possible arrangements would correspond to minima in the potential energy of the system. The various shapes which a molecule can assume by means of free rotation around a bond are known as conformations of the molecule (Orville-Thomas, 1974). Conformational analysis is concerned with the detailed arrangement in space of the atoms comprising a molecule. Clearly, therefore, internal rotation has important consequences for molecular structure and dynamics, making it an old, yet new field of research.

The scientific literature relating to internal rotation is enormous and includes many excellent reviews and books: Mizushima (1954), Wilson (1959), Lowe (1968), Wilson (1972), Orville-Thomas (1974), Lister et al. (1978) and Long (1985). The first concepts of stereochemistry were put forward by Van't Hoff and Le Bel in the latter part of the nineteenth century. Van't Hoff postulated a tetrahedral model for the bonds associated with a saturated carbon atom (Long, 1985). He made one further great contribution to the theory of stereochemistry. In his view, free rotation could occur around any carbon-

carbon single bond, but free rotation around a double bond was restricted (Long, 1985). As time passed, the concept of free rotation around single bonds became suspect, as indications were obtained that in many compounds free rotation was restricted and that the molecule existed to a greater or lesser extent as a number of preferred rotamers.

An important characteristic of a potential function for internal rotation is the height of the barrier. In 1936, Kemp and Pitzer estimated the barrier height in ethane to be 3.15 kcal/mol, using third-law entropy effects from calorimetric data. The recent accurate value is 2.90 kcal/mol obtained by Hirota and others (1979), using microwave spectroscopy. Soon after the work of Kemp and Pitzer it became apparent that rotation about single bonds is in fact restricted in many molecules. However, the barriers are only of the order of a few kcal/mol and are not large enough to permit chemical isolation of rotamers. A barrier of about 15 kcal/mol is required to allow isolation of rotational isomers at room temperature (Long, 1985). Although the barriers to free rotation are small, the existence of restricted rotation has been found to have far reaching implications for chemistry. A wide variety of methods has been developed for the study of this phenomenon, and the physical origin of the restricting barriers has proved a stern test of theoretical chemistry (Orville-Thomas, 1974; Morino, 1985; Hirota, 1985 and Pitzer, 1983).

In 1957, Wilson published a paper on the origin of potential barriers to internal rotation in molecules. The conclusion to which the arguments lead was that potential barriers to internal rotation, at least in the case where one group carries only hydrogen atoms, must in some way be an inherent property of the axial bond itself and not due in any substantial measure to direct forces between the attached atoms. Pauling (1958) developed a simple theory

carbon single bond, but free rotation around a *double bond* was restricted (Long, 1985). As time passed, the concept of free rotation around *single bonds became suspect*, as indications were obtained that in many compounds free rotation was restricted and that the *molecule existed* to a greater or lesser extent as a number of preferred rotamers.

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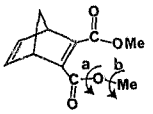
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for these potential barriers. According to his theory, the potential barriers are not a property of the axial bond itself, but result from the exchange interactions of electrons involved in the other bonds (adjacent bonds) formed by each of the two atoms, as determined by the overlap between the parts of the adjacent bond orbitals that extend from each of the two atoms toward each other. However, recent developments indicate that this approach advocated by Pauling is not really tenable, although it has an intuitive appeal to many chemists (Lowe, 1973; Lister et al. 1978; Dunlap 1986). Other points of view have been taken in attempts to understand internal rotational barriers, and extensive references to these may be found in the reviews by Dale (1966); Lowe (1968) and by Payne and Allen (1977).

If the rotational isomerism in a certain molecule differs significantly from that in related molecules it is usual to seek an explanation in terms of intramolecular forces which are present in the molecule. In doing this it is assumed that the conformational energies and barriers can be expressed as sums of terms due to different types of forces. Apart from the usual "barrier forces" other types of forces which may be invoked in special situations include:

1. double bond character due to resonance
2. hydrogen bonding
3. steric repulsion.

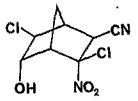
Double bond character is particularly important in situations where a single bond occurs between two double bonds, such as in the bicyclic ester, 4, below



4

The barrier to internal rotation about bond a in most simple esters is between 6 and 12 kcal/mol, whereas the barrier about bond b is about 1 kcal/mol and largely threefold (Jones and Owen, 1973).

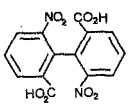
Intramolecular hydrogen bonding has been used to explain conformational behaviour and energies in molecules ranging from simple acids and alcohols to large and complicated systems such as proteins, nucleic acids and carbohydrates. Intramolecular hydrogen bonding between nitro and hydroxy groups in the norbornyl system, 5, was investigated by Boeyens, Denner and Michael (1984). In conformational terms, the orientations of both the nitro and hydroxy groups were rather surprising, but understandable in view of the hydrogen bonding.



5

Steric repulsions occur when two atoms are separated by a distance less than the sum of their van der Waal's radii.

and are expected to play an important part in determining rotational isomerism and barriers in instances where bulky atoms or groups are attached to the atoms of the bond about which internal rotation is taking place. The first compounds for which rotational isomerism was shown to occur were substituted derivatives of biphenyl, where steric interactions between the side groups are so large that interconversion of conformers at room temperature is not possible. Christie and Kenner (1922) succeeded in the resolution of the two optically active isomers of 2,2'-dinitrodiphenyl-6,6'-dicarboxylic acid, 6.



6

2.2 Dynamic Nuclear Magnetic Resonance Spectroscopy

One of the methods rapidly emerging as the most powerful tool to study barriers to rotation and rates of rotameric interconversions in solution is the method of dynamic nuclear magnetic resonance spectroscopy (DNMR). Although this project deals with the crystallographic and computational parts of internal rotation, it is felt that a brief description of DNMR would be in order. Numerous articles and reviews deal with this area of chemistry: Oki (1985), Kessler (1970) and by Rubiralta (1986). Briefly then, from the coalescence of resonances, rate data and the free energy of activation for the exchange can be calculated. Gutowsky and Holm (1956) solved the Bloch equation and obtained equation (1), which affords the rate constant (k_c) of the exchange at the coalescence temperature (T_c), where delta is the difference in

chemical shifts of the two sites with equal populations.

$$k_c = \frac{\pi}{\sqrt{2}} \Delta\upsilon \qquad 1$$

If this rate constant substituted into the Eyring equation (2), the free energy of activation (3) is obtained for the exchange.

$$k = \frac{k_b}{h} \exp\left[\frac{-\Delta G}{RT}\right] \qquad 2$$

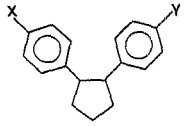
Thus:

$$\Delta G = 4.57\, T_c \left[9.97 + \log\frac{T_c}{\Delta\upsilon}\right] \qquad 3$$

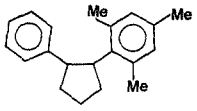
The coalescence method described above affords the free energy of activation at one temperature, T_c. To gain further insight into the exchange process, it may be desirable to perform a complete lineshape analysis.

2.3 General Examples of Restricted Rotation

In a search for isomerism due to restriction of rotation of neighbouring aromatic rings, several cis-1,2-di(p-substituted phenyl)cyclopentanes, 7, were investigated by Curtin and Dayagi (1964).

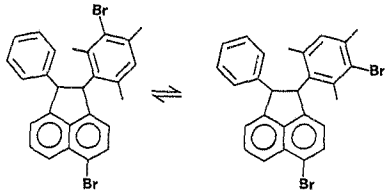


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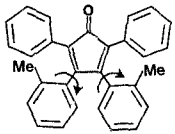
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NMR studies showed that the phenyl rings in these compounds rotate rapidly on the NMR time scale at room temperature. The same authors later published work dealing with cis-1-mesityl-2-phenyl-cyclopentane, 8, which was found to undergo slow ring rotation with a barrier of 11 kcal/mol (Curtin and co-workers, 1971). The important factor relevant to the observed low barriers of 7 and 8, is not the staggering of aromatic rings in the rotational ground state, but the fact that the five-membered ring is torsionally *soft*. Miller and Curtin (1976) isolated the rotamers 9 and 10, with a barrier of 25.6 kcal/mol hindering the interconversion. Therefore gusseting the *five-membered* ring (rather than further increasing the rotor size) was predicted to increase dramatically the rotational barrier of adjacent aryl groups.

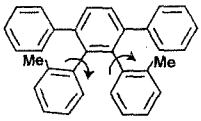


9 10

The effect of ring size on restricted inter-annular rotation in biphenyl-like molecules like 11 and 12, was investigated by Haywood-Farmer et al. (1971). The NMR spectra of both compounds at ambient temperature showed that the signal for the methyl protons in each compound were separated into two lines. This occurrence of two methyl signals was ascribed to the presence of two diastereomers in either the cis or trans arrangement.

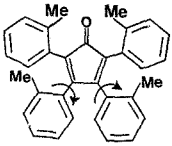


11



12

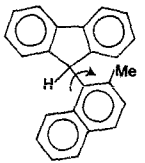
The calculated barriers were 21.8 and >25.6 kcal/mol for 11 and 12, respectively. In 1981, Willem and co-workers published a paper on the static and dynamic stereochemistry of tetra-o-tolylcyclopentadienone, 13, as examined by topological approaches.



13

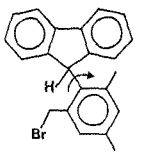
DNMR spectra revealed two coalescence regions, below and above room temperature, ascribed to two different modes of rearrangement. The low-temperature coalescence was assigned to an uncorrelated one-ring rotation of an alpha-aryl ring while the high temperature coalescence was due to an uncorrelated one-ring rotation of a beta-ring. A barrier in the region of 23 kcal/mol was found. An X-ray crystal structure determined by *Nishinaga et al.* (1978) on 2,5-di-tertbutyl-3-(4-chlorophenyl)cyclopentadienone showed that the aryl ring is nearly perpendicular to the central ring.

Oki (1976) presented a study of unusually high barriers involving the tetrahedral carbon atom. He found that high barriers to rotation appear to require not only high energy transition states of rotation but also low potential energies in the ground state. A high potential in the transition state is frequently associated with a high potential in the ground state, but this necessarily lowers the barrier to rotation. Siddall and Stewart (1969) were able to concentrate one rotamer of 9-(2-methyl-1-naphtyl)fluorene, 14, up to >90% purity and another to 58% purity by fractional crystallisation. The free energy of activation was found to be 29.2 kcal/mol.



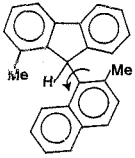
14

Nakamura and Oki (1974) succeeded in isolating the pure rotamers of 15, with a reported barrier of 27 kcal/mol.



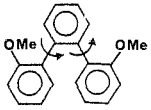
15

Introduction of a methyl group in position 1 of the fluorene nucleus appears to raise the barrier considerably. Ford et al. (1975) isolated compound 16, in two forms and the free energy of activation was estimated to be 33.3 kcal/mol.

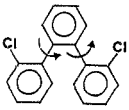


16

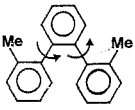
Adams and Yuan, in their review on the stereochemistry of biphenyls in 1933, pointed out that suitably substituted o-terphenyls should be capable of existing as isomers. Huang in 1954 synthesised 2,2"-dimethoxy-o-terphenyl, 17, and then in 1959, 2,2"-dichloro-o-terphenyl, 18, in an attempt to substantiate this postulate, but was unable to isolate separate isomers.



17



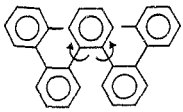
18



19

In a paper published in 1980, Mitchell and Yan reported the measurement of rotational potential barriers for 2,2"-dimethyl-o-terphenyl, 19, and a number of 3,3"-substituted examples. A barrier of 15 kcal/mol was found for 19.

Hammerschmidt et al. (1980) reported the barrier for bis-o-terphenyl, 16.1, to be 18 kcal/mol.

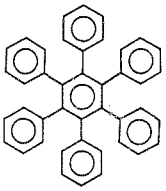


16.1

An X-ray crystal structure analysis was performed on o-terphenyl in 1978 by Aikawa and Maruyama. The two phenyl groups were reported as twisted in the same direction with respect to the central ring. The dihedral angles between the mean plane of the central ring and those of the two phenyl rings were 62.1 and 42.5 degrees. This was followed by a neutron diffraction study by Brown and Levy in 1979. One of the rings is twisted 42.1 and the other 62.1 degrees. The inter-ring angles are opened to 123.6 and 123.0 degrees. A ground state conformational analysis has been carried out on p-, m-, and o-terphenyl using a recently developed semi-empirical calculation method (CS-CINDO) by Baraldi and Fonterini (1985). The potential energy surface was calculated as a function of the torsion angles of the two terminal phenyl groups relative to the central one. Their results showed two conformers for p-, and m-terphenyl and an equilibrium conformation for o-terphenyl of phi(1) = phi(2) = 48 degrees. This is quite different from the crystal geometry. Maybe it is analogous to the classical example of biphenyl, where the twist angle between the two phenyl rings is about 40 degrees in the gas phase and 0 degrees in the crystalline phase (Simonetta, 1974; Bastiansen and Samdal, 1985 and references therein).

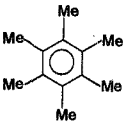
The twisting effects in polyphenyl benzene derivatives

have always been a point of interest. Bart (1968) performed an X-ray single crystal analysis on hexaphenylbenzene, 20. The peripheral rings are not perpendicular to the central ring but are twisted by about 25 degrees from this position. The molecule is highly distorted as the result of out-of-plane bending of the exocyclic bonds. The result from electron diffraction (Almenningen et al., 1958) gave an interplanar angle of 90 degrees.



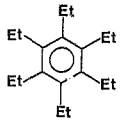
20

Iroff (1980) performed empirical force field calculations on hexamethylbenzene, 21, to elucidate the internal motions of the methyl group. When the benzene ring was constrained to be planar, the methyl groups went through a geared, disrotary movement with a barrier of between 1.6 and 1.9 kcal/mol. A recent calculation with the Boyd force field found a barrier of 2.4 kcal/mol for strictly synchronous, geared rotation (Maverick, Trueblood and Bokoe, 1978). These authors ascribed the apparent planarity of the hexamethylbenzene in the crystal structure to dynamic disorder.



21

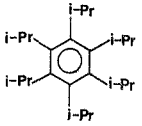
Blount, Hunter and Mislow (1984) reported the stereodynamics of ethyl group rotation in coordinated hexaethylbenzene, 22, by means of crystallography and DNMR. The crystal structure is disordered, which is not surprising in view of the up-down alternation of ethyl groups actually observed in hexaethylbenzene. This is only one of eight possible arrangements in which the ethyl groups are located on one side or other of the benzene ring plane.



22

In 1983, Siegel and Mislow published experimental evidence for a high barrier to internal rotation in hexaisopropylbenzene, 23. The six isopropyl groups form a tightly interlocking tongue-and-groove arrangement by virtue of cooperative nonbonded repulsions. To measure the isopropyl group rotation barrier, of ca. 22 kcal/mol, the ground-state symmetry of 23 was lowered by complexation with a transition metal. These findings indicated that 23 exhibits conformational rigidity and suggested the possibility of separating cycloenantiomers.

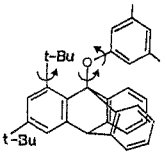
The communication was followed by a full paper (Siegel et al., 1986) on the static and dynamic stereochemistry of 23, a gear-meshed hydrocarbon of exceptional rigidity. Crystallographic orientational disorder around the molecular six-fold axes was modelled with the help of simple structural and geometrical considerations. The relative energies of the nine conformers of 23 were estimated by molecular mechanical calculations.



23

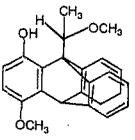
Coupled rotation of two or more parts of a molecule often becomes energetically more feasible than independent rotation of a single group.

In many cases, torsional motion around single bonds is one of the most effective modes of transmitting structural relaxation. Typical examples may be found in the stable atropisomers of bridgehead-substituted triptycenes (Kawada and Iwamura, 1983; Hounshell et al., 1980; Bürgi et al., 1983; Kawada, Okamoto and Iwamura, 1983 and Yamamoto and Oki (1981). Yamamoto and Oki (1986) investigated internal rotation in 1,3-ditert-butyl-9-(3,5-dimethylphenoxy)-triptycene, 24, by means of low temperature NMR. This study revealed restricted rotation of the 1-tert-butyl group with an energy barrier of 9.2 kcal/mol. C-O restricted rotation occurs above room temperature with an energy barrier of 17.6 kcal/mol.



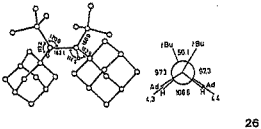
24

Kawada et al. (1983) studied stereoisomerism in molecular level-gears. The rotational motions around the two C-X bonds at the bridgehead of molecules (9-triptycyl)$_2$ X (X=O or CH_2) are rapid and disrotatorily coupled. These authors were able, by labelling one of the benzene rings on each triptycene unit in these triptycyl compounds, to generate new stereoisomers due to the different phase relationship between the labelled rings. Resolution of the dl-isomers of bis(2-and 3-chloro-9-triptycyl)-methanes and ethers into the optical antipodes was achieved by HPLC using a column of silanised silica gel coated with chiral poly(triphenylmethyl methacrylate). The electrostatic and hydrogen bonding effects on the rotamer distribution in 1-oxy-substituted 9-(1-methoxyethyl)triptycene, 25, were studied by Yamamoto, Tanaka and Oki (1983). They concluded that hydrogen bonding effects are the crucial factors governing the rotamer distribution.



25

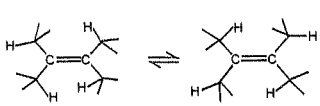
In the course of work on the consequences of strain for the structure of aliphatic molecules, Rüchardt and Beckhaus (1985), were able to isolate two rotamers of d,l-3,4-di(1-adamantyl)-2,2,5,5- tetramethylhexane, 26, this being the first isolation of a rotamer pair of an aliphatic hydrocarbon. Structures of the two rotamers of 25, with important bond lengths (pm) and angles (degrees) derived from crystal structure analyses are shown below:



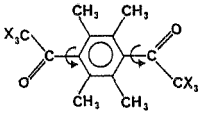
26

The two conformationally stable rotamers could even be separated by manual crystal selection.

Ermer (1983) studied the conformational inversion of tetraisopropylethylene, 27, via a no cogwheel (gearing) mechanism with an inversion barrier of 17 kcal/mol. With the help of molecular mechanical calculations, he was able to map the interconversion pathway, which proceeds stepwise such that the four isopropyl groups rotate consecutively via three intermediate minima.

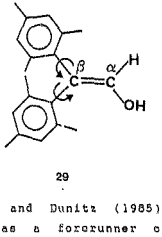


Pinkus and Kalyanam (1978) presented results on the cis-trans isomerism in bis(trihalogenoacetyl)polymethylbenzenes, 28, resulting from restricted rotation about carbonyl groups. NMR evidence suggested an angle of 90 degrees between the plane through the carbonyl group and the mean benzene plane. The free energy of activation was determined as 18.8 kcal/mol.



28

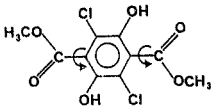
Nugiel et al. (1984) investigated correlated rotation in 2,2-dimesitylethenols like 29. Diaryl- and triarylvinyl systems exist in a propeller conformation. Correlated rotation in molecular propellers is commonly analysed in terms of flip mechanisms, involving helicity reversal. They concluded that the threshold rotational mechanism of 29, is the beta-ring flip which exchanges the two pairs of diastereotopic groups on the beta ring and has a barrier of 10.4 kcal/mol.



29

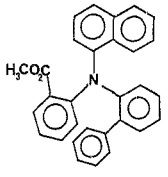
Yang, Richardson and Dunitz (1985) studied internal molecular motion as a forerunner of a phase change

involving conformational isomerisation. They accepted the notion that the vibrational behaviour of a molecule in its electronic ground state is related to the pathways for its unimolecular reactions as intuitively appealing. In their paper they described variable temperature X-ray diffraction results that showed a connection between internal molecular motion and the conformational isomerisation for the dimethyl-3,6-dichloro-2,5-dihydroxyterephthalate, 30.



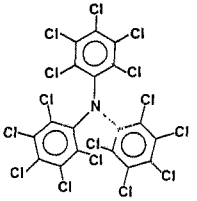
30

Residual diastereoisomerism in a maximally labelled triarylamine, methyl N-(biphenyl-2-yl)-N-(1-naphthyl)-anthramilate, 31, was demonstrated for the first time by Glaser, Blount and Mislow (1980). The two stereoisomers were obtained by manual separation of crystals, and their structures were determined by X-ray analysis. All single-step stereoisomerisations of these systems involve correlated torsional motion of the three rings, resulting in a reversal of propeller helicity. In 1976, Mislow gave a full account on the stereochemical consequences of correlated rotation in molecular propellers.



31

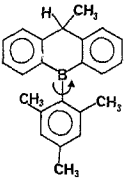
Hayes et al. (1980), in the course of their studies on molecular propellers, used empirical force field calculations to estimate the energy requirements for enantiomerisation of compounds in which three mesityl groups are attached to a common center. The structure of perchlorotriphenylamine, 32, was determined by X-ray analysis. Partial resolution was achieved by chromatography on microcrystalline cellulose triacetate. The racemisation barrier was calculated to be 26.0 kcal/mol.



32

Finocchiaro et al. (1980) dealt with the static and dynamic stereochemistry of propeller-like molecules. In order to have more information on the ground state geometry of 9-mesityl-9,10-dihydro-9-boraanthracene, 33, they turned to an X-ray structure determination. From

DNMR experiments, they were able to calculate a barrier of 12.9 kcal/mol for the B-C rotation in 33.



33

A less direct, but possibly more general approach to extracting dynamic information from crystallographic data is known as the structure-correlation method (Bürgi and Dunitz, 1983). The basic assumption behind the structure-correlation method is that a distribution of sample points corresponding to observed structures will tend to be concentrated in low-lying regions of the potential energy surface.

In this brief account of the role of hindered internal rotation in organic compounds, examples have been considered from a variety of compounds. Much interesting work has also been carried out on molecules by means of microwave spectroscopy, dipole measurements, relaxation methods, kinetics and various spectroscopic methods. Indeed, with increasing computer facilities, the time may be close that quite accurate structural data may be derived for macromolecules based on the experimental findings for the constituent monomeric entities.

In conclusion it should be noted that the considerable interest that scientists have in the details of the molecular structure of simple compounds, may be appreciated when one considers that the same forces which dictate the preferred conformation of N-methyl acetamide probably contribute substantially to the structure of the

alpha-helix found in proteins.

CHAPTER 3

EXPLORATORY STUDIES

3.1 Introduction

In the course of this study on internal rotation, conformation in different physical states, and potential barriers, numerous crystallographic studies of organic and organo-metallic compounds, of which only a few are reported in this thesis, were examined. The purpose of investigating such a variety of structure determinations was *firstly* to *become familiar* with the fundamentals of crystallography, and secondly, to provide an in depth look at the fascinating subject of molecular conformation. Any crystal structure corresponds to a free energy minimum which can usually be identified with a potential energy minimum. An additional entropy term must be included for disordered structures. Many conformations of a molecule may also be *energetically equivalent*, or nearly so, and one should be aware of the possibility of a number of conformations appearing in different crystal structures, or even in the same crystal structure. Certain packing modes are more favourable than others and this may lead to a predominance of one conformation in a crystal structure, while in solution a different conformation may be present. Thus, the potential energy *minimum represents a* balance between a wide variety of attractive and repulsive interactions. Hydrogen bonding is a very important force governing molecular conformation, and in the pages that follow, a few structures that exhibit hydrogen bonding will be discussed.

3.2 The Structure of Cis-4a,9a-dihydroxy-7-methyl-1,2,3,4,4a,9a-hexahydro-9(10H)-acridone

CHAPTER 3

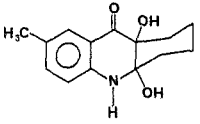
EXPLORATORY STUDIES

3.1 Introduction

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3.2 The Structure of Cis-4a,9a-dihydroxy-7-methyl-1,2,3,4,4a,9a-hexahydro-9(10H)-acridone

A hydrogen bond is a bond between a functional group A-H and an atom or group of atoms B in the same or different molecule. Hydrogen bond strengths are generally estimated to be 5-10 kcal/mol per hydrogen bond. The structure of cis-4a,9a-dihydroxy-7-methyl-1,2,3,4,4a,9a-hexahydro-9(10H)-acridone, OR24, was determined to establish the effects of hydrogen bonding on the conformation of the molecule (Boeyens, Denner, Marais and Staskun, 1986).



OR24

A yellow, prismatic single crystal of OR24 was used for the diffraction work. Preliminary Weissenberg and oscillation photographs indicated that the crystal was monoclinic, space group $P2_1/c$. Accurate lattice parameters were determined from 25 accurately measured theta values on a CAD4 automatic Nonius diffractometer (See experimental section in Chapter 8 for operational conditions). The necessary crystal data and crystallographic information are outlined in Table 1. Tables of crystallographic details, atomic coordinates and bond lengths and angles are listed in Appendix III. Three standard reflections monitored every hour showed only statistical fluctuations. The data were corrected for Lorentz-polarisation factors and absorption.

The structure was solved by direct methods using a prerelease version of the program SHELXS86 (Sheldrick, 1985) and refined by full-matrix least squares, using the program SHELX76 (Sheldrick, 1976). Some of the hydrogen atoms could not be located on a difference Fourier map

and were placed in calculated positions. A common isotropic temperature factor was refined for all hydrogen atoms. The refined atomic coordinates are in Table 2, with relevant bond lengths and angles in Table 3. Anisotropic thermal parameters, hydrogen atom coordinates and observed and calculated structure factors are listed in Appendix II. The molecular structure and atomic numbering scheme are shown in Figure 2. A packing diagram, showing the hydrogen bonds, is depicted in Figure 3.

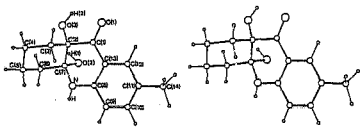


Fig.2. Molecular structure of OR24.

The calculated geometrical parameters are in agreement with accepted literature values (Kennard et al., 1972). The six-membered puckered ring, A, has the symmetrical flattened half-chair conformation, $^{2}H_{5}$ according to the parameters of pucker (Roeyens, 1978); phi = 95 degrees, theta = 49 degrees, with an amplitude Q = 0.41 angstrom. The cyclohexane ring, B, is in the chair conformation $^{1}C_{4}$, with puckering parameters: phi = 89 degrees, theta = 4 degrees and amplitude Q = 0.57 angstrom.

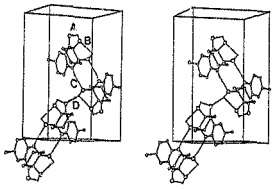


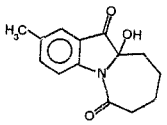
Fig.3. Packing diagram for OR24.

An interesting system of hydrogen bonds (Figure 3), four in total, exists within the crystal. Two intramolecular and two intermolecular hydrogen bonds can be identified. A strong hydrogen bond [A, O(1)...O(3) = 2.653 and O(1)...H(2) = 2.068 angstrom] binds the ketone oxygen with the alpha-hydroxy group. The second intramolecular hydrogen bond [B, O(2)...O(3) = 2.748 and O(3)...H(1) = 2.429 angstrom] is found between the hydroxy groups of the cis-diol. Furthermore, two molecules are held together across a centre of symmetry by the dimeric hydrogen bond [C, O(2)...N = 3.048 and O(2)...H = 2.103 angstrom]. The fourth hydrogen bond [D, O(1)...O(2) = 2.905 and O(1)...H(1) = 2.012 angstrom] occurs between two adjacent molecules in the crystal lattice, located at equivalent positions x,y,z and -x,y+0.5,-z+0.5. It is interesting to note that O(1) forms a bifurcated hydrogen bond, through H(1) and H(2) as intramolecular and intermolecular interactions respectively. This is a good example of how intramolecular hydrogen bonding can effect the conformation of a hydroxyl group.

3.3 The Structure of the lactam, OR43

Crystallography

A suitable crystal of OR43 was mounted on a glass fibre and attached to a goniometer. Approximate cell parameters, crystal system and the space group were deduced from oscillation and Weissenberg photographs. The crystal was transferred to an Enraf Nonius CAD4 diffractometer (See Chapter 8 for operational details), equipped with a graphite crystal monochromator and using Mo-K(alpha) radiation. Accurate cell parameters were obtained by refinement of 25 high order reflections. The relevant crystal data and details of the crystallographic analysis appear in Table 4. The collected intensity data were corrected for background, Lp and absorption.



OR43

The structure was solved by direct methods using the program SHELXS86 (Sheldrick, 1985) and refined by full-matrix least-squares using the program SHELX76 (Sheldrick, 1976). The 18 non-hydrogen atoms were assigned anisotropic temperature factors during the last cycles of refinement. Some of the hydrogen atoms could not be identified on a difference Fourier map. These hydrogen atoms were placed in calculated positions and an overall isotropic temperature factor was assigned to all hydrogen atoms. The final atomic coordinates appear in Table 5, and the bond lengths and angles are listed in Table 6. The supplementary material, calculated and observed structure amplitudes, thermal parameters for non-hydrogen atoms, and fractional atomic coordinates for hydrogen atoms, appear in Appendix II.

Results and Discussion

A stereoscopic drawing of OR43 showing the numbering scheme is depicted in Figure 4.

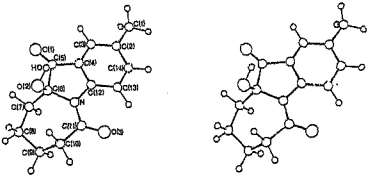


Fig.4. Molecular Structure of OR43.

The observed structural parameters agree well with that observed elsewhere (Kennard et al., 1972). The five-membered ring [N-C(12)-C(4)-C(5)-C(6)] has puckering parameters of Q = 0.01 angstrom and phi = 71.2 degrees. The seven membered ring [N-C(6)-C(7)-C(8)-C(9)C(10)-C(11)] is in the C4 conformation with puckering parameters: Q(2) = 0.40 angstrom, phi(2) = 41.4 degrees and Q(3) = 0.64 angstrom, phi(3) = 76.0 degrees.

There are two intramolecular hydrogen bonds. The first is between O(1) and OH [O(1)...O(2) = 2.964 angstrom and O(1)...HO = 2.898 angstrom. This is a weak hydrogen bond. The second hydrogen bond is between the amide oxygen and a hydrogen atom on the aromatic ring [O(3)...H(131) = 2.242 angstrom]. The packing of the molecules in the crystal is strengthened by a strong intermolecular hydrogen bond, as is depicted in the packing diagram of OR43 in Figure 5.

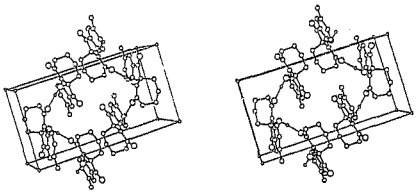
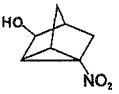


Fig.5. Packing diagram of OR43. All hydrogen atoms (except hydroxyl proton) have been omitted for clarity).

The strong intermolecular hydrogen bond is between the hydroxyl proton and the ketone oxygen atom [O(2)...O(3) = 2.759 and O(3)...HO = 1.809 angstrom].

3.4 The Structure of (1R*,3S*)-1-Nitrotricyclo-[2.2.1.0^{2,6}]-heptan-3-ol

The strain, nitro group conformation and hydrogen bonding characteristics of (1R*,3S*)-1-nitrotricyclo-[2.2.1.0^{2,6}]-heptan-3-ol, OR03, were investigated by means of single crystal X-ray analysis (Boeyens, Denner and Michael, 1986).



OR03

Bicyclo[2.2.1]heptanes provide a popular and readily accessible class of compounds in which interactions between appended functional groups may be examined free from the conformational complications that arise when systems containing less rigid skeletons are studied. Our

own interest is in the hydrogen bonding capabilities of less common donor or acceptor groups attached to the bicyclo[2.2.1]heptane framework.

Crystals of OR03 were grown by slow diffusion of hexane vapour into a solution of the compound in chloroform. The crystal used for data collection was a colourless prism. The space group and preliminary lattice constants were obtained from oscillation and Weissenberg photographs. Data were collected on an Enraf Nonius CAD4 diffractometer with Mo-K(alpha) radiation monochromated by a square graphite crystal. The necessary operating conditions used for the CAD4, are discussed in Chapter 8. The relevant crystal and data collection parameters are given in Table 7. The structure using the SHELX76 (Sheldrick, 1978) routine for centrosymmetric structures. The best set of signs, in terms of figures of merit, produced an E-map showing all the non-hydrogen atoms of the three molecules in the asymmetric unit. Their positions and individual isotropic temperature factors were adjusted by cycles of full-matrix least-squares refinement using SHELX76 (Sheldrick, 1978). In the final cycles, anisotropic temperature factors were assigned to all non-hydrogen atoms. A few hydrogen atoms could be located from a difference Fourier synthesis. The remaining hydrogen atoms, except for those of the three hydroxyl groups, were placed in calculated positions. The isotropic temperature factor of all the hydrogen atoms was refined as a single parameter.

The fractional atomic coordinates and bond lengths and angles are given in Tables 8 to 9. Anisotropic thermal parameters, hydrogen atom coordinates and calculated and observed structure factors appear in Appendix II. Figure 6 shows a diagram of the asymmetric unit in the structure of OR03, as well as the numbering scheme used.

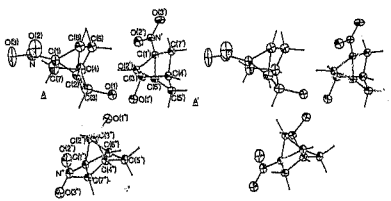


Fig. 6. The asymmetric unit of OR03. Thermal ellipsoids are at 50% probability.

The three molecules of OR03 (labelled A, A' and A" for convenience), linked sequentially by OH...OH hydrogen bonds, form the asymmetric unit. The hydroxylic protons did not appear during refinement, and their location in the hydrogen bonds is inferred from parameters to be discussed below.

Molecule A in the asymmetric unit belongs to one enantiomeric series, while A' and A" belong to another. The geometries of the three molecules are not quite identical, the biggest discrepancies (up to 0.05 angstrom in C-C bond lengths, and 4.3 degrees in bond angles) occurring with molecule A. The cyclopropyl rings in all three molecules approximate equilateral triangles. In fact, the only noteworthy differences between the three molecules in the asymmetric unit relate to the geometry of the nitro-groups. While those in molecules A' and A" are symmetrical (average N-O bond length, 1.216 angstrom), that in molecule A is noticeably unsymmetrical [N-O(2) = 1.269(8) angstrom, and N-O(3) = 1.172(7) angstrom]. At the same time, the temperature factors for the oxygen atoms in this group are substantially the largest in the whole system, indicating possible rotational disorder.

The plane of the nitro-group in A nearly coincides with the crystallographic (110) plane; both the a and b axes are very much shorter than the c axis, and thus the much smaller number of data points collected in this zone leads to less precision in the refinement. The pronounced elongation of the thermal ellipsoids of the oxygen atoms in the c direction are consistent with some rotary motion of the nitro-group, though why the phenomenon should be manifested with molecule A alone is unclear.

A more fascinating feature is the arrangement of molecules within the crystal (Figure 7).

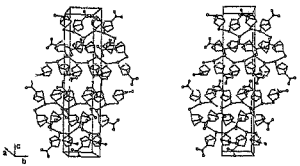


Fig.7. Stereoscopic packing diagram of OR03 showing the hydrogen bonds between the oxygen atoms. Hydrogen atoms have been omitted for clarity.

From the short O(1)...O(1) distances [O(1)...O(1') = 2.708 angstrom, O(1")...O(1) = 2.678 angstrom], we infer that hydrogen bonding between alcohol groups is the chief intermolecular force which dictates the mode in which molecules pack. The distances quoted all compare well with the mean O...O distance of 2.772 angstrom found in a statistical analysis of 45 crystal structures containing 196 OH...OH hydrogen bonds (Kroon et al., 1975). Most significantly, in terms of the aims of this study, there are no OH...O_2N interactions whatsoever. The hydrogen bonds show the thermodynamically favourable "cooperative effect" in that they link up to form infinite

(...OH...OH...) chains (Jeffrey and Takagi, 1978). The chains, four per unit cell, are propagated in the b direction; when viewed down the b axis, each chain can be seen to curve around in a helix of triangular cross-section. The sequence of the molecules in the helix is A...A'...A", and each asymmetric unit goes on to associate only with its neighbour in the same symmetry position of the contiguous unit cell in the b direction. The arrangement of hydrogen bonds with respect to the connecting O-C bonds, shown schematically in Figure 8, displays a systematic dissymmetry, consistent with a set of interaction vectors of uniform orientation along the ...OH...OH...OH... helix.

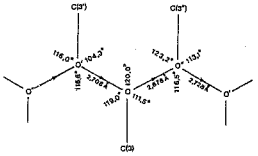


Fig.8. Schematic diagram showing the hydrogen bonds in OR03.

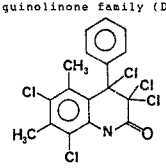
On the usual VSEPR assumption that the presence of lone pairs on an atom causes steric compression of other bond angles around that atom, the small angle at each oxygen is interpreted to represent the angle C-O-H.

In order to achieve the compact structure shown in Figure 6, the molecules pack into the crystal in a complex fashion. While molecules A and A' lie to one side of the hydrogen-bonded helix, A" lies to the other side. In (100) projection asymmetric units are perceived to stack pairwise in the [001] direction, in a sort of triple-decker sandwich, with two hydrogen-bonded helixes acting as filling between three layers of tricycloheptane skeleton. The last point of interest concerns the relative conformations of the nitro-groups and the

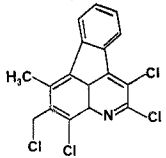
cyclopropane rings. In all three molecules the N-O(3) bond comes within 5 degrees of eclipsing the C(1)-C(7) bond, implying that the plane of the nitro-group bisects the C(2)-C(1)-C(6) angle. Crystallographic precedents for this phenomenon already exists, and the Cambridge Structural Database (November 1984 update) contains nine structures possessing twelve nitrocyclopropyl fragments between them. Geometrical data are not retrievable for all of these compounds, but seven of the fragments have conformations that match the present example closely (Kai et al., 1982; Beintema, 1976; Schenk and Benci, 1972; and Stam and Evers, 1965).

3.5 The Structures of Two Indeno[1,2,3-de]quinolinones.

X-ray crystallography was used to determine the solid state structures of OR07 and OR12, two members of the quinolinone family (Denner et al., 1985).



OR07



OR12

Preliminary cell constants and space group information were obtained by standard photographic techniques. Refined cell parameters and subsequent data collection were recorded on a Philips PW1100 diffractometer. Crystal data and crystallographic parameters are listed in Tables 10 and 11.

The structures of OR07 and OR12 were solved using the direct methods package in the program SHELX76 (Sheldrick, 1976). The structures were refined by full-matrix least-squares, employing anisotropic thermal parameters for all

non-hydrogen atoms and a common isotropic temperature factor for all the hydrogen atoms. The hydrogen atoms that were not apparent on the difference Fourier maps were placed in calculated positions. To ensure meaningful refinement of the structure of OR12, the C(12)-C(13) bond length was constrained to a value of 1.54 angstrom rendering the estimated standard deviation for this bond statistically meaningless.

Discussion

The molecular structures of OR07 and OR12 are shown in Figures 9 and 10, together with the crystallographic numbering schemes.

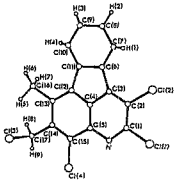


Fig.9. The crystal structure of OR07.

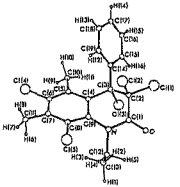


Fig.10. The crystal structure of OR12.

The atomic coordinates and bond lengths and angles are listed in Tables 12 to 15. Anisotropic thermal parameters, hydrogen atom coordinates and calculated and observed structure factors are listed in Appendix II. The bond lengths and angles for these compounds agree well with accepted literature values (Kennard et al., 1972). The structure of OR07 is interesting in that it is essentially planar, except for the Cl(3)-atom which is displaced from the molecular plane, owing to the steric repulsion of the substituents on C(13) and C(15), respectively. This relief of steric crowding can clearly be seen in the packing diagram in Figure 11.

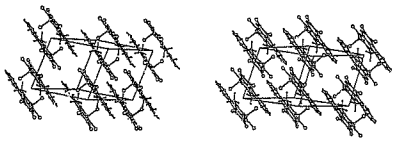


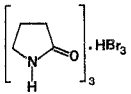
Fig.11. Packing diagram for OR07.

The structure of OR12 is dictated by the conformation of the ethyl group and the phenyl group bonded to C(3). The two hydrogen atoms on C(12) are diasterotopic because of the hindered rotation around the N-C(12) bond, leaving these two hydrogen atoms, H(4) and H(5), in magnetically non-equivalent environments. Any substitution on C(3) is sterically demanding due to the presence of three bulky chlorine atoms as well as an adjacent methyl group. Thus, the only degree of freedom to reduce the strain in OR12 is torsional mobility around the C(3)-C(14) bond. The primary steric demand is interaction between the phenyl group and Cl(1) and Cl(2). Therefore, the almost

equidistant non-bonded contacts for C(15)...Cl(1) and C(19)...Cl(2) of 4.20 and 4.23 angstrom, respectively, are a good balance between these steric interactions.

3.6 The Structure of Tris(2-pyrrolidinone) Hydrogen Tribromide

The reaction of the cyclic amide 2-pyrrolidinone with bromine and hydrogen bromide in methanol, produces a compound of composition $(C_4H_7NO)_3HBr_3$, OR05, (Boeyens, Denner, Howard and Michael, 1986).



OR05

This compound, now commercially available, is a valuable reagent for the bromination of ketones. Exactly how the bromine is incorporated into the structure of the compound is, however, still only open to speculation. A crystallographic investigation of the title compound was done in order to clear up present uncertainties about the structure, and also to study the effects of hydrogen bonding on the solid state structure of OR05.

Dark-red distorted hexagonal prisms were used for the diffraction studies. Preliminary camera work indicated appreciable decomposition on exposure to X-rays, and the crystals also proved to be hygroscopic. A fairly large, freshly recrystallised crystal picked from the batch was cut into a cube and then ground with a Nonius crystal grinder into a sphere of diameter 0.11 mm. This crystal was sealed into a Lindemann tube. Cell constants initially obtained from Weissenberg and oscillation photographs were refined from centred settings of 25 high theta-angle reflections on an Enraf Nonius CAD4

diffractometer using graphite-monochromated Mo-K(alpha) radiation. The relevant crystal and data collection parameters are given in Table 16, and operating conditions of the diffractometer in Chapter 8.

The structure was solved by standard heavy-atom techniques using the program SHELX76 (Sheldrick, 1976). The positions of the remaining non-hydrogen atoms were obtained from the first difference Fourier synthesis, and were refined by full-matrix least-squares methods with anisotropic temperature factors for the bromine atoms only. The hydrogen atoms were placed in calculated positions (C-H and N-H = 1.08 angstrom) riding with an overall isotropic temperature factor. The final atomic parameters and lengths and angles are given in Tables 17 to 1 ies of observed and calculated structur factors, as well as anisotropic temperature factors and hydrogen atom coordinates appear in Appendix II.

The asymmetric unit of OR05, consisting of three pyrrolidinone molecules (labelled A, A' and A" for convenience), a hydrogen-bonded proton, and an indisputable tribromide ion, is illustrated in Figure 12, which also shows the numbering scheme adopted.

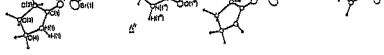


Fig.12. The asymmetric unit of OR05.

What is immediately apparent is that the tribromide ion is not quite symmetrical [Br(1)-Br(2) = 2.524(2) angstrom, and Br(2)-Br(3) = 2.545(3) angstrom], and slightly bent [Br(1)-Br(2)-Br(3) = 177.9(1) degrees]. This is not unusual, however. Fewer than half of the tribromide structures retrieved from the Cambridge Structural Database have a perfectly symmetrical tribromide ion; only four are linear, reflecting the fact that the central Br atom lies in a special position (Andressen et al., 1962; Lawton et al., 1977; Lawton, 1973; and Lawton, 1968). The present Br-Br bond lengths agree well with the average published bond length of 2.54 angstrom. The arrangements of the tribromide ions is also interesting. They are arranged in nearly colinear chains, and are separated by a distance of 3.49 angstrom. This is less than the sum of the van der Waals radii of two Br atoms (2 x 1.85 angstrom, Bondi, 1964), indicating some secondary interaction between the tribromide ions. Furthermore, the structure is a clathrate; the tribromide chains are located in channels formed by a matrix of 2-pyrrolidinone [101] and [010] directions of the unit cell. One such channel is shown in Figure 13.

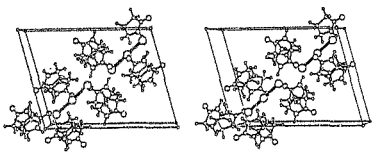


Fig.13 Stereoscopic view of the tribromide ion-containing channel in the [101] direction of the unit cell.

Stereoscopic view of the tribromide ion-containing channel in the [101] direction of the unit cell.

The *pyrrolidinone molecules* are held together by a complex series of intermolecular hydrogen bonds which will be

described below. The channels when viewed down the cavity (Figure 14), have a square cross-section, with A and A' of the asymmetric unit lying along the channel wall opposite to that which contains A".

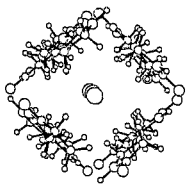


Fig.14. Cross-section of a channel in the lattice of OR05.

The distance across the channel is approximately 6.8 angstrom - large enough to accommodate a bromine, as measured by its van der Waals radius, with ease. The shortest non-bonded contact between a ring atom and a bromine atom, C(3') to Br(2), is 3.66 angstrom, and the closest N...Br contact is 3.78 angstrom. There is thus no hydrogen bonding between pyrrolidinone molecules and tribromide ions. The colinear arrangement of tribromide ions and the channel structure for the compound are, to our knowledge, unique amongst published tribromide structures. Similar situations are rare even with the more numerous triiodides; a recent case in point is found in the structure of tetrabutylammonium triiodide (Herbstein et al., 1981). It is interesting that the other structure in this publication, that of bis(benzamide) hydrogen triiodide, contains roughly rectangular channels through which run double strands of triiodide ions. The most fascinating feature of this tribromide structure is the nature and arrangement of the hydrogen bonds holding the 2-pyrrolidone matrix together.

These are shown in the accompanying packing diagram Figure 15.

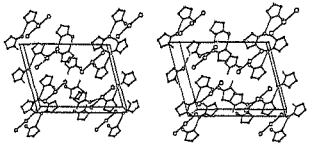


Fig.15. Stereoscopic packing diagram of OR05 showing the four different hydrogen bonding interactions. C-H bonds have been omitted for clarity.

No fewer than four different hydrogen-bonding arrangements can be identified, and the hydrogen bonds are of three distinct types. The normal amide-amide dimeric interaction involving C=O...H-N occurs in two situations. The first is between pyrrolidinone molecule A and the nearby molecule A in the asymmetric unit at symmetry position -x,-y,-z. The non-bonded distance is 2.96 angstrom for both N(1)...O(1) contacts, and the N(1)-H(1)...O(1) hydrogen bond lengths are 1.96 angstrom. The hydrogen atoms have, admittedly, been placed in calculated positions. Even so, the hydrogen bonds are very short, comparing favourably with the average length of 1.934 angstrom reported by Taylor et al. (1984) in a systematic survey of 597 such bonds. The hydrogen bonds are somewhat bent [N-H...O = 161.3 degrees]. The second intermolecular interaction is between pyrrolidinone molecule A" and its neighbour A" located at symmetry position 1-x,1-y,-z. In this case, the hydrogen bonds are marginally weaker; both N(1")...O(1") contacts are 3.01 angstrom, the N(1")-H(1")...O(1") hydrogen-bond lengths are 1.95 angstrom, and the N-H...O angles are 168.6 degrees.

The second type of hydrogen bond is again of the C=O...H-N variety, but is is a singly-bridging interaction. Pyrrolidinone molecules A and A' in the same asymmetric unit are arranged such that the O(1)...N(1') distance is 3.76 angstrom. The O(1)...H(1')-N(1') hydrogen bond is a strong one, as evidenced by the very short O...H length of 1.68 angstrom. In this case, the hydrogen bond is nearly linear [O...H-N = 173.9 degrees], and its orientation is approximately in the same direction as the channel axis.

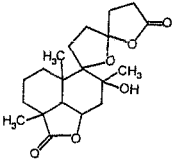
The third type of hydrogen bond involves the "free" proton which, unfortunately, did not become apparent during refinement due to the presence of heavy bromine atoms. The previously mentioned $Br_3^-...Br_3^-$ distance of 3.49 angstrom is short enough to allow proton bridging, but a similar suggestion involving triiodide ions (Reddy et al., 1964) has been convincingly refuted (Herbstein et al., 1981). However, if we consider the remarkably short distance of 2.45 angstrom between the two carbonyl oxygen atoms O(1') and O(1") of pyrrolidinones A' and A" at symmetry position -x,y+0.5,0.5-z, it becomes highly probable that the missing proton bridges these functionalities (Herbstein, 1985) thereby producing a very strong, possibly symmetrical, linear hydrogen bond. This situation is comparable with that in the structure of bis(benzamide) hydrogen triiodide, reported by Herbstein and his co-workers (1981), in which the O...O distance is 2.41 angstrom. As far as we are aware, ours is the first tribromide to incorporate a more-or-less free proton as cation.

It is worth mentioning, in conclusion, that most of the reported tribromides are unstable on exposure to X-rays, as experience confirms in the present case as well. This behaviour is presumably due to the loss of molecular bromine from the tribromide anions.

3.7 The Structure of 9a,13(S)-Epoxy-8-beta-hydroxy-labdane-6-beta,19;16,15-diolide

X-Ray Crystallography

A colourless, prismatic crystal of the compound OR42 was used for crystallographic measurements at room temperature. The crystal was mounted on an Enraf Nonius CAD4 Kappa-axis diffractometer, equipped with a graphite monochromator and a scintillation counter (Mo-K(alpha)) radiation, (See Chapter 8 for operational details). The necessary crystal data and details of the crystallographic refinement are reported in Table 19. Systematic absences were consistent with the space group $P2_12_12_1$.



OR42

The intensities and their standard deviations were corrected for Lorentz and polarisation effects. An empirical absorption correction was applied to the data and there was no evidence of crystal decomposition.

The structure was solved by direct methods using a prerelease version of SHELXS86 (Sheldrick, 1985) which gave an E map showing all 26 of the non-hydrogen atoms. The structure was refined with the program SHELX76 (Sheldrick, 1976) using the full-matrix least-squares procedure, first with isotropic and later with anisotropic temperature factors. At this stage some of the hydrogen atoms could be located from a difference Fourier map;

however, for simplicity they were all treated by the same procedure. All hydrogen atoms were therefore placed in calculated positions and refined subject to the constraint that the C-H vectors were constant in magnitude and direction but not position (i.e., a riding model). A common isotropic temperature factor was used for the hydrogen atoms. Final atomic parameters for non-hydrogen atoms are in Table 20. A list of bond lengths and angles appears in Table 21. Anisotropic thermal parameters for non-hydrogen atoms, and a list of hydrogen atom coordinates along with a Table of observed and calculated structure amplitudes, are listed in Appendix II.

Discussion

The structure of OR42 and the atomic numbering scheme are illustrated in Figure 16. Puckering parameters for rings A-E are given in Table 22. In the system of fused rings both six-membered rings are distorted from the energetically favoured chain conformation, although in one instance only slightly so. The envelope conformation of the five-membered rings and the orientation of the 18-methyl group force and twist conformation for the A-ring. The B-ring is distorted towards a half-chair.

Table 22. Puckering parameters* of the rings in OR42.

Ring	Q	Theta	Phi	Conf.
A C1-C10-C5-C4-C3-C2	0.78	94.9	267.4	$^2T_6$
B C5-C10-C9-C8-C7-C6	0.52	158.4	265.0	$^1C_4$
C O1-C9-C11-C12-C13	0.28	-	273.4	$^4T_3$
D O2-C19-C4-C5-C6	0.38	-	290.5	^4E
E O3-C15-C14-C13-C16	0.39	-	284.0	^4E-$^4T_3$

*Puckering parameters are given in the units: Q and angstrom, Theta and Phi in degrees.

however, for simplicity they were all treated by the same procedure. All hydrogen atoms were therefore placed in calculated positions and refined subject to the constraint that the C-H vectors were constant in magnitude and direction but not position (i.e., a riding model). A common isotropic temperature factor was used for the hydrogen atoms. Final atomic parameters for non-hydrogen atoms are in Table 20. A list of bond lengths and angles appears in Table 21. Anisotropic thermal parameters for non-hydrogen atoms, and a list of hydrogen atom coordinates along with a Table of observed and calculated structure amplitudes, are listed in Appendix II.

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Table 22. Puckering parameters* of the rings in OR42.

Ring	Q	Theta	Phi	Conf.
A C1-C10-C5-C4-C3-C2	0.78	94.9	267.4	$^2T_8$
B C5-C10-C9-C8-C7-C6	0.52	158.4	265.0	$^1C_4$
C O1-C9-C11-C12-C13	0.28	-	273.4	$^4T_3$
D O2-C19-C4-C5-C6	0.38	-	290.5	^4E
E O3-C15-C14-C13-C16	0.39	-	284.0	^4E-$^4T_3$

*Puckering parameters are given in the units: Q and angstrom, Theta and Phi in degrees.

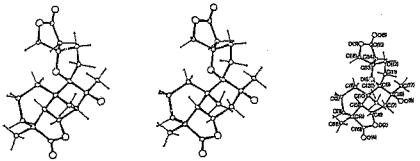


Fig.16. Molecular structure of OR42.

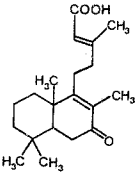
There is no evidence of intramolecular hydrogen bonding and the only intermolecular hydrogen bond occurs between O(6)-H...O(4), as evidenced by the oxygen separation of 3.08 angstrom. It is noted that the hydroxyl hydrogen atom could not be observed experimentally.

3.8 The Structure of (7-Oxo-labda-8,13-dien-15(E)oic acid)

Crystallography

Suitable single crystals of compound OR28 were obtained by recrystallisation from benzene-petroleum ether. Approximate unit cell parameters were determined from oscillation and Weissenberg photographs. Intensity data were collected on an automatic kappa-axis Enraf Nonius diffractometer, equipped with a graphite monochromator using Mo-K(alpha) radiation (See Chapter 8 for operational details). Refined unit cell dimensions were obtained by a least-squares fit of the theta angles of 25 high order reflections, widely separated in reciprocal space. Relevant crystallographic details appear in Table 23. The space group, P1 was inferred from X-ray photographs and the known chirality of the compound. The intensities of

three standard reflections monitored every hour remained essentially constant throughout the data collection. Lorentz and polarization factors, as well as an empirical absorption correction were applied.



OR28

The structure was solved by direct methods with the program SHELXS86 (Sheldrick, 1985). An E map, computed with the phases of highest figure of merit, revealed all the non-hydrogen atoms of the two molecules in the asymmetric unit, which were refined, first isotropically, then anisotropically using the program SHELX76 (Sheldrick, 1976). A difference synthesis located some of the hydrogen atoms in stereochemically feasible positions, but all hydrogen atoms, except for the two acidic protons, were eventually placed in calculated positions and refined with a common isotropic temperature factor. No significant residual electron density was present. Observed and calculated structure factors, anisotropic thermal parameters for the non-hydrogen atoms and fractional coordinates of the hydrogen atoms are listed in Appendix II.

Results and Discussion

A stereoscopic drawing of OR28 is given in Figure 17. It shows the absolute configuration of the acid. Only its relative configuration can be deduced from our X-ray data. The X-ray results fully confirm the structure which we suggested for OR28.

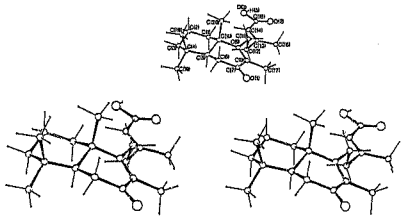


Fig.17. Molecular structure of OR26.

The final atomic parameters for the non-hydrogen atoms are listed in Table 24. Interatomic distances and bond angles are shown in Table 25.

The conformation of the six-membered rings was established by calculation of their appropriate puckering parameters. The rings (C1-C2-C3-C4-C5-C10) in both molecules have chair conformations (phi = 6 degrees and 5 degrees respectively, Q = 0.56 and 0.58 angstrom). The other rings (C5-C6-C7-C8-C9-C10) have almost identical envelope shapes, ^{1}E with puckering parameters phi = 0.4 and 0.5 degrees; theta = 57 and 57 degrees; Q = 0.51 and 0.52 angstrom.

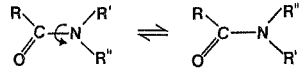
CHAPTER 4

CRYSTALLOGRAPHIC STUDY OF SIMPLE AMIDES AND THIOAMIDES

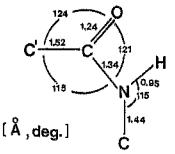
4.1 Introduction: Literature Survey

Much of the interest in the amide group is no doubt related to the fact that it is the repeating unit in the biologically important polypeptide macromolecules. Since the amide groups within the polypeptide molecule appear to be only weakly interacting, they retain their character to a large extent as isolated amide groups, and for this reason the study of isolated amides has been intense (Robin, 1971).

The simplest amide, formamide, has been shown to have a planar molecular framework and NMR spectroscopy and other techniques have shown quite unequivocally that internal rotation about the C-N bond in many simple amides is hindered by a fairly high potential barrier of the order of 10 kcal/mol (Kessler, 1970). This potential barrier is predominantly twofold in character, with the result that an asymmetrically disubstituted amide is able to exist as either of two rotamers, each with a planar skeletal framework, as shown below:



X-ray structure analyses of a variety of crystalline amides show a fairly constant geometry for the amide group as illustrated below (Robin et al., 1971; Hagler et al., 1976).



Amides have been studied by NMR spectroscopy more extensively than any other class of compounds, especially by DNMR from which barrier heights can be calculated. Most of the NMR studies of amides are concerned with the partial double-bond character of the amide C-N bond. The double bond character arises from the contribution of resonance structure.

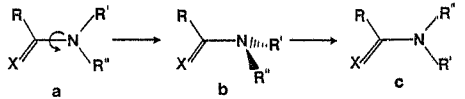


All of these considerations, however, will not be discussed in this survey. The reader is referred to extensive review articles and papers dealing with the NMR methodology of the amide linkage (Stewart and Siddall, 1970; Bourn et al., 1964; Hallam and Jones, 1970; Jennings and Saket, 1985; Lipshutz and co-workers, 1984; Fatoftah et al., 1985; Yamagami et al., 1986).

4.2 Stereochemistry of the C-N Fragment

The geometry of the C-N molecular fragment was studied in 90 X-ray crystallographically determined molecules containing such a group (amides, thioamides, enamines etc.) by Gilli and his co-workers (1986). They found that the amide group displays a definite tendency to be planar. In spite of this generic tendency towards planarity, several crystal structures have been found where the amide

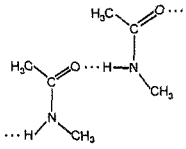
group undergoes out-of-plane distortions by rotation around the C-N bond and/or nitrogen pyramidalisation. (Winkler and Dunitz, 1971; Mühlebach et al., 1986). The basic idea of these authors was to analyse the out-of-plane distortions in a large number of molecules containing the C-N fragment, with the aim of finding the geometrical modifications along the path of what could be considered the reaction of cis, trans-isomerisation (a to b to c) of the group itself.



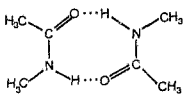
This is an application of the so-called structure correlation method (Bürgi and Dunitz, 1983).

Substituents are generally believed to affect the rotational barriers in amides and thioamides by some combination of steric and electronic effects. A multiple substituent parameter analysis of the effects of substituents at nitrogen on the barriers in amides was carried out by Yoder and Gardner in 1981. Their studies revealed that the steric effect was of considerably greater importance in explaining the barrier than the inductive effect. Generally, increasing conjugation capacity of a group will lower the C-N barrier. In the planar state, crowding will lower the barrier, and in the transition state the effect is opposite (Isaksson et al., 1967).

Of particular interest have been the N-mono-substituted amides since they constitute the simplest models for studying the peptide link. Hydrogen bonding plays an important role in both solid and solution conformations. N-mono-substituted amides and thioamides can associate via linear multimers (A) or through the formation of cyclic dimers (B).



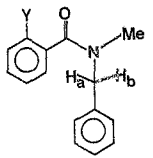
A



B

Several theoretical calculations of the number of possible conformations of a polypeptide chain have been made, in particular by Scheraga (1968) and Brant and Flory (1965), which indicate the importance of steric restrictions in reducing conformational freedom. In all these approaches the planar-trans amide conformation is used for the peptide unit although the possibility of non-planar distortion of the group is recognised.

Recently there has been considerable discussion in the literature regarding the origin of the chemical shift non-equivalence of geminal methylene hydrogen atoms in the N-alkyl groups (Berg and co-workers, 1980; Siddall, 1966; Shvo et al., 1967). Rotation around the C(X)-N bond in 34 is normally slow on the NMR time-scale at around ambient temperature as shown by the observation of two sets of N-alkyl signals.



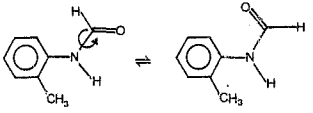
34

Additionally, steric interactions between the ortho Y or H substituents and the proximate $N-CH_2$ group or oxygen/sulfur atom normally force the aryl ring to twist out of the amide/thioamide plane. Provided that rotation

of the aryl ring through the amide/thioamide plane is slow, the molecule is chiral on the NMR time-scale and the paired geminal hydrogen atoms attached to the prochiral methylene carbons are diastereotopic and potentially anisochronous.

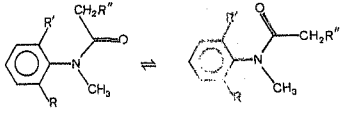
In view of the vast literature on amides, thioamides and selenoamides, this survey is restricted to the actual isolation in the crystalline form of systems exhibiting restricted internal rotation around a C-N bond.

Perhaps the most significant study that has recently been reported on rotamers is that of Siddall et al. (1968) on o-methyl-formanilide, 35. They reported the isolation of both cis and trans conformers of this amide and ... that this is the first time both pure cis and trans ... had been isolated for a secondary amide. The ... equilibrium cis:trans isomer ratio was found to be 3:1.



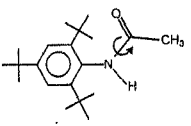
35 Z

Chupp and Olin (1967) were able to isolate stable rotamers of 2,6-disubstituted N-(haloacetyl)-N-methylanilines, 36, and to show that their reactivities were quite different:



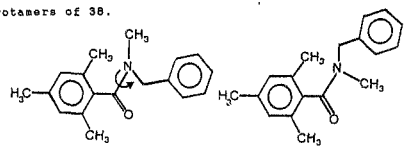
36

Another example was provided by Kessler and Rieker (1967), who isolated rotamers of 2,4,6-tri-tert-butylacetanilide, 37. The potential barrier to isomerisation was found by DNMR spectroscopy to be 23.9 kcal/mol.



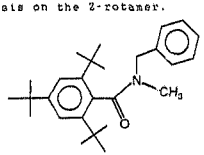
37

Mannschreck (1965) discussed the formation and isolation of rotamers of 38.



38

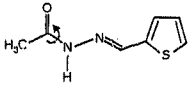
A sterically more crowded molecule analogous to 38, namely N-methyl-N-benzyl-2,4,6-tri-tert-butylbenzamide, 39, was separated in its rotameric forms by Jungk and Schmidt (1971). These authors performed an X-ray crystal structure analysis on the Z-rotamer.



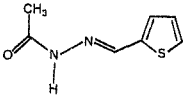
39

N-arylhydrazones of methyl pyruvate and of aromatic aldehydes show considerable chelating power with transition metals, and can be used to recover and analyse metals selectively as hydrazone complexes (Palia et al., 1986). These authors recently found, using chromatographic techniques, that several hydrazones of

methyl pyruvate existed in solution as mixtures of E and Z geometrical isomers, whose ratio depended on the nature of the solvent and influenced their chelating capability. This picture was confirmed by X-ray structural analyses carried out on two crystalline rotamers of 2-formylthiophene-N-acetylhydrazone, 40, (Domiano et al., 1986).

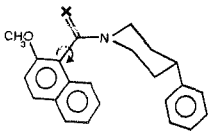


⇅

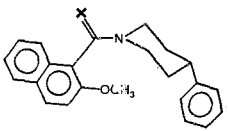


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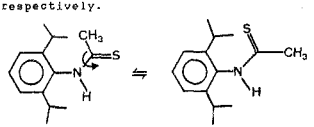
When the amide oxygen is replaced by sulphur or selenium, the amide rotational barriers increase (Stewart and Siddall, 1970). For analogous amides and thioamides the increase can be as much as 5 kcal/mol, although a 2-3 kcal/mol is more typical. The increase is probably due to increased electron delocalisation and increased size. Recently, Fraser and Taymaz (1976) reported the measurement of high barriers to rotation about the Ar-C=O bond of a benzamide and the Ar-C=S bond of a thioamide. The two compounds under study were 2-methoxy-1-naphthoyl-4-phenylpiperidine, 41, and 2-methoxy-1-thionaphthoyl-4-phenylpiperidine, 42. The barriers for isomerisation of 8 and 9 are 20.7 and 23.8 kcal/mol.



⇵ X=O 41
X=S 42



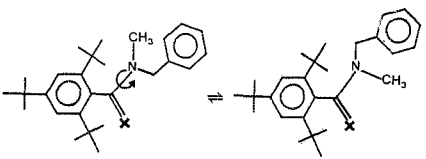
The E- and Z-isomers of 2,6-di-isopropylthioacetanilide, 43, have been isolated by fractional crystallisation (Walter and Becker, 1971). The E to Z and Z to E barriers to internal rotation were found to be 24.16 and 23.74 kcal/mol, respectively.



43

Mannschreck (1965) achieved the separation of rotamers of amides, *thioamides and nitrosamines*. For two different substituents on the N-atom, two barriers need to be specified. He found barriers of 27.3 and 26.8 kcal/mol for 44. The corresponding oxygen compounds, 45, gave values of 22.9 and 22.3 kcal/mol, respectively. The crystalline substances were stable, but in solution an

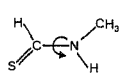
equilibrium mixture was formed.



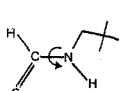
X = O 44

X = S 45

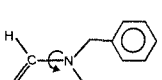
In 1971, Walter and Schauman succeeded in the separation of geometrical isomers and determination of rotational barriers of N-alkylformamides. The separation into the different crystalline forms was achieved by preparative thin layer chromatography. N-methyl-, 46, N-neopentyl-, 47, and N-benzylthioformamide, 48, as well as N-methylthioacetamide, 49, were separated into their respective rotameric forms. DNMR spectroscopy revealed the following barrier heights in kcal/mol for Z to E and E to Z isomerisations measured in deuterated chloroform: 46, (24.5 and 23.2); 47, (24.9 and 24.7); 48, (24.2 and 23.2); 49, (22.2 and 20.4).



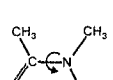
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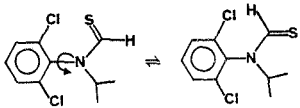


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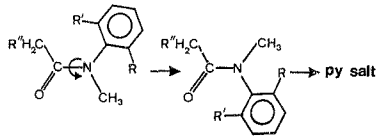
49

Walter and Becker (1972) investigated the barrier to rotation of N-isopropyl-N-(2,6-dichlorophenyl)thioformamide, 50. Its Z form crystallised, but on dissolution in carbon tetrachloride it slowly isomerised. The free energy of activation was 22.02 kcal/mol for the Z to E process and 22.31 kcal/mol for the reverse.



50

A number of atropisomers reported in the literature are discussed in this chapter. Obviously many more systems that can give rise to stable rotamers may be found both by chance and by systematic study. Although, in general one can say that such a system should have a relaxed ground state and a highly congested transition state for rotation, it is not an easy task at present to predict what systems may give stable atropisomers. Also, knowledge of the reactivities of rotamers is becoming increasingly important (Oki 1984), esperially because, by aiming at highly selective reactions, synthetic organic chemists utilise low reaction temperatures and/or complexation with metals to lock molecules into desired conformations. It is anticipated that, in future, the reactivity of a locked conformation of this type might be predicted. Atropisomers might serve as models in this area. As an example, Chupp and Olin (1967) examined the separate reactivities of stable rotamers of the haloacetamide derivative below in Menschutkin reactions with pyridine. They found that although the Z form reacted directly with pyridine, the reactivity of the E form was so low that it had to rotate to the Z form to react. Therefore, the rate determining step in the reaction of the E form is essentially the internal rotation.

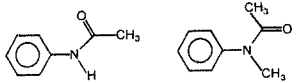


E Z

This example of differential reactivities of rotamers suggests that the reactivity is controlled by a steric factor.

4.3 Results and Discussion

Amides represent very simple, but intriguing, models for studying conformational behaviour of molecules. The amide moiety is particularly sensitive towards steric effects of substituents. For example, acetanilide, 51, (Brown, 1966) prefers the E conformation, whereas N-methyl-acetanilide, 52, (Pedersen, 1967) is found in the Z conformation. The conformation of the phenyl ring with respect to the amide framework is also very different in 51 and 52. In 52 it is perpendicular to the amide plane and in 51 it is not.

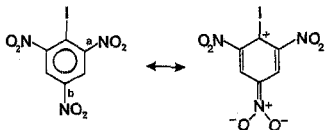


51 52

Furthermore, the amount of Z isomer increases for acetanilides as the bulk of ortho substituents increases (Stewart and Siddall, 1970). This increase is paralleled by an increase in the barrier to rotation.

These interesting findings on relatively simple structural models inspired a brief study of some of the conformational parameters of several para-substituted acetanilides. The reason for choosing para-substituted derivatives lies in the question of how the resonance effect would influence bond lengths in these delocalised

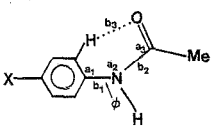
systems. By analogy, bond lengths and angles for the o- and p-nitro groups in picryl iodide, 53, are quite different (*Wepster*, 1958).



53

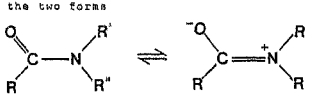
Distance a is 1.45 angstrom, whereas b is 1.35 angstrom. The obvious explanation is that the oxygens of the p-nitro group are in the plane of the ring and thus in resonance with it, so that b has partial double-bond character, while the oxygens of the o-nitro groups are forced out of the plane by the large iodine atom (March, 1977).

The parameters chosen are shown in the diagram below.



The diagnostic parameters are listed in Table 26.

A significant feature of these molecules is the b_2 bond length (of the order of 1.34 angstrom) which is much shorter than the sum of the covalent atomic radii (1.475 angstrom) for a single bond. In view of the resonance between the two forms



Compound R factor	Parameters* b_1	b_2	b_3	a_1	a_2	a_3	phi	Reference
Acetanilide R = 0.0[illegible]	1.413(3)	1.354(3)	-	123.8(2)	127.6(2)	123.1(2)	17.6	*Brown, 1966.*
p-Hydroxyacetanilide (Monoclinic form) R = 0.072	1.425(3)	1.340(3)	2.38(3)	124.3(2)	128.3(2)	122.8(3)	21.1	Haisa et al., 1976.
p-Hydroxyacetanilide (Orthorhombic form) R = 0.077	1.422(5)	1.341(6)	2.34(4)	123.7(3)	129.8(4)	122.8(4)	17.7	Haisa et al., 1974.
p-Methylacetanilide (Monoclinic form) R = 0.084	1.416(3)	1.349(3)	2.35(4)	124.2(2)	127.9(2)	123.5(2)	18.7	Haisa et al., 1977.
p-Methylacetanilide (Orthorhombic form) R = 0.088	1.409(8)	1.352(9)	2.24(5)	125.0(6)	128.6(6)	123.5(6)	5.7	Haisa et al., 1977.
p-Aminoacetanilide R = 0.084	1.421(4)	1.344(4)	2.57(4)	123.3(3)	128.0(3)	123.1(3)	34.4	Haisa et al., 1977.
p-Ethoxyacetanilide R = 0.088	1.40(1)	1.34(1)	-	123.5(7)	127.0(7)	123.2(8)	29	Patel et al., 1983.
p-Chloroacetanilide R = 0.14	1.43(3)	1.34(3)	-	126(3)	127(3)	121(3)	5	Subramanian, 1966.
p-Bromoacetanilide R = 0.082	1.44(3)	1.30(3)	-	124.7(2)	128.9(2)	123.8(3)	5.8	Andreetti et al., 1968.

* Bond lengths in angstrom and angles in degrees.

Table 26.

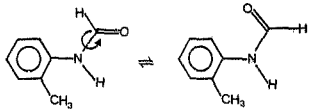
the bond is expected to show considerable double bond character. In fact, the b_2 bond length would seem to indicate that there is substantial contribution by the structure B to the resonance. However, if we exclude the structures of p-chloroacetanilide and the orthorhombic form of p-methylacetanilide from our analysis, it would appear that the effect of para-substitution on the bond lengths b_1 and b_2 is insignificantly small, except for p-bromoacetanilide, with bond lengths of b_1 = 1.44(3) and b_2 = 1.30(3) angstrom, respectively. The answer to this lies in the angle, phi = 5.8 degrees. It is common knowledge that resonance is maximised for planar structures. For the other structures the angle phi varies from 17.6 to 34.4 degrees.

Another very interesting feature that emerged from this analysis is the values of the three angles a_1, a_2 and a_3; all these angles are larger than 120 degrees. The mean values for the seven structures investigated are: a_1 = 123.9(6); a_2 = 128.2(7) and a_3 = 123.2(8) degrees. The reason for this distortion must be due to the acetyl group being very nearly coplanar with the rest of the molecule. The steric hindrance caused by the proximity of the ortho-hydrogen of the benzene ring and the oxygen of the same molecule (b_3) tends to increase the angles a_1, a_2 and a_3. The distance b_3 is significantly smaller than the sum of the van der Waal's radii. It is important to note that all the above mentioned molecules are involved in intermolecular hydrogen bonding.

4.4 Monosubstituted Amides

Very few studies of the rates of rotation around the amide bond in monosubstituted amides have been reported (Stewart and Siddall, 1970). In part this has been due to the preponderance of the E form over the Z form. Isolation, by crystallisation, of the pure isomers of o-methylformanilide, OR09, allowed study of the rotation

rate by equilibration (Siddall et al., 1968). However, owing to the strong intermolecular association of monosubstituted amides, the overall process may involve making and breaking of hydrogen bonds as well as rotation. The detailed explanation for the effects of varying the ortho substituents may be quite complicated. The ortho-substituted ring is out of the amide plane in its ground state.



OR09 OR10

All of this, as well as some of the questions raised in Section 4.2, prompted the synthesis and determination of single-crystal structures of OR09 and its rotameric form OR10. Compound OR09 can be recrystallised to produce either isomer in substantially pure form. When the crude product is recrystallised, the trans form comes out as large, rectangular plates with the corners clipped off diagonally. The cis form crystallises as agglomerates of very small crystals whose individual shape is plate-like under the microscope. Microscopic examination is a fairly reliable method for the identification of isomers and even gives some indication of isomer purity.

Single crystals of OR09 and OR10 were mounted in Lindemann tubes and sealed off. Preliminary oscillation and Weissenberg photographs indicated the space group $P2_1/c$ for both crystals. Intensity data were collected on an Enraf Nonius CAD4 diffractometer, equipped with a graphite-monochromated Mo-K(alpha) radiation. Accurate cell constants were obtained by fitting 25 high angle reflections by means of a least square procedure. These and other crystallographic details appear in Table 27 and 28 for OR09 and OR10, respectively. A summary of the

general data collection procedure appears in Chapter 8.

The structures of OR09 and OR10 were solved with direct methods using a prerelease version of the computer package SHELXS86 (Sheldrick, 1985) and refined by full-matrix least-squares using SHELX76 (Sheldrick, 1976). All the hydrogen atoms of OR09 could be located from a difference synthesis and were refined freely with isotropic temperature factors. Some of the hydrogen atoms of OR10 could be seen on a difference map, but instead all the hydrogen atoms were placed and refined in calculated positions. All the non-hydrogen atoms in both structures were refined anisotropically. The reason for the relatively high R factor for OR10 is not clear. There is no appreciable disorder in the structure of OR10.

The final atomic coordinates and bond lengths and angles appear in Tables 29 to 32. Anisotropic thermal parameters, hydrogen atom coordinates, and observed and calculated structure factors are listed in Appendix ii. The numbering scheme, molecular structure of OR09 and packing diagram are shown below in Figures 18 and 19.

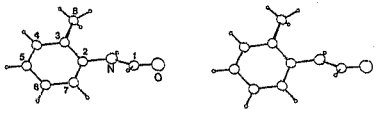


Fig.18. Molecular structure and numbering scheme for OR09.

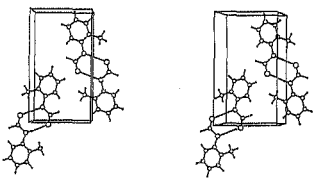


Fig.19. Packing diagram for OR09.

The numbering scheme and molecular structure of OR10 are shown in Figure 20. The molecular packing diagram is depicted in Figure 21.

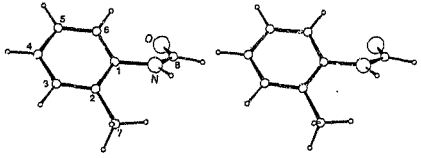


Fig.20. Molecular structure and numbering scheme of OR10.

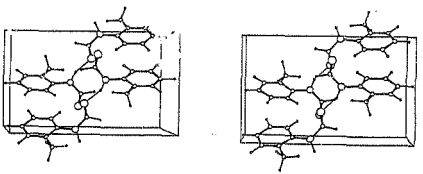


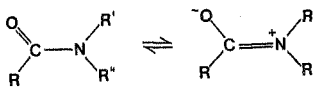
Fig.21. Packing diagram for OR10.

The molecular parameters of OR09 and OR10 agree well with accepted literature values (Kennard et al., 1972). A comparison of some parameters is given below:

Parameter	OR09	OR10
C(1)-N (angstrom)	1.426(4)	1.439(9)
C(8)-N	1.342(4)	1.358(10)
C(8)=O	1.213(4)	1.194(9)
C(1)-N-C(8) (degrees)	124.7(3)	123.9(8)
N-C(8)=O	124.5(4)	128.6(8)
C(6)-C(1)-N	119.5(3)	121.1(7)
C(2)-C(1)-N	119.2(3)	117.1(7)
C(2)-C(1)-N-C(8)	144.8(9)	145.0(9)
C(1)-N-C(8)=O	176.2(9)	2.6(9)

It appears from the structural studies that in spite of nonplanarity, there exists appreciable conjugation and double bond character in the C-N linkage. In fact, the C(8)-N distances of 1.342(4) and 1.358(10) angstrom are intermediate between the single bond C-N distance of 1.47 angstrom and the double bond distance of 1.24 angstrom. However, in the same molecules, one finds the C(8)=O distances of 1.213(4) and 1.194(9) angstrom to be just as expected for a pure C=O double bond, the intermediate length being approximately 1.31 angstrom. Thus, there appears to be some factor perturbing the sigma-electron systems of amides which affects the bond lengths, and which does not allow a simple pi-electron explanation of

the reciprocal relationship shown in the figure below, if indeed, such a relationship exists at all (Robin et al., 1971).



The best mean planes through the fragment C(1) to C(7) were calculated. The N atoms were found to be displaced by 0.10 angstrom and 0.33 angstrom out of this plane, respectively.

An interesting feature of the structure of OR10 is the short non-bonded distance of 2.968 angstrom (O...H(6) = 2.465 angstrom, C(6)-H(6) = 1.08 angstrom). To relieve this strain the angle C(6)-C(1)-N opens to 121.1(7) degrees and the angle C(2)-C(1)-N contracts to 117.1(7) degrees. The bisecting position is adopted in OR09 (and presumably also in OR10 for which hydrogen atomic positions were calculated) by the methyl group with respect to the N-H group.

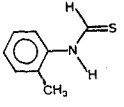
A reason for the successful isolation of the rotamers OR09 and OR10 definitely lies in their bifunctional(ty with respect to hydrogen bond formation. OR09 forms centrosymmetrical intermolecular hydrogen bonded dimers as shown in Figure 19. The hydrogen-bond length is 2.151 angstrom for O...H(6) [O...N=2.935 angstrom]. OR10 forms infinite chains of intermolecular hydrogen bonds with a length of 1.800 angstrom for O...H [O...N=2.827, N-H=1.08 angstrom]. The N-H...O=C hydrogen bonds in the crystalline state tend to be aligned in approximately the directions of the conventionally viewed sp^2 lone pairs. The errors associated with X-ray determinations of N-H...O=C hydrogen-bond geometries have recently been analysed by Taylor and Kennard (1983) and by Taylor, Kennard and Versichel (1983). The analysis was based on a

comparison of X-ray and neutron diffraction results. They found that the random errors in X-ray N-H and H...O distances could be "normalised". This involves moving the hydrogen atom position along the observed N-H bond direction until the N-H distance is equal to a standard value of 1.03 angstrom.

The structures of OR09 and OR10 are, to the best of our knowledge, the first X-ray structure determinations on rotamers of simple mono-substituted amides.

4.5 The Structure of o-Methylthioformanilide

The title compound was prepared by treating o-methylformanilide with P_2S_5 in benzene.



OR26

A great deal of experimental effort afforded us only with the E-isomer. An evenly-shaped crystal of OR26 was mounted on a glass fibre and photographed using standard oscillation and Weissenberg techniques, revealing an orthorhombic crystal system and the space group Pccn, uniquely defined by its systematic absences. The crystal was transferred to an Enraf Nonius CAD4 diffractometer fitted with graphite-monochromated Mo-K(alpha) radiation (See Chapter 8 for operational conditions). After accurate unit cell dimensions were determined from high theta scans, intensity data were collected. The necessary parameters are listed in Table 33. The structure was solved by the centrosymmetric direct methods package of a prerelease version of SHELXS86 (Sheldrick, 1985), and refined by full-matrix least-squares with SHELX76 (Sheldrick, 1976). All the hydrogen atoms were apparent on a difference Fourier map and were refined with

individual isotropic temperature factors. All the non-hydrogen atoms were refined anisotropically.

The molecular structure is shown in Figure 22. Final atomic coordinates and bond lengths and angles appear in Tables 34 to 35. Supplementary material is listed in Appendix II.

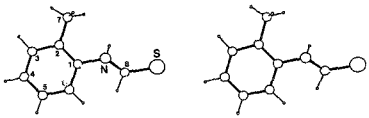
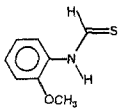


Fig.22. Molecular structure and numbering scheme for OR26

The molecular structure of OR26 can be compared to that of the published 2-methoxythioformanilide, 54 (Jarchow and Schmalle, 1977). Compound 54 crystallises in the space group $P2_1/a$ and was refined to a final R index of 0.072.

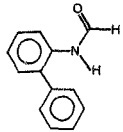


54

C(6)=S		
C(1)-N-C(8)	OR26	54
C(2)-C(1)-N		
C(6)-C(1)-N angstrom)	1.423(3)	1.409(5)
C(1)-N	1.325(3)	1.317(5)
C(8)=S	1.645(3)	1.632(4)
C(1)-N-C(8) (degrees)	124.2(2)	126.9(3)
C(2)-C(1)-N	119.6(2)	116.7(3)
C(6)-C(1)-N	119.0(2)	123.2(3)

4.6 The Structure of o-Phenylformanilide

The effect of ortho-substitution on the benzene-to-nitrogen torsion angle in monosubstituted amides was investigated by synthesising OR15 and determining its single-crystal structure.



OR15

Preliminary diffraction experiments on a Weissenberg *camera showed* that OR15 crystallises in the monoclinic space group $P2_1$. Accurate unit cell dimensions were obtained on an Enraf Nonius CAD4 diffractometer using graphite monochromated Mo-K(alpha) radiation, after which intensity data were recorded. Information regarding the crystal and data collection is summarised in Table 36, and that of operational conditions for the CAD4 in Chapter 8. The structure was solved by non-centrosymmetric direct methods with a prerelease version of SHELXS86 (Sheldrick, 1985). Full-matrix least-squares refinement was performed with the program SHELX76 (Sheldrick, 1978). Anisotropic thermal parameters were assigned to all non-hydrogen atoms, but. although a number of hydrogen atoms were apparent after a difference synthesis, they were all placed in calculated positions and refined with a common isotropic temperature factor. The high value for the R-factor is a direct consequence of using data at the OMIT 1 level. Observed and calculated structure factors and anisotropic temperature factors appear in Appendix II.

The molecular structure and numbering scheme are shown below. Note that OR15 is in the E conformation, in contrast with the most stable Z conformations of OR09 and OR26.

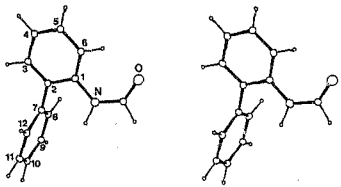


Fig.23. Molecular structure of OR15 showing the numbering scheme used.

The final atomic coordinates and bond lengths and angles are listed in Tables 37 and 38. Some selected structural parameters of OR15 are tabulated below.

Parameter	OR15
C(1)-N (angstrom)	1.417(7)
C(13)-N	1.316(9)
C(2)-C(7)	1.446(10)
C(1)-N-C(13) (degrees)	127.7(7)
N-C(13)=O	127.6(9)
C(2)-C(1)-N	115.6(7)
C(6)-C(1)-N	122.6(8)
C(1)-C(2)-C(7)	122.7(7)
C(2)-C(1)-N-C(13)	163.2(9)
C(1)-N-C(13)=O	2.8(9)
C(7)-C(2)-C(1)-N	0.0(9)
C(1)-C(2)-C(7)-C(8)	95.2(9)
C(13)=O	1.227(8)
O...C(6) (angstrom)	2.902
O...H(6)	2.266

The analysis shows an unusual opening-up of the angle C(6)-C(1)-N to 122.6(8) degrees and the closing-up of the angle C(2)-C(1)-N to 115.6(7) degrees. This must be due to the relief of steric strain between O and H(6) [O...C(6)=2.902 and O...H(6)=2.266 angstrom, C(6)-

H(6)=1.08 angstrom]. The strain in OR15 is further reduced by the twisting of the ortho-phenyl substituent [C(1)-C(2)~C(7)-C(8)=95.2(9) degrees]. This is totally different from the angle in biphenyl. The steric congestion around the ortho substituent contributes to the fact that the E form of the amide is the most stable. Again, linear chains of intermolecular hydrogen-bonded molecules are found throughout the crystal as is illustrated in the packing diagram below.

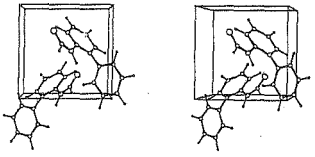
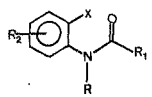


Fig.24. Packing diagram for OR15.

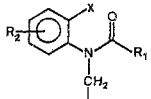
The bond distances are: O...H = 1.966 angstrom and O...N = 2.858 angstrom, between molecules at symmetry positions x,y,z and 1-x,y+0.5,1-z.

4.7 Crystallographic Characterisation of Single-ortho, N-substituted Acetanilide Derivatives.

The relative spatial dispositions of the moieties attached to the N atom in anilides of type 55 and 56, has been of long-standing interest (Siddall and Prohaska, 1966; Baert et al., 1984).



55a R = H
b R ≠ H



56a R = CH_3
57b R ≠ CH_3

Numerous X-ray and proton-NMR studies have established the conformation of the carbonyl group as endo in N-unsubstituted 55a, and as exo in N-substituted 55b, both in the solid state and in solution (Stewart and Sidall, 1970). The less common single-ortho, N-substituted anilides of types 56a and 57a, have been analysed principally by NMR spectroscopy and the inferred stereochemistry for 57a is depicted in Figure 25 (Brown et al., 1968; Staskun, 1981). To our knowledge no X-ray structural work has been done on anilide of types 56a and 57a, an omission attributed in part to the dearth of suitable crystalline material.

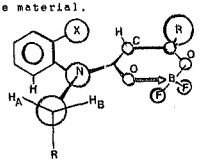


Fig.25. Lowest energy conformation for 57a in solution.

The main thrust of this investigation was to determine the solid state structures of several representatives of 56 for *comparison* with the stereochemistry derived from solution studies. These are not necessarily the same (Hazell and Hazell, 1977). We present here crystallographic characterisations of three single-ortho, N-substituted amide derivatives, bearing markedly dissimilar acyl groups, viz. acet-2'-bromo-N-(p-nitrobenzyl)anilide, OR04, (Figure 26), 3',5'-dimethyl-N-ethyl-2,2,2',4'-tetrachlorobenzoylacetanilide, OR22, (Figure 27), and difluoro[[1-phenyl-2-(o-ethylphenyl-N-benzyl-carbamoyl)vinyl]oxy]borane, OR01, (Figure 28) (Boeyens, Denner, Painter and Staskun, 1987).

Crystallographic analyses were based on X-ray diffraction data obtained with a Nonius CAD4 diffractometer (See Chapter 8 for operational conditions). Accurate cell constants were derived by least-squares refinement against sets of 25 accurately measured theta-values. The structures were solved by conventional Patterson and direct methods and refined by full-matrix least-squares using the program SHELX76 (Sheldrick, 1978).

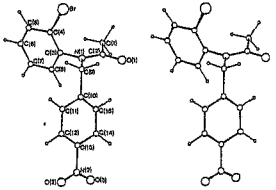


Fig.26. Molecular structure of OR04.

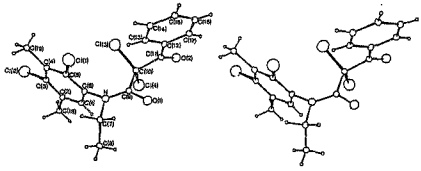


Fig.27. Molecular structure of OR22.

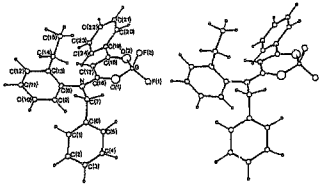


Fig.28. Molecular structure of OR01.

Crystal data and details of the crystallographic analyses are summarised in Tables 39 to 41. *Refined* atomic coordinates of non-hydrogen atoms are in Tables 42 to 44, and tables of bond lengths and angles appear in Tables 45 to 47. Some of the *hydrogen* atoms were located by difference Fourier techniques and all others were placed in geometrically calculated positions. The supplementary material *is listed in* Appendix II.

All three molecules have the same gross relative disposition of the N- and ortho-substituents. In each: (a) the amide carbonyl oxygen is pointing away from the arylamido ring, i.e., in the exo conformation, (b) the dihedral angle (phi) between the benzene ring plane and the plane of the amido group (with the N atom common to both planes) is approximately orthogonal; (c) the R-substituent in OR04, OR22, and OR01, is disposed essentially trans to, and is relatively far removed from the 2'-X substituent, and (d), one of the geminal protons lies in the vicinity of the deshielding region of the amide carbonyl oxygen, while the other resides near the shielding face of the orthogonal benzene ring (Nicole et al., 1980).

The aforementioned substituent dispositions in these three N-o-anilides as determined in the solid state are thus essentially like those in solution. Hydrogen bonding effects in OR04, OR22, and OR01 are likely to be minimal and the observed (X-ray) conformational preferences would seem to be largely determined by steric interactions.

The X-ray structure of OR01, (Figure 28) shows a 6-membered, virtually planar, true chelate with the sp^3 boron atom in a symmetrical, approximately tetrahedral environment, and covalently bonded to both oxygen atoms.

CHAPTER 5

Molecular Stereodynamics Studied by Molecular Mechanics

5.1 Introduction

Molecular structure and conformation are interrelated. Consideration of the three-dimensional geometries of molecules is fundamental to analysis of equilibria and reactivities. For many years, chemists performed conformational analysis using mechanical molecular models. Inspection of models, though often useful in the assessment of conformational ground-state properties, can be grossly misleading when extended to the dynamics of conformational interconversion. It may even fail as a reliable gauge of ground-state properties.

Theoretical calculations have been widely applied to different fields in chemistry. Different approaches have been used to describe molecular structures. Computer programs using the ab initio method require very large amounts of computer time. This requirement increases in the number of orbitals in a molecule. Semi-empirical quantum mechanical methods such as CNDO, INDO and MINDO are faster, but less reliable. However, the molecular mechanics technique is able to calculate the geometries of a wide variety of molecules remarkably well. Thus, when one is investigating chemical systems which may exhibit a variety of conformations, it is recommended to apply the current method of molecular mechanics in order to explore conformational space.

Molecular stereodynamics represents a major application of molecular mechanics calculations (Ivanov and Osawa, 1984). Most empirical force fields in current use are suitable for the calculation of minimum strain-energy conformations of molecules. This application of molecular mechanics,

however, lies outside the scope of this survey. Excellent reviews on molecular mechanics have been published by Williams et al. (1966), Englar et al. (1973), Altona and Faber (1974), Dunitz and Bürgi (1976), Hursthouse et al. (1978), Kitaigorodsky (1978), De Tar (1977), White (1978), Beagley (1978), Ermer (1981), Burkert et al. (1982), Boeyens (1986) and many more.

Although there are often considerable differences in the techniques used for specific applications, the common approach is to express the molecular strain energy as a sum of contributions from interactions such as bond-stretching, valence-angle deformation, torsional angle variation and non-bonded interactions. Normally all of these interactions are specified by various empirical valence-force potentials which are suitably parameterised. For stretching and bending motions harmonic forces are often used and a periodic function for torsional mobility. Non-bonded interactions may be specified by functions such as the Lennard-Jones or Buckingham potentials. The total potential for the molecule is then expanded about some assumed geometry and minimised iteratively until a self-consistent geometry is found. From the eigenvalues associated with this geometry one can obtain the vibrational spectrum.

Although previous computational studies in structural chemistry for the most part have been concerned with equilibrium structures (minima on the potential energy surface), many studies are now appearing that deal with movements across saddle points on the energy surface, simulating reaction pathways (Burkert et al., 1982). Geometry optimisation is essential if the energies at the transition state and along the interconversion pathway are to be meaningful. It is desirable therefore to move continuously along the potential surface from one energy minimum to another over the lowest saddle point along the minimum energy pathway. When conformational

interconversions for organic molecules have been studied by molecular mechanics, the torsion angle driving method has been used exclusively. In this method, one or more torsion angles are chosen as a reaction coordinate, and is driven incrementally through a range of values, all other internal degrees of freedom being optimised at each increment. Wiberg and Boyd (1972) first reported a method that can be applied to "driver" calculations. In this case, a strong torsional "driving" potential,

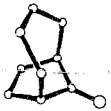
$$V = c_1(1 + \cos i(\omega + c_2))$$

is added to the force field, with a large potential constant c_1 which makes this torsion angle omega much stiffer than the others in the molecule, and with its potential minimum at the offset value of c_2. After geometry optimisation, the energy of the configuration obtained in this way is recalculated with the original force field, without the strong driving potential. The torsion angle driving method is, however, not free of pitfalls as was pointed out by Burkert and Allinger (1982). The elucidation of the full interconversion pathways by torsion angle driving depends on the assumption that one torsion angle is a good approximation to the reaction coordinate, which is rarely the case. This method also suffers from lagging effects, which is an inherent property of the driving method as used to explore energy surfaces. Forced steering in the wrong direction eventually leads to a sudden jump into a different domain of the surface. Quite a few published torsional energy profiles obtained with the one-bond drive method indeed contain discontinuous curves involving sharp maxima and a sudden drop after the maximum edge is passed (Baas et al., 1980 and Osawa, 1979). This feature violates the principle of microscopic reversibility.

A few computational examples of two-parametric, three-dimensional energy surfaces in the literature will now

follow. The dimensions of many chemical systems are generally higher than two, but the reader is referred to an excellent paper by Nørskov-Lauritsen and Bürgi (1985) who applied cluster analysis to analyse observed conformations for molecular fragments with more than three torsional degrees of freedom. The method is an adaption of statistical cluster analysis to multidimensional, symmetric, periodic distributions of data points.

Osawa (1982) studied the mechanisms of conformational chirality inversion in bicyclo[4.2.1]nonan-9-one, 58, and bicyclo[4.2.2]decane, 59, and presented the merits of rotating at least two bonds simultaneously.



58

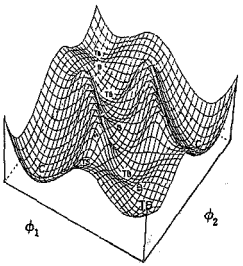


59

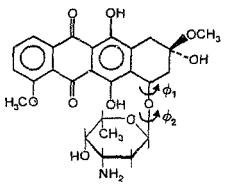
He concluded by stating that it appears desirable to re-examine, by the two-bond drive method, discontinuous torsional profiles recorded in the past. Schneider and co-workers (1979) addressed the problem of full conformational relaxation in some cyclohexanes, bicyclo[2.2.1]heptanes and related compounds using the Allinger MMI force field (1976). The figure illustrates their potential surface as calculated for cyclohexane, 60, as a function of phi$_1$ and phi$_2$ (C = chair, TB = twist boat, B = boat).



60

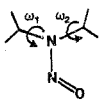


The conformation of the anti-cancer drug daunomycin, 61, has been investigated in detail by potential energy calculations by Islam and Neidle (1983). The flexibility around the ether linkage, connecting the anthracycline chromophore and the amino sugar group, has been evaluated using several types of potential energy function. The two bond torsion angles phi$_1$ and phi$_2$ were varied, while the rest of the molecule were held in the crystal geometry. The results largely support the hypothesis that the crystallographically observed conformation is the most stable one, although considerable detailed variation with respect to potential function was found.

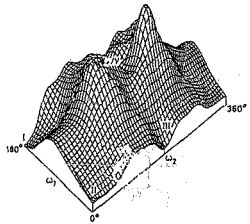


61

Lunazzi and co-workers (1982) used molecular mechanics to determine the *energy minima* and *interconversion* mechanism for di-isopropyl-nitrosoamine, 62. Evidence of the *existence of correlated* intramolecular orientation (gear effect) was observed. The interconversion pathway is summarised in the scheme below, where the surface corresponding to the dependence of the computed energy upon the torsional angles $omega_1$ and $omega_2$ is reported.



62

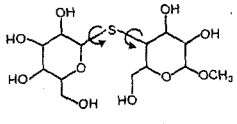


Bushweller et al. (1982) did a complete study using molecular mechanics on the stereodynamics of diethylmethylamine and triethylamine. A 5000 point optimised energy surface was computed as a function of two dihedral angles in diethylmethylamine, with the lone pair labeled as position number 1, the two pertinent dihedral angles are 1-2-7-8 and 1-2-14-15, as defined in structure 63.



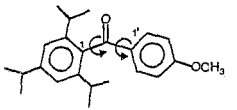
63

Perez and Vergelati (1984) reported the structure and conformational analysis of methyl alpha-thio-maltoside, 64.



64

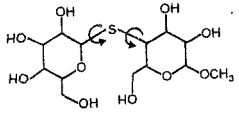
Energy maps were computed as a function of phi$_1$ and phi$_2$ at intervals of 5 degrees by including partitioned contributions arising from the van der Waals, torsional and hydrogen-bond contributions. The hindered rotation in 2,4,6-triisopropylbenzophenones like 65, was studied by Ito and co-workers (1981) using DNMR and molecular mechanics calculation. The calculation revealed that the most energetically feasible rotation around the C(1)-C(7) bond in, 65, involves the concomitant rotation of the C(7)-C(1') bond in the same molecule.



65

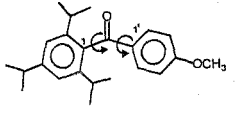
Roussel and co-workers (1976) studied the gear effect as a model for conformational transmission. They showed with the aid of a simple model that polyhedral substituents like alkyl groups can be involved in a "gear system" which may transmit long-range conformational changes. The strain energy calculations were performed in a rather simplified manner, taking only the van der Waal's interactions into account by a potential function of the Buckingham exp-6 type. Furthermore, the methyl groups in, 66 and 67, were treated as "combined atoms".

The van der Waal's energy was calculated as a function of the angles phi$_1$ and phi$_2$ with all other geometric



64

Energy maps were computed as a function of phi_1 and phi_2 at intervals of 5 degrees by including partitioned contributions arising from the van der Waals, torsional and hydrogen-bond contributions. The hindered rotation in 2,4,6-triisopropylbenzophenones like 65, was studied by Ito and co-workers (1981) using DNMR and molecular mechanics calculation. The calculation revealed that the most energetically feasible rotation around the C(1)-C(7) bond in, 65, involves the concomitant rotation of the C(7)-C(1') bond in the same molecule.

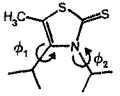


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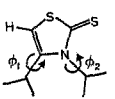
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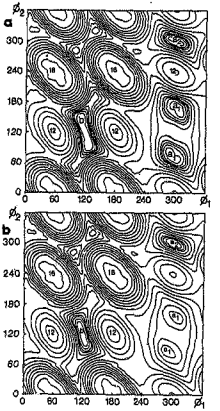
parameters held fixed. The energies are represented in the maps with iso-energetic curves.



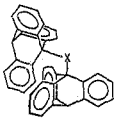
66



67



Disrotatory coupling of the internal rotational degrees of freedom in double rotor molecules like bis(9-triptycyl) methanes and bis-(9-triptycyl)ethers was studied by Iwamura (1985) by means of molecular mechanics. Independent rotation around the bonds connecting the bridgehead carbon atoms to the central carbon or oxygen atom in 66, respectively, is a most unlikely process. Torsional motions seem to be possible only in a gear-meshed fashion.

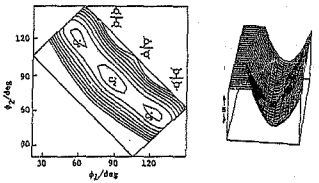


Tp_2X (X = CH_2, O)

68

The higher limit of the barrier to rotation from one form to the other was estimated to be 7 - 8 kcal/mol.

A partial torsional energy surface of 68, is shown. The two energy minima lie in a deep valley of the Ramachandran diagram representing the gear-meshing motions of this molecule.



Finally, a review on theoretical investigation of reaction paths on multidimensional energy surfaces has been published by Müller (1980). The difficulties and problems encountered with this approach are discussed in this review.

In conclusion, it is interesting to note that the Boyd-Wiberg driver technique is used almost exclusively by chemists doing dynamic molecular mechanics calculations. One should always be very careful when torsional angles are chosen for a structural problem, or if a function of many variables is projected onto a space of smaller dimension, that the chosen mapping parameters represent the reaction coordinate sufficiently closely.

5.2 Conformational Analysis by means of Molecular Mechanics

The complexities of isomerism and isomerisation in molecules possessing one, two or more aryl or other bulky groups in sterically crowded environments, are typically functions of internal rotations and amenable to analysis by the static and dynamic stereochemistry of such molecules, utilising the approach of full relaxation molecular mechanical calculations.

5.2.1 Description of Empirical Force Field

For the purposes of molecular mechanics calculations the molecule is treated as an isolated set of static nuclei held together by elastic linkages. Molecular mechanics force-fields are based on the assumption that the intramolecular potential energy (strain energy) of a molecule may be represented as the sum of terms which are functions of the differences between actual and suitable reference values of the parameters describing the molecular geometry (usually internal co-ordinates and non-bonded distances). Molecular mechanics minimises this strain energy by systematically adjusting the co-ordinates. The collection of the individual potential functions and their necessary parameters is called the "force field".

The steric energy, E_s, of a molecule may be separated into the following independent contributions (Westheimer, 1956).

$$E_s = E_r(r) + E_\theta(\theta) + E_\varphi(\varphi) + E_\delta(\delta) + E_{nb}(d)$$

where $E_r(r)$ represents the bond stretching component, $E_\theta(\theta)$ the bond angle bending component, $E_\varphi(\varphi)$ the torsional effects, $E_{NB}(d)$ the non-bonded interactions, and $E_\delta(\delta)$ the out-of-plane deviation. Because of the difficulties associated with the calculation of

electrostatic interactions arising from the choice of values for the effective dielectric constant and from the fact that it is difficult to estimate reliably where the dipoles should be located, this component of E_m has not been considered (Walter and Imbert, 1975). Some elucidation of the potential functions used is necessary. Bond lengths and angle deformations are assumed to follow a quadratic harmonic potential:

$$E_r(r) = 0.5k_r(r - r_o)^2$$

$$E_\theta(\theta) = 0.5k_\theta(\theta - \theta_o)^2$$

r_o and θ_o are reference values of r and θ in a hypothetical strain-free molecule and k_r and k_θ are force constants. It is well known that the non-bonded interactions alone cannot account for torsional effects (Scheraga, 1968). Thus, a separate contribution for the intrinsic internal rotation to the total energy must be considered. The torsional contributions to steric strain are assumed to arise from interaction between all bonding electron groups adjacent to the reference bond. In delocalised systems this interaction is attractive and favours a torsion angle of zero. Non-planar systems are therefore torsionally strained. All other torsional interactions are repulsive and assumed to approach zero as phi tends to 60 degrees. Both types of interaction are conveniently computed by using the standard expression commonly used for calculating threefold barriers to rotation (Bosyens, Cotton and Han, 1965).

$$E_\varphi(\varphi) = 0.17k_\varphi(1 + \cos 3\varphi)$$

The problems due to the choice of non-bonded potential have been discussed in detail (Williams et al., 1968 and Williams, 1981) and it is probably the most critical part of the parameterisation. In a classical way, and in analogy with intermolecular interactions, we have used a Buckingham exp-6 potential,

$$E_{nb}(d) = A\exp(-Bd) - Cd^{-6}$$

The attractive part depends on the constant C, which is a function of the polarisabilities and valence electron densities of the atom pair (Scott and Scheraga, 1965). The constant B is a shielding function, and A is chosen to ensure that the attractive and repulsive parts balance to produce a minimum at a separation corresponding to the sum of the van der Waal's radii. A large set of tested parameters is now available for interactions in saturated compounds. Finally, the delta angle accounts for out-of-plane bending according to the potential,

$$E_{\delta}(\delta) = 0.5k_{\delta}(\delta)^2$$

The strain energy calculations were performed with the computer program MOLBLD3 (Boyd, 1968). Energy minimisation is accomplished by means of an iterative Newton-Raphson procedure, whereby the co-ordinates of the atoms corresponding to the conformation are adjusted to converge upon the equilibrium values for the minimum energy conformation (White and Guy, 1975). The literature contains numerous investigations of various degrees of sophistication which are concerned with the application of molecular-mechanical calculations to the study of the structure of compounds containing aromatic rings (See Androse and Mislow, 1974 and references cited therein). In Boyd's method, the preadjusted parameterisation treats the delocalised system as a transferable structural unit, and the relevant parameters can change during the minimisation process (Boyd et al., 1971; Chang et al., 1970; Shieh, McNally and Boyd, 1969; Carballeira et al., 1982; Maverick, Smith, Korerski, Anet and Trueblood, 1975; Carballeira et al., 1984, and by Osawa and Musso, 1983).

5.2.2 Dynamical Molecular Mechanical Calculations

Theoretical investigation of rotational isomerism requires

a detailed knowledge of the potential energy of the molecular system. As a first step in such a study, minimum energy reaction paths have to be mapped on a generally multidimensional molecular potential surface. By contrast, the experimental situation is less straightforward. The transition state as well as the region around it are generally not amenable by direct experimental observation. In order to identify conformers sterically allowed with a certain margin of confidence, as well as to establish the order of magnitude of the barriers hindering the conversion from one conformer to another, a new type of driver subroutine was added to the MOLBLD3 program.

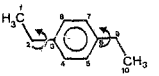
The standard MOLBLD3 program provides a facility to drive one, or several torsional angles simultaneously, at different rates, through a specified range while calculating strain energy at regular intervals (Wiberg and Boyd, 1972; Boyd and Breitling, 1972(a) and Boyd and Breitling, 1972(b). This torsional angle driving method of Wiberg and Boyd is used in almost all calculations pertaining to dynamic molecular mechanics. The subroutine NDRIVE in Allinger's MM force field also rotates according to the Wiberg-Boyd method (Osawa, 1979, and Allinger, 1977).

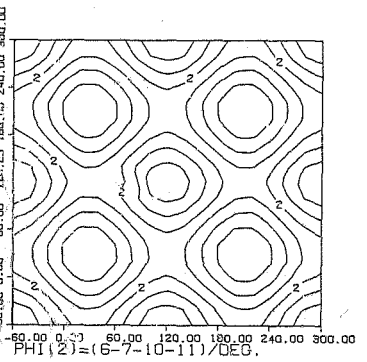
The conformational analysis of systems like the fulgides or 1,8-di-arylnaphthalenes by molecular mechanics, requires a systematic adjustment of two independent torsion angles that determine the orientation of the aryl substituents. This cannot be achieved any of the existing computer programs, that was available to us at the time, and necessitated the modification to the existing computer program MOLBLD3 to provide this facility (Allenspach-Wasser, Boeyens and Denner, 1986). A procedure to compare the steric energy of all conformations in a selected two-dimensional surface, representing rotation about two chemical bonds, has

therefore been incorporated into the standard molecular mechanics program MOLBLD3 of Boyd. No modification of the existing Newton-Raphson minimisation algorithm was introduced and the changes amounted only to a programmed procedure to step two selected torsion angles through a specified range, with full relaxation empirical force field calculation, with the exception that the two dihedral angles were held constant during energy minimisation, at each incremented step. In the driving routine of MOLBLD3 the refined co-ordinates at any step are used as input co-ordinates for the next step. This is done to avoid unrealistic distortion of the molecular structure, that could happen if the adjustment of torsion angle is too sudden. A succession of small steps ensures a gradual change in conformation and eliminates the danger of distortions. Since the same is true for the simultaneous adjustment of two torsion angles, standard Fortran DO-loops would not be suitable unless the inner loop has a fixed range of 360 degrees. This is not always practical and to avoid large jumps the inner loop was defined with a change of increment sign at both ends of the specified range. This causes the changing conformation to zig-zag through the field in small steps. Other two-bond drive schemes have been used by Osawa (1982). A subroutine to plot the output in the form of a two-dimensional contour map was added. This facility to map conformational energies as a function of two dihedral angles is foreseen to find general application in the study of rotational barriers.

Finally, an example of the abovementioned two-dimensional driver as applied to the conformational analysis of 1,4-diethylbenzene is given. The torsion angles phi_1 and phi_2 were driven through 360 degrees in steps of 10 degrees, i.e. phi_1 = 10 degrees, phi_2 = 0 to 360 degrees in steps of 10 degrees, followed by phi_1 = 20 degrees, phi_2 = 0 to 360 degrees in steps of 10 degrees, etc. The conformational space of diethylbenzene is depicted in the

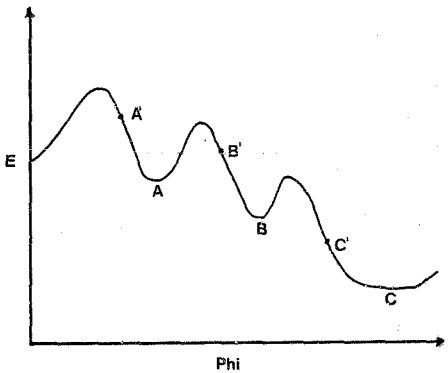
contour map with contour intervals of 0.3 kcal/mol.





5.2.3. Exploring Two Dimensional Steric Energy Surfaces with Molecular Mechanics.

The minimisation scheme used in most empirical force-fields relies on the Newton-Raphson method to find the minimum strain energy. Consider the graph below which shows a torsional coordinate versus strain energy.



This graph shows three minima A,B and C, with C being the global minimum. If the trial coordinates for this structure is chosen to be at either A', B' or C', the Newton-Raphson method will give three minimum strain energy conformations at minima A,B and C, respectively.

A second point to note when using a refinement technique

is the criteria for convergence. In MOLBLD3 the convergence criterion is the root-mean-sguare deviation of the shifts. The minimisation method used, although it generally converges rapidly, does not guarantee convergence. For instance, the convergence of conformer A in the above diagram will be much faster than conformer C, because a big shift in phi is not accompanied with a large change in energy. A typical value for the root-mean-square deviation of the shifts in a molecule is 0.005.

Naturally, chemists are interested in studying the conformations of molecules at the global minima of the respective molecules. However, in certain molecules the idea of exploring the entire conformational energy surface is more useful, especially in cases where rotamers are suspected to exist. Strictly speaking, all possible torsional angles should be varied to cover the entire conformational energy space and in doing so identify all possible energy minima and barrier heights.

The stereodynamics in this thesis deal exclusively with two-dimensional conformational problems such as 1,8-diphenylnaphthalene. The approach used in this work is described in the previous section. The strategy for all two-dimensional "driver" calculations were as follows: The torsional mobility of a molecule was determined by identifying two torsion angles which defined the major conformational effects of the molecule. These two torsion angles were then driven each through 360 degrees, with refinement at each grid point (See previous Section). Since, this can be an enormous amount of calculation for even small to medium molecules, it was decided to do these conformational runs with a larger convergence limit, typically 0.01.

Because of this all steric energy plots in this thesis are not 100% symmetrical in nature. Nevertheless, the main conformational features of these steric energy maps are

still revealed. All minima and maxima are well defined. The second phase of the calculations involved the identification of these conformers. These conformers were then refined with a much better convergence criteria, namely 0.002 - 0.005. The strain energies obtained in these calculations were used to calculate the barrier heights.

This approach differ significantly from other approaches in the literature (Burgi et al, 1983). These workers applied symmetry constrains to all molecules during conformational interconversion, to produce perfectly symmetrical two-dimensional strain energy maps.

CHAPTER 6

Conformational Analysis of Peri-substituted Naphthalenes.

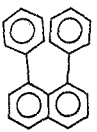
6.1 Introduction

Highly congested molecules always present challenges to chemists. On the one hand, there is the organic chemist's problem of trying to synthesise such molecules. On the other hand, theoretical chemists can test the limits of their predictions against the effect of intramolecular strain on ground-state structure and internal mobility.

Alkyl or aryl substituents at the 1,8 positions (the peri-positions) of naphthalene are in close proximity to one another and provide many interesting opportunities for the study of conformations and barriers to rotation about the extracyclic bonds. The essence of conformational analysis of peri-substituted naphthalene is to study how these molecules accommodate strain.

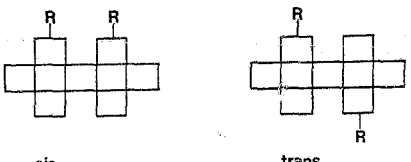
6.2 Literature Survey

Substituted naphthalenes with peri-substituents are a case in point and much work has been done on the subject. It has been reviewed by Balasubramaniyan (1966). Clough and Roberts (1978) studied molecular models of, 69, and suggested that a 180 degrees rotation about a phenyl-naphthyl bond is expected to be effectively blocked for derivatives of peri-diphenylnaphthalenes, in which steric requirements force the phenyl rings to assume a face-to-face conformation.

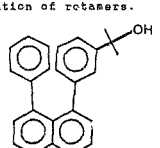


69

This was confirmed by an X-ray diffraction study of Ogilvie (1971). As a result, House and co-workers (1970) prepared several derivates of 1,8-diphenylnaphthalene (like 70) having a substituent at one meta position of each phenyl ring, with the expectation that they would be able to isolate cis and trans isomers as shown schematically below:

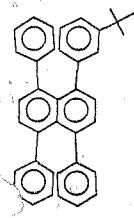


However, it was not possible to obtain separate isomers, and, instead, a single crystalline compound was isolated. Subsequent NMR measurements on the derivative, 70, showed that the barrier to rotation was only 16.4 kcal/mol, which is too low for isolation of rotamers.



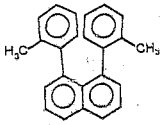
70

Clough and Roberts (1978) reported the synthesis and measurement of the rotational barrier of the highly strained, 71, which has two sets of crowded peri-phenyl groups.



71

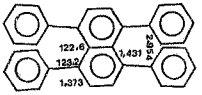
DNMR studies and visual matching using computer-generated spectra allowed them to calculate the barrier for rotation, to be 14.9 kcal/mol. More recently, Clough and Roberts (1976) found that 1,8-di-o-tolylnaphthalene, 72, could be resolved into a cis and trans isomer pair due to the restricted rotations around the tolyl-naphthyl bonds.



72

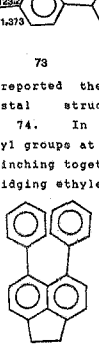
The two isomers were found to be stable in the crystalline state, but interconversible in solution having a half-life of about one day at room temperature. The publication of this communication sparked off a whole series of structural analyses of internally crowded naphthalene derivatives to explain the magnitude of rotational barriers as well as the effects that the peri-substituents have on the naphthalene nucleus.

An X-ray crystallographic study of 1,4,5,8-tetraphenylnaphthalene, 73, published by Evrard et al. (1972) revealed a fairly warped structure for the naphthalene moiety. This study supported the idea that relief of the steric stress is accomplished by splaying of the bonds to the peri-substituents as well as by surprisingly large in-plane and out-of-plane deformations of the naphthalene nucleus. Relevant bond lengths, angles and non-bonded distances are depicted in the drawing.



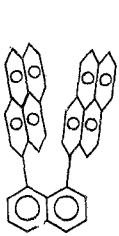
73

Clough et al. (1976) reported the synthesis, ^1H NMR spectrum and crystal structure of peri-diphenylacenaphthalene, 74. In this compound, the overcrowding of the phenyl groups at the 1 and 8 positions is accompanied by a pinching together at the 4 and 5 positions, due to the bridging ethylene groups.

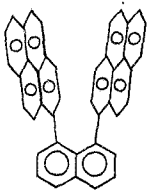


74

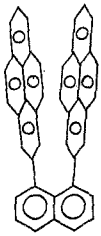
Wahl et al. (1984) reported the syntheses, molecular structure and spectroscopic properties of three 1,8-dipyrenylnaphthalenes 75 to 77.



75

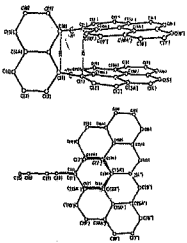


76



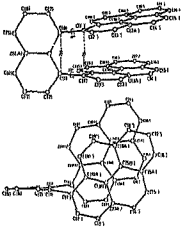
77

In order to establish kinetic parameters for the interconversion of stereoisomers 75 and 76 these authors invoked the crystallographic result that 76 crystallises in the chiral space group $P2_1$. Thus, a separation of enantiomers were possible by picking out single crystals. By determining the decrease of the optical rotations with time the rate constants for the cis-trans isomerisation were measured at various temperatures. From these measurements an Arrhenius plot was obtained from which the activation energy E_A = 24.8 kcal/mol was derived. A few structural features of these compounds need to be emphasised. In the projection of, 75, on the naphthalene plane the peri-bonds C(1)-C(1') and C(8)-C(1") are spread by 13 degrees. Furthermore, these bonds deviate from the naphthalene plane by 7 and 9 degrees in an opposite up-and-down fashion. Due to these deformations the bond angles C(1')-C(1)-C(8A), C(1)-C(8A)-C(8) and C(8A)-C(8)-C(1") are increased to 123.2, 125.4 and 124.4 degrees respectively. The pyrenyl groups are rotated against the naphthalene plane at angles of 85 and 90 degrees, respectively.



76

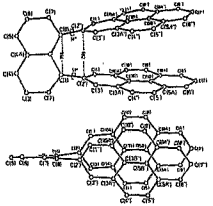
In contrast to, 75, the spreading of the peri-bonds C(1)-C(1') and C(8)-C(1") in, 76, occurs mainly as an in-plane bending of about 16 degrees. The out-of-plane deviations of these bonds as well as of C(1) and C(8) are negligible. The bond angles C(1')-C1)-C(8A), C(1)-C(8A)-C(8) and C(8A)-C(1") are widened to 125.2, 125.3 and 124.5 degrees respectively. The pyrene planes are both twisted against the naphthalene plane by an angle of 75 degrees; the inclination of the two pyrene planes towards each other is 6.8 degrees.



75

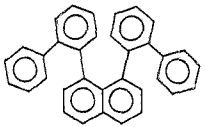
The in-plane and out-of-plane deformations of the naphthalene peri-bonds C(1)-C(1') and C(8)-C(1") of 75 are similar as in 75 (16 and 10 degrees, respectively). The

angles C(2')-C(1)-C(8A), C(1)-C(8A)-C(8), and C(8A)-C(8)-C(2") are widened to 123.6, 126.2 and 126.3. The most interesting feature in the structure of 77 is the fact that the pyrene planes are not nearly orthogonal to the naphthalene plane, but form torsional angles of only 56 and 59 degrees respectively with the central nucleus.

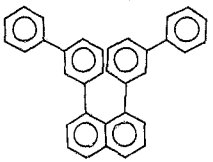


77

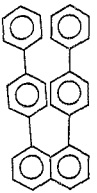
Ibuki et al. (1982) described the preparation and conformational properties of several 1,8-diarylnaphthalenes, 78 to 83.



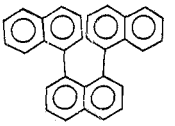
78



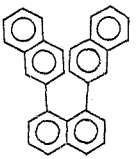
79



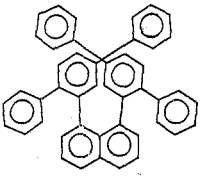
80



81



82



83

In addition, these authors describe the characterisation and interconversion of the stable *cis* and *trans* rotamers of 1,8-di-1-naphthylnaphthalene. Both these rotamers are quite stable in solution at room temperature for over two weeks. The stability seems to be remarkable as compared with the half-life of only about one day in solution with respect to the interconversion between cis and trans 1,8-di-tolylnaphthalene at room temperature. Each of the other diarylnaphthalenes, 78 to 80 and 82 to 83, proved to be single substances indicating low energy barriers to rotation of the two rings around the bonds at the peri-positions.

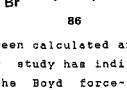
A crystal structure analysis and strain energy minimisation calculations on 1,8-dimethylnaphthalene, 84, have been performed by Bright and co-workers (1973). The crystal structure revealed that the most marked

distortions occur at the junctions of the methyl groups with the naphthalene nucleus. The inner angles, C(9)-C(1)-C(11) and C(9)-C(8)-C(12), are increased from the ideal 120 degrees to almost 125 degrees while the outer angles, C(2)-C(1)-C(11) and C(7)-C(8)-C(12), are decreased to 116 degrees.



84

Surprisingly, the carbon skeleton was found to be planar. No carbon atom deviates from the best least-squares plane through the ring carbon atoms by >0.02 angstrom. One might expect that substituent interaction could be reduced by bending one methyl above and one below the naphthalene plane, but this was not observed. This is in contrast with the situation in 1,4,5,8-tetrachloronaphthalene, 85, reported by Gafner and Herbstein (1962), where the chlorine atoms were found to be 0.2 Angstrom out of the molecular plane, in addition, a significant buckling of the naphthalene nucleus was observed. In 1,8-diphenylnaphthalene the nucleus is planar, but the phenyl carbon atoms attached to the nucleus are displaced by 0.06 angstrom above and below this plane (Ogilvie and Parkes, 1973). A buckling of the naphthalene nucleus was also reported for 3-bromo-1,8-dimethylnaphthalene, 86, by Jameson and Penfold (1965).

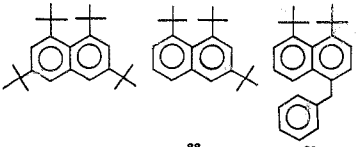


86

The comparison between calculated and observed structures were good. Their study has indicated that the strain parameters of the Boyd force-field developed for *cyclophanes* are indeed applicable to other systems, at

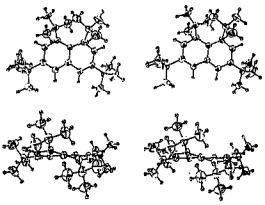
least to provide an understanding of the gross effects of overcrowded molecules.

In order to get a better understanding of peri-interactions in phenylnaphthalene derivatives, it was necessary to do a literature survey encompassing many different functional groups in the 1 and 8 positions of naphthalene. Anderson et al. (1972) reported considerable twisting in 1,8-di(-tert-butyl)naphthalene derivatives, 87 to 89, where the tert-butyl groups are found on opposite sides of the mean plane of the naphthalene ring.



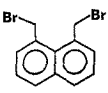
87 88 89

The barrier to flipping of these groups to give the enantiomer conformation is greater than 24 kcal/mol. The same steric interactions give rise, in contrast, to a particularly low barrier to rotation about the tert-butylnaphthalene bonds of about 6.5 kcal/mol. These workers concluded by stating that steric interactions may occasionally produce low rather than high barriers of rotation. Handal et al. (1977), described the detailed molecular structure of a peri-di(tert-butyl)naphthalene, 90, as determined by X-ray crystallography and molecular mechanics calculation. Both methods produced a highly distorted non-planar framework.



90

It was concluded from the observation of diastereotopic hydrogen atoms in the NMR spectrum of the benzylnaphthalene that the aromatic framework was non-planar and chiral. Furthermore from *coalescence studies*, it was determined that the free energy of the barrier to racemisation was greater than 20 kcal/mol. The tert-butyl groups and the *peri-carbon atoms must pass* through the mean plane of the molecule for racemisation to occur. Roberts et al. (1974) determined the conformation of 1,8-di(*bromo*-methyl)naphthalene, 91, in the crystalline state and they found that the molecule has essentially twofold symmetry about the C(9)-C(10) bond with the bromine atoms located above and below the plane of the ring. The strain in the molecule is largely relieved both by in-plane and out-of-plane bending of the peri bonds as well as some skeletal distortion in the ring. They explained the relief of steric strain in terms of a buttressing effect exerted by the 2,7-hydrogen atoms which tends to favor out-of-plane distortions.

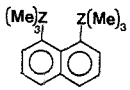


91

Low-temperature proton NMR studies of, 91, and some

related compounds showed no evidence of barriers to rotation about the peri bonds, large enough to be detectable.

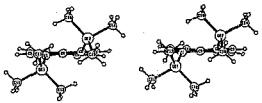
Blount et al. (1980) published the X-ray structures of 1,8-bis(trimethylgermyl)naphthalene, 92, and 1,8-bis(tri-methylstannyl)naphthalene, 93.



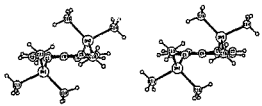
Z = Ge 92
Z = Sn 93
Z = Si 94

Empirical force-field calculations yielded structures in good agreement with those obtained by X-ray diffraction; these calculations also predicted that the conformation of the yet unknown 1,8-bis(trimethylsilyl)naphthalene, 94, resembles that of 92 and 93. The stereoviews of 92 and 93, below show the distortions.

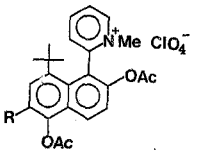
92



93



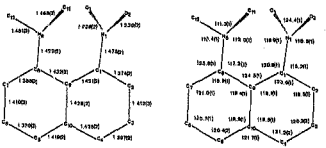
Fields and Regan (1971) presented the synthesis of 6-substituted 2,5-diacetoxy-8-tert-butyl-(-2-pyridyl)-naphthalene, 95. Low-temperature NMR spectra of the N-methyl quaternary salt of 95 indicated the existence of two isomers and were interpreted in terms of skewing of the naphthalene framework.



95

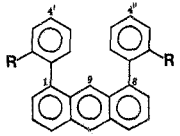
While the two conformational isomers were found to be in rapid equilibrium above -50 degrees celsius, the rate of ring inversion slows sufficiently below this temperature to allow the separate N-methyl resonances to be observed. A value for the barrier of 12.0 kcal/mol was obtained.

Egli, Wallis and Dunitz (1986) studied the *structure of* N,N-dimethyl-8-nitro-1-naphthaleneamine, 96, in seven crystalline environments. *They found*, in all seven conformations, that the lone pair of the amino N-atom is directed *approximately towards* the nitro N-atom, which is slightly pyramidalised towards the amino group. This trend *to close-up* rather than open-up the peri bonds on the naphthalene ring is the exact opposite of all the *examples* mentioned above. This point reconfirm the importance of conformational analysis.



96

House et al. (1986) in their studies on 1,8-diphenylanthracene and its derivatives, used the fact that these compounds exist largely in conformations with the two phenyl rings approximately parallel and approximately perpendicular to the plane of the anthracene ring, as a useful synthetic advantage. Such conformers possess a molecular cavity bounded on the bottom by the anthracene ring and on the two sides by phenyl rings. This cavity is of sufficient size to allow reagents to enter and engage in chemical reactions at the bottom of the cavity, namely the C(9) position. Consideration of this geometry lead them to the conclusion that 1,8-diarylanthracene derivatives like, 97, with two unsymmetrically substituted aryl rings, could exist as two geometrical isomers, a cis form, 97a, and a trans form, 97b.



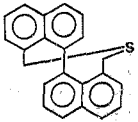
cis 97a

trans 97b

Furthermore, the trans form, 97b, would be composed of two non-superimposable mirror images (enantiomers). Resolution of an appropriate set of enantiomers possessing a functional group at the C(9) position would provide two molecules, each with a chiral cavity and a functional group at the bottom of the cavity. Such molecules have the potential to serve as "synthetic enzymes" that could convert achiral substrates to chiral products or could discriminate between the two enantiomers of a racemic mixture. To explore possibilities like these, more information about rotational barriers, rotamers and conformation is needed.

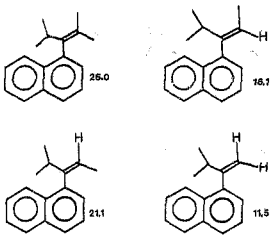
Studies on binaphthyl derivatives revealed some

interesting facts too. Korp et al. (1981) determined the crystal structure of (-)-8,8'-(2-thiatrimethylene)-1,1-binaphthyl, 98. The molecules were found to have axial chirality R(helicity M), while each individual naphthalene unit was twisted with P helicity. The angle between the main planes of the naphthalene pairs is 92.5 degrees and they are badly buckled.



98

Anderson and Hazlehurst (1980) reported a study on the hindered rotation in 1-alkenyl-naphthalene analogues of binaphthyl using DNMR. The following compounds were studied:



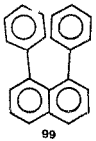
(Barriers are free energies of activation in kcal/mol). An analogous series with an ethyl group instead of an isopropyl group attached to C(11), was also investigated. The barrier to rotation for binaphthyl is 23.5 kcal/mol according to Cooke and Harris (1967).

A theoretical study of configurational inversion in 1,1'-binaphthyl by molecular mechanics was done by Carter and Liljefors (1976). Pathways connecting local minima were calculated using an approach similar to that described by Wiberg and Boyd, 1972. A one-fold torsional function was used for the pivot bond C(1)-C(1'). The preferred pathway was found to be one on the anti side of the molecule involving one point of H...H non-bonded contact in each of two identical transition states, separated by a very shallow minimum. These low barriers to rotation for congested molecules are undoubtedly due to the energy of the ground states of the molecules being raised relative to the transition states so as to make the energy gap smaller. Therefore, it is necessary to explore compounds that are less congested in the ground state, but possess severe interaction between the parts of the molecule in the transition state for rotation, to make it possible to isolate atropisomers at room temperature.

6.3 Conformational Analysis of 1,8-Di-arylnaphthalenes by Two-dimensional Mapping of Steric Energy

Introduction

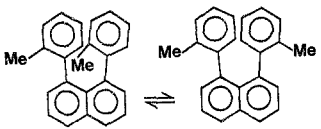
It is commonly assumed (Balasubramaiyan, 1966) that the favoured conformation of 1,8-diphenylnaphthalene, **99**, occurs with its two parallel phenyl rings perpendicular to the plane of the naphthalene ring, and with substantial energy barrier to rotation of the phenyl rings.



99

It was therefore surprising to find [illegible]e and Baske, 1965) that cis and trans rotamers of 1,8-di(ortho-or meta-

substituted phenyl)-naphthalenes could not be isolated, and this was ascribed (House, Campbell and Gall, 1970) to a relatively low barrier to rotation which amounts to about 9.6 kcal/mol for phenyl rings. Resolution of 1,8-di-o-tolylnaphthalene, 100, however, was subsequently achieved by column chromatography.



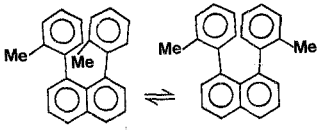
100

The trans isomer was found to be the more stable by 0.7 kcal/mol. The barrier to interconversion was calculated to be 24 kcal/mol and a lower observed barrier of 9 kcal/mol was interpreted in terms of the flipping of aromatic rings from side to side of the naphthalene planes. These calculations were based on DNMR data. This difference in opinion has never been resolved and the actual structures of the relevant rotamers have neither been determined nor calculated. To settle the question two-dimensional steric energy surfaces have been calculated by molecular mechanics to elucidate possible pathways of interconversion between rotamers.

Results

In order to elucidate the mechanism of conformational changes in di-arylnaphthalenes the conformational energy of 1,8-di-phenylnaphthalene, 1,8-di-tolylnaphthalene and 1,8-di(m-methylphenyl)naphthalene was calculated as a function of aromatic ring rotations, using the default force-field of the program MOLBLD3 (Boyd, 1968). An X-ray single crystal analysis on an analogous compound, 1-tolyl-naphthalene, OR44, is also presented together with the first results obtained from a crystal of cis-1,8-

substituted phenyl)-naphthalenes could not be isolated, and this was ascribed (House, Campbell and Gall, 1970) to a relatively low barrier to rotation which amounts to about 9.6 kcal/mol for phenyl rings. Resolution of 1,8-di-o-tolylnaphthalene, 100, however, was subsequently achieved by column chromatography.



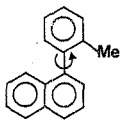
100

The trans isomer was found to be the more stable by) kcal/mol. The barrier to interconversion was calc to be 24 kcal/mol and a lower observed barrie of 9 kcal/mol was interpreted in terms of the flipping of aromatic rings from side to side of the naphthalene planes. These calculations were based on DNMR data. This difference in opinion has never been resolved and the actual structures of the relevant rotamers have neither been determined nor calculated. To settle the question two-dimensional steric energy surfaces have been calculated by molecular mechanics to elucidate possible pathways of interconversion between rotamers.

Results

In order to elucidate the mechanism of conformational changes in di-arylnaphthalenes the conformational energy of 1,8-di-phenylnaphthalene, 1,8-di-tolylnaphthalene and 1,8-di(m-methylphenyl)naphthalene was calculated as a function of aromatic ring rotations, using the default force-field of the program MOLBLD3 (Boyd, 1968). An X-ray single crystal analysis on an analogous compound, 1-tolyl-naphthalene, OR44, is also presented together with the first results obtained from a crystal of *cis*-1,8-

di-tolylnaphthalene.



OR44

The results are presented as two-dimensional surfaces and calculated minimum energy conformations.

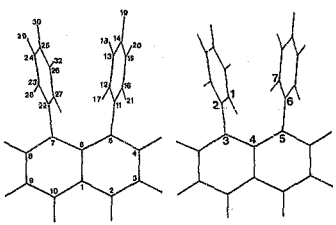


Fig.28. Stereoscopic drawing of the minimum energy arrangement of 1,8-diphenylnaphthalene obtained by molecular mechanics.

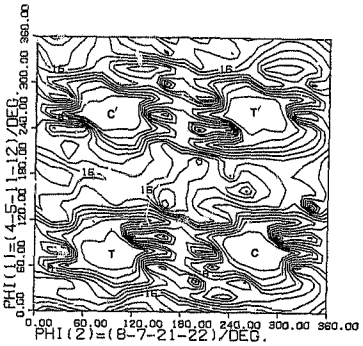


Fig.29. Two dimensional contour map of the conformational energy of 1,8-diphenylnaphthalene as a function of the rotation of the two phenyl rings. Contour intervals are 2 kcal/mol.

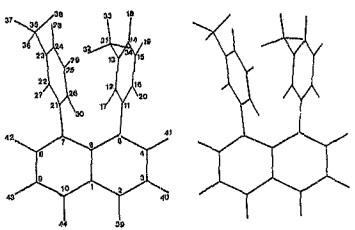


Fig.30. Stereoscopic drawing of the minimum energy arrangement of 1,8-di(meta-methylphenyl) naphthalene obtained by molecular mechanics.

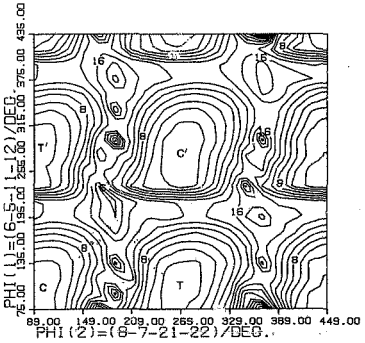


Fig.31. Two dimensional contour map of the conformational energy of 1,8-di(meta-methylphenyl)naphthalene as a function of the rotation of the two phenyl rings. Contour intervals are 2 kcal/mol.

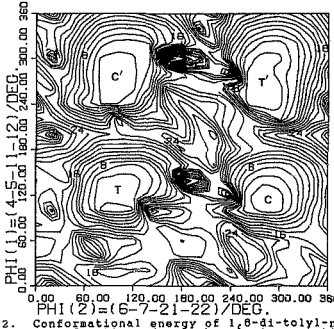


Fig.32. Conformational energy of 1,8-di-tolyl-naphthalene as a function of two phenyl ring rotations. Contours are drawn at 2 kcal/mol.

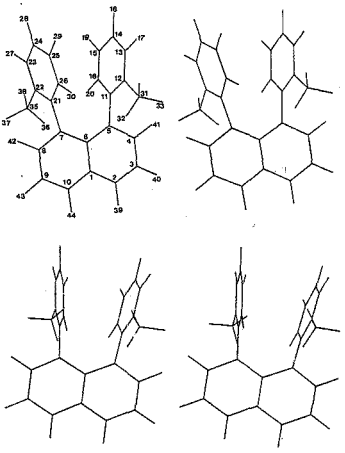


Fig.33. Stereoscopic drawings of the cis and trans forms of 1,8-di-tolylnaphthalene as obtained by molecular mechanics.

It is important to note that a complete energy minimisation was performed after each rotational step (See Chapter 5 for detailed discussion of this method). Any rotation that leads to severe steric clashing therefore produces a gradual distortion of the naphthalene skeleton to relieve the accumulating strain. For instance, when two aryl rings rotate together from an energy-minimum arrangement, a high-energy transition state, corresponding to a flattened naphthalene nucleus is approached. As they rotate past each other, the system relaxes into a different, low-energy puckered conformation. This type of

pathway occurs along the diagonals of the two-dimensional steric energy maps. Although conformational flip between enantiomeric configurations is therefore not explicitly provided for in the calculation, it is produced automatically by the facility to minimise conformational strain at all points on the surface. It follows that the effects produced by the rotations of a given aryl ring depend quite critically on the orientation of the second aryl ring, during the course of the rotation. With the second ring in a favourable orientation, the first can slide past fairly easily and without distorting the naphthalene nucleus. Another orientation of the second ring however, may produce a high-energy encounter that causes skeletal distortion and eventually conformational flip. This accounts for the fact that the calculated contour surfaces are not totally symmetrical.

The contour interval in Figure 29 is about 2 kcal/mol and the steric energy surface dufines four pronounced minima, corresponding to two pairs of enantiomeric conformations. Chirality results from distortion of the naphthalene nucleus and offset of the aryl groups in opposite directions with respect to the mean naphtaalene plane. Reversal of the offset inverts the sense of the helix defined by the bonds between the points of attachments on the aryl rings. This corresponds to flipping between enantiomers. Because of the absence of any substituents on the phenyl rings however, nominal geometric isomers are identical. The conformations marked T and C are indistinguishable and the barrier to interconversion simply represents the steric barrier to rotation of a phenyl ring. The calculated value is about 9 kcal/mol, in good agreement with previous estimates (House and Baske, 1965). The interconversion to an enantiomeric conformation (either T' or C') involves not only the rotation of one or both rings, but also the conformational flip, first described by Clough and Roberts (1975). This transition requires ca. 15 kcal/mol, from which the

barrier to conformational flip is obtained as 5.9 kcal/mol. An upper limit of 8.8 kcal/mol for flipping and a barrier to rotation of 16 kcal/mol were measured before (Clough and Roberts, 1975).

The results for 1,8-di(meta-methylphenyl)naphthalene is depicted in Figure 31. The contours were drawn also at intervals of 2 kcal/mol. Four distinct minima can be clearly identified, namely at positions of *phi(1)* and phi(2) of (90,90), (90,270), (270,90) and (270,270). These positions are denoted by the symbols C,T,T' and C' on the map. The barrier to rotation of one phenyl ring is calculated to be about 12 kcal/mol. This is ca. 3 kcal/mol higher than in the unsubstituted case of 1,8-diphenylnaphthalene. The interconversion from C to C' involves not only the rotation of one or both rings, but also a conformational flip, which was calculated to require ca. 20 kcal/mol, from which the b[illegible] of the conformational flip was obtained as ca. 6 kc[illegible]. These barrier heights explain the experimental diffi[illegible]es that House and co-workers (1970) experienced in trying to separate rotamers of 1,8-meta-substituted phenyl naphthalene derivatives.

The results for 1,8-di-tolylnaphthalene (Figure 32) are equally interesting though more complicated due to the presence of the o-methyl substituents. The contour interval in Figure 32 was kept the same as of Figures 29 and 31 to emphasise the increased strain in the 1,8-di-tolylnaphthalene system. Several modes of rotation must now be considered, depending on how the aromatic rings move past each other. Rotations that lead to direct clashing of the methyl groups would encounter enormous barriers, but the contour map shows that saddle points exist and that, by avoiding any direct clash, the barrier is reduced to about 16 kcal/mol. The barrier to enantiomer inversion with rotation now amounts to about 22 kcal/mol, which is in good agreement with the

experimentally measured value of 24 kcal/mol reported before (Clough and Roberts, 1975). The energy barrier to flipping follows as 6 kcal/mol, as for the previous two cases.

Discussion

The results are in total agreement with the measurements of both House (1970) and Roberts (1975). There are three barriers to conformational change: a high barrier to rotation with configurational flip; a low barrier without flip; and a simple configurational flip, without rotation. House and co-workers (1970) observed the low barriers to rotation (15 and 9.6 kcal/mol) at room temperature. These account for the rather rapid interconversion of rotamers in solution at room temperature. Clough and Roberts (1975) observed the high barriers (24 and 16 kcal/mol) to rotation at 40 degrees celsius, interpreted to be in line with the existence of separable rotamers of di-tolylnaphthalene. Their observed low barrier (9 kcal/mol) for this compound was correctly interpreted in terms of conformational flip.

House and co-workers (1965) observed the low rotational barriers. The implications are clear. Interconversion between geometric forms can occur with or without enantiomeric flip. The two types of barrier differ by 6 kcal/mol and whereas transition over the low barrier occurs at room temperature, rotation with flip is activated only at somewhat higher temperatures. The lower barrier allows equilibration in room-temperature solutions, but are sufficient to stabilise different rotamers in the solid state.

At the time this thesis was written the structure of one of the rotamers of 1,8-di-tolylnaphthalene, namely the cis-compound was determined by X-ray crystallography. Only the space group and cell constants obtained are

listed below, due to poor nature of the data brought about by poor crystal quality, decay and disorder in the crystal leaving the obtained structure planar.

Space Group: $P2_1/c$
Cell Constants: a = 16.560 angstrom
b = 7.565 angstrom
c = 15.382 angstrom
beta = 117.21 degrees

Work is in progress in an attempt to resolve the disorder that seems to be present. The calculated structures are in line with splaying of the bonds to the peri substituents and some deformation of the naphthalene nucleus. Details of these distortions are summarised in Table 49, in comparison with corresponding values, previously measured (Evrard et al., 1972) crystallographically in related structures. The agreement is good.

Table 49. Calculated bond parameters (angstrom and degrees) of di-arylnaphthalenes and the corresponding observed values from tetraphenylnaphthalene. As expected, the latter structure is more distorted but of the same conformational type. The numbering scheme is defined in Figure 28.

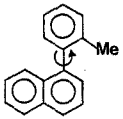
	Compounds				
Parameter*	1	2	3	4	Experimental
2-6	2.86	2.85	2.87	2.86	2.95
3-5	2.47	2.46	2.46	2.47	2.51
2-3-4	125.4	124.5	125.4	125.0	123.2
3-4-5	123.3	123.2	123.4	123.3	122.6
4-5-6	125.3	124.4	125.5	124.7	123.2
1-2-3-4	79.4	76.1	83.8	-81.5	-52.0
2-3-4-5	8.1	10.9	7.9	-9.4	-19.6
3-4-5-6	8.9	12.3	6.2	-10.1	-20.2
4-5-6-7	79.0	74.6	84.7	-76.9	-51.7

1: Di-phenyl-naphthelene
2: Di-(m-methylphenyl)naphthalene
3: Trans-di-o-tolylnaphthalene
4: Cis-di-o-tolylnaphthalene
* Bond lengths in angstrom and angles in degrees.

The calculated orthogonal coordinates for structures in this chapter, as well as a full list of calculated bond lengths and angles appear in Appendix I.

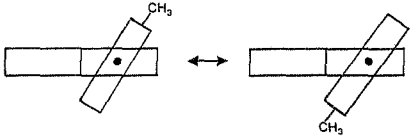
6.4 The Structure of 1-(o-Methyl)naphthalene

During the investigations on the structure of 1,8-diarylnaphthalene derivatives, an analogous compound 1-tolyl naphthalene, OR44, was also at hand in the crystalline form.



OR44

The structure of compound OR44 posed two interesting questions namely: (i) What is the orientation of the tolyl group with respect to the naphthalene nucleus; (ii) Would this orientation cause any distortions in the framework of the naphthalene skeleton. It is clear from the diagram below that if the torsion angle, phi lies in the region between 0 and 90 degrees that the resulting structure would consist of two stereoisomers.



Crystallography

The crystal of OR44 was mounted in a Lindemann tube and aligned on an Enraf Nonius CAD4 diffractometer equipped with a graphite monochromator and Mo-K(alpha) radiation. (See Chapter 8 for details). Accurate cell constants were

obtained from refinement of 25 high angle reflections. The necessary crystal data and crystallographic parameters appear in Table 50. The intensity data collected were corrected for Lorentz and polarisation effects, but not for absorption.

The structure was solved by direct methods using the TREF routine in the prereleased version of SHELXS86 (Sheldrick, 1985). The resulting E-map revealed all the non-hydrogen atoms. The structure was refined with full matrix-least-squares using the program SHELX76 (Sheldrick, 1976). Some, but not all, of the hydrogen atom positions were apparent on a difference Fourier map, and a riding model was used for all hydrogen atoms. The refined atomic coordinates and bond length and angles appear in Tables 51 and 52. Supplementary material is listed in Appendix II.

Discussion

The molecular structure of OR44 together with the numbering scheme (the hydrogen atoms numbering follows that of the carbon atoms they are bonded to) used, are shown in Figure 34.

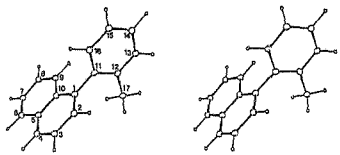


Fig.34. The molecular structure of OR44.

The bond lengths and angles agree well with accepted

literature values (Kennard et al., 1972). The main conformational feature is orientation of the tolyl group. It has a torsion angle of 90 degrees relative to the naphthalene framework. The packing diagram (Figure 35) shows no unusual non-bonded contacts.

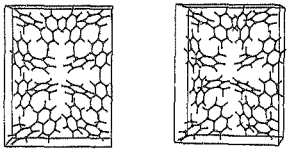


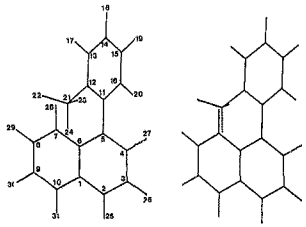
Fig.35. Packing diagram for OR44.

Simulated Structure of OR44

The unexpected result from the crystal structure regarding the orientation of the tolyl group (tolyl group orthogonal with naphthalene ring) prompted an investigation into the stereodynamics of 1-tolylnaphthalene by means of molecular mechanics calculation. The reason for this was to explore possible other orientations of the tolyl group which could exist possibly in solution (e.g. a possible biphanyl-like conformation of ca. 42 degrees).

Results

The minimum strain energy structure obtained for 1-tolylnaphthalene is depicted below (Geometric parameters and coordinates appear in Appendix I).



The two dimensional conformational energy map with contours drawn at 3 kcal/mol interval appear in Figure 36.

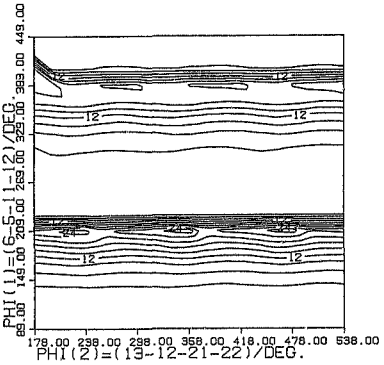


Fig.36. Conformational energy map for 1-tolylnaphthalene as calculated by molecular mechanics.

The contour map shows that rotation of the methyl group

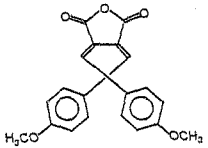
has little effect on conformational energy during the interconversion. It is shown that ca. 21 kcal/mol energy is required for the methyl-side of the tolyl group to pass over the naphthalene ring whereas only about 18 kcal/mol is required by the ortho-hydrogen side of the tolyl group.

CHAPTER 7

Conformational Analysis of Fulgides and Related Compounds

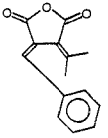
7.1 Introduction; Literature Survey

The derivatives of dimethylenesuccinic anhydride are known as "fulgides" (Stobbe, 1905). The aromatic fulgides are strongly coloured, yellow, orange or red when solid, and give intense yellow solutions. This old class of overcrowded molecules has been the point of focus of thermal and photochemical investigations, but never, as far as we could ascertain, has it been studied in any depth, structurally. The only crystal structure that we could find in the literature is that of di-p-anisylidenefulgide(di-p-anisylidenesuccinic anhydride), 101, published by Cohen and co-workers in 1970. They synthesised the trans-trans, cis-cis and the cis-trans isomers. The structure of the trans-trans isomer , 101, is depicted below. The benzene rings are not parallel, the angle between the vectors normal to the planes of the two rings were found to be 18.8 degrees.

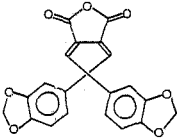


101

Swoboda et al. (1967) succeeded to synthesise the fulgides 102 and 103. The assignment of stereochemistry was done by NMR spectroscopy.

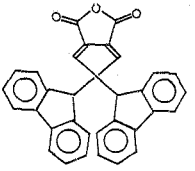


102

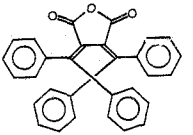


103

In 1972, Hart and Heller suggested that the stereochemistry of many bis-arylmethylenesuccinic anhydrides reported in the literature is incorrect. They were able readily to obtain stable high melting crystalline compounds, bis-diphenylmethylenesuccinic anhydride, 105 , and bis-fluorenylidenesuccinic anhydride, 104 .

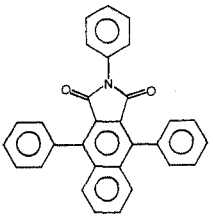


104



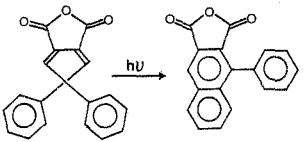
105

A few 1,2-dihydronaphthalene derivatives were also isolated, including 106.



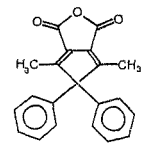
106

Crescente et al. (1979) showed that irradiation of (E,E)-bis(benzylidene)succinic anhydride, 108, in toluene gave 1-phenyl-1,4-dihydronaphthalene-2,3-dicarboxylic anhydride, 109.



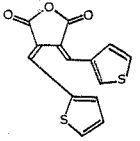
108 109

The synthesis, stereochemistry and isomerisation of alpha-phenylethylidenesuccinic esters and related compounds were studied by Heller and Szewczyk (1974). One of the many fulgides studied was, 110.



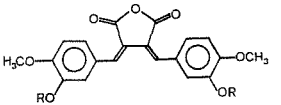
110

The synthesis of 4-(2-thienyl)benzothiophen-5,6-dicarbonylic anhydride, 111, was published by Abdel-Wahhab et al. (1971).

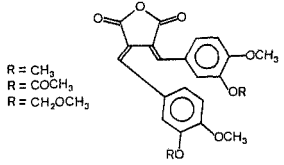


111

The photochemical cis-trans isomerisation of substituted dibenzylidene succinic anhydrides, 112 and 113, were investigated by Brunow and Tylli (1968). The observed downfield shift of the vinyl proton signals in the NMR spectra was attributed to the decreased conjugation in the fulgide molecule. To get a rough theoretical estimate of this effect, the pi-electron charge densities at the carbon atoms bearing the vinyl hydrogen atoms were calculated by the Hückel molecular orbital method.



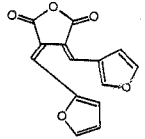
112



113

R = CH_3
R = $COCH_3$
R = CH_2OCH_3

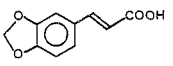
Titherley and Spencer (1904) obtained the disubstituted anhydride 114.



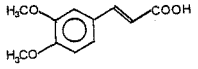
114

The study of fulgide conformation and internal rotation was initiated by analysing the possible conformations of the different subunits, that appear in the fulgides.

To establish why planar aromatic molecules tend to crystallize in highly overlapped structures, Desiraju and co-workers (1984) published the crystal structures of 3,4-methylenedioxycinnamic acid, 115, and that of 3,4-dimethoxycinnamic acid, 116. Their aim was to do crystal engineering via non-bonded interactions involving oxygen.

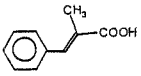


115

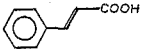


116

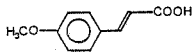
So varied and subtle are the factors governing the crystal structures of organic compounds that even a minor change in the chemical structure can often result in a considerably different crystal structure. Many crystal structure analyses on derivatives of cinnamic acid were investigated, namely, alpha-methyl-trans-cinnamic acid, 117, (Bryan and White, 1982), alpha-trans- and p-methoxycinnamic acids, 118 and 119, (Bryan and Freyberg, 1975), 2-coumaric acid, 120, (Raghunathan et al., 1979), and 3,4-dimethoxycinnamic acid, 121, (Desiraju and Sarma, 1983).



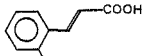
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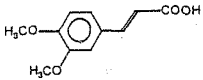
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119



120

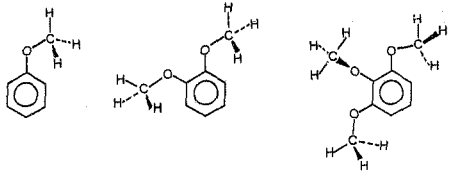


121

The aim of looking at all these crystal structures in terms of fulgide conformation was to determine whether the cinnamic acid framework is planar or not. In all cases the fragment was found to be planar within a 3 to 4 degrees margin.

Since our work dealt with p-methoxyphenyl- and 3,4-dimethoxyfulgides, it was thought appropriate to review the possible conformations that methoxy groups can adapt on a benzene ring. A crystal structure survey of the geometry of the methoxyphenyl group appeared in the recent literature (Nyburg and Faerman, 1986). The Cambridge Structural Database was used to retrieve the geometry of the methoxyphenyl group as determined in 212 crystal structure analyses by X-ray diffraction. The experimental data were found to be in accord with recent STO-3G basis MO calculations in that the preferred conformation is planar with a standard deviation of 5.0 degrees. The O-CH_3 and O-C(aromatic) bond lengths are 1.425 and 1.371 angstrom, respectively.

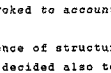
Cosse-Barbi and Dubois (1986) did a conformational dynamics study on the correlated and non-correlated rotations of methoxy groups in anomeric structures. They *suggested* that the rotation of one -OMe group induces a similar rotation of the other distant rotor. Karle et al. (1984) investigated the conformations of five tetra- and pentamethoxylated phenyl derivatives. In three of the compounds studied, the methyl carbon atoms were placed alternately above and below the plane of the aryl group in a regular fashion, while in two very similar compounds there were unexpected irregularities in the rotations about the C(aromatic)-O bonds and consequent crowding of adjacent methoxy groups. Preferred conformations of methoxy groups on phenyl rings for mono-o-di-, and o-trimethoxy substitution are shown below:



A survey of monomethoxy- and o-dimethoxyphenyl structures has been published by Anderson et al. (1979). Photoelectron spectroscopy and ab initio STO-3G calculations provided evidence that o-dimethoxybenzene and derivatives *exist in predominantly* non-planar conformations in the gas phase. By contrast, the preferred conformations of methoxybenzene and m- and p-dimethoxybenzene are planar, and the non-planar structures are significantly less stable. Partition coefficient, dipole moment, and dielectric relaxation measurements on o-dimethoxybenzene are consistent with the presence of *non-planar conformations* in solution. On the other hand, the crystal structures of compounds bearing o-dimethoxy

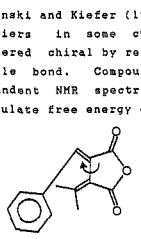
substituents indicate a preference for planar conformations: of 32 examples studied by these authors in the literature, only two structures contained a non-planar methoxy group. In the absence of steric hindrance, these substituents with lone pairs attached to aromatic rings prefer planar conformations over gauche or perpendicular conformations. Optimisation of conjugation between the substituent p-type lone pair orbital and the aromatic system is commonly invoked to account for this preference.

In view of the absence of structural data on fulgide conformations it was decided also to look at examples of strained anhydrides in the literature. Empirical force field calculations to evaluate the gem-dialkyl effect on succinic anhydride derivatives, like 121, resulted in a torsion angle of 25.8 degrees (Ivanov and Pojarlieff, 1984). Hence, in anhydrides, substitution leads to a twist around the C(2)-C(3) bond due to steric repulsion.

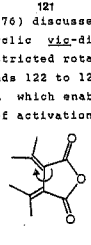


121

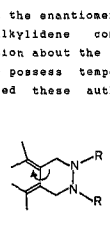
Jelinski and Kiefer (1976) discussed the enantiomerisation barriers in some cyclic vic-dialkylidene compounds, rendered chiral by restricted rotation about the sp^2-sp^2 single bond. Compounds 122 to 123 possess temperature-dependent NMR spectra which enabled these authors to calculate free energy of activation.



122



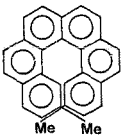
123



The helical structure of some fulgides make them in a way

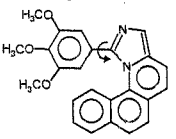
similar to the conformations of the helicenes. The helicenes are characterised by a chiral structure made up of ortho-condensed aromatic rings, by the presence of a powerful inherently chiral chromophore, and by the possibility of interactions (e.g. electronic interactions) between overlapping aromatic rings (Martin, 1974).

Rates of racemisation of several Me-substituted helicenes have been measured, like the barrier of 43.8 kcal/mol reported for 1,14-dimethylhexahelicene, 124. From an analysis by these authors (Borkent and Laarhoven, 1978), a suggestion was made that the conformation of highest energy during racemisation of 124 is that of which the terminal rings in an orthogonal position.



124

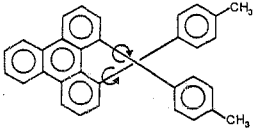
The rotational energy barrier in the 1-aryl derivative of benz[h]imidazo[1,5-a]quinoline, 125, was determined by computer line-shape analysis by Schmidt et al. (1977) to be 11.8 kcal/mol. They postulated that it is likely that, as suggested by a Dreiding model, the aryl substituent cannot undergo 360 degrees rotation but rather changes conformations by a rocking motion.



125

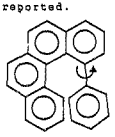
This could be the value for a helicity reversal rather than a ring rotation.

The 4,5-disubstituted phenanthrenes are known to exist in chiral, non-planar conformation. It is therefore possible to study the rates of racemisation by introducing an appropriate substituent. Chirality and conformational changes in 4,5-diaryltriphenylenes were studied by *Tinnemans and Laarhoven* (1979). The barrier for the rotation of the phenyl substituents in, 126, was determined as 13.4 kcal/mol.



126

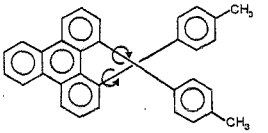
In a recent paper concerning the chirality and conformational changes in 1-phenylbenzo[c]phenanthrene, 127, Laarhoven et al. (1978) demonstrated that rotation of the phenyl group can occur independently of racemi _tion. The rotation of the phenyl group is hindered by a barrier of 13.0 kcal/mol. During racemisation the phenyl group turns around the opposite end of the benzo[c]phenanthrene skeleton in a rotatory movement. This is accompanied by a simultaneous inversion of helical conformation. For this process a barrier of ca. 16 kcal/mol was reported.



127

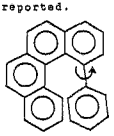
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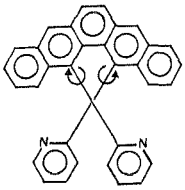
126

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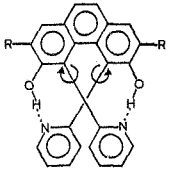
127

Previously a similar study has been described for N-methyl and N,N'-dimethylpyridinium derivatives derived from 13,14-bis(2-pyridyl)pentathene, 128, (Fields et al., 1971a). It was shown that rotational and inversional barriers can be separated. The exchange processes in these compounds were ascribed to an unexpectedly facile pentaphene ring inversion, accompanied by a synchronous rotation of both pyridinium rings.



128

In 1971b, Fields and Regan published a paper on a new class of sterically hindered compounds, namely the 4,5-bis(2-pyridyl)phenanthrene-3,6-diols, 129, which if completely planar would have the two pyridyl rings occupying the same space.



129

Obviously considerable altering of normal bond lengths and angles is necessary to have even the minimum allowed ca. 3 angstrom separation between the pyridyl rings. The nature and extent of the deviations have been clarified through a

single-crystal X-ray analysis of a representative member 130 (Smith and Barrett, 1971), and a brief qualitative description of the results is warranted here. No major segment of the molecule is truly planar; yet all individual rings are approximately planar. The pyridyl rings have a stair-step relationship to one another (Figure 37) and are almost parallel, being inclined 11 degrees to each other.

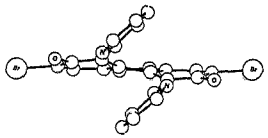


Fig.37. View of 130 along the twofold symmetry axes.

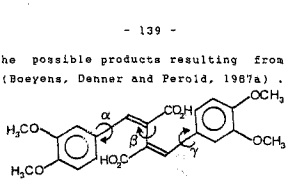
Under such steric constraint it seems reasonable that the pyridines would suffer restricted rotation, making possible two diastereomers other than that known from the X-ray study. The three isomers would differ solely in the orientation of the pyridines with respect to one another. These authors found no experimental evidence suggesting the presence of the latter two possibilities.

7.2 Stereochemistry and Conformational Analysis of Bis-(3,4-dimethoxybenzylidene) Succinic Anhydrides by X-ray Crystallography and Molecular Mechanics

7.2.1 Introduction

The dicarboxylic acid derivative, 131, presents one with an interesting stereochemical problem when one has to

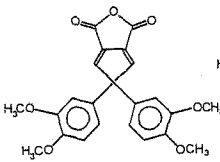
predict the possible products resulting from anhydride formation (Boeyens, Denner and Perold, 1987a).



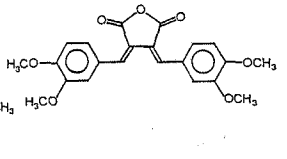
131

The governing factor in the case of compound 131 is the magnitudes of the torsion angles alpha, beta and gamma (and to a lesser extent that of the MeO-phenyl groups) at the anhydride ring closure step. This is a typical example of how single-bond torsional mobility predicts the resulting conformational preferences.

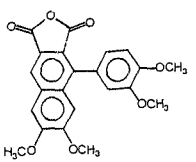
Single crystal X-ray analysis was used to study the products from the anhydride-formation reaction in an effort to develop an understanding of these interesting molecules. Crystal structures determinations of the compounds listed below were performed.



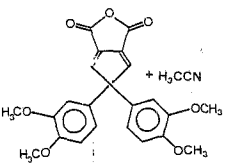
QR02



QR06

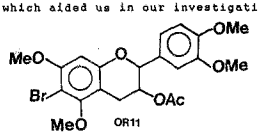


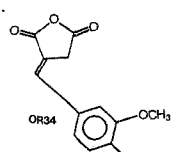
QR08



QR41

The conformations of other possible rotamers were simulated by molecular mechanics, using an empirical force-field based on the observed structure of the E,E-rotamer, described here. Furthermore, two additional crystal structures, that of OR11 and OR34, will also be discussed because they possess analogous functional groups which aided us in our investigations.





7.2.2 Exploratory Studies

7.2.2.1 The Structure of E-veratrylidene-succinic Anhydride

Crystallography

A suitable needle-like crystal of OR34 was selected for data collection and sealed in a 0.3 mm Lindemann capillary (Boeyens, Denner and Perold, 1987b). The crystal system and approximate cell dimensions were determined photographically. Accurate cell parameters were determined and reflection data collected on an Enraf Nonius CAD4 diffractometer (See Chapter 8 for operational details). Reflections were measured using graphite monochromated Mo-K(alpha) radiation. Table 53 contains the necessary crystallographic information. The data were corrected for Lorentz and polarisation factors and also for absorption.

The structure was solved by direct methods SHELXS86 (Sheldrick, 1985) which gave the positions of all the non-hydrogen atoms. The structure was refined isotropically by full-matrix least-squares using the program SHELX76

(Sheldrick, 1978). An isotropic refinement followed by a difference synthesis gave the positions of three hydrogen atoms. The rest of the hydrogen atoms were placed in calculated positions. The final atomic coordinates are given in Table 54. The molecular structure of OR34 and atomic numbering is given in Figure 38. The bond lengths and angles are listed in Table 55. Tables of anisotropic thermal parameters, hydrogen atom coordinates and temperature factors, and observed and calculated structure factors appear in Appendix II.

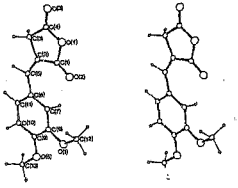


Fig.38. Molecular structure of OR34.

Discussion

The reason for determining the structure of OR34, which was at hand, was to establish the conformational relationship between the anhydride moiety and the aryl ring. This aspect was explored by calculating the best planes through the phenyl ring and the fragment consisting of the atoms C(1), C(2), O(2) and O(4) respectively. The two planes are inclined by an angle of 5.8 degrees, confirming that the two pi-systems are in virtually parallel alignment.

7.2.2.2 Crystal Structure and Absolute Configuration of (-)-3-O-Acetyl-6-bromo-3',4',5',7-tetra-O-methylepigateschin

Experimental

The title compound, OR11, crystallises in the orthorhombic space group $P2_12_12_1$ which is uniquely defined by the systematic absence of $h\phi\phi$, $\phi k\phi$, and $\phi\phi l$, reflections for h, k, l, $\neq 2n$. (Boeyens, Denner et al., 1986). Intensity data were measured at room temperature with an Enraf Nonius CAD4 single-crystal diffractometer with an incident beam graphite-crystal monochromator (See Chapter 8 for operational conditions). The crystal data and crystallographic details appear in Table 56. For the structure solution an orthorhoabic data set was recorded, and in order to determine the absolute configuration the Friedel opposite reflections at negative hkl were measured directly after each reflection of positive index. Data reduction consisted of correction for background, Lorentz polarisation, and absorption.

Results and Discussion

The structure was solved by Patterson methods and refined by the full-matrix least-squares technique, using the program SHELX76 (Sheldrick, 1978). In view of the fairly widespread practice (Jones, 1984) of basing enantiomer refinement on data sets not containing Friedel opposites, without applying absorption corrections and using unit weights, the importance of these factors was reiterated during refinement of this structure. It was found that two modes of refinement indicated different enantiomers as the most likely absolute configuration. A standard data set, consisting of 1025 reflections, not including Friedel opposites and not corrected for absorption, refined to unit weight R indices of 0.07 and 0.08 for the (2S,3S)- and (2R,3R) conformations respectively. This difference

is highly significant in terms of a Hamilton test (Hamilton, 1965). The R indices in refinement with a full data set of 2410 reflections corrected for absorption and using a weighting scheme based on counting statistics, are shown below:

	(2S,3S)	(2R,3R)
R	0.084	0.062
R_w	0.057	0.036
R_e	0.049	0.022

(R definitions - SHELX76 (Sheldrick, 1976))

There is an even more significant difference, but now favouring the opposite (2R,3R)-isomer. We accept the more detailed refinement as conclusive and as dramatic vindication of the cautionary measures advocated by Jones (1984) for the refinement of absolute structure. Some of the methyl hydrogen atoms that could not be located by difference synthesis were placed in calculated positions and the methyl groups were refined as rotating tetrahedra, using a common isotropic temperature factor for all hydrogen atoms. Anisotropic temperature thermal parameters were refined for all non-hydrogen atoms. The refined coordinates are in Table 57, listed in terms of the atomic numbering scheme defined in Figure 39. The numbering of hydrogen atoms follows the carbon atom numbers. Anisotropic temperature factors, hydrogen atom coordinates and observed and calculated structure factors are listed in Appendix II. All calculated bond lengths and angles (Table 58) compared well with accepted literature standards (Kennard et al., 1972). The puckered ring has the symmetrical half-chair conformation $^2H_3$, in terms of the crystallographic carbon atom numbering and as specified by its coordinates of pucker (Boeyens, 1978); phi = 270 degrees, theta = 126 degrees, and amplitude is 0.5 angstrom.

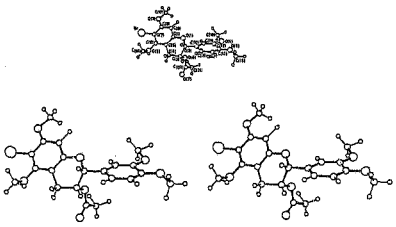


Fig.39. Molecular structure of OR11.

One aspect of particular interest to us was the conformation of methoxy groups in relation to the phenyl rings. The methoxy groups bonded to C(12) and C(13) lie in the plane of the phenyl ring. However, the presence of the bulky Br-atom attached to C(7) forces the two adjacent methoxy groups to lie above and below the plane of the aromatic system. Only one is significantly rotated out of the plane of the benzene ring.

7.2.3 Crystallographic Analysis of Bis(3,4-di-methoxy-benzylidene) Succinic Anhydrides

Crystal Data

Single crystals were examined by standard optical and photographic X-ray methods. Crystals of OR02 (Stobbe and Leuner, 1911; Boshoff and Perold, 1986a and 1986b) were well developed ruby hexagonal prisms and diffraction quality crystals were readily selected. Fulgide OR06 crystallised in well-formed orange plates or flat needles of good quality in ordinary light but showing extensive poly-crystalline domain structure in polarised light and by X-ray diffraction. Isolated crystals, suitable for

crystallographic work could be selected on two occasions from recrystallised material. Both crystals were found to correspond to the Z,Z-isomer OR06. Crystals of OR08 were small but well-developed platelets. The crystals were of relatively low quality, but suitable for crystallographic characterisation. The identity of poly-crystalline OR06, and single crystal thereof, was established by X-ray powder diffraction. Single crystals of OR02 obtained from acetonitrile were shown crystallographically to be solvated. To distinguish between the solvated and unsolvated OR02, the code OR41 was assigned to the solvated form.

Crystallography

Crystal data and details of the single-crystal analyses of compounds OR02, OR06, OR08 and OR41 are given in Tables 59 to 62. All measurements were made on an Enraf-Nonius CAD4 diffractometer with an incident beam graphite crystal monochromator using Mo-K(alpha) radiation (See Chapter 8 for operational details). The cell constants were determined by least-squares refinement, based on 25 accurately measured high theta values.

Data reduction consisted of correction for background, Lp and absorption, but no decay correction. The structures were solved by direct methods using the program SHELXS86 (Sheldrick, 1985) and refined by full-matrix least-squares using the program SHELX76 (Sheldrick, 1976), and a weighting scheme based on counting statistics. Structure OR08, by all criteria, did not refine as well as the others, because of the poorer quality crystal, but converged convincingly to an arrangement, completely commensurate with other evidence, to firmly establish the molecular structure. No hydrogen atom positions were calculated for OR08. In the other structures, hydrogen atoms were placed in calculated positions. The refined fractional coordinates of the four compounds are in Tables 63 to 66, according to the atomic numbering schemes in

figure A. Hydrogen atomic numbering follows the numbering of their associated heavy atoms. Lists of bond lengths and angles appear in Tables 67 to 70. Final observed and calculated structure factors, atom thermal parameters and hydrogen atom coordinates are listed in Appendix II.

To establish the identity of the selected single crystal from recrystallised OR06 and the bulk of the material a powder diffraction pattern was generated by computer methods (Yoon et al., 1977) from the single-crystal structure for comparison with the observed powder pattern of the bulk of OR06. The observed pattern, recorded on a Rigaku diffractometer, using Cu-K(alpha) radiation, agreed in detail with the calculated, confirming the identities of the single-crystal and the bulk of the material.

Results and Discussions

Two of the symmetrical forms were studied in detail by X-ray diffraction. In both structures, OR02 and OR06, a two-fold axis through O(1) and bisecting the C(2) - C(2'), relates the two halves of the molecule. The other symmetrical structure, OR41, does not have a two-fold axes, this is probably due to the presence of the acetonitrile solvent molecule. In addition the dehydrogenation product OR08 arising from the E,Z-form has also been studied. Stereoscopic drawings of the four X-ray structure are shown in Figure 40 to 43.

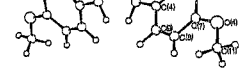


Figure A. Atomic numbering scheme.

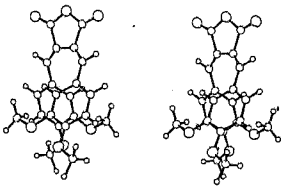


Fig.40. Molecular structure of OR02.

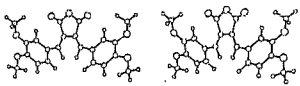


Fig.41. Molecular structure of OR06.

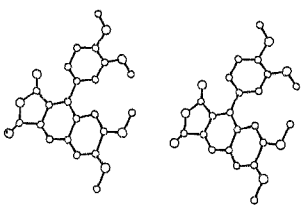


Fig.42. Molecular structure of OR08.

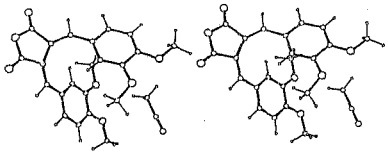


Fig.43. Molecular structure of OR41.

Comparison of the anhydride conformations in the three forms provide an interesting measure of the relative steric strain for each case. A comparison, both in terms of puckering parameters (*Cremer and Pople, 1975*) and endocyclic torsion angles is made in Table 71. It clearly indicates OR02 (and OR41) as the sterically most strained form. The strain originates from the eclipsing of the aryl rings. In the Z,Z-form, OR06, the non-bonded repulsion is eliminated by separation of the rings and in OR08 by the formation of the naphthalene structure. The observed difference in density of the two forms probably relates to the eclipsed and open nature of structures OR02 and OR06, respe

Comparison of the anhydride configurations in the X-ray structures of OR02, OR06 and OR08. The angles phi are endocyclic torsion angles in degrees. Chi and Q represent puckering mode and amplitude in degrees and angstrom respectively.

Table 71. Comp

Comparison of the anhydride configurations in the X-ray structures of OR02, OR06 and OR08. The angles phi are endocyclic torsion angles in degrees. Chi and Q represent puckering mode and amplitude in degrees and angstrom respectively.

Compound	OR02	OR06	OR08
Phi[O(1)-C(1)]	-4.8	-1.1	-2.2
Phi[C(1)-C(2)]	12.4	2.5	5.7
Phi[C(2)-C(2')]	-14.6	-3.0	-1.6
Chi	90.0	90.0	94.8
Q	0.14	0.06	0.03
Conformation	$^{3}T_{4}$	$^{3}T_{4}$	$^{3}T_{4}$

No unusual bond lengths were observed, but the bond angles at the olefinic trigonal carbon atoms, as shown in Figure 44, are worth nothing.

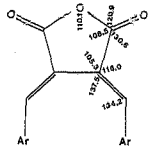
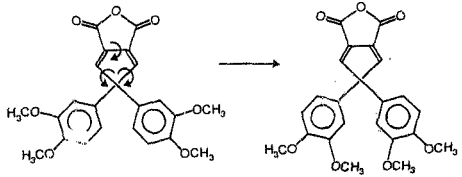


Fig.44. Some bond angles for OR02.

In order to keep C(1)-C(2)-C(2') close to the 108 degrees required for a planar pentagon, the external angle C(2')-C(2)-C(3) opens up to 137 degrees. For the same reason the angle O(2)-C(1)-C(2) is found to be 131 degrees. The opening of C(2)-C(3)-C(4) to 134 degrees occurs for less obvious reasons. It is noted that the reason why the bonds O(2)=C(1) and C(3)=C(2) do not bisect the external ring angles, is not entirely of steric origin.

7.2.4 Molecular Mechanics of Bis(3,4-di-methoxy-benzylidene) Succinic Anhydrides

The crystallographic study on OR02 revealed that it is the most strained of the fulgide structures studied here. It was decided to perform a thorough conformational study on OR02 in search for any other possible rotamers. NMR spectroscopy indicated the existence in solution of another symmetrical isomer like 132, accessible by rotation around single bonds which was best monitored by molecular mechanics.



OR02 132

The reliability of molecular mechanics predictions of configuration depends on the relevance of the force-field, rather than its theoretical basis. A force-field defined from first chemical principles could thus be less appropriate than an empirical force-field adapted to the same type of structure. In the present application an empirical force-field was parameterised against the criterion of correctly predicting the crystallographic structure of OR02. However, trial values of harmonic force constants and strain-free bond parameters [k,p$_o$] were adopted within chemical reason and should be relevant to the interpretation of electronic bond orders (Boeyens, Cotton and Han, 1985). Numerical detail of the force-field is listed in Appendix I. Currently accepted (Boeyens, Cotton and Han, 1985) non-bonded potentials were used without modification.

Free rotations that could produce *different* rotamers of OR02 are *possible* around the single bonds C(3)-C(4) and C(3')-C(4') only. Whereas these rotations do not preserve the two-fold symmetry of OR02, a limited rotation or flipping around C(2)-C(2') does. It is noted that symmetrical rotamers obtained by the C(2)-C(2') flip are also accessible by rotations around both C(3)-C(4) and C(3')-C(4') but compared to C(2)-C(2') flip this latter mode is less convenient for the sampling of all symmetrical forms. The configurational flip with conservation of symmetry requires simultaneous rotations

around the bonds C(3)-C(4), C(2)-C(2') and C(3')-C(4'). The corresponding torsion angles in the observed structure are -10, -40 and -10 degrees, respectively. If it flips to an enantiomeric arrangement at 10, 40, 10 degrees over a transition state of -90, 0, -90 degrees, rotations of -160, 80 and 160 degrees respectively are involved. The first rotation should therefore be driven at twice the rate of the second and in an opposite sense. Symmetrical OR06, Z,Z and unsymmetrical (precursor to OR08), E,Z open structures arise from forced rotations around C(2) = C(3) double bonds. These rotations have not been examined by molecular mechanics. Barriers to single-bond rotation were calculated by replacing the standard torsional parameter by an artificial incremental parameter (Boyd, 1968) to drive the rotation around the bond, sampling and refining the steric strain at each position.

Results and Discussions: Simulated Structures

Crystallographic and molecular mechanics results established the number of possible isomers at five, viz. three symmetrical [E,E(OR02); Z,Z(OR06); E,E + flip (132)] and two asymmetrical forms [E,Z(OR08); E,E + 1 phenyl (133) group rotated through 180 degrees]. The unsymmetrical open form however, is unstable and readily dehydrogenates to form a naphthalene structure.

The empirical force-field adapted to reproduce structure OR02 was assumed to be appropriate for all rotamers of this compound and for modelling barriers to rotation. Steric energy as a function of an aryl ring rotation is shown in Figure 45. A barrier of about 15.5 kcal/mol separates OR02 from an energetically almost equivalent, but unsymmetrical, form 133, reached after rotation through 180 degrees. This structure, shown stereoscopically in Figure 46, has not been observed in the course of these experiments. This is interpreted to indicate that rotation is restricted by this barrier in

around the bonds C(3)-C(4), C(2)-C(2') and C(3')-C(4'). The corresponding torsion angles in the observed structure are -10, -40 and -10 degrees, respectively. If it flips to an enantiomeric arrangement at 10, 40, 10 degrees over a transition state of -90, 0, -90 degrees, rotations of -160, 80 and 160 degrees respectively are involved. The first rotation should therefore be driven at twice the rate of the second and in an opposite sense. Symmetrical OR06. E,Z and unsymmetrical (precursor to OR08), E,Z open structures arise from forced rotations around C(2) = C(3) double bonds. These rotations have not been examined by molecular mechanics. Barriers to single-bond rotation were calculated by replacing the standard torsional parameter by an artificial incremental parameter (Boyd, 1968) to drive the rotation around the bond, sampling and refining the steric strain at each position.

Results and Discussions: Simulated Structures

Crystallographic and molecular mechanics results established the number of possible isomers at five, viz. three symmetrical [E,E(OR02); Z,Z(OR06); E,E + flip (132)] and two asymmetrical forms [E,Z(OR08); E,E + 1 phenyl (133) group rotated through 180 degrees]. The unsymmetrical open form however, is unstable and readily dehydrogenates to form a naphthalene structure.

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the experimental temperature range. The refined coordinates, bond lengths and angles of 133 appear in Appendix I.

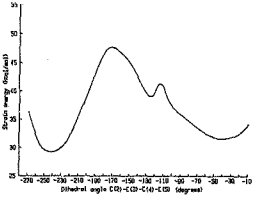


Fig.45. Steric energy of OR02 as a function of one aryl ring rotation.

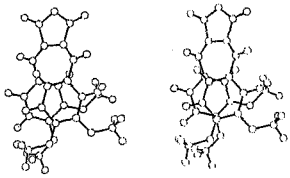


Fig.46. Calculated structure for compound 133.

The only other possible rotation is around the central bond, opposite the furyl oxygen atom. The steric energy profile for this rotation coupled with appropriate rotations around the C(3)-C(4) single bonds, is presented in Figure 47. This structure is shown stereoscopically in Figure 48. Minimum energy arrangements are predicted at angles of about -20 and 45 degrees of the torsion angle

phi (3-2-2'-3'), with a maximum near 30 degrees. The potential barrier is about 4.6 kcal/mol, low enough to allow interconversion between OR02 and 132. Orthogonalised atomic coordinates, bond lengths and angles for the simulated structure of 132 appear in Appendix I. It is suggested that a similar interconversion between the unsymmetrical structure and its enantiomer is possible.

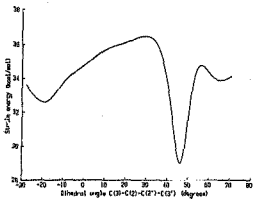


Fig.47. Steric energy of OR02 as a function of flip and two aryl ring rotations.

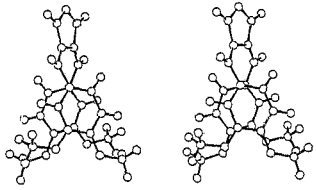


Fig.48. Calculated structure of compound 132.

The most interesting feature of structure 132, is the nearly equivalent dispositions of the methoxy groups, with respect to the eclipsing benzene rings. It is of further interest to note that rotamer 132 is calculated to be more

stable than OR02 by about 4.8 kcal/mol, although only OR02 seems to occur in the crystalline state. These two factors are probably related. In the crystal, neighbouring molecules pack in a head-to-tail fashion with major intermolecular contact between the parallel aryl rings, as shown in the packing diagram of OR02. The intermolecular pi-interactions benefit from the way in which the methoxy groups lie on the outside of the overlapping aromatic rings. If, because of its different conformation, this efficient mode of packing is not possible between molecules of 132, it could explain why crystals of OR02 have the lower lattice energy. The major conformational difference that could affect the packing is in the torsion angles C(2)-C(3)-C(4)-C(5), calculated at ca. -30 and ca. 115 degrees for isolated molecules of OR02 and 8 respectively. The former represents a flatter type of molecule that should pack together more smoothly than the more angular type of molecule 132, which in turn has its aryl rings more closely in parallel alignment. The major NMR solution signal could therefore quite possibly represent molecule132, which on crystallisation transforms into OR02.

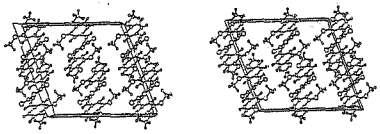


Fig.49. Packing diagram for OR02.

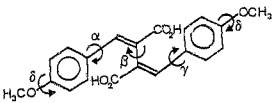
Some features of the force field required for the simulation of the eclipsed structures deserve comment.

Accepted values of bond lengths and angles are not adequate everywhere, particularly not around the double bonds to the five-membered ring. Although no adjustment of force constants was attempted during parameterisation, the reference values for bonds and angles in this vicinity clearly indicate extensive delocalisation around the conjugated system C(4-3-2-2'-3'-4') and O(2)-C(1)-C(2) etc. The O(2)-C(1)-C(2) bond angle of 131 degrees however, results from a simple steric effect. One important consequence of the delocalisation would be lowering of the barrier to rotation around the formal double bonds, thus promoting conversion to E,Z and Z,Z-isomers. The methoxy groups were modelled to be co-planar with the aromatic rings, as suggested by Ando et al. (1979), and as observed for all the structures reported here, and elsewhere (Karle *et al.*, 1984).

7.3 *Stereochemistry and Conformational Analysis of Bis-(p-methoxybenzilidene) Succinic Anhydrides by X-ray Crystallography and Molecular Mechanics*

7.3.1 Introduction

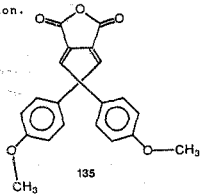
In analogy to the previous section of this Chapter, and based on the interesting conformational features that emerged from that study, we also investigated the possible products that could be formed on ring closure of bis-p-methoxybenzylidenesuccinic acid, 134, to form corresponding anhydrides (*Boeyens, Denner and Perold, 1987c*).



134

Again, relative rotational freedom about the three single bonds alpha, beta and gamma can lead to a variety of

products. *Furthermore*, the conformational analysis of products resulting from this anhydride-formation is complicated by the fairly low barrier to rotation around the MeO-phenyl (indicated as delta) bonds. Cohen et al. (1970) published the structure of 135, one possible product from this reaction.



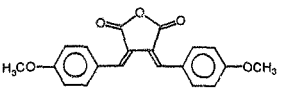
135

In addition to compound 135, we performed X-ray crystallographic analyses on two additional crystalline compounds obtained from the above-mentioned reaction.

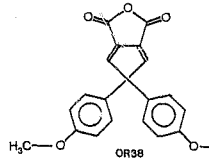
7.3.2 The Crystal Structures of Two Bis(p-methoxybenzylidene) Succinic Anhydrides

Crystallography

The single crystal structures of Z,Z-bis(p-methoxybenzylidene) succinic anhydride, OR37, and a rotameric form of E,E-bis(p-methoxybenzylidene) succinic anhydride, OR38, were determined.



OR37



OR38

The quality of crystals selected were analysed by standard oscillation and Weissenberg photographs. Although crystals of OR37 were very small and fibrous, compared to those of OR38, it still gave a reasonable diffraction pattern. The crystals were transferred onto an Enraf Nonius CAD4 diffractometer equipped with graphite monochromated Mo-K(alpha) radiation (See Chapter 8 for operational details). Accurate cell parameter were obtained from least-squares refinement of 25 high-order theta reflections, which were sufficiently spread through reciprocal space. The necessary crystal data and details of the crystallographic analyses appear in Tables 72 and 73. No crystallographic molecular symmetry was found, despite the fact that molecular two-fold axes could pass through the oxygen atoms of the anhydride rings. The data were corrected for background, Lp, and in the case of OR38, for isotropic decay.

The structures were solved by direct methods using the program SHELXS86 (Sheldrick, 1985). All of the non-hydrogen atoms were apparent on the first E-maps. The structures were refined, first isotropically and then anisotropically for the non-hydrogen atoms, by full-matrix least-squares using the program SHELX76 (Sheldrick, 1976). Hydrogen atoms were treated uniformly by placing them in calculated positions and by refining them with common isotropic temperature factors. The final atomic fractional coordinates for OR37 and OR38 appear in Tables 74 and 75, respectively. The bond lengths and angles for OR37 and OR38 are listed in Tables 76 and 77. An

isotropic thermal parameters for non-hydrogen atoms, fractional atomic coordinates for hydrogen atoms and observed and calculated structure amplitudes for OR37 and OR38 appear in Appendix II.

Results and Discussion

Stereoscopic drawings of the molecular structures obtained for OR37 and OR38, together with the numbering schemes used (Hydrogen atom numbers follow the corresponding heavy atoms they are bonded too) are depicted in Figures 49 and 51.

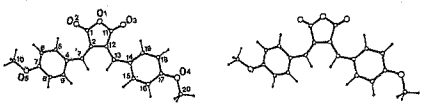


Fig.50. Molecular Structure of OR37.

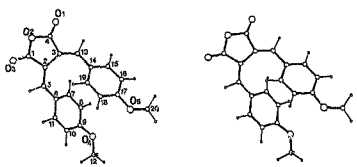


Fig.51. Molecular Structure of OR38.

The observed bond parameters agree well with accepted literature values (Cohen et al., 1979; Kennard et al., 1972). The packing diagrams for OR37 and OR38 are shown in Figures 52 and 53.

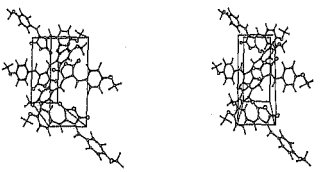


Fig.52. Packing diagram for OR37.

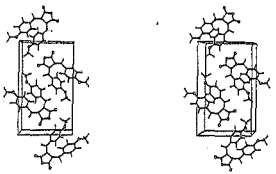
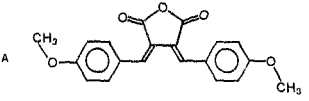
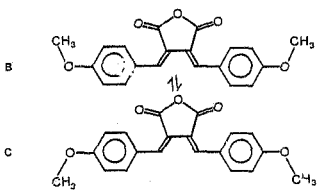


Fig.53. Packing diagram for OR38.

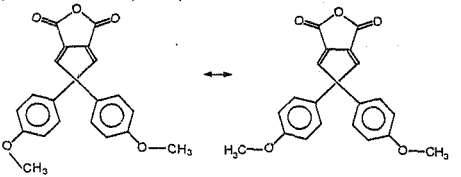
The *most interesting* feature that emerged from the structure determination of OR37 is its unexpected conformation, viz. A instead of B or C.





Furthermore the two aryl rings also do not have the same orientation with respect to the anhydride ring. This is probably a combined effect resulting from the fairly open structure and relatively free rotation about the MeO-phenyl bonds.

The observed structure for OR38 is a remarkable result. To our knowledge, this is the first time that a rotamer of this sort has been isolated and the structure determined (See Cohen et al., 1970).



The increased barrier to rotation around the MeO-phenyl bond, brought about by the increased steric demand of the eclipsed aryl groups is apparently enough to enable isolation of rotamer. In a recent paper by Steger (1985), he studied the steric interaction in E,E-bis(p-methoxy)-benzylidene succinic anhydride by means of variable temperature NMR spectroscopy in deuterated acetone in the temperature range 28 to -90 degrees celsius. The internal rotation of the p-methoxy groups were found to be frozen at -90 degrees celsius. A barrier height of 10.04

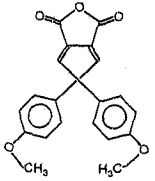
kcal/mol was calculated using the coalescence method.

A further interesting feature of the molecular structure of OR38 is that it is much less strained than that of OR02. This can be seen from the values of the torsion angle C(3-2-2'-3) which is 42 degrees for OR02 and 18 degrees for OR38. It is interesting to note that the methoxy groups in both OR37 and OR38 again lie in the same plane as the benzene rings (within 5 degrees).

7.3.3 Molecular Mechanics of E,E-Bis-(p-methoxybenzylidene) Succinic Anhydride

In view of the experimental evidence of rotameric forms obtained for OR38, it was decided to model this congested system by means of molecular mechanics in an effort to map its conformational transmission, and to look for any other possible rotamers in particular compound C, which was not observed experimentally.

C



The Force Field

The program used was that of Boyd (1968) together with the force field that was developed for 3,4-dimethoxyfulgides in Section 7.2 of this chapter. The relevant parameters are listed in Appendix I. It was decided to perform a two-driver stereodynamical calculation using the improved driver version of Boyd's program (Allenspach, Wasser, Booyens and Donner, 1987). The two torsion angles MeO-

phenyl were driven through 360 degrees in 18 steps of 20 degrees. This method is described in detail in Chapter 5. The starting coordinates were obtained by orthogonalising the fractional atomic coordinates of the crystal structure of OR38.

Results and Discussions

The map of conformational steric energy is shown in Figure 54.

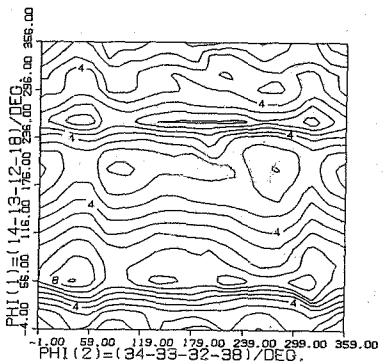


Fig.54. Steric energy surface for R,R-bis(p-methoxy) fulgide. Contour interval is 1 kcal/mol.

The three regions of interest to us on this steric energy surface are the positions for [phi(1), phi(2)] of (0,0), (0,180) or (180,180) degrees respectively. The crystal structure of OR38 represents the position at (0,0) degrees, whereas the published structure by Cohen et al.

(1970) is found at (0,180) or (180,0) degrees. The only rotameric form that was not observed experimentally is the structure at the position of (180,180) degrees. As a matter of fact, the strain energy at this point is very comparable with that found at (0,0) and (180,0) or (0,180) degrees, which would seem to indicate that the (180,180) degrees rotamer could exist. It is quite possible that a mixture of all three of these rotamers exists in solution at a specific temperature. This structure then awaits experimental isolation at the correct conditions.

Stereoscopic drawings of the three rotamers (0,0), (180,0) and (180,180), together with the numbering scheme used, are shown below in Figures 55 to 57. The refined orthogonalised atomic coordinates, bond lengths and angles appear in Appendix I.

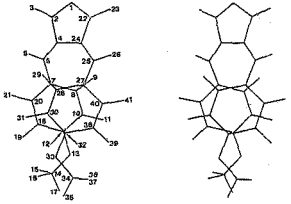


Fig.55. Calculated structure of rotamer (0,0).

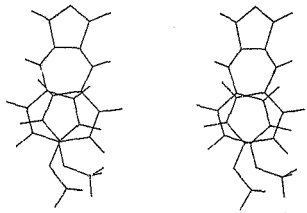


Fig.56. Calculated structure of rotamer (180,0).

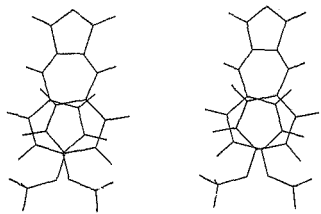


Fig.57. Calculated structure of rotamer (180,180).

In conclusion, the analyses of structures like OR37 and OR38 again re-emphasised the importance of conformational analysis of internal rotations. These surprisingly simple structural models can prove to be a stern test for conformational theories.

CHAPTER 6

EXPERIMENTAL

6.1 Introduction

This chapter deals with the experimental diffraction work as well as a short description of programs used in the processing of the crystallographic data. A table of the crystallographic analysis appears with the discussion of each crystal structure in this thesis. It is however, necessary to elaborate on some of the procedures followed during analyses of the crystal structures.

6.2 Geometric Data Collection

Stable crystals were mounted on the end of glass capillaries using cyanoacrylate glue. The capillary was in turn fixed to a brass holder with wax. The crystals were then aligned with the aid of a goniometer on a Huber Weissenberg camera. A Seifert generator with Cu-K(alpha) radiation and a Ni-filter was used as radiation source. The quality of the crystal was checked by recording oscillation, zero- and first-layer Weissenberg photographs. From these the unit cell parameters as well as some indication of possible space groups could be found.

Unstable crystals were mounted inside Lindemann tubes with silicon grease, after which both ends of the tube were sealed off. Good quality single crystals were only examined with a polarising microscope before being transferred to the diffractometer.

6.3 Intensity Data Collection

All the intensity data were collected on an Enraf Nonius

CAD4 diffractometer by the author, except for the data of OR07 and OR12, which were collected on a Philips PW1100 four-circle diffractometer by Jon Albain, NIMR, CSIR, Pretoria.

The CAD4 data collection routine provides a large number of features which enable the user to configure the data collection process to his needs (See CAD4 operators manual). After alignment, the intensity measurement begins with a pre-scan using a user specified speed. Pre-scan data are used to determine the final scan speed and thus the quality of the final scan data. Given only a starting point the goniometer scans through reciprocal space to find and centre up to 25 reflections. The unit cell parameters were obtained from the indexing of these reflections. This was followed by a trial data collection in the theta-range 16 - 20 degrees. With the aid of good reflections found in these runs, the orientation matrices were refined by a least-squares procedure.

The type of scan used for both pre-scan and final scan were selected after analysis of a reflection shape was completed. This was followed by choosing an omega scan angle as well as aperture size. The scan width for each reflection was calculated as omega = DOMA + 0.34tan theta (DOMA depends on the crystal shape and mosaicity and on the divergence of the beam). The vertical aperture was set at 4.0 mm and the horizontal aperture varied according to the scan width, in the range 1.3 to 5.9 mm. Intensity checks on three standard reflections were performed each hour to monitor crystal deterioration. The orientation of the crystal was monitored with two reflections every 120 reflections, and where necessary a crystal re-orientation was done before the data collection was restarted.

8.4 Data Reduction

Data reduction consisted of correction for background,

Lorentz and polarisation effects, absorption, and a decay correction, if necessary. The corrections were performed on a PDP11/23+ computer using the programs BEGIN, PSI, EAC, STDPLT and DECAY in the Structure Determination Package supplied by Enraf Nonius. A decay correction was only made when the decay was isotropic and larger than 5%. Absorption corrections were determined empirically (North, Philips and Mathews, 1968) from scans of nine suitable reflections at chi 90 degrees, in 37 different orientations around the diffraction vector, 10 degrees apart.

All computations were done on a Control Data, Cyber 760 computer of the National Institute for Informatics, CSIR, Pretoria.

REFERENCES

Abdel-Wahhab S.M. and El-Rayyes N.R. (1971). J. Chem. Soc.(B), 3171.

Adams R. and Yuan H.C. (1933). Chem. Rev., 12, 261.

Aikawa S. and Maruyama Y. (1978). Acta Cryst., B34, 2901.

Allenspach-Wasser C.E., Boeyens J.C.A. and Denner L. (1987). S. Afr. J. Chem., 40, 47.

Allenspach-Wasser C.E., Boeyens J.C.A. and Denner L. (1986). S. Afr. J. Sci., 82, 336.

Allinger N.L. (1977). J. Am. Chem. Soc., 99, 8127.

Allinger N.L. (1976). Adv. Phys. Org. Chem., 13, 1.

Almenningen A., Bastiansen O. and Skancke P.N. (1958). Acta Chem. Scand., 12, 1215.

Altona C. and Faber D.H. (1974). Fortschr. Chem. Forsch., 45, 1.

Anderson J.E. and Hazlehurst C.J. (1980). J.C.S., Chem. Comm., 1186.

Anderson J.E., Franck R.W. and Mandella W.L. (1972). J. Am. Chem. Soc., 94, 4608.

Anderson III G.H., Kollman P.A., Domelsmith L.N. and Houk K.N. (1979). J. Am. Chem. Soc., 101, 2344.

Androse J.D. and Mislow K. (1974). J. Am. Chem. Soc., 96, 2168.

Andreetti G.D., Cavalca L., Domiano P. and Musatti A. (1968). Acta Cryst., B24, 1195.

Andresen O. and Rømming C. (1962). Acta Chem. Scand., 16, 1862.

Baas J.M.A., van de Graaf B., van Rantwijk B. and van Veen A. (1979). Tetrahedron, 35, 421.

Baert F., Lamiot J., Couturier D., Roussel D. and Ricart G. (1984). Acta Cryst., C40, 1071.

Balasubramaniyan V. (1966). Chem. Rev., 66, 567.

Baraldi I. and Ponterini G. (1985). J. Mol. Struct. (Theochem), 122, 287.

Bart J.C.J. (1968). Acta Cryst., B24, 1277.

Bastiansen O. and Samdal S. (1985). J. Mol. Struct., 128, 115.

Beagley B. (1978). Molecular Structure by Diffraction Methods, Vol. 6, p.63, Bartholomew Press, Dorking.

Beintema J. (1976). Acta Cryst., B32, 1631.

Berg U., Sandström, Jennings W.B. and Randall D. (1980). J.C.S., Perkin Trans. II, 949.

Blount J.F., Hunter G. and Mislow K. (1984). J.C.S., Chem. Comm., 170.

Blount J.F., Cozzi F., Damewood J.R. [illegible] off L.D., Sjostrand U. and Mislow K. (1980). J. [illegible] Soc., 102, 99.

Boeyens J.C.A. (1966). Structure and Bonding, 63, 65.

Boeyens J.C.A. (1978). J. Cryst. Mol. Struct., 8, 317.

Boeyens J.C.A., Cotton F.A. and Han S. (1985). Inorg. Chem., 24, 1750.

Boeyens J.C.A., Denner L., Howard A.S. and Michael J.P. (1986). S. Afr. J. Chem., 39, 217.

Boeyens J.C.A., Denner L., Kolodziej H., Ferreira D. and Roux D.G. (1986). J. Chem. Soc. Perkin. Trans. II, 301.

Boeyens J.C.A., Denner L., Marais J.L.C. and Staskun B. (1986). S. Afr. J. Chem., 39(4), 221.

Boeyens J.C.A., Denner L. and Michael J.P. (1986). S. Afr. J. Chem., 39, 233.

Boeyens J.C.A., Denner L. and Michael J.P. (1984). J.C.S., Perkin Trans. II, 767.

Boeyens J.C.A., Denner L., Orchard S.W., Rencken I. and Rose B.G. (1986). S. Afr. J. Chem., 39(4), 229.

Boeyens J.C.A., Denner L., Painter S. and Staskun B. (1987). S. Afr. J. Chem., 40, 60.

Boeyens J.C.A., Denner L. and Perold G.W. (1987a). Submitted for publication.

Boeyens J.C.A., Denner L. and Perold G.W. (1987b). S. Afr. J. Chem., 40, 44.

Boeyens J.C.A., Denner L. and Perold G.W. (1987c). Submitted for publication.

Bondi A. (1964). J. Phys. Chem., 64, 441.

Berkent J.H. and Laarhoven W.H. (1978). Tetrahedron, 34, 2565.

Boshoff P.R. and Perold G.W. (1986a). J. Chem. Res.(S)., 80.

Boshoff P.R. and Perold G.W. (1986b). J. Chem. Res.(M)., 735.

Bourn J.R., Gillies D.G. and Randall E.W. (1964). Tetrahedron, 20, 1811.

Boyd R.H. (1968). J. Chem. Phys., 49, 3574.

Boyd R.H. and Breitling S.M. (1972a). Macromolecules, 5, 1.

Boyd R.H. and Breitling S.M. (1972b). Macromolecules, 5, 279.

Boyd R.H., Sanwal S.N., Shary-Tehrany S. and McNally D. (1971). J. Phys. Chem., 75, 1264.

Brant D.A. and Flory P.J. (1965). J. Am. Chem. Soc., 87, 2788.

Brelliere J.C. and Lehn J.M. (1965). J.C.S., Chem. Comm., 426.

Bright D., Maxwell I.E. and de Boer J. (1973). J.C.S., Perkin Trans. II, 2101.

Brown C.J. (1966). Acta Cryst., 21, 442.

Brown G.M. and Levy H.A. (1979). Acta Cryst., B35, 785.

Brown R.F.C., Radom L., Sternhell S. and Rae I.D. (1968).

Can. J. Chem., 46, 2577.

Brunow G. and Tylli H. (1968). Acta Chem. Scand., 22, 590.

Bryan R.F. and White D.H. (1982). Acta Cryst., B38, 1332.

Bryan R.F. and Freyberg D.P. (1975). J.C.S., Perkin Trans. II, 1835.

Bürgi H.B. and Dunitz J.D. (1983). Acc. Chem. Res., 16, 153.

Bürgi H.B., Hounshell W.D., Nachbar R.B. (Jr.) and Mislow K. (1983). J. Am. Chem. Soc., 105, 1427.

Burkert U. and Allinger N.L. (1982). Molecular Mechanics, ACS Monograph, No. 177.

Burkert U. and Allinger N.L. (1982). J. Comput. Chem., 3, 40.

Bushweller C.H., Fleischman S.H., Grady G.L., McGoff P., Rithner C.D., Whalon M.R., Brennan J.G., Marcantonio R.P. and Domingue R.P. (1982). J. Am. Chem. Soc., 104, 6224.

Carballeira L., Pereiras A.J. and Rios M.A. (1984). J. Chem. Phys., 80, 4997.

Carballeira L., Casado, Gonzalez E. and Rios M.A. (1982). J. Chem. Phys., 77, 5655.

Carter R.E. and Liljefors T. (1976). Tetrahedron, 32, 2915.

Chang S.-J., McNally D., Shary-Tehrany S., Hickey M.J. and Boyd R.H. (1970). J. Am. Chem. Soc., 92, 3109.

Christie G.H. and Kenner J. (1922). J. Chem. Soc., LXXI, 614.

Chupp J.P. and Olin J.F. (1967). J. Org. Chem., 32, 2297.

Clough R.L. and Roberts J.D. (1976). J. Org. Chem., 43(7), 1328.

Clough R.L., Kung W.J., Marsh R.E. and Roberts J.D. (1976). J. Org. Chem., 41(22), 3603.

Clough R.L. and Roberts J.D. (1976). J. Am. Chem. Soc., 98, 1018.

Cohen M.D., Kaufman H.W., Sinnreich D. and Schmidt G.M.J. (1970). J. Chem. Soc. (B), 1032.

Cooke A.S. and Harris M.M. (1967). J. Chem. Soc. (C), 988.

Cossa-Barbi A. and Dubois J.-E. (1986). Tetrahedron Lett., 27(30), 3501.

Cremer D. and Pople J.A. (1975) J. Am. Chem. Soc., 97, 1358.

Crescente O., Heller H.G. and Olivier S. (1979). J.C.S., Perkin Trans. I., 150.

Curtin D.Y., Bender P.E. and Hetzel D.S. (1971). J. Org. Chem., 36(4), 565.

Curtin D.Y. and Dayagi S. (1964). Can. J. Chem., 42, 867.

Dale J. (1966). Tetrahedron, 22, 3373.

Denner L., Marais J.L.C. and Staskun B. (1985). Tetrahedron, 41(23), 5615.

Desiraju G.R., Kamala R., Kumari B. H. and Jagarlapudi A.

R. P. S. (1984). J.C.S., Perkin Trans. II, 181.

Desiraju G.R. and Sarma J.A.R.P. (1983). J.C.S., Chem. Commun., 45.

De Tar D.F. (1977). Comput. Chem., 1, 139.

Domiano P., Predieri G., Lanfranchi M., Tarasconi P. and Palla G. (1986). J.C.S., Perkin Trans. II, 521.

Dunitz J.D. and Bürgi H.B. (1976). MTP Int. Rev. Sci., London, Butterworths, p. 81 ff.

Dunlap B.I. and Cook M. (1986). Int. J. Quant. Chem. 1, 767.

Egli M., Wallis J.D. and Dunitz J.D. (1986). Helv. Chim. Acta, 69, 255.

Engler E.M., Androse J.D. and Schleyer P. von R. (1973). J. Am. Chem. Soc., 95, 8005.

Ermer O. (1983). Angew. Chem. Suppl. 1353.

Ermer O. (1981a). Aspekte von Kraftfeldrechnungen, München, Wolfgang Baur Verlag.

Ermer O. (1981b). Structure and Bonding, 27, 161.

Evrard F.G., Piret P. and Meerssche M.V. (1972). Acta Cryst., B28, 497.

Fatoftah Z.A., Ibrahim M.R. and Al-sa'id N.H. (1985). J. Mol. Struct., 127, 308.

Fields D.L., Regan T.H. and Graves R.E. (1971a). J. Org. Chem., 36, 2995.

Fields D.L. and Regan T.H. (1971b). J. Org. Chem., 36(20), 2986.

Fields D.L. and Regan T.H. (1971c). J. Org. Chem., 36, 2991.

Finocchiaro P., Recca A., Bottino F.A., Bickelhaupt F., van Veen R., Schenk H. and Schagen J.D. (1980). J. Am. Chem. Soc., 102, 5594.

Ford W.T., Thompson T.B., Snoble K.A.J. and Timko J.M. (1975). J. Am. Chem. Soc., 97, 98.

Fraser R.R. and Taymaz K. (1976). Tetrahedron Lett., 4573.

Gafner G. and Herbstein F.H. (1962). Acta Cryst., 15, 1081.

Gilli G., Bertolasi V., Bellucci F. and Ferretti V. (1986). J. Am. Chem. Soc., 108, 2420.

Glaser R., Blount J.F. and Mislow K. (1980). J. Am. Chem. Soc., 102, 2777.

Gutowsky H.S. and Holm C.H. (1956). J. Chem. Phys., 25(6), 1228.

Hagler A.T., Leiserowitz L. and Tuval M. (1976). J. Am. Chem. Soc., 98, 4600.

Haisa M., Kashino S., Matsuzaki Y., Kawai R. and Kunitomi K. (1977). Acta Cryst., B33, 2449.

Haisa M., Kashino S., Kawai R. and Maeda H. (1976). Acta Cryst., B32, 1283.

Haisa M., Kashino S. and Maeda H. (1974). Acta Cryst.,

B30, 2510.

Hamilton W.C. (1965). Acta Cryst., 18, 502.

Hallam H.E. and Jones C.M. (1970). J. Mol. Struct. 5, 1.

Handal J., White J.G., Franck R.W. Yuh Y.H. and Allinger N.L. (1977). J. Am. Chem. Soc., 99, 3345.

Hart R.J. and Heller H.G. (1972). J.C.S., Perkin Trans. I, 1321.

Hayes K.S., Nagumo M., Blount J.F. and Mislow K. (1980). J. Am. Chem. Soc., 102, 2773.

Haywood-Farmer J. and Battiste M.A. (1971). Chem. and Industry, 1232.

Hazell A.C. and Hazell R.G. (1977). Acta Cryst., B33, 1102.

Heller H.G. and Szewczyk M. (1974). J.C.S., Perkin Trans. I, 1483.

Herbstein F.H. (1985). Personal communication.

Herbstein F.H., Kaftory M., Kapon M., and Saenger W. (1981). Z. Kristallogr., 154, 11.

Hirota E. (1985). J. Mol. Struct., 126, 25.

Hirota E., Saito S. and Endo Y. (1979). J. Chem. Phys., 71, 1183.

Hounshell W.D., Johnson C.A., Guenzi A., Cozzi F. and Mislow K. (1980). Proc. Natl. Acad. Sci. USA, 77(12), 6961.

House H.O., Wrabie J. A. and Vanderveer D. (1986). J. Org. Chem., 51, 921.

House H.O., Campbell W.J. and Gall m. (1970). J. Org. Chem., 35(6), 1815.

House H.O. and Baske R.W. (1965). J. Org. Chem., 30, 2942.

Huang R.L. and Lee K.H. (1959). J. Chem. Soc., 923.

Huang R.L. (1954). J. Chem. Soc., 3655.

Hursthouse M.B., Moss G.P. and Sales K.D. (1978). Annu. Rep. Progr. Chem. Soc., B, 75, 23.

Ibuki T., Ozasa S., Fujioka Y. and Mizutani H. (1982). Bull. Chem. Soc. Jpn., 55, 845.

Iroff L.D. (1980). J. Comput. Chem., 1(1), 76.

Isaksson G. and Sandstrom J. (1967). Acta Chem. Scand., 21(6), 1605.

Islam S.A. and Neidle S. (1983). Acta Cryst., B39, 114.

Ito Y., Umehara Y., Nakamura K., Yamada Y. and Matsuura T. (1981). J. Org. Chem., 46(22), 4359.

Ivanov P.M. and Osawa E. (1984). J. Comp. Chem., 5(4), 307.

Ivanov P.M. and Pojarlieff I.G. (1984). J.C.S., Perkin Trans. II, 245.

Iwamura H. (1985). J. Mol. Struct., 126, 401.

Jameson M.B. and Penfold B.R. (1965). J. Chem. Soc., 525.

Jarchow V.O. and Schmalle H.W. (1977). Acta Cryst., B33, 1610.

Jeffrey G.A. and Takagi S. (1978). Acc. Chem. Res., 11, 264.

Jelinski L.W. and Kiefer E.F. (1976). J. Am. Chem. Soc., 98, 281.

Jennings W.Ba. and Saket B.M. (1985). J.C.S., Perkin Trans. II, 1005.

Jones G.I.L. and Owen N.L. (1973). J. Mol. Struct., 18, 1.

Jones P.G. (1984). Acta Cryst., A40, 663.

Jungk A.E. and Schmidt M.J. (1971). Chem. Ber., 104, 3289.

Kai Y., Knochel P., Kwaitkowski S., Dunitz J.D., Oth J.F.M., Seebach D. and Kalinowski H.-O. (1982). Helv. Chim. Acta, 65, 137.

Karle I.L., Flippen-Anderson J.L., Chiang J.F. and Lowrey A.H. (1984). Acta Cryst., B40, 500.

Kawada Y. and Iwamura H. (1983). J. Am. Chem. Soc., 105, 1449.

Kawada Y., Okamoto Y. and Iwamura H. (1983). Tetrahedron Lett., 24(48), 5359.

Kawada Y., Iwamura H. and Okazaki M. (1983). Tetrahedron Lett., 24(8), 791.

Kemp J.D. and Pitzer K.S. (1936). J. Chem. Phys., 8, 410.

Kennard O., Watson D.G., Allen F.J., Isaacs N.W., Motherwell W.D.S., Pettersen R.C. and Town W.G. (1972). Molecular Structure and Dimensions, Vol A1, Crystallographic Data Centre, Cambridge.

Kessler H. (1970). Angew. Chem. Int. Ed., 9(3), 219.

Kessler H. and Rieker A. (1967). Liebigs Ann. Chem., 708, 57.

Kitaigorodsky A.I. (1978). Chem. Soc. Rev., 1, 133.

Korp J.D., Bernal I., Harris M.M. and Patel P.K. (1981). J.C.S., Perkin Trans. II, 1621.

Kroon J., Kanters J.A., Van Duijneveldt-van de Rijdt J.G.C.M. and Vliegenhart J.A. (1975). J. Mol. Struct., 24, 109.

Laarhoven W.H., Peters W.H.M. and Tinnemans A.H.A. (1978). Tetrahedron, 34, 769.

Lawton S.L., Hoh D.M., Johnson R.C. and Knisely A.S. (1977). Inorg. Chem., 12, 1277.

Lawton S.L., McAfee E.R., Benson J.E. and Jacobson R.A. (1973). Inorg. Chem., 12, 2939.

Lawton S.L. and Jacobson R.A. (1968). Inorg. Chem., 7, 2124.

Lipshutz B.H., McCarthy K.E. and Hungate R.W. (1984). J. Org. Chem., 49, 1218/

Lister D.G., Macdonald J.M. and Owen N.L. (1978). Internal Rotation and Inversion, Academic Press, London.

Long D.A. (1985). J. Mol. Struct., 126, 9.

Lowe J.P. (1973). Science, 179, 527.

Lunazzi L., Guerra M., Macciantelli D. and Cerioni G. (1982). J.C.S., Perkin Trans. II, 1527.

Mannschreck A. (1965). Tetrahedron Lett., 19, 1341.

Mannschreck A. (1965). Angew. Chem. Int. Ed., 4(11), 985.

March J. (1977). Advanced Organic Chemistry: Reactions, Mechanisms, and Structure, Second Ed., McGraw-Hill, London.

Martin R.H. (1974). Angew. Chem. Int. Ed., 13(10), 649.

Maverick E., Trueblood K.N. and Bekoe D.A. (1978). Acta Cryst., B34, 2777.

Maverick E., Smith S., Kozerski L., Anet F.A.L. and Trueblood K.N. (1975). Acta Cryst., B31, 805.

Miller A.R. and Curtin D.Y. (1976). J. Am. Chem. Soc., 98, 1860.

Mislow K. (1976). Acc. Chem. Res., 9, 26.

Mitchell R.H. and Yan J.S.-H. (1980). Can. J. Chem., 58, 2584.

Mizushima S. (1954). Structure of Molecules and Internal Rotation, Academic Press, London and New York.

Morino Y. (1985). J. Mol. Struct., 126, 1.

Mühlebach A., Lorenzi G.P. and Gramlich V. (1986). Helv. Chim. Acta, 69, 389.

Müller K. (1980). Angew. Chem. Int. Ed., 19(1), 1.

Nakamura M. and Oki M. (1974). Tetrahedron Lett., 505.

Nicole D., Delpuech J.-J., Wierzbicki M. and Cagniant D. (1980). Tetrahedron, 36, 3233.

Nishinaga T., Itahara T., Matsuura T., Rieker A., Koch D., Albert K. and Hitchcock P. (1978). J. Am. Chem. Soc., 100, 1826.

North A.C.T., Phillips D.C., and Mathews F.S. (1968). Acta Cryst., A24, 351.

Nørskov-Lauritsen L. and Bürgi H.B. (1985). J. Comput. Chem., 6(5), 215.

Nugiel D.A., Biali S.E. and Rappoport Z. (1984). J. Am. Chem. Soc., 106, 3357.

Nyburg S.C. and Faerman C.H. (1986). J. Mol. Struct., 140, 347.

Ogilvie R.A. and Parkes A. (1973). Personal communication to Bright et al, see J.C.S., Perkin Trans. II, 1973, 2101.

Ogilvie R.A. (1971). Thesis, Massachusetts Institute of Technology.

Oki M. (1985). Applications of Dynamic NMR Spectroscopy to Organic Chemistry, VCH Publishers, Inc., Deerfield Beach.

Oki M. (1984). Acc. Chem. Res., 17, 154.

Oki M. (1976). Angew. Chem. Int. Ed., 15(2), 87.

Orville-Thomas W.J. (Ed.) (1974). Internal Rotation in Molecules, Wiley, Chichester and New York.

Osawa E. and Musso H. (1983). Angew. Chem. Int. Ed.,

22(1), 1.

Osawa E. (1982). J. Comput. Chem., 3(3), 400.

Osawa E., Aigami K. and Inamoto Y. (1979). J.C.S., Perkin Trans. II, 172.

Osawa E. (1979). J. Am. Chem. Soc., 101, 5523.

Palla G., Predieri G., Domiano P., Vignali C. and Turner W. (1986). Tetrahedron, 42(13), 3649.

Patel U., Patel T.C. and Singh T.P. (1983). Acta Cryst., C39, 1445.

Pauling L. (1958). Proc. Natl. Acad. Sci., USA, 44, 211.

Payne P.W. and Allen L.C. (1977). Methods of Electronic Structure Theory, Schaefer, III, H.-F. (Ed.), Plenum, New York.

Pedersen B.F. (1967). Acta Chem. Scand., 21, 1415.

Perez S. and Vergelati C. (1984). Acta Cryst., B40, 294.

Pinkus A.G. and Kalyanam V. (1978). J.C.S., Chem. Comm., 201.

Pitzer R.M. (1983). Acc. Chem. Res., 16, 207.

Raghunathan S. and Pattabhi V. (1979). Acta Cryst., B35, 214.

Reddy A.M., Knox K. and Robin M.B. (1964). J. Chem. Phys., 40, 1082.

Robert J.-B., Sherfinski J.S., Marsh R.E. and Roberts J.D. (1974). J. Org. Chem., 39(8), 1152.

Robin M.B., Bovey F.A. and Basch H. (1971). in "The Chemistry of the Amide Group" ed. S. Patai, Interscience, New York.

Roussel C., Liden A., Chanon M., Metzger J. and Sandström J. (1976). J. Am. Chem. Soc., 98, 2847.

Rubiralta M., Feliz M., Jaime C. and Giralt E. (1986). Tetrahedron, 42(14), 3957.

Rüchardt C. and Beckhaus H.-D. (1985). Angew. Chem. Int. Ed., 24(7), 529.

Schenk H. and Benci P. (1972). Acta Cryst., B28, 538.

Scheraga H.A. (1968). Advan. Phys. Org. Chem., 6, 103.

Schmidt J.C., Benson H.D., Macomber R.S., Weiner B. and Zimmer H. (1977). J. Org. Chem., 42(11), 2003.

Schneider H.-J., Gschwendtner W. and Weigand E.F. (1979). J. Am. Chem. Soc., 101, 7195.

Scott R.A. and Scheraga H.A. (1965). J. Chem. Phys., 42(6), 2209.

Sheldrick G.M. (1985). Personal communication.

Sheldrick G.M. (1978). in "Computing in Crystallography", H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld and G.C. Bassi (eds.) Delft University Press.

Shieh C.-F., McNally D. and Boyd R.H. (1969). Tetrahedron, 25, 3653.

Shvo Y., Taylor E.C., Mislow K. and Raban M. (1967). J. Am. Chem. Soc., 89, 4910.

Siddall, III, T.H. and Stewart W.E. (1969). J. Org. Chem., 34, 233.

Siddall, III, T.H., Stewart W.E. and Marston A.L. (1968). J. Phys. Chem., 72(6), 2135.

Siddall, III, T.H. (1966). J. Org. Chem., 31, 3719.

Siddall, III, T.H. and Prohaska C.A. (1966). J. Am. Chem. Soc., 88, 1172.

Siegel J., Gutierrez A., Schweizer W.B., Ermer O. and Mislow K. (1986). J. Am. Chem. Soc., 108, 1569.

Siegel J. and Mislow K. (1983). J. Am. Chem. Soc., 105, 7763.

Simonetta M. (1974). Acc. Chem. Res., 7, 345.

Smith D.L. and Barett E.K. (1971). Acta Cryst., B27, 419.

Stam C.H. and Evers H. (1965). Recl. Trav. Chim. Pays-Bas, 84, 1496.

Staskun B. (1981). J. Org. Chem., 46, 1643.

E. Steger, (1985), Z. Chem., 25, 144.

Stewart W.E. and Siddall, III, T.H. (1970). Chem. Rev., 70(5), 517.

Stobbe H. and Leuner K. (1911). Liebigs Ann. Chem., 380, 75.

Stobbe H. (1905). Ber., 38, 3673.

Subramanian E. (1966). Z. Kristallographie, 123.

Swoboda G.A., Wang K.T. and Weinstein B. (1967). J. Soc. (C), 161.

Taylor R. and Kennard O. (1983). Acta Cryst., B39, 133.

Taylor R., Kennard O. and Versichel W. (1984). Acta Cryst., B40, 280.

Taylor R., Kennard and Versichel W. (1983). J. Am. Chem. Soc., 105, 5761.

Tinnemans A.H.A. and Laarhoven W.H. (1979). Tetrahedron, 35, 1537.

Titherley A.W. and Spencer J.F. (1904). J. Chem. Soc., 85, 183.

Van de Graaf B., Baas J.M.A. and van Veen A. (1980). Recl. Trav. Chim. Pays-Bas, 99, 175.

Wahl P., Krieger C., Schweitzer D. and Staab H.A. (1984). Chem. Ber., 117, 260.

Walter W. and Imbert J.P. (1975). J. Mol. Struct., 29, 253.

Walter W. and Becker R.F. (1971). Liebigs Ann. Chem., 753, 187.

Walter W. and Schauman E. (1971). Chem. Ber., 104, 3361.

Wepster B.M. (1956). Prog. Stereochem., 2, 99.

Westheimer F.H. (1956). in M.S. Newman (Ed.), Steric effects in Organic Chemistry, S.524, Wiley, New York.

White D.N.J. (1978). in "Molecular Structure by

Diffraction Methods", Vol. 6, p.38, Bartholomew Press, Dorking.

White D.N.J. and Guy M.H.P. (1975). J.C.S., Perkin Trans. II, 43.

Wiberg K.B. and Boyd R.H. (1972). J. Am. Chem. Soc., 94, 8426.

Williams D.E. (1981). in R.M. Metzger (Ed.), Topics in Current Physics, 26, 3.

Williams J.E., Stang P.J. and Schleyer Paal vonR. (1968). Annu. Rev. Phys. Chem., 19, 531.

Willem R., Pepermans H., Hoogzand F., Hallenga K. and Gielen M. (1981). J. Am. Chem. Soc., 103, 2297.

Wilson E.B. (1972). Chem. Soc. Rev., 1, 293.

Wilson (Jr.) E.B. (1957). Proc. Natl. Acad. Sci., USA, 43, 816.

Winkler F.K. and Dunitz J.D. (1971). J. Mol. Biol., 59, 169.

Yamagami C., Takao N. and Takeuchi Y. (1986). Aust. J. Chem., 39, 457.

Yamamoto G. and Oki M. (1986). Tetrahedron Lett., 27(1), 49.

Yamamoto G. and Oki M. (1981). Bull. Chem. Soc. Jpn., 54, 481.

Yamamoto G., Tanaka Y. and Oki M. (1983). Bull. Chem. Soc. Jpn., 56, 3028.

Yang Q.-C., Richardson M.F. and Dunitz J.D. (1985). J. Am. Chem. Soc., 107, 5535.

Yoder C.H. and Gardner R.D. (1981). J. Org. Chem., 46, 64.

Yoon K., Jeuteschko P. and Parthé I. (1977). J. Appl. Cryst., 10, 73.

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APPENDIX I

Force Constants

Force constants used were taken from the literature (Boeyens, Cotton and Han, 1965; Allenspach-Wasser, Boeyens and Denner, 1987; Allenspach-Wasser, Boeyens and Denner, 1986; Boyd et al., 1971 and Boyd, 1968). The additional force constants used for the fulgides in Chapter 7 are listed below:

Details of the force field: force constants, k/mdyne/angstrom or k/mdyne/radians and characteristic, strain-free parameters for bond (r_e/angstrom) or angle (theta/degrees) distortions.

Bond or angle	k	Strain-free bond length or angle
C-O(furyl)	4.40	1.39
C=O	7.65	1.19
C(1)-C(2)	4.40	1.47
C(3)-C(4)	4.40	1.45
C(2)=C(3)	6.85	1.35
C-C(aromatic)	7.65	1.40
C-O(Me)	4.40	1.35
O-C(Me)	4.40	1.43
H-C	5.00	1.08
H-C(sp^3)-R'	0.62	109.5
H-C(sp^2)-R'	0.62	120
C-O-C	0.80	109.5
C-O-C(Me)	0.80	120
C-C-C(aromatic)	1.00	120
C(2)-C(2')=C(3)	0.62	130
C(1)-C(2)=C(3)	0.62	116
C(2)=C(3)-C(4)	0.62	128

Simulated Structures

The calculated orthogonalised atomic coordinates and bon lengths and angles of the simulated structures in Chapters 6 and 7 are listed below:

Coordinates for 1,8-diphenylnaphthalene.

Atom	x	y	z
1	.00000	2.02077	1.37451
2	-.00030	2.69436	2.64510
3	-.09967	4.01216	2.74901
4	-.19201	4.77701	1.59664
5	-.14601	4.18600	.33826
6	-.07495	2.79072	.21113
7	-.07778	2.12996	-1.02577
8	.05524	.74672	-1.08072
9	.11378	-.00663	.08105
10	.08361	.63384	1.30930
11	-.40906	5.14785	-1.78340
12	-1.71080	5.34289	-1.23163
13	-1.96470	6.24611	-2.25738
14	-.91693	6.95402	-2.83516
15	.38443	6.76036	-2.38614
16	.63932	5.85772	-1.35990
17	-2.53179	4.78581	-.77892
18	-2.96543	6.39692	-2.61967
19	-1.11559	7.65957	-3.64278
20	1.20677	7.31949	-2.84133
21	1.68868	5.70545	-1.00701
22	.02372	2.79528	-2.92845
23	-1.11566	2.97827	-3.14409
24	-1.01662	3.57863	-4.39391
25	.22190	3.99495	-4.86873
26	1.36166	3.81000	-4.09443
27	1.26259	3.21034	-2.84422
28	2.08584	2.68123	-2.77110
29	-1.91116	3.72631	-5.00006
30	.29900	4.46924	-5.84783
31	2.33307	4.13800	-4.46626
32	2.15581	3.06637	-2.22533
33	.06362	2.03018	3.55095
34	-.12370	4.48948	3.72939
35	-.30847	5.85623	1.68164
36	.11566	.24569	-2.04775
37	.20142	-1.09252	.02875
38	.14013	.04147	2.22341

Bond lengths for 1,8-diphenylnaphthalene in angstrom.

I= 1	J= 2	RIJ=1.41099	I=24	J=29	RIJ=1.09062
I= 2	J= 3	RIJ=1.38528	I=25	J=30	RIJ=1.09065
I= 3	J= 4	RIJ=1.38617	I=26	J=31	RIJ=1.09063
I= 4	J= 5	RIJ=1.39123	I=27	J=32	RIJ=1.09057
I= 5	J= 6	RIJ=1.40257	I= 2	J=33	RIJ=1.09073
I= 6	J= 7	RIJ=1.40233	I= 3	J=34	RIJ=1.09067
I= 7	J= 8	RIJ=1.39071	I= 4	J=35	RIJ=1.09079
I= 8	J= 9	RIJ=1.38568	I= 8	J=36	RIJ=1.09079
I= 9	J=10	RIJ=1.38554	I= 9	J=37	RIJ=1.09068
I= 1	J= 6	RIJ=1.39710	I=10	J=38	RIJ=1.09073
I= 5	J=11	RIJ=1.50190	I=10	J= 1	RIJ=1.39097
I=11	J=12	RIJ=1.39046	I=15	J=20	RIJ=1.09062
I=12	J=13	RIJ=1.39011	I=16	J=21	RIJ=1.09053
I=13	J=14	RIJ=1.39025	I= 7	J=22	RIJ=1.50191
I=14	J=15	RIJ=1.39021	I=22	J=23	RIJ=1.39043
I=15	J=16	RIJ=1.39006	I=23	J=24	RIJ=1.39007
I=16	J=11	RIJ=1.39043	I=24	J=25	RIJ=1.39022
I=12	J=17	RIJ=1.09057	I=25	J=26	RIJ=1.39026
I=13	J=18	RIJ=1.09062	I=26	J=27	RIJ=1.39012
I=14	J=19	RIJ=1.09065	I=27	J=22	RIJ=1.39048
I=33	J=26	RIJ=1.09064			

Coordinates for 1,8-diphenylnaphthalene.

Atom	x	y	z
1	.00000	2.02077	1.37451
2	-.00030	2.63496	2.64510
3	-.09967	4.01216	2.74901
4	-.19201	4.77701	1.59664
5	-.14001	4.16600	.33826
6	-.07495	2.79072	.21113
7	-.07778	2.12996	-1.02577
8	.05524	.74672	-1.08072
9	.11376	-.00663	.08105
10	.08361	.63384	1.30930
11	-.40908	9.14785	-.76940
12	-1.71080	5.34289	-1.23163
13	-1.96470	6.24611	-2.25798
14	-.91693	6.95402	-2.83516
15	.38445	6.76036	-2.38614
16	.63832	5.85772	-1.35990
17	-2.53179	4.78881	-.77892
18	-2.98543	6.39692	-2.61967
19	-1.11559	7.65957	-3.64278
20	1.20677	7.91348	-2.84193
21	1.65888	5.70545	-1.00701
22	.02372	2.79528	-2.66465
23	-1.11566	2.97827	-3.14409
24	-1.01662	3.57869	-4.39991
25	.22100	3.99495	-4.86873
26	1.36166	3.81000	-4.09443
27	1.26259	3.21034	-2.84422
28	-2.09684	2.68123	-4.77110
29	-1.91116	3.72631	-5.00006
30	.29900	4.46924	-5.84783
31	2.33307	4.13800	-4.46625
32	2.15581	3.08467	-2.29531
33	.06362	2.03018	3.55095
34	-.12370	4.48948	3.72939
35	-.30847	5.85823	1.68164
36	.11566	.24569	-2.04775
37	.20142	-1.09252	.02675
38	.14013	.04147	2.22341

Bond lengths for 1,8-diphenylnaphthalene in angstrom.

I= 1	J= 2	RIJ=1.41099	I=24	J=29	RIJ=1.09062
I= 2	J= 3	RIJ=1.38528	I=25	J=30	RIJ=1.09065
I= 3	J= 4	RIJ=1.38617	I=26	J=31	RIJ=1.09063
I= 4	J= 5	RIJ=1.39123	I=27	J=32	RIJ=1.09057
I= 5	J= 6	RIJ=1.40257	I= 2	J=33	RIJ=1.09073
I= 6	J= 7	RIJ=1.40233	I= 3	J=34	RIJ=1.09067
I= 7	J= 8	RIJ=1.39071	I= 4	J=35	RIJ=1.09079
I= 8	J= 9	RIJ=1.38588	I= 8	J=36	RIJ=1.09079
I= 9	J=10	RIJ=1.38554	I= 9	J=37	RIJ=1.09068
I= 1	J= 6	RIJ=1.39710	I=10	J=38	RIJ=1.09073
I= 5	J=11	RIJ=1.50190	I=10	J= 1	RIJ=1.39097
I=11	J=12	RIJ=1.39048	I=15	J=20	RIJ=1.09062
I=12	J=13	RIJ=1.39011	I=16	J=21	RIJ=1.09058
I=13	J=14	RIJ=1.39025	I= 7	J=22	RIJ=1.50191
I=14	J=15	RIJ=1.39021	I=22	J=23	RIJ=1.39043
I=15	J=16	RIJ=1.39006	I=23	J=24	RIJ=1.39007
I=16	J=11	RIJ=1.39043	I=24	J=25	RIJ=1.39022
I=12	J=17	RIJ=1.09057	I=25	J=26	RIJ=1.39026
I=13	J=18	RIJ=1.09062	I=26	J=27	RIJ=1.39012
I=14	J=19	RIJ=1.09065	I=27	J=22	RIJ=1.39048
I=23	J=28	RIJ=1.09054			

Bond angles for 1,8-diphenylnaphthalene in degrees.

I=	4	J=	5	K=11	A=	13.376	I=11	J=12	K=13	A=120.000
I=	4	J=	5	K=11	A=	13.290	I=11	J=12	K=17	A=119.953
I=	6	J=	6	K= 7	A=	.271	I=13	J=12	K=17	A=120.047
I=	6	J=	7	K=22	A=128.426		I=12	J=13	K=14	A=119.990
I=	7	J=	7	K=22	A=	13.604	I=12	J=13	K=18	A=119.986
I=	2	J=	1	K= 6	A=120.676		I=14	J=13	K=18	A=120.021
I=	2	J=	1	K=10	A=119.413		I=13	J=14	K=15	A=120.020
I=	6	J=	1	K=10	A=120.910		I=14	J=14	K=19	A=119.992
I=	1	J=	2	K= 3	A=120.005		I=15	J=14	K=19	A=119.988
I=	1	J=	2	K=33	A=120.462		I=14	J=15	K=16	A=119.993
I=	3	J=	2	K=33	A=119.528		I=14	J=15	K=20	A=120.022
I=	2	J=	3	K= 4	A=119.420		I=16	J=15	K=20	A=119.984
I=	2	J=	3	K=34	A=120.284		I=15	J=16	K=11	A=120.001
I=	4	J=	3	K=34	A=120.286		I=15	J=16	K=21	A=120.032
I=	3	J=	4	K= 5	A=121.002		I=11	J=16	K=21	A=119.967
I=	3	J=	4	K=35	A=119.292		I= 7	J=22	K=23	A=120.107
I=	5	J=	4	K=35	A=119.707		I= 7	J=22	K=27	A=119.890
I=	4	J=	5	K= 6	A=120.419		I=23	J=22	K=27	A=119.996
I=	5	J=	6	K= 1	A=118.375		I=22	J=23	K=24	A=120.001
I=	7	J=	6	K= 1	A=118.354		I=22	J=23	K=28	A=119.967
I=	6	J=	7	K=22	A=120.219		I=24	J=23	K=28	A=120.033
I=	7	J=	8	K= 9	A=120.770		I=23	J=24	K=25	A=119.993
I=	7	J=	8	K=36	A=119.814		I=23	J=24	K=29	A=119.984
I=	9	J=	8	K=36	A=119.416		I=25	J=24	K=29	A=120.023
I=	8	J=	9	K=10	A=119.402		I=24	J=25	K=26	A=120.020
I=	8	J=	9	K=37	A=120.291		I=24	J=25	K=30	A=119.988
I=10	J=	9	K=37	A=120.297		I=26	J=25	K=30	A=119.992
I=	9	J=10	K= 1	A=120.250		I=25	J=26	K=27	A=119.990
I=	9	J=10	K=38	A=119.390		I=25	J=26	K=31	A=120.021
I=	1	J=10	K=38	A=120.354		I=27	J=26	K=31	A=119.989
I=	5	J=11	K=16	A=119.887		I=26	J=27	K=22	A=119.999
I=	5	J=11	K=16	A=120.110		I=26	J=27	K=32	A=120.048
I=12	J=11	K=16	A=119.996		I=22	J=27	K=32	A=119.953

Coordinates for 1,8-di(m-methylphenyl)naphthalene.

Atom	x	y	z
1	.00000	2.02077	1.37451
2	-.00140	2.63436	2.64510
3	-.17222	4.00498	2.74901
4	-.33471	4.76111	1.59882
5	-.26651	4.17864	.33873
6	-.13723	2.76429	.21254
7	-.13204	2.12329	-1.02476
8	.08180	.78019	-1.08066
9	.19297	.00080	.07973
10	.14990	.63956	1.30825
11	-.67501	5.09364	-.77769
12	-2.00269	5.13379	-1.18785
13	-2.39255	5.99082	-2.21206
14	-1.44826	6.81201	-3.22120
15	-.12108	6.77419	-3.18093
16	.26210	5.91529	-2.15727
17	-2.73765	4.49090	-.70159
18	-1.77419	7.68542	-4.23416
19	.61788	7.41592	-2.99020
20	1.39732	5.98471	-1.06445
21	-.02688	2.81080	-2.35590
22	-1.14886	2.94158	-3.16550
23	-1.04736	3.56903	-4.40341
24	.16330	4.05811	-4.83000
25	1.30998	3.92281	-4.04228
26	1.20238	3.30005	-2.78494
27	-2.10690	2.54976	-2.62246
28	.26910	4.55240	-5.77839
29	2.27020	4.30788	-4.35962
30	2.08238	3.19663	-2.14914
31	-3.83019	6.01991	-2.66570
32	-4.43740	6.55849	-1.92735
33	-4.21916	5.00542	-2.73015
34	-5.93091	6.61362	-3.62257
35	-2.25939	3.73017	-5.29050
36	-3.13357	3.25468	-4.83398

37	-2.0w373	3.27546	-6.25610
38	-2.46051	4.78856	-5.42463
39	.11367	2.03676	3.64961
40	-.20473	4.48184	3.72932
41	-.52235	5.83197	1.66690
42	.16650	.25425	-2.04836
43	.33895	-1.07869	.02567
44	.25268	.05167	2.22122

Bond lengths for 1,8-di(m-methylphenyl)naphthalene in angstrom.

I= 1	J= 2	RIJ=1.41099	I=23	J=24	RIJ=1.39130
I= 1	J= 3	RIJ=1.38613	I=24	J=25	RIJ=1.39014
I= 3	J= 4	RIJ=1.38603	I=25	J=26	RIJ=1.38967
I= 4	J= 5	RIJ=1.39114	I=26	J=21	RIJ=1.39017
I= 5	J= 6	RIJ=1.40301	I=22	J=27	RIJ=1.09041
I= 6	J= 7	RIJ=1.40283	I=24	J=28	RIJ=1.09062
I= 7	J= 8	RIJ=1.39072	I=25	J=29	RIJ=1.09063
I= 8	J= 9	RIJ=1.38575	I=26	J=30	RIJ=1.09056
I= 9	J=10	RIJ=1.39533	I=13	J=31	RIJ=1.50482
I= 1	J= 6	RIJ=1.39713	I=31	J=32	RIJ=1.09053
I=10	J= 1	RIJ=1.39090	I=31	J=33	RIJ=1.09061
I= 5	J=11	RIJ=1.50205	I=31	J=34	RIJ=1.09038
I=11	J=12	RIJ=1.39037	I=23	J=35	RIJ=1.50478
I=12	J=13	RIJ=1.39116	I=35	J=36	RIJ=1.09071
I=13	J=14	RIJ=1.39179	I=35	J=37	RIJ=1.09076
I=14	J=15	RIJ=1.39026	I=35	J=38	RIJ=1.09085
I=15	J=16	RIJ=1.38975	I= 2	J=39	RIJ=1.09074
I=16	J=11	RIJ=1.39010	I= 3	J=40	RIJ=1.09063
I=12	J=17	RIJ=1.09057	I= 4	J=41	RIJ=1.09074
I=14	J=18	RIJ=1.09055	I= 8	J=42	RIJ=1.09072
I=15	J=19	RIJ=1.09063	I= 9	J=43	RIJ=1.09066
I=16	J=20	RIJ=1.09054	I=10	J=44	RIJ=1.09073
I= 7	J=21	RIJ=1.50197	I=21	J=22	RIJ=1.39041
I=22	J=23	RIJ=1.39154			

Bond angles for 1,8-di(m-methylphenyl)naphthalene in degrees.

I= 2	J= 1	K= 6	A=120.743	I=11	J=12	K=17	A=119.669
I= 2	J= 1	K=10	A=118.349	I=13	J=12	K=17	A=120.166
I= 6	J= 1	K=10	A=120.907	I=12	J=13	K=14	A=119.774
I= 1	J= 2	K= 3	A=119.867	I=12	J=13	K=31	A=119.772
I= 1	J= 2	K=39	A=120.530	I=14	J=13	K=31	A=120.514
I= 3	J= 2	K=39	A=119.594	I=13	J=14	K=15	A=120.123
I= 2	J= 3	K= 4	A=119.474	I=13	J=14	K=18	A=120.314
I= 2	J= 3	K=40	A=120.249	I=15	J=14	K=18	A=119.564
I= 4	J= 3	K=40	A=120.286	I=14	J=15	K=19	A=120.051
I= 3	J= 4	K= 5	A=121.196	I=14	J=15	K=19	A=119.996
I= 3	J= 4	K=41	A=119.258	I=16	J=15	K=19	A=119.953
I= 5	J= 4	K=41	A=119.546	I=15	J=16	K=11	A=119.938
I= 4	J= 5	K= 6	A=120.222	I=15	J=16	K=20	A=120.052
I= 4	J= 5	K=11	A=113.744	I=11	J=16	K=20	A=120.000
I= 6	J= 5	K=11	A=124.351	I= 7	J=21	K=22	A=120.142
I= 1	J= 6	K= 7	A=123.161	I= 7	J=21	K=26	A=119.800
I= 5	J= 6	K= 1	A=116.436	I=22	J=21	K=26	A=120.052
I= 7	J= 6	K= 1	A=116.402	I=21	J=22	K=23	A=120.099
I= 6	J= 7	K= 8	A=120.281	I=21	J=22	K=27	A=119.506
I= 6	J= 7	K=21	A=124.486	I=23	J=22	K=27	A=120.395
I= 8	J= 7	K=21	A=113.924	I=22	J=23	K=24	A=119.722
I= 7	J= 8	K= 9	A=120.938	I=22	J=23	K=35	A=120.534
I= 7	J= 8	K=42	A=119.767	I=24	J=23	K=35	A=119.742
I= 9	J= 8	K=42	A=119.294	I=23	J=24	K=25	A=120.152
I= 8	J= 9	K=10	A=120.387	I=23	J=24	K=28	A=120.077
I= 8	J= 9	K=43	A=120.297	I=25	J=24	K=28	A=119.771
I=10	J= 9	K=43	A=120.297	I=24	J=25	K=26	A=119.999
I= 9	J=10	K= 1	A=120.230	I=24	J=25	K=29	A=120.016
I= 9	J=10	K=44	A=119.395	I=26	J=25	K=29	A=119.985
I= 1	J=10	K=44	A=120.366	I=25	J=26	K=21	A=119.944
I= 5	J=11	K=12	A=119.787	I=25	J=26	K=30	A=120.072
I= 5	J=11	K=16	A=120.196	I=21	J=26	K=30	A=119.984
I=12	J=11	K=16	A=120.010	I=13	J=31	K=32	A=110.157
I=11	J=12	K=13	A=120.164	I=13	J=31	K=33	A=110.382
I=13	J=31	K=34	A=111.003	I=23	J=35	K=37	A=110.258
I=32	J=31	K=33	A=108.582	I=23	J=35	K=38	A=110.138

I=32	J=31	K=34	A=108.487	I=36	J=35	K=37	A=108.315
I=33	J=31	K=34	A=108.156	I=36	J=35	K=38	A=108.342
I=23	J=35	K=36	A=111.116	I=37	J=35	K=38	A=108.600

Coordinates for 1,8-di-tolylnaphthalene.

Atom	x	y	z
1	.00000	2. 977	1.37451
2	.00171	2.47736	2.64510
3	.13317	4.09909	2.74901
4	.26560	4.73995	1.59779
5	.31909	4.17008	.34323
6	.15840	2.78149	.21319
7	.14970	2.11767	-1.02369
8	.04632	.73153	-1.07140
9	-.12848	-.00879	.06659
10	-.15185	.63974	1.31025
11	.28504	5.15195	-.79683
12	-.92488	5.58499	-1.94083
13	-.91717	6.50320	-2.39731
14	.28548	6.98945	-2.88635
15	1.48832	6.56226	-2.93849
16	1.49628	5.64631	-1.29403
17	-1.85596	6.84778	-2.52239
18	.28504	7.70428	-3.71011
19	2.43289	6.94155	-2.73021
20	2.43080	5.32054	-.85747
21	.80713	2.74546	-2.34385
22	1.83867	2.91728	-3.72439
23	2.13105	3.49127	-3.95859
24	1.10598	3.88812	-4.80919
25	-.21867	3.70958	-4.43117
26	-.51594	3.13717	-3.20081
27	3.16759	3.63284	-4.26659
28	1.34150	4.34241	-5.77234
29	-1.02412	4.02211	-5.09679
30	-1.55610	2.98733	-2.91025
31	-2.24429	5.08703	-.80592
32	-2.19000	4.02453	-.86962
33	-3.04015	5.22887	-1.69717
34	-2.50817	5.62917	.10224
35	2.96963	2.47880	-1.82801
36	3.12554	1.40397	-1.92140
37	2.74737	2.70550	-.78550
38	3.89660	2.98726	-2.09270
39	-.11419	2.93531	3.36211
40	.11039	4.49147	3.72692
41	.32335	5.85519	1.67967
42	.10822	.21715	-2.03129
43	-.22433	-1.09411	.03680
44	-.27600	.05568	2.22303

Bond lengths for 1,8-di-tolylnaphthalene in angstrom.

I= 1	J= 2	RIJ=1.41099	I=23	J=24	RIJ=1.38989
I= 2	J= 3	RIJ=1.38491	I=24	J=25	RIJ=1.38904
I= 3	J= 4	RIJ=1.38576	I=25	J=26	RIJ=1.38918
I= 4	J= 5	RIJ=1.39124	I=26	J=21	RIJ=1.39086
I= 5	J= 6	RIJ=1.41490	I=23	J=27	RIJ=1.09057
I= 6	J= 7	RIJ=1.40369	I=24	J=28	RIJ=1.09065
I= 7	J= 8	RIJ=1.39781	I=25	J=29	RIJ=1.09063
I= 8	J= 9	RIJ=1.38548	I=26	J=30	RIJ=1.09032
I= 9	J=10	RIJ=1.38510	I=19	J=31	RIJ=1.50829
I= 1	J= 6	RIJ=1.39730	I=31	J=32	RIJ=1.08981
I=10	J= 1	RIJ=1.39084	I=31	J=33	RIJ=1.09006
I= 5	J=11	RIJ=1.50497	I=31	J=34	RIJ=1.09009
I=11	J=12	RIJ=1.39549	I=22	J=35	RIJ=1.50826
I=12	J=13	RIJ=1.39222	I=36	J=35	RIJ=1.09009
I=13	J=14	RIJ=1.38992	I=36	J=37	RIJ=1.08978
I=14	J=15	RIJ=1.38905	I=36	J=38	RIJ=1.09006
I=15	J=16	RIJ=1.38920	I= 2	J=39	RIJ=1.09074
I=16	J=11	RIJ=1.39089	I= 3	J=40	RIJ=1.09065
I=13	J=17	RIJ=1.09057	I= 4	J=41	RIJ=1.09075
I=14	J=18	RIJ=1.09067	I= 8	J=42	RIJ=1.09073
I=15	J=19	RIJ=1.09065	I= 9	J=43	RIJ=1.09068

I=16 J=20 RIJ=1.09033
I= 7 J=21 RIJ=1.50498
I=22 J=23 RIJ=1.39219

I=10 J=44 RIJ=1.09074
I=21 J=22 RIJ=1.39547

Bond angles for 1,8-di-tolylnaphthalene in degrees.

I= 2	J= 1	K= 6	A=120.766	I=15	J=14	K=18	A=119.987
I= 2	J= 1	K=10	A=118.264	I=14	J=15	K=16	A=119.871
I= 6	J= 1	K=10	A=120.968	I=14	J=15	K=19	A=120.089
I= 1	J= 2	K= 3	A=119.987	I=16	J=15	K=19	A=120.040
I= 1	J= 2	K=39	A=120.494	I=15	J=16	K=11	A=120.290
I= 3	J= 2	K=39	A=119.543	I=15	J=16	K=20	A=119.786
I= 2	J= 3	K= 4	A=119.414	I=11	J=16	K=20	A=119.923
I= 2	J= 3	K=40	A=120.286	I= 7	J=21	K=22	A=121.145
I= 4	J= 3	K=40	A=120.206	I= 7	J=21	K=26	A=118.901
I= 3	J= 4	K= 5	A=121.111	I=22	J=21	K=26	A=119.945
I= 3	J= 4	K=41	A=119.260	I=21	J=22	K=23	A=119.592
I= 5	J= 4	K=41	A=119.628	I=21	J=22	K=35	A=121.173
I= 4	J= 5	K= 6	A=120.329	I=23	J=22	K=35	A=119.292
I= 4	J= 5	K=11	A=113.663	I=22	J=23	K=24	A=120.386
I= 6	J= 5	K=11	A=124.928	I=22	J=23	K=27	A=120.232
I= 5	J= 6	K= 7	A=123.372	I=24	J=23	K=27	A=119.412
I= 5	J= 6	K= 1	A=118.325	I=23	J=24	K=25	A=120.006
I= 7	J= 6	K= 1	A=118.303	I=23	J=24	K=28	A=120.007
I= 6	J= 7	K= 8	A=120.197	I=25	J=24	K=28	A=119.988
I= 6	J= 7	K=21	A=125.044	I=24	J=25	K=26	A=119.671
I= 8	J= 7	K=21	A=113.780	I=24	J=25	K=29	A=120.089
I= 7	J= 8	K= 9	A=120.677	I=26	J=25	K=29	A=120.039
I= 7	J= 8	K=42	A=119.739	I=25	J=26	K=21	A=120.286
I= 9	J= 8	K=42	A=119.584	I=25	J=26	K=30	A=119.787
I= 8	J= 9	K=10	A=119.362	I=21	J=26	K=30	A=119.923
I= 8	J= 9	K=43	A=120.306	I=12	J=31	K=32	A=110.605
I=10	J= 9	K=43	A=120.316	I=12	J=31	K=33	A=110.965
I= 9	J=10	K= 1	A=120.268	I=32	J=31	K=33	A=110.961
I= 9	J=10	K=44	A=119.374	I=32	J=31	K=34	A=107.980
I= 1	J=10	K=44	A=120.362	I=33	J=31	K=34	A=108.439
I= 5	J=11	K=16	A=121.156	I=23	J=21	K=34	A=108.507
I= 5	J=11	K=16	A=118.091	I=22	J=35	K=36	A=110.058
I=12	J=11	K=16	A=119.944	I=22	J=35	K=37	A=110.801
I=11	J=12	K=13	A=119.630	I=22	J=35	K=38	A=110.970
I=11	J=12	K=31	A=121.181	I=36	J=35	K=37	A=108.436
I=13	J=12	K=31	A=119.285	I=36	J=35	K=38	A=106.510
I=12	J=13	K=14	A=120.568	I=37	J=35	K=38	A=107.983
I=12	J=13	K=31	A=120.831	I=14	J=13	K=17	A=119.411
I=13	J=14	K=15	A=120.005	I=13	J=14	K=18	A=120.006

Coordinates for 1-tolylnaphthalene.

Atom	x	y	z
1	.00000	2.02077	1.31451
2	-.00003	2.69436	2.64510
3	-.15654	4.01103	2.74901
4	-.31580	4.77941	1.60228
5	-.32021	4.17547	.36000
6	-.14898	2.79933	.25142
7	.15145	2.19391	-1.02225
8	.00569	.81744	-1.13235
9	.16494	.04209	-.00969
10	.16220	.64339	1.26260
11	-.48386	5.02940	-.87583
12	-1.73941	5.26900	-1.49515
13	-1.84250	6.06631	-2.57116
14	-.70514	6.62079	-3.14647
15	.54364	6.38076	-2.58700
16	.65246	5.58593	-1.45267
17	-2.81990	6.25963	-3.01523
18	-.79300	7.24425	-4.09713
19	1.43696	6.81525	-3.03737
20	1.63276	5.39679	-1.01362
21	-2.99782	4.68555	-.84360
22	-2.77814	4.05206	.01520
23	-3.51779	4.08079	-1.58667
24	-3.66477	5.48352	-.51667
25	.12207	2.09448	3.54779
26	-.15591	4.46809	3.72964

27	-.44062	5.85973	1.68453
28	-.27702	2.78797	-1.12787
29	.00397	.34560	-2.11572
30	.28977	-1.03799	-.07705
31	.28532	.02912	2.15543

Bond lengths for 1-tolylnaphthalene in angstrom.

I= 1	J= 2	RIJ=1.41099	I=16	J=11	RIJ=1.39057
I= 2	J= 3	RIJ=1.38943	I=13	J=17	RIJ=1.09072
I= 3	J= 4	RIJ=1.38952	I=14	J=18	RIJ=1.09073
I= 4	J= 5	RIJ=1.39031	I=15	J=19	RIJ=1.09071
I= 5	J= 6	RIJ=1.39226	I=16	J=20	RIJ=1.09065
I= 6	J= 7	RIJ=1.39221	I=12	J=21	RIJ=1.50795
I= 7	J= 8	RIJ=1.38978	I=21	J=22	RIJ=1.08955
I= 8	J= 9	RIJ=1.38952	I=21	J=23	RIJ=1.09021
I= 9	J=10	RIJ=1.38973	I=21	J=24	RIJ=1.09017
I= 1	J= 6	RIJ=1.39067	I= 2	J=25	RIJ=1.09069
I=10	J= 1	RIJ=1.39140	I= 3	J=26	RIJ=1.09069
I= 5	J=11	RIJ=1.50287	I= 4	J=27	RIJ=1.09061
I=11	J=12	RIJ=1.39523	I= 7	J=28	RIJ=1.09033
I=12	J=13	RIJ=1.39170	I= 8	J=29	RIJ=1.09073
I=13	J=14	RIJ=1.38998	I= 9	J=30	RIJ=1.09073
I=14	J=15	RIJ=1.38927	I=10	J=31	RIJ=1.09070
I=15	J=16	RIJ=1.38935			

Bond angles for 1-tolylnaphthalene in degrees.

I= 2	J= 1	K= 6	A=119.783	I= 9	J=10	K=31	A=119.610
I= 2	J= 1	K=10	A=120.193	I= 1	J=10	K=31	A=120.320
I= 6	J= 1	K=10	A=120.019	I= 5	J=11	K=12	A=121.673
I= 1	J= 2	K= 3	A=119.862	I= 5	J=11	K=16	A=118.475
I= 1	J= 2	K=25	A=120.404	I=12	J=11	K=16	A=120.022
I= 3	J= 2	K=25	A=119.713	I=11	J=12	K=13	A=119.490
I= 2	J= 3	K= 4	A=119.940	I=11	J=12	K=21	A=121.521
I= 2	J= 3	K=26	A=120.037	I=13	J=12	K=21	A=118.689
I= 4	J= 3	K=26	A=120.023	I=12	J=13	K=14	A=120.384
I= 3	J= 4	K= 5	A=120.232	I=12	J=13	K=17	A=120.116
I= 3	J= 4	K=27	A=119.910	I=14	J=13	K=17	A=119.601
I= 5	J= 4	K=27	A=119.859	I=13	J=14	K=15	A=119.994
I= 4	J= 5	K= 6	A=120.176	I=13	J=14	K=18	A=120.006
I= 4	J= 5	K=11	A=119.223	I=15	J=14	K=18	A=120.000
I= 6	J= 5	K=11	A=120.386	I=14	J=15	K=16	A=119.871
I= 5	J= 6	K= 7	A=120.396	I=14	J=15	K=19	A=120.072
I= 5	J= 6	K= 1	A=119.768	I=16	J=15	K=19	A=120.057
I= 7	J= 6	K= 1	A=119.813	I=15	J=16	K=11	A=120.241
I= 6	J= 7	K= 8	A=120.102	I=15	J=16	K=20	A=119.886
I= 6	J= 7	K=28	A=119.764	I=11	J=16	K=20	A=119.873
I= 8	J= 7	K=28	A=119.134	I=12	J=21	K=22	A=111.464
I= 7	J= 8	K= 9	A=120.033	I=12	J=21	K=23	A=110.195
I= 7	J= 8	K=29	A=119.980	I=12	J=21	K=24	A=110.180
I= 9	J= 8	K=29	A=119.987	I=22	J=21	K=23	A=108.121
I= 8	J= 9	K=10	A=119.955	I=22	J=21	K=24	A=108.214
I= 8	J= 9	K=30	A=120.020	I=23	J=21	K=24	A=108.584
I=10	J= 9	K=30	A=120.025	I= 9	J=10	K= 1	A=120.070

Coordinates for E,E-bis(3,4-dimethoxybenzylidene)-succinic anhydride.

Atom	x	y	z
1	6.55418	9.58654	3.35880
2	7.47219	8.78409	4.05072
3	8.39535	9.24579	4.55664
4	7.03590	7.37209	3.94202
5	7.53612	6.46432	4.62881
6	8.22564	6.85992	5.55612
7	6.97814	5.16047	5.24547
8	5.64354	4.81501	5.01316
9	4.97507	5.53729	4.58308
10	5.18087	3.51374	5.29185
11	3.88110	3.18177	5.00210
12	2.87360	4.18136	4.75879
13	2.99967	4.80996	3.88606
14	2.73356	4.88869	5.56122
15	1.88096	3.78118	4.60314

16	6.07885	2.55917	5.82967
17	5.67666	1.27309	6.09041
18	6.61649	.21456	6.32464
19	6.17461	-.76276	6.45398
20	7.33902	.05891	5.53362
21	7.22132	.32027	7.21836
22	7.40464	2.94710	6.10468
23	8.11756	2.24920	6.51552
24	7.84789	4.24539	5.82184
25	8.88233	4.50096	5.99961
26	5.56996	8.75932	2.75946
27	4.71066	9.24745	2.06270
28	5.85222	7.37330	3.08520
29	5.27346	6.40765	2.31993
30	4.64821	6.74349	1.50879
31	5.70017	5.03692	2.05204
32	6.99528	4.57597	2.33298
33	7.72780	5.27598	2.08975
34	7.33598	3.21600	2.19291
35	8.60062	2.79712	2.52357
36	9.69795	3.71850	2.67226
37	9.60442	4.42502	3.48430
38	9.88664	4.33555	1.48762
39	10.65224	3.28251	2.67456
40	6.35320	2.29789	1.74787
41	6.63571	.96132	1.61776
42	5.60096	-.03007	1.47299
43	5.95622	-1.05074	1.44244
44	4.86651	-.04463	2.26629
45	5.01333	.04364	.56998
46	5.06600	2.77399	1.43112
47	4.29112	2.10526	1.08995
48	4.74207	4.12822	1.58330
49	3.73297	4.45660	1.38056

Bond lengths for E,E-bis(3,4-dimethoxybenzylidene)-succinic anhydride in angstrom.

I= 1	J= 2	RIJ=1.40193	I=26	J=27	RIJ=1.19733
I= 2	J= 3	RIJ=1.19727	I=26	J=28	RIJ=1.48106
I= 1	J=26	RIJ=1.40130	I=28	J=29	RIJ=1.36351
I= 2	J= 4	RIJ=1.48166	I=29	J=30	RIJ=1.07785
I= 4	J= 5	RIJ=1.36406	I=29	J=31	RIJ=1.46040
I= 4	J=28	RIJ=1.45691	I=31	J=32	RIJ=1.40311
I= 5	J= 6	RIJ=1.07746	I=31	J=48	RIJ=1.40122
I= 5	J= 7	RIJ=1.46056	I=32	J=33	RIJ=1.07419
I= 7	J= 8	RIJ=1.40310	I=32	J=34	RIJ=1.40897
I= 7	J=24	RIJ=1.40109	I=34	J=35	RIJ=1.37262
I= 8	J= 9	RIJ=1.07402	I=34	J=40	RIJ=1.41663
I= 8	J=10	RIJ=1.40891	I=35	J=36	RIJ=1.44055
I=10	J=11	RIJ=1.37243	I=36	J=37	RIJ=1.08043
I=10	J=16	RIJ=1.41662	I=36	J=38	RIJ=1.08101
I=11	J=12	RIJ=1.44046	I=36	J=39	RIJ=1.08108
I=12	J=13	RIJ=1.08049	I=40	J=41	RIJ=1.37228
I=12	J=14	RIJ=1.08104	I=40	J=46	RIJ=1.40850
I=12	J=15	RIJ=1.08110	I=41	J=42	RIJ=1.44032
I=16	J=17	RIJ=1.37249	I=42	J=43	RIJ=1.08116
I=16	J=22	RIJ=1.40849	I=42	J=44	RIJ=1.08119
I=17	J=18	RIJ=1.43610	I=42	J=45	RIJ=1.08073
I=18	J=19	RIJ=1.08119	I=46	J=47	RIJ=1.07891
I=18	J=20	RIJ=1.08125	I=46	J=48	RIJ=1.40072
I=18	J=21	RIJ=1.08072	I=48	J=49	RIJ=1.08038
I=22	J=23	RIJ=1.07894	I=22	J=24	RIJ=1.40073
I=24	J=25	RIJ=1.08027			

Bond angles for E,E-bis(3,4-dimethoxybenzylidene)-succinic anhydride in degrees.

I= 1	J= 2	K= 3	A=122.240	I=19	J=18	K=20	A=103.355
I= 1	J=26	K=27	A=122.363	I=19	J=18	K=21	A=102.440
I= 1	J= 2	K= 4	A=108.446	I=20	J=18	K=21	A=104.201
I= 1	J=26	K=28	A=108.348	I=22	J=24	K=25	A=119.279
I= 2	J= 1	K=26	A=110.207	I=23	J=22	K=24	A=117.861
I= 2	J= 4	K= 5	A=118.585	I=26	J=28	K=29	A=116.072
I= 2	J= 4	K=28	A=106.317	I=27	J=26	K=28	A=129.212
I= 3	J= 2	K= 4	A=129.226	I=28	J=29	K=30	A=116.753
I= 4	J= 5	K= 6	A=115.416	I=28	J=29	K=31	A=130.106
I= 4	J= 5	K= 7	A=129.075	I=30	J=29	K=31	A=108.896
I= 4	J=28	K=26	A=106.596	I=31	J=32	K=33	A=118.806
I= 4	J=28	K=29	A=132.344	I=31	J=32	K=34	A=121.358
I= 5	J= 4	K=28	A=132.697	I=31	J=48	K=49	A=120.266
I= 5	J= 7	K= 8	A=123.032	I=31	J=48	K=46	A=120.345
I= 6	J= 7	K=24	A=117.830	I=32	J=31	K=48	A=119.016
I= 6	J= 5	K= 7	A=112.902	I=32	J=34	K=35	A=119.563
I= 7	J= 8	K= 9	A=118.851	I=32	J=34	K=40	A=119.277
I= 7	J= 8	K=10	A=121.344	I=33	J=32	K=34	A=119.810
I= 7	J=24	K=22	A=120.346	I=34	J=35	K=36	A=122.107
I= 7	J=24	K=25	A=120.281	I=34	J=40	K=41	A=121.211
I= 8	J= 7	K=24	A=118.995	I=34	J=40	K=46	A=119.051
I= 8	J=10	K=11	A=115.515	I=35	J=34	K=40	A=115.560
I= 8	J=10	K=16	A=119.294	I=35	J=36	K=38	A=115.080
I= 9	J= 8	K=10	A=119.732	I=35	J=36	K=39	A=114.584
I=10	J=11	K=12	A=122.049	I=37	J=36	K=38	A=104.120
I=10	J=16	K=17	A=121.184	I=37	J=36	K=39	A=103.571
I=10	J=16	K=22	A=119.026	I=38	J=36	K=39	A=103.409
I=11	J=12	K=13	A=117.371	I=40	J=41	K=42	A=122.143
I=11	J=12	K=15	A=117.371	I=40	J=46	K=47	A=121.196
I=11	J=12	K=15	A=114.172	I=40	J=46	K=48	A=120.911
I=11	J=16	K=16	A=121.164	I=41	J=42	K=43	A=114.619
I=13	J=12	K=14	A=103.443	I=41	J=42	K=44	A=115.066
I=13	J=12	K=15	A=102.053	I=41	J=42	K=45	A=115.326
I=14	J=12	K=15	A=103.207	I=43	J=42	K=44	A=103.359
I=16	J=17	K=18	A=121.370	I=43	J=42	K=45	A=102.675
I=16	J=22	K=23	A=121.209	I=44	J=42	K=45	A=104.201
I=16	J=22	K=24	A=120.912	I=46	J=48	K=49	A=119.366
I=17	J=18	K=19	A=114.617	I=47	J=46	K=48	A=117.873
I=17	J=18	K=20	A=115.376	I=29	J=31	K=32	A=122.773
I=17	J=18	K=21	A=115.350	I=29	J=31	K=48	A=118.052
I=17	J=16	K=22	A=119.789	I=35	J=34	K=40	A=121.136
I=41	J=40	K=46	A=119.738	I=11	J=12	K=14	A=114.661

Coordinates for compound 133.

Atom	x	y	z
1	6.55418	9.58654	3.35880
2	7.47523	8.78409	4.05072
3	8.40711	9.22565	4.65664
4	7.01989	7.38360	3.95456
5	7.44728	6.46467	4.86053
6	8.13779	6.77861	5.62697
7	6.73954	5.22773	5.13829
8	7.39760	3.99350	5.08214
9	8.45062	3.99287	4.84850
10	6.70614	2.78730	5.31808
11	7.37611	1.58976	5.27745
12	8.81219	1.51311	5.20979
13	9.36753	1.98137	6.01017
14	9.25641	1.93658	4.32477
15	9.20168	.50461	5.20951
16	5.31597	2.82781	5.59416
17	4.59822	3.67258	5.78306
18	3.17982	1.67796	6.00669
19	2.73768	.69831	6.12367
20	2.85620	2.19852	6.89708
21	2.59238	2.12041	5.21428
22	4.66911	4.07660	5.68212
23	3.61562	5.14833	5.90346
24	5.37633	5.26849	5.48183
25	4.86223	6.21567	5.55951
26	5.49385	8.80185	2.87370

27	4.58480	9.25676	2.24278
28	5.77933	7.38882	3.20292
29	5.14302	6.39511	2.52838
30	4.41549	6.69069	1.78973
31	5.66400	5.07924	2.17922
32	7.04300	4.82923	2.10409
33	7.72419	5.64530	2.27675
34	7.53656	3.54364	1.78077
35	8.88973	3.35307	1.65788
36	9.84838	4.39686	1.91567
37	9.82935	4.80308	2.91711
38	9.76373	5.26608	1.27871
39	10.88072	4.10257	1.78777
40	6.62227	2.48780	1.56972
41	7.06522	1.22357	1.27040
42	6.16499	.11799	1.06876
43	6.64824	-.82288	.84510
44	5.52626	-1.12286	1.91014
45	5.46804	.23607	.25098
46	5.23979	2.74397	1.66676
47	4.51341	1.95992	1.51664
48	4.76367	4.02867	1.96066
49	3.69979	4.20068	2.03762

Bond lengths for compound 133 in angstrom.

I= 1	J= 2	RIJ=1.40393	I=26	J=27	RIJ=1.19641
I= 2	J= 3	RIJ=1.19604	I=28	J=28	RIJ=1.47870
I= 1	J=26	RIJ=1.40547	I=26	J=29	RIJ=1.39917
I= 2	J= 4	RIJ=1.41579	I=29	J=30	RIJ=1.07809
I= 4	J= 5	RIJ=1.35910	I=29	J=31	RIJ=1.45769
I= 4	J=28	RIJ=1.45051	I=31	J=32	RIJ=1.40349
I= 5	J= 6	RIJ=1.07836	I=31	J=48	RIJ=1.40061
I= 5	J= 7	RIJ=1.45513	I=32	J=33	RIJ=1.07694
I= 7	J= 8	RIJ=1.40268	I=32	J=34	RIJ=1.40907
I= 7	J=24	RIJ=1.40050	I=34	J=35	RIJ=1.37288
I= 8	J= 9	RIJ=1.07863	I=34	J=40	RIJ=1.41703
I= 8	J=10	RIJ=1.41021	I=35	J=36	RIJ=1.44047
I=10	J=11	RIJ=1.37281	I=36	J=37	RIJ=1.08086
I=10	J=18	RIJ=1.41790	I=36	J=38	RIJ=1.08094
I=11	J=12	RIJ=1.43971	I=36	J=39	RIJ=1.08106
I=12	J=13	RIJ=1.08097	I=40	J=41	RIJ=1.37261
I=12	J=14	RIJ=1.07699	I=40	J=46	RIJ=1.40936
I=13	J=15	RIJ=1.08110	I=41	J=42	RIJ=1.43992
I=16	J=17	RIJ=1.37310	I=42	J=43	RIJ=1.08110
I=16	J=22	RIJ=1.40913	I=42	J=44	RIJ=1.08113
I=17	J=18	RIJ=1.43693	I=42	J=45	RIJ=1.08094
I=18	J=19	RIJ=1.08115	I=46	J=47	RIJ=1.07903
I=18	J=20	RIJ=1.08097	I=46	J=48	RIJ=1.40119
I=18	J=21	RIJ=1.08110	I=48	J=49	RIJ=1.08064
I=22	J=23	RIJ=1.07898	I=22	J=24	RIJ=1.40032
I=24	J=25	RIJ=1.08050			

Bond angles for compound 133 in degrees.

I= 1	J= 2	K= 3	A=123.354	I=20 J=18 K=21	A=104.122
I= 1	J=26	K=27	A=122.885	I=22 J=24 K=25	A=119.706
I= 1	J= 2	K= 4	A=107.931	I=23 J=22 K=24	A=117.769
I= 3	J=26	K=26	A=128.121	I=26 J=28 K=29	A=119.853
I= 2	J= 4	K= 5	A=110.239	I=27 J=26 K=28	A=120.062
I= 2	J= 4	K=28	A=120.069	I=28 J=29 K=30	A=117.097
I= 5	J= 4	K=28	A=107.112	I=28 J=29 K=31	A=127.701
I= 4	J= 5	K= 6	A=128.591	I=30 J=29 K=31	A=108.940
I= 4	J= 5	K= 7	A=118.585	I=31 J=32 K=33	A=118.550
I= 6	J= 5	K= 7	A=123.227	I=31 J=32 K=34	A=121.212
I= 4	J=28	K=24	A=119.494	I=31 J=48 K=49	A=120.154
I= 4	J=28	K=29	A=130.915	I=31 J=48 K=46	A=120.143
I= 5	J= 4	K=28	A=128.078	I=32 J=31 K=48	A=119.305
I= 5	J= 7	K= 8	A=120.541	I=32 J=34 K=35	A=119.723
I= 5	J= 7	K=24	A=119.832	I=32 J=34 K=40	A=119.253
I= 8	J= 5	K= 7	A=115.176	I=33 J=32 K=34	A=120.223
I= 7	J= 8	K= 9	A=118.127	I=34 J=35 K=36	A=122.424
I= 7	J= 8	K=10	A=120.913	I=34 J=40 K=41	A=120.960
I= 7	J=24	K=22	A=119.960	I=34 J=40 K=46	A=119.107
I= 7	J=24	K=25	A=120.279	I=35 J=36 K=37	A=115.241
I= 9	J= 7	K=24	A=119.622	I=36 J=36 K=38	A=115.156

I= 8	J=10	K=11	A=120.125	I=35	J=36	K=39	A=114.651
I= 8	J=10	K=16	A=119.267	I=37	J=36	K=38	A=104.026
I= 9	J= 8	K=10	A=120.989	I=37	J=36	K=39	A=103.223
I=10	J=11	K=12	A=122.319	I=38	J=36	K=39	A=102.942
I=10	J=16	K=17	A=121.015	I=40	J=41	K=42	A=122.409
I=10	J=16	K=22	A=119.179	I=40	J=46	K=47	A=121.252
I=11	J=12	K=13	A=117.041	I=40	J=46	K=48	A=120.949
I=11	J=12	K=13	A=117.041	I=41	J=42	K=43	A=114.691
I=11	J=12	K=15	A=114.145	I=41	J=42	K=44	A=115.068
I=11	J=16	K=16	A=120.608	I=41	J=42	K=45	A=115.161
I=13	J=12	K=14	A=103.076	I=43	J=42	K=44	A=103.144
I=13	J=12	K=15	A=102.663	I=43	J=42	K=45	A=103.107
I=14	J=12	K=15	A=102.550	I=44	J=42	K=45	A=104.095
I=16	J=17	K=18	A=122.318	I=46	J=48	K=49	A=119.691
I=16	J=22	K=23	A=121.330	I=47	J=46	K=48	A=117.799
I=16	J=22	K=24	A=120.900	I=29	J=31	K=32	A=121.654
I=17	J=18	K=19	A=114.671	I=29	J=31	K=48	A=118.998
I=17	J=18	K=20	A=115.201	I=35	J=34	K=40	A=121.019
I=17	J=18	K=21	A=115.096	I=41	J=40	K=46	A=119.932
I=17	J=16	K=22	A=119.805	I=11	J=12	K=14	A=116.411
I=19	J=18	K=20	A=102.991	I=19	J=18	K=21	A=103.175

Coordinates for compound 132.

Atom	x	y	z
1	6.55418	9.58654	3.35880
2	7.47564	8.78409	4.05072
3	8.40276	9.23686	4.65664
4	7.12474	7.36746	3.81167
5	8.03422	6.38503	4.56293
6	8.98592	6.70471	4.45522
7	8.09673	5.06149	3.44861
8	8.65497	4.00966	4.16204
9	9.08388	4.23827	5.14494
10	8.60332	2.67988	3.71182
11	9.13058	1.66606	4.47623
12	9.73936	1.90443	5.75923
13	9.12015	2.35115	6.52450
14	10.61056	2.54390	5.73383
15	10.10892	1.01336	6.24738
16	7.97967	2.40423	-.47348
17	7.88533	1.12066	1.39559
18	7.24626	.81224	.74683
19	7.23267	-.23883	.49419
20	6.20456	1.09369	-.68056
21	7.69662	1.26769	-.12418
22	7.43935	3.47036	1.72878
23	6.95028	3.30168	.78186
24	7.49862	4.78545	2.20618
25	7.05203	5.56864	1.61528
26	5.60571	6.78641	2.21097
27	4.67662	9.23675	2.10140
28	5.93603	7.36937	2.97665
29	4.99420	6.40652	2.77567
30	4.09153	6.72301	2.47736
31	4.87434	5.11459	3.44545
32	4.24825	4.06333	2.76227
33	3.82249	4.27115	1.79961
34	4.22059	2.75649	3.29156
35	3.63142	1.73977	2.56194
36	3.02892	1.95049	1.29104
37	3.69959	2.31552	.52571
38	2.30643	2.65176	1.27673
39	2.61697	1.06399	.84133
40	4.83012	2.50491	4.54594
41	4.84793	1.24396	5.08806
42	5.47249	.95568	6.35370
43	5.42113	-.06075	6.65682
44	6.52863	1.17445	6.40593
45	5.05453	1.48022	7.20159
46	5.49767	3.57095	5.23672
47	5.91931	3.41866	6.19034
48	5.46875	4.86174	4.69436
49	5.95610	5.64381	5.24432

Bond lengths for compound 132 in angstrom.

I= 1	J= 2	RIJ=1.40417		I=26	J=27	RIJ=1.19684
I= 2	J= 3	RIJ=1.19653		I=26	J=28	RIJ=1.47827
I= 1	J=26	RIJ=1.40377		I=28	J=29	RIJ=1.36183
I= 2	J= 4	RIJ=1.47887		I=29	J=30	RIJ=1.07925
I= 4	J= 5	RIJ=1.36224		I=29	J=31	RIJ=1.46035
I= 4	J=28	RIJ=1.45263		I=31	J=32	RIJ=1.40276
I= 5	J= 6	RIJ=1.07793		I=31	J=48	RIJ=1.40189
I= 5	J= 7	RIJ=1.46017		I=32	J=33	RIJ=1.07870
I= 7	J= 8	RIJ=1.40268		I=32	J=34	RIJ=1.41020
I= 7	J=24	RIJ=1.40197		I=34	J=35	RIJ=1.37275
I= 8	J= 9	RIJ=1.07866		I=34	J=40	RIJ=1.41716
I= 8	J=10	RIJ=1.41016		I=35	J=36	RIJ=1.44012
I=10	J=11	RIJ=1.37267		I=36	J=37	RIJ=1.08114
I=10	J=16	RIJ=1.41719		I=36	J=38	RIJ=1.08103
I=11	J=12	RIJ=1.43998		I=36	J=39	RIJ=1.08111
I=12	J=13	RIJ=1.08103		I=40	J=41	RIJ=1.37270
I=12	J=14	RIJ=1.08101		I=40	J=46	RIJ=1.40816
I=12	J=15	RIJ=1.08118		I=41	J=42	RIJ=1.44038
I=16	J=17	RIJ=1.37296		I=42	J=43	RIJ=1.08112
I=16	J=22	RIJ=1.40830		I=42	J=44	RIJ=1.08088
I=17	J=18	RIJ=1.43612		I=42	J=45	RIJ=1.08094
I=18	J=19	RIJ=1.08112		I=46	J=47	RIJ=1.07906
I=18	J=20	RIJ=1.08084		I=46	J=48	RIJ=1.40024
I=18	J=21	RIJ=1.08097		I=48	J=49	RIJ=1.07772
I=22	J=23	RIJ=1.07907		I=22	J=24	RIJ=1.40036
I=24	J=25	RIJ=1.07777				

Bond angles for compound 132 in degrees.

I= 1	J= 2	K= 3	A=122.822	I=20	J=18	K=21	A=104.026
I= 1	J=26	K=27	A=122.917	I=22	J=24	K=25	A=118.540
I= 1	J= 2	K= 4	A=108.168	I=23	J=22	K=24	A=117.723
I= 3	J=26	K=28	A=108.067	I=26	J=28	K=29	A=119.659
I= 2	J= 1	K=26	A=110.280	I=27	J=26	K=28	A=128.926
I= 2	J= 4	K= 5	A=120.185	I=28	J=29	K=30	A=117.404
I= 2	J= 4	K=28	A=116.612	I=28	J=29	K=31	A=127.910
I= 3	J= 2	K= 4	A=128.909	I=30	J=29	K=31	A=110.056
I= 4	J= 5	K= 6	A=116.281	I=31	J=32	K=33	A=117.959
I= 4	J= 5	K= 7	A=126.864	I=31	J=32	K=34	A=121.307
I= 4	J=28	K=26	A=106.649	I=31	J=48	K=46	A=121.066
I= 4	J=28	K=29	A=130.523	I=31	J=48	K=46	A=120.332
I= 5	J= 4	K=28	A=130.777	I=32	J=31	K=48	A=119.059
I= 5	J= 7	K= 8	A=118.302	I=32	J=34	K=35	A=119.991
I= 5	J= 7	K=24	A=122.456	I=32	J=34	K=40	A=119.284
I= 6	J= 5	K= 7	A=112.982	I=33	J=32	K=34	A=120.654
I= 7	J= 8	K= 9	A=117.972	I=34	J=35	K=36	A=122.310
I= 7	J= 8	K=10	A=121.304	I=34	J=40	K=41	A=121.217
I= 7	J=24	K=22	A=120.324	I=34	J=40	K=46	A=119.044
I= 7	J=24	K=25	A=121.100	I=35	J=34	K=40	A=118.714
I= 8	J= 7	K=24	A=119.048	I=35	J=36	K=36	A=115.162
I= 8	J=10	K=11	A=119.965	I=36	J=36	K=39	A=114.670
I= 8	J=10	K=16	A=120.697	I=37	J=36	K=38	A=109.097
I= 9	J= 8	K=10	A=120.631	I=37	J=36	K=39	A=103.078
I=10	J=11	K=12	A=122.262	I=38	J=36	K=39	A=103.140
I=10	J=16	K= 7	A=121.166	I=40	J=41	K=42	A=122.444
I=10	J=16	K=22	A=119.022	I=40	J=46	K=47	A=117.290
I=11	J=12	K=13	A=117.193	I=40	J=46	K=48	A=120.954
I=11	J=12	K=14	A=117.193	I=41	J=42	K=43	A=114.679
I=11	J=12	K=15	A=114.222	I=41	J=42	K=44	A=111.219
I=11	J=10	K=16	A=120.726	I=41	J=42	K=45	A=115.109
I=13	J=12	K=14	A=103.521	I=43	J=42	K=44	A=103.197
I=13	J=12	K=15	A=102.517	I=43	J=42	K=45	A=103.078
I=14	J=12	K=15	A=102.878	I=44	J=42	K=45	A=104.028
I=16	J=17	K=18	A=122.265	I=46	J=48	K=49	A=118.561
I=16	J=22	K=23	A=121.309	I=47	J=46	K=48	A=117.747
I=16	J=22	K=24	A=120.960	I=29	J=31	K=32	A=118.423
I=17	J=18	K=19	A=114.683	I=29	J=31	K=48	A=122.914
I=17	J=18	K=20	A=116.223	I=35	J=34	K=40	A=120.715
I=17	J=18	K=21	A=115.127	I=41	J=40	K=46	A=119.738
I=17	J=16	K=22	A=119.611	I=11	J=12	K=14	A=114.688
I=19	J=18	K=20	A=103.123	I=19	J=18	K=21	A=103.064

Coordinates for Rotamer (0,0).

Atom	x	y	z
1	6.55418	9.58654	3.35880
2	7.47561	8.76409	4.05072
3	8.39965	9.24338	4.65694
4	7.05587	7.37110	3.91423
5	6.65236	6.46418	4.66758
6	8.42841	6.73667	5.93725
7	7.13824	5.08235	5.01152
8	5.76738	4.77521	4.97796
9	9.03865	5.53969	4.74971
10	5.33133	3.45523	5.16944
11	4.27505	3.23289	5.11477
12	6.25771	2.42332	5.40267
13	5.79121	1.14567	5.52625
14	6.69531	.03652	5.65108
15	6.21660	-.93056	5.71807
16	7.38768	-.08634	4.83031
17	7.33324	.05806	6.52394
18	7.62799	2.73958	5.41265
19	9.37066	1.97273	5.63380
20	8.06148	4.06159	5.28917
21	9.12034	4.27647	5.29777
22	5.68594	6.78621	2.73354
23	4.73843	9.24940	2.01941
24	5.66489	7.97211	3.07202
25	5.22642	6.38194	2.36698
26	4.52257	6.68341	1.62831
27	5.66987	5.01897	2.11093
28	7.02310	4.64592	2.17642
29	7.76861	5.37794	2.27091
30	7.39855	3.30379	2.01632
31	8.44309	3.03428	2.08553
32	6.42912	2.31338	1.78037
33	6.83582	1.01671	1.65349
34	5.90269	-.05747	1.44200
35	6.25131	-1.03823	1.36515
36	5.16509	-.18340	2.22247
37	5.31504	.01772	.59752
38	8.07539	2.69114	1.69483
39	4.30212	1.95687	1.52745
40	4.70118	4.03441	1.85739
41	3.65399	4.30004	1.82919

Bond lengths for Rotamer (0,0) in angstrom.

I= 1	J= 2	RIJ=1.40418	I=22	J=23	RIJ=1.19773
I= 2	J= 3	RIJ=1.19663	I=22	J=24	RIJ=1.48056
I= 1	J=22	RIJ=1.40320	I=24	J=25	RIJ=1.36285
I= 2	J= 4	RIJ=1.48032	I=25	J=26	RIJ=1.07789
I= 4	J= 5	RIJ=1.36317	I=25	J=27	RIJ=1.45963
I= 4	J=24	RIJ=1.45868	I=27	J=28	RIJ=1.40524
I= 5	J= 6	RIJ=1.07764	I=27	J=40	RIJ=1.40438
I= 5	J= 7	RIJ=1.45940	I=28	J=29	RIJ=1.07823
I= 7	J= 8	RIJ=1.40525	I=28	J=30	RIJ=1.40282
I= 7	J=20	RIJ=1.40407	I=30	J=31	RIJ=1.08100
I= 8	J= 9	RIJ=1.07853	I=30	J=32	RIJ=1.40584
I= 8	J=10	RIJ=1.40326	I=32	J=33	RIJ=1.36517
I=10	J=11	RIJ=1.08081	I=32	J=38	RIJ=1.40805
I=10	J=12	RIJ=1.40620	I=33	J=34	RIJ=1.43915
I=12	J=13	RIJ=1.36575	I=34	J=35	RIJ=1.08123
I=12	J=18	RIJ=1.40804	I=34	J=36	RIJ=1.08122
I=13	J=14	RIJ=1.43638	I=34	J=37	RIJ=1.08124
I=14	J=15	RIJ=1.08116	I=38	J=39	RIJ=1.07941
I=14	J=16	RIJ=1.08103	I=38	J=40	RIJ=1.40386
I=14	J=17	RIJ=1.08134	I=40	J=41	RIJ=1.08072
I=18	J=19	RIJ=1.07962	I=18	J=20	RIJ=1.40332
I=20	J=21	RIJ=1.08047			

Bond angles for Rotamer (0,0) in degrees.

I=	1	J=	2	K=	3	A=	122.472	I=16	J=14	K=16	A=103.131
I=	1	J=22		K=23		A=	122.524	I=15	J=14	K=17	A=103.292
I=	1	J=	2	K=	4	A=	108.299	I=16	J=14	K=17	A=103.729
I=	1	J=22		K=24		A=	108.233	I=16	J=20	K=21	A=119.278
I=	2	J=	1	K=22		A=	110.269	I=19	J=16	K=20	A=118.435
I=	2	J=	4	K=	5	A=	120.134	I=22	J=24	K=25	A=119.390
I=	2	J=	4	K=24		A=	106.504	I=23	J=22	K=24	A=129.184
I=	3	J=	2	K=	4	A=	129.199	I=24	J=25	K=26	A=117.162
I=	4	J=	5	K=	6	A=	116.084	I=24	J=25	K=27	A=129.136
I=	4	J=	5	K=	7	A=	128.211	I=26	J=25	K=27	A=109.052
I=	4	J=24		K=22		A=	105.648	I=27	J=28	K=29	A=120.765
I=	4	J=24		K=25		A=	132.239	I=27	J=28	K=30	A=120.425
I=	5	J=	4	K=24		A=	132.596	I=27	J=40	K=41	A=120.053
I=	5	J=	7	K=	8	A=	122.411	I=27	J=40	K=38	A=120.523
I=	5	J=	7	K=20		A=	118.258	I=28	J=27	K=40	A=119.115
I=	6	J=	5	K=	7	A=	112.747	I=28	J=30	K=31	A=119.327
I=	7	J=	8	K=	9	A=	120.766	I=28	J=30	K=32	A=120.871
I=	7	J=	8	K=10		A=	120.363	I=29	J=28	K=30	A=118.799
I=	7	J=20		K=18		A=	120.505	I=30	J=32	K=33	A=118.608
I=	7	J=20		K=21		A=	120.091	I=30	J=32	K=38	A=118.957
I=	8	J=	7	K=20		A=	119.161	I=32	J=33	K=34	A=121.922
I=	8	J=10		K=11		A=	119.359	I=32	J=38	K=39	A=121.042
I=	8	J=10		K=12		A=	120.544	I=32	J=38	K=40	A=120.394
I=	9	J=	8	K=10		A=	119.834	I=33	J=34	K=35	A=114.719
I=10		J=12		K=13		A=	118.486	I=33	J=34	K=36	A=115.067
I=10		J=12		K=18		A=	118.982	I=33	J=34	K=37	A=115.073
I=11		J=10		K=12		A=	120.083	I=35	J=34	K=36	A=103.221
I=12		J=13		K=14		A=	121.013	I=35	J=34	K=37	A=103.246
I=12		J=18		K=19		A=	121.153	I=36	J=34	K=37	A=103.955
I=12		J=18		K=20		A=	120.379	I=38	J=40	K=41	A=119.370
I=13		J=14		K=15		A=	114.682	I=39	J=38	K=40	A=118.548
I=13		J=14		K=16		A=	115.236	I=13	J=14	K=17	A=115.220
I=13		J=12		K=18		A=	122.550				

Coordinates for Rotamer (0,180).

Atom	x	y	z
1	6.55418	9.58654	3.35880
2	7.47363	8.78409	4.05072
3	8.39955	9.24069	4.65664
4	7.04255	7.37340	3.92725
5	7.62517	6.40939	4.69426
6	8.40335	6.73970	5.36236
7	7.08335	5.10457	5.05898
8	5.70566	4.83026	5.04297
9	4.99061	5.60924	4.83121
10	5.23991	3.52442	5.25890
11	4.17819	3.32737	5.22003
12	6.14316	2.47379	5.49683
13	5.64624	1.21068	5.64689
14	6.52354	.07940	5.75782
15	6.02214	-.87401	5.84950
16	7.41619	-.07103	4.91742
17	7.18781	.09251	6.61056
18	7.52122	2.75707	5.54517
19	8.24650	1.97461	5.70819
20	7.98449	4.06558	5.33938
21	9.04796	4.25505	5.33368
22	5.56243	6.78835	2.73680
23	4.73971	9.24274	2.01823
24	5.84942	7.37558	3.08908
25	5.19916	6.38292	2.41681
26	4.49615	6.66229	1.65086
27	5.62521	5.01018	2.16184
28	6.97156	4.61712	2.24075
29	7.74711	5.34202	2.42880
30	7.33014	3.26791	2.10126
31	8.37295	3.00189	2.18370
32	6.34575	2.28927	1.87023
33	6.65188	.96432	1.74748
34	7.99863	.48007	1.90061
35	8.10831	-.59085	1.80207
36	8.70867	.86714	1.18364

37	5.45487	.68964	2.85809
38	5.00107	2.68808	1.76802
39	4.22942	1.95040	1.60040
40	4.64272	1.03756	1.91225
41	3.60094	4.31727	1.67221

Bond lengths for Rotamer (0,180) in angstrom.

I= 1	J= 2	RIJ=1.40266	I=22	J=23	RIJ=1.19707
I= 1	J= 3	RIJ=1.19707	I=22	J=24	RIJ=1.48031
I= 1	J=22	RIJ=1.40296	I=24	J=25	RIJ=1.36289
I= 2	J= 4	RIJ=1.48024	I=25	J=26	RIJ=1.07766
I= 3	J= 5	RIJ=1.36274	I=25	J=27	RIJ=1.46013
I= 4	J=24	RIJ=1.45812	I=27	J=26	RIJ=1.40476
I= 5	J= 6	RIJ=1.07752	I=27	J=40	RIJ=1.40415
I= 5	J= 7	RIJ=1.45907	I=28	J=29	RIJ=1.07712
I= 7	J= 8	RIJ=1.40485	I=28	J=30	RIJ=1.40299
I= 7	J=20	RIJ=1.40371	I=30	J=31	RIJ=1.07936
I= 8	J= 9	RIJ=1.07846	I=30	J=32	RIJ=1.40718
I= 8	J=10	RIJ=1.40313	I=32	J=33	RIJ=1.36539
I=10	J=11	RIJ=1.08055	I=32	J=38	RIJ=1.40629
I=10	J=12	RIJ=1.40580	I=33	J=34	RIJ=1.43935
I=12	J=13	RIJ=1.36561	I=34	J=35	RIJ=1.08105
I=12	J=18	RIJ=1.40771	I=34	J=36	RIJ=1.08089
I=13	J=14	RIJ=1.43869	I=34	J=37	RIJ=1.08095
I=14	J=15	RIJ=1.08112	I=36	J=39	RIJ=1.08061
I=14	J=16	RIJ=1.08074	I=38	J=40	RIJ=1.40342
I=14	J=17	RIJ=1.08109	I=40	J=41	RIJ=1.08039
I=18	J=19	RIJ=1.07928	I=18	J=20	RIJ=1.40327
I=20	J=21	RIJ=1.08023			

Bond angles for Rotamer (0,180) in degrees.

I= 1	J= 2	K= 3	A=122.576	I=15	J=14	K=16	A=103.154
I= 1	J=22	K=23	A=122.533	I=15	J=14	K=17	A=103.226
I= 2	J= 1	K= 3	A=108.248	I=16	J=14	K=17	A=103.788
I= 1	J=22	K=24	A=108.213	I=18	J=20	K=21	A=119.298
I= 2	J= 1	K=22	A=110.916	I=19	J=18	K=20	A=118.445
I= 2	J= 4	K= 5	A=120.180	I=22	J=24	K=25	A=119.474
I= 2	J= 4	K=24	A=106.548	I=23	J=22	K=24	A=129.204
I= 3	J= 2	K= 4	A=129.123	I=24	J=25	K=26	A=117.112
I= 4	J= 5	K= 6	A=116.161	I=24	J=25	K=27	A=129.204
I= 4	J= 5	K= 7	A=127.915	I=26	J=25	K=27	A=109.112
I= 4	J=24	K=22	A=106.607	I=27	J=28	K=29	A=120.737
I= 4	J=24	K=25	A=132.224	I=27	J=28	K=30	A=120.661
I= 5	J= 4	K=24	A=132.410	I=27	J=40	K=41	A=120.127
I= 5	J= 7	K= 8	A=122.411	I=27	J=40	K=38	A=120.420
I= 5	J= 7	K=20	A=116.260	I=28	J=27	K=40	A=119.084
I= 6	J= 5	K= 7	A=112.787	I=28	J=30	K=31	A=116.446
I= 7	J= 5	K= 9	A=120.756	I=28	J=30	K=32	A=120.420
I= 7	J= 8	K=10	A=120.368	I=29	J=28	K=30	A=118.691
I= 7	J=20	K=18	A=120.605	I=30	J=32	K=33	A=122.194
I= 7	J=20	K=21	A=120.074	I=30	J=32	K=38	A=118.921
I= 8	J= 7	K=20	A=119.160	I=32	J=33	K=34	A=121.781
I= 8	J=10	K=11	A=119.362	I=32	J=38	K=39	A=120.032
I= 8	J=10	K=12	A=120.551	I=32	J=38	K=40	A=120.577
I= 9	J= 8	K=10	A=118.642	I=33	J=34	K=35	A=114.799
I=10	J=12	K=13	A=118.429	I=33	J=34	K=36	A=115.086
I=10	J=12	K=18	A=118.973	I=33	J=34	K=37	A=115.089
I=11	J=10	K=12	A=120.074	I=35	J=34	K=36	A=103.150
I=12	J=13	K=14	A=120.991	I=35	J=34	K=37	A=103.304
I=12	J=18	K=19	A=121.141	I=36	J=34	K=37	A=103.940
I=12	J=18	K=20	A=120.384	I=38	J=40	K=41	A=119.400
I=13	J=14	K=15	A=114.706	I=39	J=38	K=40	A=119.976
I=13	J=14	K=16	A=115.1[illegible]	I=25	J=27	K=28	A=122.197
I=13	J=14	K=17	A=115.266	I=25	J=27	K=40	A=116.590
I=13	J=12	K=18	A=122.586	I=31	J=30	K=32	A=121.131
I=33	J=32	K=38	A=118.884				

Coordinates for Rotamer (180,180).

Atom	x	y	z
1	6.55416	9.58654	3.35880
2	7.47321	6.76409	4.05072
3	8.39777	9.24034	4.65664
4	7.03721	7.37489	3.93215
5	7.55800	6.43069	4.76701
6	8.27632	6.78446	5.58928
7	7.00694	5.12346	5.11081
8	5.64766	4.81203	4.92777
9	4.94766	5.56492	4.60055
10	5.16207	3.50339	5.12946
11	4.13873	3.29755	4.95844
12	6.07027	2.48481	5.53024
13	5.67295	1.19155	5.71743
14	4.37256	.74652	5.29127
15	4.18045	-.30685	5.44868
16	3.53572	1.22414	5.78550
17	4.16458	.88347	4.23698
18	7.42234	2.80945	5.75249
19	8.12062	2.04171	6.08819
20	7.88552	4.11927	5.55152
21	8.93682	4.33834	5.68977
22	5.66178	6.78190	2.63363
23	4.87027	9.23486	1.85644
24	5.92257	7.36835	2.98854
25	5.33289	6.37783	2.25982
26	4.69384	6.68149	1.44574
27	5.74882	4.99336	2.05502
28	7.06586	4.56122	2.30070
29	7.83570	5.26458	2.57869
30	7.39651	3.19958	2.22045
31	8.41435	2.90279	2.43598
32	6.41485	2.24800	1.87772
33	6.69411	.91317	1.80392
34	7.95706	.37661	2.24061
35	8.04489	-.69928	2.11878
36	8.82163	.73627	1.69975
37	8.20161	.56989	3.27710
38	5.10553	2.68687	1.59997
39	4.33653	1.97150	1.33869
40	4.77467	4.04792	1.58899
41	3.75178	4.35727	1.51681

Bond lengths for Rotamer (180,180) in angstrom.

I= 1	J= 2	RIJ=1.40260	I=22	J=23	RIJ=1.19820
I= 2	J= 3	RIJ=1.19587	I=22	J=24	RIJ=1.48057
I= 1	J=22	RIJ=1.40346	I=24	J=25	RIJ=1.36377
I= 2	J= 4	RIJ=1.47987	I=25	J=26	RIJ=1.07857
I= 4	J= 5	RIJ=1.36372	I=25	J=27	RIJ=1.46003
I= 4	J=24	RIJ=1.46043	I=27	J=28	RIJ=1.40775
I= 5	J= 6	RIJ=1.07834	I=27	J=40	RIJ=1.40599
I= 5	J= 7	RIJ=1.45970	I=28	J=29	RIJ=1.07918
I= 7	J= 8	RIJ=1.40646	I=28	J=30	RIJ=1.40951
I= 7	J=20	RIJ=1.40818	I=30	J=31	RIJ=1.08191
I= 8	J= 9	RIJ=1.07895	I=30	J=32	RIJ=1.40948
I= 8	J=10	RIJ=1.40331	I=32	J=33	RIJ=1.36573
I=10	J=11	RIJ=1.08080	I=33	J=38	RIJ=1.40856
I=10	J=12	RIJ=1.40910	I=33	J=34	RIJ=1.44002
I=12	J=13	RIJ=1.36581	I=34	J=35	RIJ=1.08157
I=12	J=16	RIJ=1.40815	I=34	J=36	RIJ=1.08153
I=13	J=14	RIJ=1.43898	I=34	J=37	RIJ=1.08235
I=14	J=15	RIJ=1.08185	I=38	J=39	RIJ=1.08231
I=14	J=16	RIJ=1.08291	I=38	J=40	RIJ=1.40852
I=14	J=17	RIJ=1.08330	I=40	J=41	RIJ=1.08243
I=16	J=19	RIJ=1.08188	I=18	J=20	RIJ=1.40376
I=20	J=21	RIJ=1.08140			

Bond angles for Rotamer (180,180) in degrees.

I=	1	J=	2	K=	3	A=	122.565	I=13	J=12	K=18	A=118.423
I=	1	J=22		K=23		A=	122.578	I=15	J=14	K=16	A=103.120
I=	1	J=	2	K=	4	A=	108.193	I=15	J=14	K=17	A=103.184
I=	1	J=22		K=24		A=	108.150	I=16	J=14	K=17	A=103.889
I=	2	J=	1	K=	22	A=	110.092	I=18	J=20	K=21	A=119.321
I=	2	J=	4	K=	5	A=	119.851	I=19	J=18	K=20	A=119.317
I=	2	J=	4	K=24		A=	106.314	I=22	J=24	K=25	A=119.287
I=	3	J=	2	K=	4	A=	129.174	I=23	J=22	K=24	A=129.254
I=	4	J=	5	K=	6	A=	116.931	I=24	J=25	K=26	A=117.066
I=	4	J=	5	K=	7	A=	128.321	I=24	J=25	K=27	A=129.629
I=	4	J=24		K=22		A=	106.561	I=26	J=25	K=27	A=109.267
I=	4	J=24		K=25		A=	132.727	I=27	J=28	K=29	A=120.815
I=	5	J=	4	K=24		A=	133.151	I=27	J=28	K=30	A=120.547
I=	5	J=	7	K=	8	A=	122.175	I=27	J=40	K=41	A=120.264
I=	6	J=	7	K=20		A=	118.543	I=27	J=40	K=38	A=120.350
I=	6	J=	8	K=	9	A=	112.891	I=28	J=27	K=40	A=119.159
I=	7	J=	8	K=	9	A=	120.799	I=28	J=30	K=31	A=116.449
I=	7	J=	8	K=10		A=	120.523	I=28	J=30	K=32	A=120.319
I=	7	J=20		K=18		A=	120.359	I=29	J=28	K=30	A=116.620
I=	7	J=20		K=21		A=	120.226	I=30	J=32	K=33	A=122.045
I=	8	J=	7	K=20		A=	119.194	I=30	J=32	K=38	A=119.012
I=	8	J=10		K=11		A=	116.406	I=32	J=33	K=34	A=121.812
I=	8	J=10		K=12		A=	120.424	I=32	J=38	K=39	A=120.141
I=	9	J=	8	K=10		A=	118.652	I=32	J=38	K=40	A=120.582
I=10		J=12		K=13		A=	122.700	I=33	J=34	K=35	A=114.745
I=10		J=12		K=18		A=	118.876	I=33	J=34	K=36	A=115.186
I=11		J=10		K=12		A=	121.164	I=33	J=34	K=37	A=114.962
I=12		J=13		K=14		A=	121.006	I=35	J=34	K=36	A=103.164
I=12		J=18		K=19		A=	120.048	I=35	J=34	K=37	A=103.390
I=12		J=18		K=20		A=	120.619	I=36	J=34	K=37	A=103.818
I=13		J=14		K=15		A=	114.789	I=38	J=40	K=41	A=119.329
I=13		J=14		K=16		A=	115.264	I=39	J=38	K=40	A=119.260
I=13		J=14		K=17		A=	115.000	I=25	J=27	K=28	A=122.217
I=25		J=27		K=40		A=	118.475	I=31	J=30	K=32	A=121.230
I=33		J=32		K=38		A=	118.943				

APPENDIX II

SUPPLEMENTARY MATERIAL

Tables of observed and calculated structure factors as well as anisotropic temperature factors for non-hydrogen atoms and fractional atomic coordinates for hydrogen atoms appear on microfilm in the envelope at the back of this thesis. The hydrogen atoms are numbered according to the heavy atoms they are bonded to. This medium of presentation was chosen in view of the large volume of the supplementary tables.

APPENDIX III

Tables of crystal data and details of the crystallographic analyses, fractional atomic coordinates for non-hydrogen atoms, bond lengths and angles.

Table 1. Crystal data and details of the crystallographic analysis for OR24.

Formula	$C_{14}H_{17}NO_5$
M	247.29
Space group	$P2_1/c$
a/angstrom	7.454(1)
b	15.402(2)
c	9.988(3)
α /degrees	90.00
β	96.02(2)
γ	90.00
V/Å^3	1214.4(4)
Z	4
Dc	1.35
F(000)	528
μ(Mo-K$_\alpha$)/cm^{-1}	0.56
Scan range/°	3 to 30
Scan type	ω/2θ
h	-10 to 10
k	0 to 22
l	0 to 14
N(obs)	2814
Cut-off criterion	F > 4σ(F)
Number of parameters	209
Maximum Δ/σ	0.03
Residual electron density/eÅ^{-3}	0.39
R	0.0716
R$_w$	-
Weighting scheme	unit weights

Table 2. Fractional coordinates (x10^4) and equivalent isotropic temperature factors (Å^2, x10^3) for non-hydrogen atoms of OR24.

	x/a	y/b	z/c	U(eq)
O(1)	896(3)	2746(1)	2654(2)	48(1)
O(2)	499(3)	1153(1)	-117(2)	37(1)
O(3)	-1265(3)	2574(1)	387(2)	44(1)
N	-800(3)	472(1)	1556(2)	33(1)
C(1)	321(4)	2052(2)	2492(3)	35(1)
C(2)	-1242(4)	1936(2)	1345(3)	31(1)
C(3)	-3015(4)	1950(2)	2047(3)	42(1)
C(4)	-4646(5)	1828(2)	1013(4)	50(1)
C(5)	-4512(4)	1007(2)	315(4)	46(1)
C(6)	-2749(4)	995(2)	-375(3)	36(1)
C(7)	-1084(4)	1125(2)	601(3)	31(1)
C(8)	407(4)	559(2)	2697(3)	37(1)
C(9)	1069(4)	-130(2)	3419(3)	41(1)
C(10)	2172(4)	-41(2)	4598(3)	47(1)
C(11)	2713(4)	728(2)	5118(3)	46(1)
C(12)	2119(4)	1403(2)	4379(3)	41(1)
C(13)	968(4)	1333(2)	3179(3)	34(1)
C(14)	3823(5)	802(3)	6455(4)	62(1)

APPENDIX III

Tables of crystal data and details of the crystallographic analyses, fractional atomic coordinates for non-hydrogen atoms, bond lengths and angles.

Table 1. Crystal data and details of the crystallographic analysis for OR24.

Formula	$C_{16}H_{17}NO_3$
M	247.29
Space group	$P2_1/c$
a/ångstrom	7.454(1)
b	16.402(2)
c	9.988(3)
α/degrees	90.00
β	96.02(2)
γ	90.00
V/Å^3	1214.4(4)
Z	4
D_c	1.35
F(000)	528
μ(Mo-Kα)/cm^{-1}	0.56
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-10 to 10
k	0 to 22
l	0 to 14
I(obs)	2814
Cut-off criterion	$I > 4\sigma(I)$
Number of parameters	209
Maximum Δ/σ	0.03
Residual electron density/eÅ^{-3}	0.39
R	0.0716
R_w	-
Weighting scheme	unit weights

Table 2. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR24.

	x/a	y/b	z/c	U(eq)
O(1)	896(3)	2746(1)	2654(2)	46(1)
O(2)	499(3)	1153(1)	-117(2)	37(1)
O(3)	-1265(3)	2574(1)	387(2)	44(1)
N	-800(3)	472(1)	1556(3)	33(1)
C(1)	321(4)	2052(2)	2435(3)	35(1)
C(2)	-1242(4)	1936(2)	1345(3)	31(1)
C(3)	-3015(4)	1960(2)	2047(3)	42(1)
C(4)	-4646(5)	1825(2)	1013(4)	50(1)
C(5)	-4512(4)	1007(2)	315(4)	46(1)
C(6)	-2749(4)	955(2)	-375(3)	38(1)
C(7)	-1084(4)	1125(2)	601(3)	31(1)
C(8)	407(4)	559(2)	2697(3)	33(1)
C(9)	1069(4)	-130(2)	3419(3)	41(1)
C(10)	2122(4)	-41(2)	4598(3)	47(1)
C(11)	2713(4)	728(2)	5116(3)	46(1)
C(12)	2119(4)	1402(2)	4379(3)	41(1)
C(13)	966(4)	1333(2)	3179(3)	34(1)
C(14)	3823(5)	802(3)	6455(4)	62(1)

Table 3. Bond lengths (Å) and angles (°) of OR24.

O(1)-C(1)	1.228(3)	O(2)-C(7)	1.444(3)
O(3)-C(2)	1.417(3)	N-C(7)	1.435(3)
N-C(8)	1.362(4)	C(1)-C(2)	1.521(4)
C(1)-C(13)	1.449(4)	C(2)-C(3)	1.561(4)
C(2)-C(7)	1.536(4)	C(3)-C(4)	1.526(5)
C(4)-C(5)	1.520(5)	C(5)-C(6)	1.549(4)
C(6)-C(7)	1.521(4)	C(8)-C(9)	1.403(4)
C(8)-C(13)	1.405(4)	C(9)-C(10)	1.371(5)
C(10)-C(11)	1.407(5)	C(11)-C(12)	1.377(5)
C(11)-C(14)	1.500(5)	C(12)-C(13)	1.404(4)
C(7)-N-C(8)	120.6(2)	O(1)-C(1)-C(2)	118.1(3)
O(1)-C(1)-C(13)	124.9(3)	C(2)-C(1)-C(13)	117.0(2)
O(3)-C(2)-C(1)	110.2(2)	O(3)-C(2)-C(3)	109.6(2)
C(1)-C(2)-C(3)	107.2(2)	O(3)-C(2)-C(7)	108.0(2)
C(1)-C(2)-C(7)	111.3(2)	C(3)-C(2)-C(7)	110.5(2)
C(2)-C(3)-C(4)	110.1(3)	C(3)-C(4)-C(5)	110.6(3)
C(4)-C(5)-C(6)	110.7(3)	C(5)-C(6)-C(7)	112.2(2)
O(2)-C(7)-N	106.4(2)	O(2)-C(7)-C(2)	108.7(2)
N-C(7)-C(2)	109.8(2)	O(2)-C(7)-C(6)	110.0(2)
N-C(7)-C(6)	110.1(2)	C(2)-C(7)-C(6)	111.7(2)
N-C(8)-C(9)	120.3(3)	N-C(8)-C(13)	121.4(2)
C(9)-C(8)-C(13)	118.4(3)	C(8)-C(9)-C(10)	120.2(3)
C(9)-C(10)-C(11)	122.4(3)	C(10)-C(11)-C(12)	117.3(3)
C(10)-C(11)-C(14)	120.8(4)	C(12)-C(11)-C(14)	121.9(4)
C(11)-C(12)-C(13)	121.7(3)	C(1)-C(13)-C(8)	119.2(2)
C(1)-C(13)-C(12)	120.8(3)	C(8)-C(13)-C(12)	119.9(3)

Table 4. Crystal data and details of the crystallographic analysis for OR43.

Formula	$C_{15}H_{13}NO_3$
M	245.28
Space group	$P2_1/c$
a/angstroms	9.690(4)
b	6.578(4)
c	16.453(3)
α, β, γ/degrees	90.00 95.69(2) 90.00
V/Å^3	1044.4(8)
Z	4
D_c	1.33
F(000)	520
μ(Mo-K$_\alpha$)/cm^{-1}	0.56
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-12 to 12
k	0 to 12
l	0 to 23
I(obs)	1279
Cut-off criterion	$I > 3\sigma(I)$
Number of parameters	163
Maximum Δ/σ	0.05
Residual electron density/eÅ^{-3}	0.29
R	0.0655
R_w	-
Weighting scheme	unit weights

Table 5. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors (Å^2, $\times10^2$) for non-hydrogen atoms of OR43.

	x/a	y/b	z/c	U(eq)
N	4997(4)	624(5)	6468(2)	35(1)
O(1)	3082(4)	4159(4)	5930(3)	68(1)
O(2)	2562(4)	1356(4)	6921(2)	42(1)
O(3)	6439(4)	-1432(5)	6929(3)	60(1)

C(1)	9305(7)	5572(8)	6230(4)	79(2)
C(2)	8225(6)	4230(7)	6300(3)	53(1)
C(3)	6630(6)	4435(6)	6148(3)	47(1)
C(4)	5680(5)	3175(6)	6220(3)	38(1)
C(5)	3974(6)	3119(6)	6113(3)	43(1)
C(6)	3471(5)	1432(5)	6267(3)	34(1)
C(7)	2558(6)	842(6)	5493(3)	41(1)
C(8)	1775(6)	-727(6)	5573(3)	46(1)
C(9)	2869(6)	-2057(6)	5787(3)	48(1)
C(10)	3751(6)	-1919(6)	6648(3)	44(1)
C(11)	5153(6)	-901(6)	6698(3)	49(1)
C(12)	6213(5)	1700(6)	6435(3)	36(1)
C(13)	7918(5)	1450(7)	6585(3)	45(1)
C(14)	8752(6)	2793(7)	6508(3)	54(1)

Table 6. Bond lengths (Å) and angles (°) of OR43.

N-C(6)	1.503(6)	N-C(11)	1.365(6)
N-C(12)	1.408(6)	O(1)-C(5)	1.200(5)
O(2)-C(6)	1.397(5)	O(2)-HO	.95(6)
O(3)-C(11)	1.231(6)	C(1)-C(2)	1.497(8)
C(2)-C(3)	1.389(7)	C(2)-C(14)	1.394(8)
C(3)-C(4)	1.395(7)	C(4)-C(5)	1.476(7)
C(4)-C(12)	1.382(6)	C(5)-C(6)	1.540(7)
C(6)-C(7)	1.520(6)	C(7)-C(8)	1.520(7)
C(8)-C(9)	1.504(7)	C(9)-C(10)	1.546(7)
C(10)-C(11)	1.494(7)	C(12)-C(13)	1.409(6)
C(13)-C(14)	1.381(8)		
C(6)-N-C(11)	124.3(4)	C(6)-N-C(12)	109.8(4)
C(11)-N-C(12)	125.8(4)	C(6)-O(2)-HO	108(3)
C(3)-C(2)-C(14)	117.2(5)	C(2)-C(3)-C(4)	118.9(5)
C(3)-C(4)-C(5)	120.6(5)	C(3)-C(4)-C(12)	123.5(4)
C(5)-C(4)-C(12)	107.9(4)	O(1)-C(5)-C(4)	128.4(5)
O(1)-C(5)-C(6)	123.5(4)	C(4)-C(5)-C(6)	108.0(4)
N-C(6)-O(2)	111.3(4)	N-C(6)-C(5)	102.1(4)
O(2)-C(6)-C(5)	111.2(4)	N-C(6)-C(7)	114.0(4)
O(2)-C(6)-C(7)	110.0(4)	C(5)-C(6)-C(7)	106.0(4)
C(6)-C(7)-C(8)	115.0(4)	C(7)-C(8)-C(9)	114.5(4)
C(8)-C(9)-C(10)	113.6(4)	C(9)-C(10)-C(11)	115.0(4)
N-C(11)-O(3)	120.1(5)	N-C(11)-C(10)	119.1(4)
O(3)-C(11)-C(10)	120.8(5)	N-C(12)-C(4)	122.1(4)
N-C(12)-C(13)	128.9(5)	C(4)-C(12)-C(13)	119.0(5)
C(12)-C(13)-C(14)	116.4(5)	C(2)-C(14)-C(13)	125.1(5)

Table 7. Crystal data and details of the crystallographic analysis for OR43.

Formula	$C_9H_{11}NO_3$
M	156.16
Space group	$P2_1/c$
a/angstrom	11.3779(3)
b	6.509(5)
c	29.864(9)
α /degrees	90.00
β	91.96(4)
γ	90.00
V/Å^3	2121.2(1.8)
Z	12
Dc	1.42
F(000)	996
μ(Mo-Kα)/cm^{-1}	0.70
Scan range/°	3 to 27
Scan type	ω/2θ
h	-14 to 14
k	0 to 8
l	0 to 38
I(obs)	3094
Cut-off criterion	F > 3σ(F)
Number of parameters	332
Maximum Δ/σ	0.30
Residual electron density/eÅ^{-3}	0.55
R	0.0800
R$_w$	-
Weighting scheme	unit weights

Table 8. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times 10^3$) *for non-hydrogen* atoms of OR03.

	x/a	y/b	z/c	U(eq)
O(1)	3067(3)	3636(5)	3624(1)	60(1)
O(2)	-606(5)	210(10)	4431(2)	142(2)
O(3)	-1622(4)	3029(9)	4324(2)	150(2)
N	-725(5)	2129(11)	4355(2)	96(2)
C(1)	976(4)	3223(8)	4253(2)	62(1)
C(2)	1328(4)	2168(8)	3984(2)	61(1)
C(3)	1885(5)	3696(8)	3733(2)	68(1)
C(4)	1709(5)	5637(9)	4090(2)	77(2)
C(5)	2242(5)	4568(9)	4489(2)	73(1)
C(6)	1536(4)	2617(10)	4489(2)	77(2)
C(7)	388(5)	5386(10)	4132(3)	96(2)
O(1')	4433(3)	7008(5)	3512(1)	63(1)
O(2')	5521(3)	13031(6)	4666(1)	80(1)
O(3')	5540(3)	10956(6)	5084(1)	78(1)
N'	6048(3)	11406(7)	4732(1)	55(1)
C(1')	6944(4)	9883(6)	4377(1)	43(1)
C(2')	4962(4)	9616(7)	4073(1)	49(1)
C(3')	4974(4)	7365(7)	3945(1)	48(1)
C(4')	6935(4)	7077(7)	3953(2)	49(1)
C(5')	5735(5)	8876(8)	5663(2)	59(1)
C(6')	6061(4)	10556(7)	3894(1)	50(1)
C(7')	6664(4)	7847(8)	4428(2)	50(1)
O(1")	3360(3)	10316(5)	3131(1)	66(1)
O(2")	751(3)	4095(6)	2469(2)	93(1)
O(3")	404(3)	5597(7)	1635(1)	91(1)
N"	929(3)	5426(7)	2198(2)	62(1)
C(1")	1845(3)	6973(7)	2299(1)	44(1)
C(2")	2126(4)	7545(7)	2774(1)	45(1)
C(3")	2656(4)	9762(7)	2749(2)	51(1)
C(4")	3121(4)	9502(8)	2294(2)	58(1)
C(5")	3923(4)	7719(8)	2354(2)	60(1)
C(6")	2994(4)	6260(7)	2531(2)	51(1)
C(7")	2079(6)	8757(9)	2001(2)	90(1)

Table 9. Bond lengths (Å) and angles (°) of OR03.

O(1)-C(3)	1.417(6)	O(2)-N	1.270(8)
O(3)-N	1.171(7)	N-C(1)	1.475(7)
C(1)-C(2)	1.526(7)	C(1)-C(6)	1.509(6)
C(1)-C(7)	1.483(8)	C(2)-C(3)	1.491(7)
C(2)-C(6)	1.529(7)	C(3)-C(4)	1.570(7)
C(3)-C(5)	1.498(7)	C(4)-C(7)	1.509(7)
C(5)-C(6)	1.499(7)	O(1')-C(3')	1.431(5)
O(2')-N'	1.226(5)	O(3')-N'	1.212(5)
N'-C(1')	1.452(5)	C(1')-C(2')	1.505(5)
C(1')-C(6')	1.506(6)	C(1')-C(7')	1.504(6)
C(2')-C(3')	1.514(6)	C(2')-C(6')	1.497(6)
C(3')-C(4')	1.546(6)	C(4')-C(5')	1.533(6)
C(4')-C(7')	1.539(6)	C(5')-C(6')	1.512(7)
O(1")-C(1")	1.432(5)	O(2")-N"	1.214(5)
O(3")-N"	1.213(5)	N"-C(1")	1.468(5)
C(1")-C(2")	1.490(5)	C(1")-C(6")	1.521(5)
C(1")-C(7")	1.491(6)	C(2")-C(3")	1.526(6)
C(2")-C(6")	1.493(6)	C(3")-C(4")	1.522(6)
C(4")-C(5")	1.530(7)	C(4")-C(7")	1.542(6)
C(5")-C(6")	1.522(6)		
O(2)-N-O(3)	126.3(6)	O(2)-N-C(1)	115.1(6)
O(3)-N-C(1)	118.1(6)	N-C(1)-C(2)	120.5(5)
N-C(1)-C(6)	120.0(5)	C(2)-C(1)-C(6)	60.5(3)
N-C(1)-C(7)	122.4(5)	C(2)-C(1)-C(7)	107.0(5)
C(6)-C(1)-C(7)	110.6(5)	C(1)-C(2)-C(3)	103.3(4)
C(1)-C(2)-C(6)	59.2(3)	C(3)-C(2)-C(6)	107.2(4)
C(3)-C(2)-C(2)	115.6(4)	O(1)-C(3)-C(4)	111.8(4)
C(2)-C(3)-C(4)	98.4(4)	C(3)-C(4)-C(5)	98.9(4)
C(3)-C(4)-C(7)	96.4(5)	C(5)-C(4)-C(7)	104.8(5)

C(4)-C(5)-C(6)	100.8(4)	C(1)-C(6)-C(2)	60.3(3)
C(5)-C(6)-C(1)	103.6(5)	O(2')-C(6)-C(5)	103.7(4)
C(2')-C(7')-C(4)	98.3(5)	O(2')-N'-O(3')	123.9(4)
O(2')-N'-C(1')	118.7(4)	O(3')-N'-C(1')	117.4(4)
N'-C(1')-C(6')	120.1(4)	N'-C(1')-C(4')	120.0(4)
C(2')-C(1')-C(6')	59.6(3)	N'-C(1')-C(7')	122.5(4)
C(2')-C(1')-C(7')	108.9(3)	C(6')-C(1')-C(7')	109.5(4)
C(6')-C(1')-C(3')	104.4(4)	C(1')-C(2')-C(6')	60.2(3)
C(3')-C(2')-C(6')	106.9(4)	C(1')-C(3')-C(2')	112.2(4)
C(1')-C(3')-C(4')	112.7(4)	C(2')-C(3')-C(4')	97.6(3)
C(3')-C(4')-C(5')	102.5(4)	C(3')-C(4')-C(7')	100.5(3)
C(3')-C(4')-C(7')	101.8(4)	C(4')-C(5')-C(6')	97.6(3)
C(2')-C(6')-C(2')	60.2(3)	C(1')-C(6')-C(5')	104.4(4)
C(2')-C(6')-C(1')	107.6(4)	C(5')-C(6')-C(4')	95.5(3)
O(2")-N"-O(3")	125.2(4)	O(2")-N"-C(1")	118.5(4)
O(3")-N"-C(1")	116.3(4)	N"-C(1")-C(2")	119.9(4)
N"-C(1")-C(6")	118.0(4)	N"-C(1")-C(4")	59.4(3)
N"-C(1")-C(7")	123.1(4)	C(2")-C(1")-C(7")	109.6(4)
C(6")-C(1")-C(7")	110.1(4)	C(1")-C(2")-C(3")	104.3(3)
C(1")-C(2")-C(6")	61.3(3)	C(3")-C(2")-C(6")	106.9(3)
C(1")-C(3")-C(2")	113.3(4)	C(1")-C(3")-C(4")	114.5(4)
C(2")-C(3")-C(4")	97.0(4)	C(3")-C(4")-C(5")	102.4(4)
C(3")-C(4")-C(7")	101.3(4)	C(5")-C(4")-C(7")	102.8(4)
C(4")-C(5")-C(6")	97.4(3)	C(1")-C(6")-C(2")	59.2(3)
C(1")-C(6")-C(5")	109.7(4)	C(2")-C(6")-C(5")	106.8(4)
C(1")-C(7")-C(4")	95.0(4)		

Table 10. Crystal data and details of the crystallographic analysis for OR07.

Formula	$C_{14}H_{10}NOCl_3$
M_r	314.60
Space group	$P2_1/n$
a/angstrom	13.818(1)
b	10.714(1)
c	9.214(2)
α /degrees	90.00
β	99.49(2)
γ	90.00
V/Å^3	1929.9(5)
Z	4
D_c	1.55
F(000)	920
μ(Mo-Kα)/cm^{-1}	6.91
Scan range/°	2 to 25
Scan type	$\omega/2\theta$
h	-15 to 15
k	0 to 8
l	0 to 7
I(obs)	2472
Cut-off criterion	$F > 1\sigma(F)$
Number of parameters	216
Maximum Δ/σ	0.06
Residual electron density/eÅ^{-3}	0.20
R	0.0655
R_w	0.0450
Weighting scheme	$N = 1/\sigma^2(F)$

Table 12. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times 10^3$) for non-hydrogen atoms of OR07.

	x/a	y/b	z/c	U(eq)
Cl(4)	791(1)	6621(1)	223(1)	62(1)
Cl(3)	4381(1)	6030(1)	5284(1)	67(1)
Cl(2)	2514(1)	6671(1)	2187(1)	63(1)
O(1)	3025(1)	4548(1)	3866(2)	94(1)
Cl(5)	1111(1)	4124(2)	5394(1)	97(1)
C(10)	4026(3)	-768(5)	6231(6)	45(1)
C(7)	4061(2)	248(4)	2315(5)	44(1)

(O)	1178(2)	-246(4)	3445(4)	102(1)
C(9)	4074(2)	217(4)	3828(4)	39(1)
C(3)	1732(2)	971(4)	1798(5)	55(1)
C(19)	2387(2)	2932(4)	-308(5)	44(1)
C(4)	1157(2)	3038(4)	1185(4)	33(1)
C(9)	3947(2)	-2008(4)	2336(4)	40(1)
N	973(2)	1828(4)	3381(4)	58(1)
C(3)	1396(2)	1855(4)	535(4)	42(1)
C(11)	4121(4)	1475(7)	1528(9)	80(2)
C(5)	3959(2)	-847(4)	4597(4)	33(1)
C(16)	2843(3)	3085(5)	-1267(6)	56(1)
C(14)	1875(2)	2040(4)	-577(4)	43(1)
C(1)	1269(3)	779(5)	2952(5)	66(1)
C(15)	1857(3)	1323(5)	-1185(5)	61(1)
C(6)	3989(2)	-862(5)	1607(4)	48(1)
C(16)	2293(3)	1500(5)	-2804(6)	73(2)
C(17)	2772(3)	2382(5)	-2517(6)	70(2)
C(12)	4570(2)	-3319(6)	731(6)	87(2)
C(13)	5266(3)	-3406(10)	1746(7)	129(3)

Table 13. Bond lengths (Å) and angles (°) of OR07.

C(10)-C(5)	1.501(6)	C(7)-C(6)	1.386(6)
C(7)-C(11)	1.518(8)	C(7)-C(8)	1.371(6)
O)-C(11)	1.210(6)	C(6)-C(5)	1.378(5)
C(2)-C(3)	1.562(6)	C(2)-C(1)	1.537(7)
C(19)-C(18)	1.374(6)	C(19)-C(14)	1.387(6)
C(4)-C(3)	1.511(5)	C(9)-C(8)	1.390(6)
N-C(1)	1.341(6)	C(3)-C(14)	1.520(5)
C(18)-C(17)	1.386(7)	C(14)-C(15)	1.387(6)
C(15)-C(16)	1.360(7)	C(16)-C(17)	1.361(8)
C(12)-C(13)	1.538(11)		
C(7)-C(11)-C(11)	120.7(5)	C(6)-C(7)-C(8)	116.0(4)
C(11)-C(7)-C(8)	123.3(5)	C(7)-C(6)-C(5)	123.6(4)
C(3)-C(2)-C(1)	111.4(4)	C(16)-C(19)-C(14)	120.4(5)
C(2)-C(3)-C(4)	109.6(3)	C(2)-C(3)-C(14)	110.1(3)
C(4)-C(3)-C(14)	115.4(3)	C(10)-C(5)-C(6)	118.2(4)
C(19)-C(18)-C(17)	120.7(6)	C(19)-C(14)-C(3)	119.3(4)
C(19)-C(14)-C(15)	116.1(4)	C(3)-C(14)-C(15)	122.6(4)
(O)-C(1)-C(2)	121.3(5)	(O)-C(1)-N	124.4(5)
C(2)-C(1)-N	114.3(4)	C(14)-C(15)-C(16)	120.8(5)
C(7)-C(8)-C(9)	123.1(4)	C(15)-C(16)-C(17)	120.2(6)
C(16)-C(17)-C(16)	119.9(6)		

Table 11. Crystal data and details of the crystallographic analysis for OR12.

Formula	$C_{19}H_{14}NCl_4$
Mr	369.10
Space group	P1
a/angstrom	10.417(1)
b	9.455(2)
c	6.555(1)
α /degrees	106.64(1)
β	90.42(2)
γ	111.51(2)
V/Å^3	744.5(3)
Z	2
Dc	1.77
F(000)	372
μ(Cu-Kα)/cm^{-1}	59.9
Scan range/°	2 to 55
Scan type	ω/2θ
h	-9 to 9
k	-8 to 8
l	0 to 6
I(obs)	2165
Cut-off criterion	$F > 1\sigma(F)$
Number of parameters	198
Maximum σ/ρ	0.07
Residual electron density/eÅ^{-3}	0.19
R	0.0640
R_w	0.0598
Weighting scheme	$W = 1/\sigma^2(F)$

Table 15. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times 10^3$) for non-hydrogen atoms of OR12.

	x/a	y/b	z/c	U(eq)
Cl(2)	4062(1)	2398(1)	2078(2)	50(1)
Cl(1)	3132(1)	4766(1)	4691(2)	56(1)
Cl(4)	-2076(1)	1666(2)	5460(2)	68(1)
Cl(1)	-5183(1)	-1565(2)	2218(2)	87(1)
N	738(4)	2455(4)	4501(4)	41(1)
C(12)	-795(4)	-1717(5)	1452(5)	39(1)
C(3)	1435(4)	282(5)	1735(5)	34(1)
C(4)	108(4)	-132(5)	2337(5)	34(1)
C(5)	-217(4)	938(5)	3565(5)	39(1)
C(15)	-1596(5)	355(6)	3961(5)	49(1)
C(2)	1953(4)	2809(5)	3789(5)	38(1)
C(10)	-913(5)	-3811(5)	-996(6)	48(1)
C(2)	2388(4)	1771(5)	2564(5)	36(1)
C(13)	-2124(4)	-2272(5)	1690(6)	46(2)
C(16)	-3133(5)	-3968(6)	1031(7)	59(1)
C(14)	-2514(4)	-1200(6)	3160(5)	50(1)
C(6)	1377(4)	2155(5)	456(5)	36(1)
C(8)	1992(5)	-2857(6)	-1755(6)	56(1)
C(7)	2368(5)	-1404(6)	-523(5)	44(1)
C(11)	13(4)	2360(5)	4250(5)	40(1)
C(9)	667(6)	-4026(6)	-1975(6)	57(2)
C(17)	-3950(5)	-1753(9)	3637(7)	64(1)

Table 15. Bond lengths (Å) and angles (°) of OR12.

Cl(2)-C(2)	1.722(4)	Cl(1)-C(1)	1.736(4)
Cl(4)-C(15)	1.729(4)	Cl(1)-C(17)	1.797(5)
N-C(5)	1.366(5)	N-C(1)	1.301(5)
C(12)-C(4)	1.416(6)	C(12)-C(13)	1.388(5)
C(12)-C(11)	1.474(6)	C(3)-C(4)	1.414(5)
C(3)-C(2)	1.365(5)	C(3)-C(6)	1.473(5)
C(4)-C(5)	1.376(5)	C(5)-C(15)	1.421(6)
C(15)-C(14)	1.367(6)	C(1)-C(2)	1.420(5)
C(10)-C(11)	1.395(6)	C(10)-C(9)	1.381(7)
C(13)-C(16)	1.512(7)	C(13)-C(14)	1.434(6)
C(14)-C(17)	1.496(6)	C(6)-C(7)	1.333(6)
C(6)-C(11)	1.428(5)	C(8)-C(7)	1.385(6)
C(8)-C(9)	1.370(7)		
C(5)-N-C(1)	116.4(3)	C(4)-C(12)-C(13)	117.3(4)
C(4)-C(12)-C(11)	106.0(3)	C(13)-C(12)-C(11)	136.7(4)
C(4)-C(3)-C(2)	116.3(3)	C(4)-C(3)-C(6)	106.7(3)
C(2)-C(3)-C(6)	136.9(4)	C(12)-C(4)-C(3)	111.4(3)
C(12)-C(4)-C(5)	126.3(4)	C(3)-C(4)-C(5)	122.3(4)
N-C(5)-C(4)	120.8(4)	N-C(5)-C(15)	123.9(4)
C(4)-C(5)-C(15)	115.3(4)	C(14)-C(15)-C(5)	117.3(3)
C(14)-C(15)-C(14)	121.8(3)	C(5)-C(15)-C(14)	120.9(4)
C(1)-C(1)-N	115.7(3)	C(1)-C(1)-C(2)	117.8(3)
N-C(1)-C(2)	126.6(4)	C(12)-C(10)-C(9)	119.1(4)
C(2)-C(2)-C(3)	121.5(3)	C(2)-C(2)-C(1)	121.0(3)
C(9)-C(2)-C(1)	117.5(4)	C(12)-C(13)-C(16)	120.3(4)
C(12)-C(13)-C(14)	118.8(4)	C(16)-C(13)-C(14)	120.9(4)
C(15)-C(14)-C(13)	121.7(4)	C(15)-C(14)-C(17)	118.6(4)
C(13)-C(14)-C(17)	119.7(4)	C(3)-C(6)-C(7)	130.8(4)
C(3)-C(6)-C(11)	107.8(3)	C(7)-C(6)-C(11)	121.7(4)
C(7)-C(8)-C(9)	121.1(5)	C(2)-C(7)-C(8)	117.9(4)
C(12)-C(11)-C(10)	133.2(4)	C(12)-C(11)-C(6)	108.5(3)
C(10)-C(11)-C(6)	118.2(4)	C(10)-C(9)-C(8)	121.9(5)
Cl(1)-C(17)-C(14)	111.3(3)		

Table 16. Crystal data and details of the crystallographic analysis for OR05.

Formula	$(C_8H_7NO)_3.HBr_3$
M_r	496.05
Space group	$P2_1/c$
a/angstrom	15.496(11)
b	10.079(6)
c	11.880(6)
α /degrees	90.00
β	105.08(4)
γ	90.00
V/Å^3	1791.8(1.9)
Z	4
D_c	1.84
F(000)	1160
μ(Mo-Kα)/cm^{-1}	66.13
Scan range/°	2 to 30
Scan type	$\omega/2\theta$
h	-21 to 21
k	0 to 14
l	0 to 16
I(obs)	1582
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	101
Maximum Δ/σ	0.05
Residual electron density/eÅ^{-3}	1.19
R	0.088
R_w	0.057
Weighting scheme	$w = 1/\sigma^2(F)$

Table 17. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR05.

	x/a	y/b	z/c	U(eq)	
Br(1)	887(1)	4969(3)	1250(2)	50(1)	
Br(2)	2403(1)	4868(2)	2693(2)	43(1)	
Br(3)	3909(1)	4815(3)	4200(2)	61(1)	
O(1)	1064(6)	1014(9)	2276(8)	31(3)	*
N(1)	-1088(8)	1076(13)	-291(12)	43(4)	*
C(1)	-609(8)	1484(16)	749(14)	26(4)	*
C(2)	-1086(9)	2486(18)	1186(14)	34(5)	*
C(3)	-2027(9)	2578(18)	276(14)	44(5)	*
C(4)	-1939(10)	1736(16)	-728(16)	52(6)	*
O(1")	2301(6)	475(10)	4149(9)	41(3)	*
N(1")	1211(8)	1793(12)	3813(11)	33(4)	*
C(1")	1973(9)	1323(15)	3685(14)	28(4)	*
C(2")	2485(9)	1838(15)	4791(14)	36(5)	*
C(3")	1862(8)	2835(16)	5064(14)	37(5)	*
C(4")	988(9)	2642(18)	4138(15)	35(5)	*
O(1')	4087(6)	5177(13)	1159(9)	50(3)	*
N(1')	4776(7)	6254(13)	1077(12)	45(4)	*
C(1')	5601(10)	5945(17)	1565(15)	37(5)	*
C(2')	5918(10)	6676(17)	2869(16)	57(6)	*
C(3')	5123(10)	7459(22)	2805(16)	65(6)	*
C(4')	4368(8)	7199(18)	1751(14)	58(5)	*

* Isotropic temperature factor

Table 18. Bond lengths (Å) and angles (°) of OR05.

Br(1)-Br(2)	2.524(2)	Br(2)-Br(3)	2.545(3)
O(1)-C(1)	1.197(14)	N(1)-C(1)	1.33(2)
N(1)-C(4)	1.45(2)	C(1)-C(2)	1.43(2)
C(2)-C(3)	1.56(2)	C(3)-C(4)	1.50(2)
O(1')-C(1')	1.29(2)	N(1')-C(1')	1.24(2)
N(1')-C(4')	1.41(2)	C(1')-C(2')	1.49(2)
C(2')-C(3')	1.48(2)	C(3')-C(4')	1.52(2)
O(1")-C(1")	1.26(2)	N(1")-C(1")	1.30(2)

N(1")-C(4")	1.47(2)	C(1")-C(2")	1.46(2)
C(2")-C(3")	1.51(2)	C(3")-C(4")	1.48(2)
Br(1)-Br(2)-Br(3)	177.9(1)	C(1)-N(1)-C(4)	115.3(14)
O(1)-C(1)-N(1)	122(2)	O(1)-C(1)-C(2)	129(2)
N(1)-C(1)-C(2)	109.9(13)	C(1)-C(2)-C(3)	102.4(13)
C(2)-C(3)-C(4)	104.7(14)	N(1)-C(4)-C(3)	103.8(14)
O(1')-N(1')-C(4')	113.9(14)	O(1')-C(1')-N(1')	125(2)
O(2')-C(1')-C(2')	122.7(13)	N(1')-C(1')-C(2')	113(2)
C(1')-C(2')-C(3')	102.6(12)	C(2')-C(3')-C(4')	105.2(14)
N(1')-C(4')-C(3')	104.3(12)	C(1")-N(1")-C(4")	114(2)
O(1")-C(1")-N(1")	127(2)	O(1")-C(1")-C(2")	123.6(14)
N(1")-C(1")-C(2")	110(2)	C(1")-C(2")-C(3")	105.7(14)
C(2")-C(3")-C(4")	100(2)	N(1")-C(4")-C(3")	104.1(13)

Table 19. Crystal data and details of the crystallographic analysis for OR42.

Formula	$C_{28}H_{32}O_6$
M	464.44
Space group	$P2_12_12_1$
a/angstrom	9.794(1)
b	11.980(2)
c	15.294(4)
α, β, γ /degrees	90.00
	90.00
	90.00
V/Å^3	1794.5(6)
Z	4
D_c	1.35
F(000)	764
μ(Mo-K_α)/cm^{-1}	9.59
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	0 to 13
k	0 to 16
l	0 to 21
I(obs)	2364
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	236
Maximum Δ/σ	0.001
Residual electron density/eÅ^{-3}	0.24
R	0.0445
R_w	-
Weighting scheme	unit weights

Table 20. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR42.

	x/a	y/b	z/c	U(eq)
O(1)	2096(2)	9863(2)	3803(1)	29(1)
O(2)	-2322(2)	11181(2)	3888(2)	39(1)
O(3)	5554(3)	9168(3)	3229(2)	57(1)
O(4)	-3197(3)	12815(2)	3046(2)	56(1)
O(5)	6286(3)	7928(3)	4208(3)	96(1)
O(6)	145(3)	11579(2)	5397(2)	48(1)
C(1)	1604(3)	11779(3)	2603(2)	28(1)
C(2)	029(4)	11872(3)	1754(2)	45(1)
C(3)	-680(4)	12185(3)	1867(2)	41(1)
C(4)	-1373(3)	11306(3)	2440(2)	34(1)
C(5)	-356(3)	10711(3)	3082(2)	27(1)
C(6)	-1300(3)	10275(3)	3789(2)	33(1)
C(7)	-628(3)	9936(3)	4638(2)	39(1)
C(8)	628(3)	10588(3)	4960(2)	34(1)
C(9)	1583(3)	10912(3)	4164(2)	28(1)
C(10)	774(3)	11520(3)	3419(2)	28(1)
C(11)	2860(3)	11565(3)	4452(2)	38(1)
C(12)	3968(3)	10674(3)	4572(3)	42(1)
C(13)	5639(3)	9751(3)	3997(2)	31(1)
C(14)	3930(3)	8671(3)	4237(3)	43(1)

C(15)	5378(4)	8479(4)	3920(3)	57(1)
C(16)	4304(3)	9798(3)	3059(2)	44(1)
C(17)	1942(4)	9866(3)	2642(2)	46(1)
C(18)	-2141(4)	10461(3)	1983(2)	45(1)
C(19)	-2394(3)	11756(3)	3128(2)	36(1)
C(20)	262(4)	12669(3)	3771(2)	38(1)

Table 21. Bond lengths (Å) and angles (°) of OR42.

O(1)-C(9)	1.461(3)	O(1)-C(13)	1.434(3)
O(2)-C(6)	1.484(4)	O(2)-C(15)	1.353(4)
O(3)-C(15)	1.352(5)	O(3)-C(16)	1.461(4)
O(4)-C(19)	1.207(4)	O(5)-C(15)	1.192(5)
O(6)-C(8)	1.441(4)	C(1)-C(2)	1.538(5)
C(1)-C(10)	1.553(4)	C(2)-C(3)	1.534(5)
C(3)-C(4)	1.547(5)	C(4)-C(5)	1.551(4)
C(4)-C(18)	1.547(5)	C(4)-C(19)	1.529(4)
C(5)-C(6)	1.515(4)	C(5)-C(10)	1.556(4)
C(6)-C(7)	1.512(4)	C(7)-C(8)	1.538(4)
C(8)-C(9)	1.585(4)	C(8)-C(17)	1.525(5)
C(9)-C(10)	1.567(4)	C(9)-C(11)	1.539(4)
C(10)-C(20)	1.555(4)	C(11)-C(12)	1.533(4)
C(12)-C(13)	1.530(4)	C(13)-C(14)	1.535(5)
C(13)-C(16)	1.539(4)	C(14)-C(15)	1.503(5)
C(9)-O(1)-C(13)	111.4(2)	C(6)-O(2)-C(15)	108.7(2)
C(15)-O(3)-C(16)	116.4(3)	C(2)-C(1)-C(10)	113.2(3)
C(1)-C(2)-C(3)	115.7(3)	C(2)-C(3)-C(4)	108.8(3)
C(3)-C(4)-C(5)	113.0(3)	C(3)-C(4)-C(18)	109.3(3)
C(5)-C(4)-C(18)	111.0(3)	C(3)-C(4)-C(19)	115.9(3)
C(5)-C(4)-C(19)	100.0(2)	C(18)-C(4)-C(19)	107.3(3)
C(4)-C(5)-C(6)	101.8(2)	C(4)-C(5)-C(10)	111.9(2)
C(6)-C(5)-C(10)	114.3(2)	O(2)-C(6)-C(5)	103.4(2)
O(2)-C(6)-C(7)	113.7(3)	C(5)-C(6)-C(7)	116.1(2)
C(6)-C(7)-C(8)	119.2(3)	O(6)-C(8)-C(7)	107.7(3)
O(6)-C(8)-C(9)	110.4(3)	C(7)-C(8)-C(9)	110.5(2)
O(6)-C(8)-C(17)	107.5(3)	C(7)-C(8)-C(17)	107.3(3)
C(9)-C(8)-C(17)	113.2(3)	O(1)-C(9)-C(8)	106.4(2)
O(1)-C(9)-C(10)	107.4(2)	C(8)-C(9)-C(10)	112.0(2)
O(1)-C(9)-C(11)	105.4(2)	C(8)-C(9)-C(11)	112.6(3)
C(10)-C(9)-C(11)	113.5(3)	C(1)-C(10)-C(5)	104.9(2)
C(1)-C(10)-C(9)	113.2(2)	C(1)-C(10)-C(20)	108.1(2)
C(1)-C(10)-C(20)	106.5(3)	C(5)-C(10)-C(20)	115.7(2)
C(9)-C(10)-C(20)	108.7(2)	C(9)-C(11)-C(12)	104.6(2)
C(11)-C(12)-C(13)	103.5(2)	O(1)-C(13)-C(12)	107.1(2)
O(1)-C(13)-C(14)	112.0(3)	C(12)-C(13)-C(14)	114.1(3)
O(1)-C(13)-C(16)	110.6(3)	C(12)-C(13)-C(16)	113.2(3)
C(14)-C(13)-C(16)	100.0(3)	C(13)-C(14)-C(15)	101.9(3)
O(3)-C(15)-O(5)	122.1(4)	O(3)-C(15)-C(14)	109.1(3)
O(5)-C(15)-C(14)	128.7(5)	O(3)-C(16)-C(13)	103.5(3)
O(2)-C(19)-C(4)	120.4(3)	O(2)-C(19)-C(4)	111.0(3)
O(4)-C(19)-C(4)	128.6(3)		

Table 23. Crystal data and details of the crystallographic analysis for OR28.

Formula	$C_{28}H_{38}O_5$
M	318.45
Space group	P1
a/angstrom	7.415(1)
b	7.540(2)
c	18.770(3)
α /degrees	101.82(2)
β	97.40(1)
γ	60.42(2)
V/Å^3	893.3(3)
Z	2
D_c	1.18
F(000)	348
μ(Mo-Kα)/cm^{-1}	0.43
Scan range/°	3. to 30
Scan type	$\omega/2\theta$
h	-9 to 9
k	-9 to 9
l	0 to 21
I(obs)	4345
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	416
Maximum Δ/σ	0.04
Residual electron density/eÅ^{-3}	0.26
R	0.0488
R_w	-
Weighting scheme	unit weights

Table 24. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR28.

	x/a	y/b	z/c	U(eq)
O(1)	7619(1)	7579(1)	7927(1)	57(1)
O(2)	1194(5)	-621(5)	8646(2)	71(1)
O(3)	-349(4)	-530(4)	7551(2)	58(1)
C(1)	2840(5)	4359(6)	6299(2)	47(1)
C(2)	2348(6)	4545(7)	5494(2)	59(1)
C(3)	2093(6)	6579(7)	5360(2)	57(1)
C(4)	3989(6)	6900(6)	5600(2)	50(1)
C(5)	4607(5)	6476(5)	6405(2)	39(1)
C(6)	6462(5)	6763(6)	6737(2)	52(1)
C(7)	6713(6)	6795(6)	7535(2)	52(1)
C(8)	5975(5)	5563(5)	7839(2)	38(1)
C(9)	5177(4)	4458(5)	7502(2)	34(1)
C(10)	4864(5)	4434(5)	6579(2)	36(1)
C(11)	4741(5)	2984(5)	7696(2)	39(1)
C(12)	2526(5)	4000(5)	7999(2)	46(1)
C(13)	2228(5)	2444(5)	8290(2)	41(1)
C(14)	1268(5)	1486(5)	7865(2)	40(1)
C(15)	765(5)	211(5)	8087(2)	39(1)
C(16)	3058(6)	2116(8)	9038(2)	60(1)
C(17)	5299(6)	5606(6)	8650(2)	51(1)
C(18)	5739(7)	5568(7)	5063(2)	63(1)
C(19)	3303(8)	9175(6)	5967(3)	73(1)
C(20)	6757(5)	2445(5)	6220(2)	44(1)
O(1')	-3569(4)	4083(4)	2955(2)	47(1)
O(2')	11555(4)	-2205(5)	3911(2)	57(1)
O(3')	12911(4)	-4124(4)	2555(2)	49(1)
C(1')	9164(5)	-1286(6)	1253(2)	43(1)
C(2')	2882(6)	-1959(6)	444(2)	51(1)
C(3')	905(6)	-2.44(7)	302(2)	59(1)
C(4')	-1071(6)	-129(7)	881(2)	55(1)
C(5')	-529(5)	908(5)	1391(2)	39(1)
C(6')	-2481(5)	2589(6)	1745(2)	44(1)
C(7')	-2099(5)	3028(6)	2547(2)	37(1)
C(8')	53(5)	2323(5)	2834(2)	36(1)
C(9')	1636(4)	1382(5)	2376(2)	34(1)

C(10')	1359(4)	848(5)	1554(2)	34(1)
C(11')	1795(5)	998(5)	2269(2)	40(1)
C(12')	5099(5)	-1006(5)	2945(2)	43(1)
C(13')	7161(5)	-1289(5)	3259(2)	38(1)
C(14')	8838(5)	-2398(5)	2846(2)	38(1)
C(15')	10995(5)	-2839(5)	3062(2)	39(1)
C(16')	7144(6)	-221(6)	4023(2)	62(1)
C(17')	282(5)	2901(5)	3649(2)	45(1)
C(18')	-1767(7)	1440(8)	75(2)	65(1)
C(19')	-2786(7)	-743(9)	567(3)	84(1)
C(20')	1337(5)	2571(5)	1220(2)	48(1)

Table 25. Bond lengths (Å) and angles (°) for OR28.

O(1)-C(7)	1.226(4)	O(2)-C(15)	1.187(5)
O(3)-C(15)	1.344(5)	C(1)-C(2)	1.527(6)
C(1)-C(10)	1.547(6)	C(2)-C(3)	1.531(6)
C(3)-C(4)	1.541(7)	C(4)-C(5)	1.556(5)
C(4)-C(18)	1.543(6)	C(4)-C(19)	1.538(6)
C(5)-C(6)	1.530(6)	C(5)-C(20)	1.550(6)
C(6)-C(7)	1.488(5)	C(7)-C(8)	1.474(6)
C(8)-C(9)	1.353(5)	C(8)-C(17)	1.506(5)
C(9)-C(10)	1.520(5)	C(9)-C(11)	1.520(6)
C(10)-C(20)	1.556(6)	C(11)-C(12)	1.551(5)
C(12)-C(13)	1.318(7)	C(13)-C(14)	1.337(6)
C(13)-C(16)	1.478(6)	C(14)-C(15)	1.477(6)
O(1')-C(1')	1.231(4)	O(2')-C(15')	1.189(5)
O(3')-C(15')	1.339(4)	C(1')-C(2')	1.524(5)
C(1')-C(10')	1.555(4)	C(2')-C(3')	1.525(7)
C(3')-C(4')	1.551(5)	C(4')-C(5')	1.559(5)
C(4')-C(18')	1.547(7)	C(4')-C(19')	1.549(9)
C(5')-C(6')	1.538(4)	C(5')-C(10')	1.556(5)
C(6')-C(7')	1.495(5)	C(7')-C(8')	1.477(4)
C(8')-C(9')	1.345(5)	C(8')-C(17')	1.514(5)
C(9')-C(10')	1.526(5)	C(9')-C(11')	1.520(5)
C(10')-C(20')	1.547(6)	C(11')-C(12')	1.543(5)
C(12')-C(13')	1.511(6)	C(13')-C(14')	1.342(4)
C(13')-C(16')	1.492(5)	C(14')-C(15')	1.489(5)
C(2)-C(1)-C(10)	112.7(4)	C(1)-C(2)-C(3)	110.4(4)
C(2)-C(3)-C(4)	114.4(4)	C(3)-C(4)-C(5)	108.4(4)
C(3)-C(4)-C(18)	110.1(3)	C(5)-C(4)-C(18)	114.6(3)
C(3)-C(4)-C(19)	106.8(3)	C(5)-C(4)-C(19)	109.6(3)
C(18)-C(4)-C(19)	107.1(4)	C(4)-C(5)-C(6)	114.7(4)
C(4)-C(5)-C(10)	117.5(3)	C(6)-C(5)-C(10)	109.4(3)
C(6)-C(5)-C(7)	111.8(4)	C(1)-C(7)-C(6)	120.6(4)
O(1)-C(7)-C(8)	120.3(4)	C(6)-C(7)-C(8)	118.9(4)
C(7)-C(8)-C(9)	120.1(3)	C(7)-C(8)-C(17)	115.6(4)
C(9)-C(8)-C(17)	124.2(4)	C(8)-C(9)-C(10)	123.2(4)
C(8)-C(9)-C(11)	119.1(3)	C(10)-C(9)-C(11)	117.1(3)
C(1)-C(10)-C(5)	106.6(3)	C(1)-C(10)-C(9)	110.7(3)
C(1)-C(10)-C(20)	109.2(3)	C(5)-C(10)-C(20)	109.6(3)
C(9)-C(10)-C(20)	114.6(3)	C(9)-C(11)-C(12)	108.2(3)
C(11)-C(12)-C(13)	113.2(4)	C(12)-C(13)-C(14)	110.4(4)
C(12)-C(13)-C(16)	118.4(3)	C(14)-C(13)-C(16)	115.9(4)
C(13)-C(14)-C(15)	125.7(5)	O(2)-C(15)-O(3)	125.3(6)
O(3)-C(15)-C(14)	122.3(4)	O(2)-C(15)-C(14)	127.8(4)
O(3)-C(15)-C(14)	110.0(3)	C(2')-C(1')-C(10')	112.4(3)
C(1')-C(2')-C(3')	110.7(4)	C(2')-C(3')-C(4')	113.5(4)
C(3')-C(4')-C(5')	107.9(3)	C(3')-C(4')-C(18')	110.9(4)
C(5')-C(4')-C(18')	115.7(4)	C(3')-C(4')-C(19')	106.1(4)
C(5')-C(4')-C(19')	107.9(4)	C(18')-C(4')-C(19')	108.1(4)
C(4')-C(5')-C(6')	113.4(2)	C(4')-C(5')-C(10')	116.7(3)
C(6')-C(5')-C(10')	109.0(3)	C(5')-C(6')-C(7')	111.1(2)
C(1')-C(7')-C(6')	120.1(3)	C(1')-C(7')-C(8')	120.7(3)
C(6')-C(7')-C(8')	119.1(3)	C(7')-C(8')-C(9')	120.3(3)
C(7')-C(8')-C(17')	115.3(3)	C(9')-C(8')-C(17')	124.3(3)
C(8')-C(9')-C(10')	123.2(3)	C(8')-C(9')-C(11')	119.3(3)
C(10')-C(9')-C(11')	117.2(3)	C(1')-C(10')-C(5')	105.8(3)
C(1')-C(10')-C(9')	110.6(2)	C(5')-C(10')-C(9')	109.1(3)
C(5')-C(10')-C(20')	110.1(3)	C(9')-C(10')-C(20')	115.1(3)
C(9')-C(10')-C(20')	106.2(3)	C(9')-C(11')-C(12')	113.9(3)
C(11')-C(12')-C(13')	110.3(3)	C(12')-C(13')-C(14')	119.1(3)
C(12')-C(13')-C(16')	115.7(3)	C(14')-C(13')-C(16')	125.3(3)
C(13')-C(14')-C(15')	126.1(3)	O(3')-C(15')-O(3')	122.8(3)
O(2')-C(15')-C(14')	127.1(3)	O(3')-C(15')-C(14')	110.4(3)

Table 27. Crystal data and details of the crystallographic analysis for OR09.

Formula	C_8H_9NO
M	135.17
Space group	$P2_1/c$
a/angstrom	7.077(1)
b	13.389(2)
c	7.933(1)
α /degrees	90.00
β	100.01(1)
γ	90.00
V/Å^3	740.2(2)
Z	4
D_c	1.21
F(000)	288
μ(Mo-Kα)/cm^{-1}	0.47
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-9 to 9
k	0 to 16
l	0 to 11
T(obs)	724
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	127
Maximum Δ/σ	0.01
Residual electron density/eÅ^{-3}	0.22
R	0.0656
R_w	0.0426
Weighting scheme	$N = 1/\sigma^2(F)$

Table 29. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR09.

	x/a	y/b	z/c	U(eq)
O	2404(3)	-400(2)	20(5)	71(1)
N	1444(4)	1213(2)	-41(5)	48(1)
C(1)	1866(5)	2255(3)	30(6)	45(1)
C(2)	570(5)	2916(3)	-898(6)	47(1)
C(3)	1061(6)	3923(3)	-822(6)	58(1)
C(4)	2763(6)	4253(3)	135(8)	68(1)
C(5)	4017(6)	3589(3)	1043(7)	65(1)
C(6)	3559(5)	2589(3)	1011(6)	54(1)
C(7)	-1290(6)	2869(4)	-1998(8)	90(1)
C(8)	2755(5)	486(3)	-4(7)	61(1)

Table 30. Bond lengths (Å) and angles (°) of OR09.

O-C(8)	1.213(4)	N-C(1)	1.426(4)
N-C(8)	1.342(4)	C(1)-C(2)	1.390(4)
C(1)-C(6)	1.384(4)	C(2)-C(3)	1.390(5)
C(2)-C(7)	1.501(5)	C(3)-C(4)	1.380(5)
C(4)-C(5)	1.369(5)	C(5)-C(6)	1.376(5)
C(1)-N-C(8)	124.7(3)	N-C(1)-C(2)	119.2(3)
N-C(1)-C(6)	119.5(3)	C(2)-C(1)-C(6)	121.2(3)
C(1)-C(2)-C(3)	117.2(3)	C(1)-C(2)-C(7)	121.9(4)
C(3)-C(2)-C(7)	120.9(4)	C(2)-C(3)-C(4)	121.5(4)
C(3)-C(4)-C(5)	120.3(4)	C(4)-C(5)-C(6)	119.6(4)
C(1)-C(6)-C(5)	120.1(4)	O-C(8)-N	124.5(4)

Table 28. Crystal data and details of the crystallographic analysis for OR10.

Formula	C_8H_9NO
M	135.17
Space group	$P2_1/c$
a/angstrom	12.778(3)
b	8.315(3)
c	7.093(1)
α /degrees	90.00
β	100.48(1)
γ	90.00
V/Å^3	741.1(3)
Z	4
D_c	1.21
F(000)	288
μ(Mo-Kα)/cm^{-1}	0.47
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-10 to 10
k	0 to 11
l	0 to 18
T(obs)	295
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	92
Maximum Δ/σ	0.001
Residual electron density/eÅ^{-3}	0.34
R	0.1104
R_w	0.0901
Weighting scheme	$N = 1/\sigma^2(F)$

Table 31. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR10.

	x/a	y/b	z/c	U(eq)
O	4711(5)	2770(8)	3707(8)	64(2)
N	4108(5)	275(9)	2603(9)	51(2)
C(1)	3010(6)	393(11)	2801(11)	48(2)
C(2)	2263(7)	-395(11)	1440(12)	55(2)
C(3)	1206(7)	-364(12)	1714(13)	68(3)
C(4)	894(7)	427(13)	3139(14)	76(3)
C(5)	1644(8)	1193(13)	4521(13)	81(3)
C(6)	2724(7)	1160(11)	4344(12)	62(3)
C(7)	2556(7)	-1218(12)	-286(12)	90(3)
C(8)	4930(7)	1477(12)	3047(11)	53(2)

Table 32. Bond lengths (Å) and angles (°) of OR10.

O-C(8)	1.194(9)	N-C(1)	1.439(9)
N-C(8)	1.358(10)	C(1)-C(2)	1.394(10)
C(1)-C(6)	1.378(10)	C(2)-C(3)	1.389(12)
C(2)-C(7)	1.508(11)	C(3)-C(4)	1.361(12)
C(4)-C(5)	1.368(12)	C(5)-C(6)	1.409(11)
C(1)-N-C(8)	123.9(8)	N-C(1)-C(2)	117.1(7)
N-C(1)-C(6)	121.0(7)	C(2)-C(1)-C(6)	121.8(7)
C(1)-C(2)-C(3)	116.8(8)	C(1)-C(2)-C(7)	122.5(8)
C(3)-C(2)-C(7)	120.6(9)	C(2)-C(3)-C(4)	122.8(9)
C(3)-C(4)-C(5)	119.4(8)	C(4)-C(5)-C(6)	120.6(9)
C(1)-C(6)-C(5)	118.7(8)	O-C(8)-N	128.6(8)

Table 33. Crystal data and details of the crystallographic analysis for OR26.

Formula	$C_{20}H_{17}NS$
M	151.23
Space group	Pccn
a/angstrom	15.381(3)
b	13.651(1)
c	7.639(9)
α /degrees	90.00
β	90.00
γ	90.00
V/Å^3	1645.9(1.9)
Z	8
D_c	1.22
F(000)	640
μ(Mo-Kα)/cm^{-1}	2.71
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	0 to 21
k	0 to 19
l	0 to 11
I(obs)	1914
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	127
Maximum σ/σ	0.05
Residual electron density/eÅ^{-3}	0.30
R	0.0447
R_w	--
Weighting scheme	unit weights

Table 34. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR26.

	x/a	y/b	z/c	U(eq)
S	3691(1)	1917(1)	-27(1)	57(1)
N	3650(2)	3815(2)	741(5)	42(1)
C(1')	3605(2)	4828(2)	329(5)	41(1)
C(2)	4085(2)	5501(2)	1280(5)	43(1)
C(3)	4053(2)	6481(2)	796(6)	56(1)
C(4)	3543(3)	6774(3)	-613(6)	69(1)
C(5)	3074(3)	6100(3)	-1514(6)	69(1)
C(6)	3089(2)	5127(2)	-1053(5)	56(1)
C(7)	4630(2)	5202(3)	2773(5)	63(1)
C(8)	3630(2)	3100(2)	-402(5)	46(1)

Table 35. Bond lengths (Å) and angles (°) of OR26.

S-C(8)	1.645(3)	N-C(1)	1.422(3)
N-C(8)	1.325(3)	C(1)-C(2)	1.393(4)
C(1)-C(6)	1.404(4)	C(2)-C(3)	1.399(4)
C(2)-C(7)	1.496(4)	C(3)-C(4)	1.401(5)
C(4)-C(5)	1.371(5)	C(5)-C(6)	1.377(4)
C(1)-N-C(8)	124.2(2)	N-C(1)-C(2)	119.6(2)
N-C(1)-C(6)	119.8(2)	C(2)-C(1)-C(6)	121.4(2)
C(1)-C(2)-C(3)	117.7(3)	C(1)-C(2)-C(7)	122.4(3)
C(3)-C(2)-C(7)	119.9(3)	C(2)-C(3)-C(4)	120.8(3)
C(3)-C(4)-C(5)	120.4(3)	C(4)-C(5)-C(6)	120.2(3)
C(1)-C(6)-C(5)	119.5(3)	S-C(8)-N	126.9(2)

Table 36. Crystal data and details of the crystallographic analysis for OR15.

Formula	$C_{12}H_{11}NO$
M	197.24
Space group	$P2_1$
a/angstrom	7.072(1)
b	7.509(4)
c	10.206(2)
α/degrees	90.00
β	91.15(1)
γ	90.00
V/A^3	541.9(3)
Z	2
D_c	1.21
F(000)	208
μ(Mo-Kα)/cm^{-1}	0.43
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-9 to 9
k	0 to 9
l	0 to 13
I(obs)	768
Cut-off criterion	$I > 1\sigma(I)$
Number of parameters	136
Maximum Δ/σ	0.01
Residual electron density/eA^{-3}	0.44
R	0.1429
R_w	0.0518
Weighting scheme	$W = 1/\sigma^2(F)$

Table 37. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($A^2 \times 10^3$) for non-hydrogen atoms of OR15.

	x/a	y/b	z/c	U(eq)
O	6319(8)	2791	9497(5)	61(2)
N	4401(7)	4989(11)	8686(5)	40(2)
C(1)	3672(12)	4322(14)	7432(7)	44(2)
C(2)	2303(11)	5173(14)	6816(8)	46(3)
C(3)	1737(10)	4576(15)	5577(9)	57(3)
C(4)	2697(12)	3139(15)	4990(8)	60(3)
C(5)	4248(11)	2352(15)	5636(8)	62(3)
C(6)	4851(10)	2943(16)	6844(7)	47(2)
C(7)	1299(10)	6629(13)	7415(8)	34(2)
C(8)	1825(11)	8402(14)	7137(7)	48(2)
C(9)	882(12)	9832(14)	7674(8)	64(3)
C(10)	-620(11)	9497(17)	8515(9)	61(3)
C(11)	-1106(11)	7803(16)	8803(8)	71(3)
C(12)	-161(12)	6348(14)	8284(8)	51(2)
C(13)	5485(11)	4218(12)	9581(9)	54(3)

Table 38. Bond lengths (Å) and angles (°) of OR15.

O-C(13)	1.227(8)	N-C(1)	1.417(7)
N-C(13)	1.316(9)	C(1)-C(2)	1.417(9)
C(1)-C(6)	1.398(9)	C(2)-C(3)	1.392(9)
C(2)-C(3)	1.446(10)	C(3)-C(4)	1.415(11)
C(4)-C(5)	1.381(9)	C(5)-C(6)	1.389(9)
C(7)-C(8)	1.413(10)	C(7)-C(12)	1.391(9)
C(8)-C(9)	1.394(9)	C(9)-C(10)	1.365(10)
C(10)-C(11)	1.381(11)	C(11)-C(12)	1.391(11)
C(1)-N-C(13)	127.7(7)	N-C(1)-C(2)	115.6(7)
N-C(1)-C(6)	122.6(8)	C(2)-C(1)-C(6)	121.8(7)
C(1)-C(2)-C(3)	117.8(8)	C(1)-C(2)-C(7)	122.7(7)
C(3)-C(2)-C(7)	119.5(8)	C(2)-C(3)-C(4)	119.9(9)
C(3)-C(4)-C(5)	121.3(9)	C(4)-C(5)-C(6)	119.6(8)
C(1)-C(6)-C(5)	119.4(8)	C(2)-C(7)-C(8)	119.6(7)
C(2)-C(7)-C(12)	122.2(8)	C(8)-C(7)-C(12)	118.2(7)
C(7)-C(8)-C(9)	120.5(7)	C(8)-C(9)-C(10)	119.2(9)

Table 36. Crystal data and details of the crystallographic analysis for OR15.

Formula	$C_{13}H_{11}NO$
M	197.24
Space group	$P2_1$
a/angstrom	7.872(1)
b	7.509(4)
c	10.204(2)
α/degrees	90.00
β	91.15(1)
γ	90.00
V/A^3	541.9(3)
Z	2
D_c	1.21
F(000)	208
μ(Mo-Kα)/cm^{-1}	0.43
Scan range/^{o}	3 to 30
Scan type	$\omega/2\theta$
h	-9 to 9
k	0 to 9
l	0 to 13
I(obs)	768
Cut-off criterion	$F > 1\sigma(F)$
Number of parameters	136
Maximum Δ/σ	0.01
Residual electron density/eA^{-3}	0.14
R	0.1429
R_w	0.0516
Weighting scheme	$w = 1/\sigma^2(F)$

Table 37. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors (A^2, $\times 10^3$) for non-hydrogen atoms of OR15.

	x/a	y/b	z/c	U(eq)
O	6319(8)	2791	9497(5)	61(2)
N	4401(7)	4989(11)	8685(5)	40(2)
C(1)	3872(12)	4322(14)	7432(7)	44(2)
C(2)	2303(11)	5173(14)	6813(8)	45(3)
C(3)	1737(10)	4576(15)	5577(9)	57(3)
C(4)	2697(12)	3139(15)	4990(8)	60(3)
C(5)	4248(11)	2362(15)	5594(8)	62(3)
C(6)	4851(10)	2943(16)	6824(7)	47(2)
C(7)	1299(10)	6629(13)	7415(8)	34(2)
C(8)	1825(11)	8402(14)	7197(7)	48(2)
C(9)	852(12)	9832(14)	7674(8)	64(3)
C(10)	-620(11)	9497(17)	8515(9)	61(3)
C(11)	-1166(11)	7863(18)	8803(8)	71(3)
C(12)	-161(12)	6348(14)	8284(8)	51(2)
C(13)	5465(11)	4218(12)	9581(9)	54(3)

Table 38. Bond lengths (A) and angles (^{o}) of OR15.

O-C(13)	1.227(8)	N-C(1)	1.417(7)
N-C(13)	1.316(9)	C(1)-C(2)	1.417(9)
N-C(1)-C(6)	1.398(9)	C(2)-C(3)	1.390(9)
C(2)-C(7)	1.446(10)	C(3)-C(4)	1.416(11)
C(4)-C(5)	1.381(9)	C(5)-C(6)	1.389(9)
C(7)-C(8)	1.413(10)	C(7)-C(12)	1.391(9)
C(8)-C(9)	1.394(9)	C(9)-C(10)	1.385(10)
C(10)-C(11)	1.351(11)	C(11)-C(12)	1.391(11)
C(1)-N-C(13)	127.7(7)	N-C(1)-C(2)	115.6(7)
N-C(1)-C(6)	122.6(8)	C(2)-C(1)-C(6)	121.8(7)
C(1)-C(2)-C(3)	117.8(8)	C(1)-C(2)-C(7)	122.7(7)
C(3)-C(2)-C(7)	119.5(8)	C(2)-C(3)-C(4)	119.9(9)
C(3)-C(4)-C(5)	121.3(9)	C(4)-C(5)-C(6)	119.6(8)
C(1)-C(6)-C(5)	119.4(8)	C(2)-C(7)-C(8)	119.6(7)
C(2)-C(7)-C(12)	122.2(8)	C(8)-C(7)-C(12)	118.2(7)
C(7)-C(8)-C(9)	120.8(7)	C(8)-C(9)-C(10)	119.2(9)

C(9)-C(10)-C(11)	120.2(9)	C(10)-C(11)-C(12)	122.1(8)
C(7)-C(12)-C(11)	119.5(8)	O-C(13)-N	127.6(9)

Table 39. Crystal data and details of the crystallographic analysis for OR04.

Formula	$C_{13}H_{12}N_2O_3Br$
M_r	349.20
Space group	$P2_1/c$
a/angstrom	12.636(2)
b	8.202(2)
c	15.205(9)
α /degrees	90.00
β	108.60(2)
γ	90.00
V/Å^3	1493.5(1.0)
Z	4
D_c	1.55
F(000)	708
μ(Mo-Kα)/cm^{-1}	26.78
Scan range/°	2 to 25
Scan type	ω-2θ
h	-15 to 15
k	0 to 9
l	0 to 18
I(obs)	931
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	91
Maximum Δ/σ	0.27
Residual electron density/eÅ^{-3}	0.66
R	0.098
R$_w$	0.063
Weighting scheme	$N = 1/\sigma^2(F)$

Table 42. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR04.

	x/a	y/b	z/c	U(eq)
Br	3553(2)	1431(3)	10140(1)	89(1)
O(1)	3874(9)	5265(15)	2330(7)	74(4)*
O(2)	-2091(13)	1363(18)	-817(9)	130(6)*
O(3)	-812(12)	2010(17)	-1421(10)	125(6)*
N(1)	2945(13)	3194(15)	3020(8)	58(5)*
N(2)	-1195(16)	1823(20)	-811(11)	90(6)*
C(1)	4914(13)	3277(17)	3057(10)	73(6)*
C(2)	3797(15)	4009(20)	2772(10)	56(5)*
C(3)	3100(11)	1696(19)	3518(10)	44(4)*
C(4)	3405(11)	1952(18)	4443(9)	43(4)*
C(5)	3529(14)	139(19)	4889(12)	65(5)*
C(6)	3436(13)	-1214(22)	4375(11)	69(5)*
C(7)	3169(13)	-1210(22)	3465(11)	73(5)*
C(8)	2997(14)	281(19)	2980(11)	65(6)*
C(9)	1794(13)	3950(18)	2778(10)	64(5)*
C(10)	1049(14)	3932(20)	1801(12)	80(6)*
C(11)	23(19)	2651(22)	1747(16)	110(8)*
C(12)	-647(17)	2106(22)	883(13)	93(7)*
C(13)	-388(16)	2329(21)	135(13)	71(6)*
C(14)	692(16)	3080(19)	157(11)	97(7)*
C(15)	1332(17)	3499(24)	1032(14)	108(7)*

* Isotropic temperature factor.

Table 45. Bond lengths (Å) and angles (°) of OR04.

O(1)-C(2)	1.21(2)	O(2)-N(2)	1.19(2)
O(3)-N(2)	1.19(2)	N(1)-C(2)	1.35(2)
N(1)-O(3)	1.42(2)	N(1)-C(9)	1.51(2)

N(2)-C(13)	1.53(2)	C(1)-C(2)	1.53(2)
C(3)-C(4)	1.34(2)	C(3)-C(8)	1.40(2)
C(4)-C(5)	1.40(2)	C(5)-C(6)	1.34(2)
C(6)-C(7)	1.32(2)	C(7)-C(8)	1.41(2)
C(9)-C(10)	1.57(2)	C(10)-C(11)	1.38(2)
C(10)-C(15)	1.34(2)	C(11)-C(12)	1.39(2)
C(12)-C(13)	1.29(2)	C(13)-C(14)	1.42(2)
C(14)-C(15)	1.39(2)		
C(2)-N(1)-C(3)	126(2)	C(2)-N(1)-C(9)	117.9(13)
C(3)-N(1)-C(9)	116.3(14)	O(2)-N(2)-O(3)	121(2)
O(2)-N(2)-C(13)	116(2)	O(3)-N(2)-C(13)	113(2)
N(1)-C(2)-N(1)	124(2)	N(1)-C(2)-C(1)	118(2)
N(1)-C(2)-C(1)	118(2)	N(1)-C(3)-C(4)	122(2)
N(1)-C(3)-C(8)	115.7(13)	C(4)-C(3)-C(8)	122(2)
C(3)-C(4)-C(5)	119(2)	C(4)-C(5)-C(6)	118(2)
C(5)-C(6)-C(7)	124(2)	C(6)-C(7)-C(8)	120(2)
C(3)-C(8)-C(7)	116(2)	N(1)-C(9)-C(10)	110.1(13)
C(9)-C(10)-C(11)	117(2)	C(9)-C(10)-C(15)	123(2)
C(11)-C(10)-C(15)	120(2)	C(10)-C(11)-C(12)	117(2)
C(11)-C(12)-C(13)	122(2)	N(2)-C(13)-C(12)	121(2)
N(2)-C(13)-C(14)	118(2)	C(12)-C(13)-C(14)	122(2)
C(13)-C(14)-C(15)	115(2)	C(10)-C(15)-C(14)	123(2)

Table 40. Crystal data and details of the crystallographic analysis for OR22.

Formula	$C_{16}H_{18}NO_2Cl_4$
M	434.17
Space group	$P2_1/n$
a/Angstrom	10.435(4)
b	9.181(1)
c	20.710(4)
α /degrees	90.00
β	92.50(3)
γ	90.00
V/Å^3	1982.2(9)
Z	4
D_c	1.45
F(000)	892
μ(Mo-Kα)/cm^{-1}	5.47
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-14 to 14
k	0 to 12
l	0 to 29
I(obs)	4283
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	303
Maximum δ/σ	0.03
Residual electron density/eÅ^{-3}	0.32
R	0.0410
R_w	0.0390
Weighting scheme	$w = 1/\sigma^2(F)$

Table 43. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors (Å^2, $\times10^3$) for non-hydrogen atoms of OR22.

	x/a	y/b	z/c	U(eq)
Cl(1)	1923(1)	-2472(1)	115(1)	58(1)
Cl(2)	1615(1)	5328(1)	-316(1)	82(1)
Cl(3)	4947(1)	-945(1)	750(1)	53(1)
Cl(4)	4966(1)	230(1)	2049(1)	70(1)
N	2326(2)	-1624(2)	1483(1)	42(1)
O(1)	3520(1)	-2895(2)	2219(1)	61(1)
O(2)	6709(2)	-2080(2)	2124(1)	69(1)
C(1)	2035(2)	971(2)	1273(1)	45(1)
C(2)	1783(2)	2130(2)	868(1)	49(1)
C(3)	1685(2)	1641(3)	210(1)	50(1)
C(4)	1718(2)	445(3)	-50.1	47(1)

C(5)	1913(2)	-686(2)	387(1)	42(1)
C(6)	2127(2)	-427(2)	1044(1)	40(1)
C(7)	1140(2)	-2336(3)	1700(1)	52(1)
C(8)	491(3)	-1485(4)	2212(1)	65(1)
C(9)	3440(2)	-2020(2)	1777(1)	44(1)
C(10)	4740(2)	-1577(2)	1587(1)	45(1)
C(11)	5845(2)	-2487(3)	1769(1)	53(1)
C(12)	5806(2)	-3960(2)	1486(1)	47(1)
C(13)	4899(3)	-4449(3)	1030(1)	54(1)
C(14)	4939(3)	-5839(3)	793(1)	64(1)
C(15)	5870(3)	-6776(3)	1008(2)	70(1)
C(16)	6777(3)	-6330(3)	1457(2)	74(1)
C(17)	6757(2)	-4941(3)	1698(1)	59(1)
C(18)	1585(4)	3634(3)	1143(2)	71(1)
C(19)	1541(3)	154(4)	-764(1)	63(1)

Table 46. Bond lengths (Å) and angles (°) of OR22.

Cl(1)-C(5)	1.734(2)	Cl(2)-C(3)	1.749(2)
Cl(3)-C(10)	1.758(2)	Cl(4)-C(10)	1.769(2)
N-C(6)	1.435(2)	N-C(7)	1.407(2)
N-C(9)	1.340(2)	O(1)-C(9)	1.217(2)
O(2)-C(11)	1.199(2)	C(1)-C(2)	1.379(3)
C(1)-C(6)	1.373(3)	C(2)-C(3)	1.389(3)
C(2)-C(16)	1.504(3)	C(3)-C(4)	1.392(3)
C(4)-C(5)	1.386(3)	C(4)-C(19)	1.506(3)
C(5)-C(6)	1.390(3)	C(7)-C(8)	1.504(3)
C(9)-C(10)	1.547(3)	C(10)-C(11)	1.572(3)
C(11)-C(12)	1.473(3)	C(12)-C(13)	1.379(3)
C(12)-C(17)	1.397(3)	C(13)-C(14)	1.376(3)
C(14)-C(15)	1.358(4)	C(15)-C(16)	1.361(4)
C(16)-C(17)	1.370(4)		
C(6)-N-C(7)	115.4(2)	C(6)-N-C(9)	126.5(2)
C(7)-N-C(9)	117.4(2)	C(2)-C(1)-C(6)	122.0(2)
C(1)-C(2)-C(3)	116.6(2)	C(1)-C(2)-C(16)	120.4(2)
C(3)-C(2)-C(16)	123.0(2)	C(2)-C(3)-C(2)	117.4(2)
Cl(2)-C(3)-C(4)	118.6(2)	C(2)-C(3)-C(4)	124.0(2)
C(3)-C(4)-C(5)	116.3(2)	C(3)-C(4)-C(19)	122.6(2)
C(5)-C(4)-C(19)	121.1(2)	Cl(1)-C(5)-C(4)	120.0(2)
Cl(1)-C(5)-C(6)	118.5(2)	C(4)-C(5)-C(6)	121.6(2)
N-C(6)-C(1)	120.5(2)	N-C(6)-C(5)	120.0(2)
C(1)-C(6)-C(5)	119.2(2)	N-C(7)-C(8)	112.9(2)
N-C(9)-O(1)	123.4(2)	N-C(9)-C(10)	122.3(2)
O(1)-C(9)-C(10)	114.3(2)	Cl(3)-C(10)-Cl(4)	108.5(1)
Cl(3)-C(10)-C(9)	108.3(1)	Cl(4)-C(10)-C(9)	106.1(1)
Cl(3)-C(10)-C(11)	104.8(1)	Cl(4)-C(10)-C(11)	109.4(1)
C(9)-C(10)-C(11)	109.5(2)	O(2)-C(11)-C(10)	118.0(2)
O(2)-C(11)-C(12)	122.5(2)	C(10)-C(11)-C(12)	119.5(2)
C(11)-C(12)-C(13)	124.9(2)	C(11)-C(12)-C(17)	117.4(2)
C(13)-C(12)-C(17)	117.8(2)	C(12)-C(13)-C(14)	120.8(2)
C(13)-C(14)-C(15)	120.4(3)	C(14)-C(15)-C(16)	120.0(3)
C(15)-C(16)-C(17)	120.5(3)	C(12)-C(17)-C(16)	120.5(3)

Table 41. Crystal data and details of the crystallographic analysis for OR01.

Formula	$C_{22}H_{18}NO_2F_2B$
M	405.30
Space group	$P2_1/n$
a/Angstrom	10.989(2)
b	14.644(3)
c	14.947(9)
α /degrees	90.00
β	100.86(5)
γ	90.00
V/Å^3	2159.4(1.5)
Z	4
D_c	1.25
F(000)	848
μ(Mo-Kα)/cm^{-1}	0.54
Scan range/θ	2 to 25
Scan type	$\omega/2\theta$
h	0 to 12
k	0 to 17
l	-16 to 16
I(obs)	2234
Cut-off criterion	$F > 2\sigma(F)$
Number of parameters	278
Maximum Δ/σ	0.05
Residual electron density/eÅ^{-3}	0.50
R	0.0980
R_w	0.0730
Weighting scheme	$w = 1/\sigma^2(F)$

Table 44. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR01.

	x/a	y/b	z/c	U(eq)
O(1)	10720(5)	75(3)	3400(3)	71(1)
O(2)	10959(4)	-969(3)	4370(3)	75(1)
N	8919(5)	841(4)	3382(4)	61(2)
F(1)	12666(4)	-293(4)	3066(3)	126(2)
F(2)	11570(5)	-1365(3)	3687(3)	123(2)
B	11484(9)	-528(7)	3032(7)	79(3)
C(1)	11038(6)	2381(6)	3947(6)	85(3)
C(2)	11411(6)	3290(7)	3819(7)	116(3)
C(3)	10634(11)	4000(7)	3979(7)	113(4)
C(4)	9496(9)	3851(7)	4252(7)	103(3)
C(5)	9122(7)	2944(6)	4370(5)	79(2)
C(6)	9879(6)	2226(5)	4207(5)	58(2)
C(7)	9469(7)	1269(4)	4344(5)	68(2)
C(8)	7641(7)	1115(5)	2916(5)	63(2)
C(9)	7528(8)	1831(5)	2248(5)	79(2)
C(10)	6300(11)	2101(6)	1835(6)	99(3)
C(11)	5256(9)	1685(7)	2087(7)	100(3)
C(12)	5392(9)	1001(6)	2757(7)	99(3)
C(13)	6613(9)	702(5)	3197(6)	87(3)
C(14)	6632(7)	26(5)	4051(6)	151(3)
C(15)	6648(13)	-909(6)	3620(8)	241(6)
C(16)	9573(7)	266(5)	2945(5)	61(2)
C(17)	9074(6)	-153(5)	2038(5)	62(2)
C(18)	9808(7)	-734(5)	1632(5)	60(2)
C(19)	9391(6)	-1146(5)	668(5)	63(2)
C(20)	9765(7)	-2044(6)	513(6)	89(3)
C(21)	9360(9)	-2453(7)	-392(7)	113(3)
C(22)	8608(9)	-1963(7)	-1134(7)	104(3)
C(23)	8255(7)	-1086(7)	-980(6)	86(3)
C(24)	8627(7)	-669(5)	-70(5)	68(2)

Table 47. Bond lengths (Å) and angles (°) of OR01.

O(1)-B	1.467(8)	O(1)-C(16)	1.302(6)
O(2)-B	1.440(8)	O(2)-C(18)	1.317(6)
N-C(7)	1.505(6)	N-C(8)	1.466(7)
N-C(16)	1.317(6)	F(1)-B	1.348(8)
F(2)-B	1.398(8)	C(1)-C(2)	1.411(10)
C(1)-C(6)	1.374(8)	C(2)-C(3)	1.378(10)
C(3)-C(4)	1.361(10)	C(4)-C(5)	1.400(8)
C(5)-C(6)	1.373(7)	C(6)-C(7)	1.491(7)
C(7)-C(9)	1.536(7)	C(8)-C(9)	1.537(6)
C(9)-C(10)	1.389(8)	C(10)-C(11)	1.376(9)
C(11)-C(12)	1.363(9)	C(12)-C(13)	1.404(8)
C(13)-C(14)	1.456(7)	C(14)-C(15)	1.395(7)
C(15)-C(17)	1.425(7)	C(17)-C(18)	1.353(7)
C(18)-C(19)	1.473(7)	C(19)-C(20)	1.404(7)
C(19)-C(24)	1.392(7)	C(20)-C(21)	1.395(8)
C(21)-C(22)	1.390(9)	C(22)-C(23)	1.367(9)
C(23)-C(24)	1.404(8)		
B-O(1)-C(16)	120.9(5)	B-O(2)-C(18)	121.2(5)
C(7)-N-C(8)	117.7(5)	C(7)-N-C(16)	122.2(5)
C(8)-N-C(16)	120.1(5)	O(1)-B-O(2)	115.1(6)
O(1)-B-F(1)	108.2(6)	O(2)-B-F(1)	110.9(6)
O(1)-B-F(2)	106.0(6)	O(2)-B-F(2)	106.3(7)
F(1)-B-F(2)	108.0(6)	C(2)-C(1)-C(6)	118.7(7)
C(1)-C(2)-C(3)	120.9(7)	C(2)-C(3)-C(4)	121.0(8)
C(3)-C(4)-C(5)	118.9(8)	C(4)-C(5)-C(6)	121.0(6)
C(1)-C(6)-C(5)	120.4(6)	C(1)-C(6)-C(7)	119.4(6)
C(5)-C(6)-C(7)	120.1(5)	N-C(7)-C(6)	110.5(5)
N-C(8)-C(9)	117.7(6)	N-C(8)-C(13)	116.8(6)
C(9)-C(8)-C(13)	125.3(6)	C(8)-C(9)-C(10)	116.7(7)
C(9)-C(10)-C(11)	121.0(7)	C(10)-C(11)-C(12)	121.1(8)
C(11)-C(12)-C(13)	120.2(8)	C(8)-C(13)-C(12)	117.6(7)
C(8)-C(13)-C(14)	125.4(7)	C(12)-C(13)-C(14)	116.5(7)
C(13)-C(14)-C(15)	106.2(7)	C(14)-C(15)-N	116.3(5)
C(14)-C(15)-C(17)	126.3(6)	N-C(15)-C(17)	117.4(5)
C(15)-C(17)-C(18)	119.9(5)	O(2)-C(18)-C(17)	121.8(5)
O(2)-C(18)-C(19)	115.2(5)	C(17)-C(18)-C(19)	123.1(6)
C(18)-C(19)-C(20)	118.7(6)	C(18)-C(19)-C(24)	121.1(6)
C(20)-C(19)-C(24)	120.2(6)	C(19)-C(20)-C(21)	119.6(7)
C(20)-C(21)-C(22)	119.8(7)	C(21)-C(22)-C(23)	120.2(7)
C(22)-C(23)-C(24)	120.5(7)	C(19)-C(24)-C(23)	119.5(6)

Table 50. Crystal data and details of the crystallographic analysis for OR44.

Formula	$C_{14}H_{12}$
M	216.30
Space group	Iba2
a/angstrom	15.334(2)
b	21.330(3)
c	7.596(8)
α/degrees	90.00
β	90.00
γ	90.00
V/Å^3	2484.5(2.7)
Z	8
D$_c$	1.17
F(000)	928
μ(Mo-Kα)/cm^{-1}	0.33
Scan range/°	2 to 30
Scan type	$\omega/2\theta$
h	0 to 10
k	0 to 30
l	0 to 21
I(obs)	1076
Cut-off criterion	$I > 2\sigma(I)$
Number of parameters	154
Maximum Δ/σ	0.02
Residual electron density/eÅ^{-3}	0.27
R	0.0801
R$_w$	
Weighting scheme	unit weights

Table 51. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2 \times 10^3$) for non-hydrogen atoms of OR44.

	x/a	y/b	z/c	U(eq)
C(1)	2037(4)	3038(3)	2473(2)	46(1)
C(2)	1944(5)	2711(3)	2798(14)	59(2)
C(3)	1351(5)	2339(4)	1808(15)	67(2)
C(4)	873(5)	2600(4)	493(15)	62(2)
C(5)	448(5)	3528(4)	-1236(13)	64(2)
C(6)	520(5)	4151(4)	-1569(14)	67(2)
C(7)	1021(5)	4533(4)	-611(14)	63(2)
C(8)	1600(5)	4268(3)	689(13)	49(2)
C(9)	962(4)	3294(3)	831(13)	50(2)
C(10)	1544(4)	3619(3)	1084(12)	41(1)
C(11)	2657(4)	3722(3)	3526(13)	50(2)
C(12)	3467(5)	3862(4)	3952(14)	56(2)
C(13)	4045(5)	4226(4)	3991(13)	66(2)
C(14)	3759(7)	4460(4)	5567(16)	79(3)
C(15)	2937(7)	4324(4)	6201(14)	76(2)
C(16)	2380(5)	3956(4)	5178(14)	64(2)
C(17)	3813(5)	3599(5)	1234(15)	84(3)

Table 52. Bond lengths (Å) and angles (°) of OR44.

C(1)-C(2)	1.367(9)	C(1)-C(10)	1.430(9)
C(1)-C(11)	1.480(9)	C(2)-C(3)	1.422(10)
C(3)-C(4)	1.359(11)	C(4)-C(5)	1.393(10)
C(5)-C(6)	1.356(10)	C(5)-C(9)	1.423(10)
C(6)-C(7)	1.399(11)	C(7)-C(8)	1.360(10)
C(8)-C(10)	1.420(8)	C(9)-C(10)	1.432(8)
C(11)-C(12)	1.378(9)	C(11)-C(16)	1.415(10)
C(12)-C(13)	1.399(10)	C(12)-C(17)	1.506(11)
C(13)-C(14)	1.390(12)	C(14)-C(15)	1.374(12)
C(15)-C(16)	1.397(11)		
C(2)-C(1)-C(10)	119.2(6)	C(2)-C(1)-C(11)	120.6(6)
C(10)-C(1)-C(11)	120.2(6)	C(1)-C(2)-C(3)	121.2(7)
C(2)-C(3)-C(4)	120.5(7)	C(3)-C(4)-C(5)	120.6(7)
C(6)-C(5)-C(9)	121.2(7)	C(5)-C(6)-C(7)	121.7(7)
C(6)-C(7)-C(8)	119.2(7)	C(7)-C(8)-C(10)	121.0(7)
C(4)-C(9)-C(5)	122.1(7)	C(4)-C(9)-C(10)	120.0(7)
C(5)-C(9)-C(10)	117.8(6)	C(1)-C(10)-C(8)	122.2(6)
C(1)-C(10)-C(9)	118.7(6)	C(8)-C(10)-C(9)	119.1(6)
C(1)-C(11)-C(12)	122.6(6)	C(1)-C(11)-C(16)	118.6(7)
C(12)-C(11)-C(16)	118.8(7)	C(11)-C(12)-C(13)	120.4(7)
C(11)-C(12)-C(17)	120.0(7)	C(13)-C(12)-C(17)	119.5(7)
C(12)-C(13)-C(14)	120.2(8)	C(13)-C(14)-C(15)	120.4(8)
C(14)-C(15)-C(16)	119.6(8)	C(11)-C(16)-C(15)	120.6(8)

Table 53. Crystal data and details of the crystallographic analysis for OR34.

Formula	$C_{15}H_{17}NO_5$
M	248.23
Space group	P2$_1$
a/angstrom	5.419(4)
b	6.979(8)
c	15.356(3)
α/degrees	90.00
β	95.53(4)
γ	90.00
V/Å^3	577.8(8)
Z	2
D_c	1.43
F(000)	260
μ(Mo-Kα)/cm^{-1}	0.69
Scan range/$^\circ$	3 to 30
Scan type	$\omega/2\theta$
h	-7 to 7
k	0 to 9
l	0 to 21
I(obs)	1380
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	172
Maximum Δ/σ	0.17
Residual electron density/eÅ^{-3}	0.29
R	0.0614
R_w	-
Weighting scheme	unit weights

Table 54. Fractional coordinates (x10^4) and equivalent isotropic temperature factors (Å^2, x10^3) for non-hydrogen atoms of OR34.

	x/a	y/b	z/c	U(eq)
O(1)	816(7)	1196(1)	4064(2)	51(1)
O(2')	-478(8)	-3959(10)	2239(3)	66(1)
O(3)	1424(8)	-7658(10)	1390(3)	63(1)
O(4)	1117(7)	-6145(9)	1222(2)	46(1)
O(5)	3932(8)	3968(9)	3984(3)	56(1)
C(1)	801(9)	-4408(10)	1687(3)	40(1)
C(2)	2953(9)	-3633(10)	1349(3)	36(1)
C(3)	3623(10)	-4957(10)	625(4)	42(1)
C(4)	1739(10)	-6470(10)	594(3)	40(1)
C(5)	4100(9)	-1992(10)	1542(3)	38(1)
C(6)	3980(9)	-445(10)	2108(3)	35(1)
C(7)	2284(9)	-418(10)	2815(3)	37(1)
C(8)	2338(9)	1069(11)	3401(3)	37(1)
C(9)	3997(10)	2576(10)	3355(3)	38(1)
C(10)	5664(10)	2571(10)	2716(3)	42(1)
C(11)	5618(9)	1073(11)	2143(4)	42(1)
C(12)	-668(11)	-447(11)	4208(4)	57(1)
C(13)	5703(13)	5497(12)	3983(4)	61(2)

Table 55. Bond lengths (Å) and angles ($^\circ$) of OR34.

O(1)-C(8)	1.373(5)	O(1)-C(12)	1.429(7)
O(2)-C(1)	1.188(6)	O(3)-C(4)	1.205(7)
O(4)-C(1)	1.437(6)	O(4)-C(4)	1.384(6)
O(5)-C(9)	1.362(6)	O(5)-C(13)	1.435(7)
C(1)-C(2)	1.439(6)	C(2)-C(3)	1.497(7)
C(2)-C(5)	1.347(7)	C(3)-C(4)	1.466(7)
C(5)-C(6)	1.456(7)	C(6)-C(7)	1.417(6)
C(6)-C(11)	1.385(7)	C(7)-C(8)	1.372(7)
C(8)-C(9)	1.398(7)	C(9)-C(10)	1.409(7)
C(10)-C(11)	1.366(8)		
C(8)-O(1)-C(12)	116.7(4)	C(1)-O(4)-C(4)	109.5(4)
C(9)-O(5)-C(13)	117.8(4)	O(2)-C(1)-O(4)	116.9(5)

O(2)-C(1)-C(2)	136.1(5)	O(4)-C(1)-C(2)	108.0(4)
O(2)-C(2)-C(3)	107.7(5)	C(1)-C(2)-C(5)	128.0(5)
C(3)-C(2)-C(5)	123.3(4)	C(2)-C(3)-C(4)	104.5(4)
O(3)-C(4)-O(4)	118.7(5)	O(3)-C(4)-C(3)	131.0(5)
O(4)-C(4)-C(3)	110.3(5)	C(2)-C(5)-C(6)	136.1(4)
C(5)-C(6)-C(7)	124.5(5)	C(5)-C(6)-C(11)	117.3(4)
C(7)-C(6)-C(11)	118.1(5)	C(6)-C(7)-C(8)	119.7(5)
C(7)-C(8)-C(9)	124.0(5)	O(1)-C(8)-C(9)	114.8(5)
C(7)-C(8)-O(1)	121.9(4)	O(5)-C(9)-C(8)	116.8(4)
O(5)-C(9)-C(10)	123.9(5)	C(8)-C(9)-C(10)	119.3(5)
C(9)-C(10)-C(11)	119.0(5)	C(6)-C(11)-C(10)	122.6(4)

Table 56. Crystal data and details of the crystallographic analysis for OR11.

Formula	$C_{21}H_{33}O_7Br$
M	477.30
Space group	$P2_12_12_1$
a/angstrom	17.984(7)
b	10.157(3)
c	11.227(8)
α/degrees	90.00
β	90.00
γ	90.00
V/Å^3	2158.0(2.1)
Z	4
D_c	1.44
F(000)	896
μ(Mo-Kα)/cm^{-1}	18.71
Scan range/θ	3 to 30
Scan type	$\omega/2\theta$
h	0 to 25
k	0 to 18
l	0 to 12
I(obs)	2410
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	312
Maximum Δ/σ	1.05
Residual electron density/eÅ^{-3}	0.70
R	0.0420
R_w	0.0360
Weighting scheme	$w = 1/\sigma^2(F)$

Table 57. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR11.

	x/a	y/b	z/c	U(eq)
Br(1)	-199(1)	-2333(1)	-2433(1)	76(1)
O(1)	1887(2)	-1206(3)	-2746(5)	49(1)
O(2)	933(3)	-1879(3)	-4911(5)	60(1)
O(3)	2563(3)	4915(4)	-3002(6)	80(2)
O(4)	3916(3)	4936(4)	-3875(5)	66(1)
O(5)	-45(3)	-664(4)	-364(5)	66(1)
O(6)	1634(3)	1[illegible](3)	-5641(5)	55(1)
O(7)	1770(3)	1[illegible](4)	-8024(5)	93(2)
C(1)	1422(3)	[illegible](4)	-2730(7)	44(2)
C(2)	475(3)	-1233(4)	-2561(10)	47(2)
C(3)	1510(5)	-2614(6)	-4713(9)	82(3)
C(4)	2963(3)	-1161(5)	-3741(7)	42(2)
C(5)	1429(3)	-329(5)	-3873(7)	40(2)
C(6)	2869(3)	2193(5)	-3738(6)	46(2)
C(7)	472(3)	-463(5)	-1482(8)	45(2)
C(8)	3596(4)	4050(5)	-3854(7)	54(2)
C(9)	954(4)	323(5)	-1542(7)	43(2)
C(10)	2491(4)	1193(4)	-3760(7)	41(2)
C(11)	3957(4)	3188(6)	-4236(9)	71(3)
C(12)	2154(4)	971(5)	-5250(8)	50(2)
C(13)	3599(4)	2254(7)	-4177(8)	64(2)
C(14)	2878(4)	4001(5)	-3374(8)	52(2)

C(15)	1895(4)	-192(4)	-5214(7)	46(2)
C(16)	2517(4)	3078(6)	-3374(9)	56(2)
C(17)	4609(5)	5114(7)	-4560(11)	85(3)
C(18)	-114(6)	200(7)	602(11)	97(3)
C(19)	1878(5)	4698(7)	-2278(12)	109(3)
C(20)	1007(4)	2683(7)	7325(11)	95(3)
C(21)	1502(5)	1767(6)	-7059(10)	71(3)

Table 58. Bond lengths (Å) and angles (°) of ORll.

Br(1)-C(2)	1.890(5)	O(1)-C(1)	1.373(6)
O(1)-C(10)	1.438(7)	O(2)-C(3)	1.421(7)
O(2)-C(4)	1.361(6)	O(3)-C(14)	1.372(7)
O(3)-C(19)	1.396(8)	O(4)-C(6)	1.373(7)
O(4)-C(17)	1.401(7)	O(5)-C(7)	1.389(6)
O(5)-C(16)	1.383(8)	O(6)-C(12)	1.459(7)
O(6)-C(21)	1.333(9)	O(7)-C(21)	1.207(8)
C(1)-C(5)	1.398(8)	C(1)-C(9)	1.375(8)
C(2)-C(4)	1.389(8)	C(2)-C(7)	1.395(8)
C(4)-C(6)	1.387(7)	C(5)-C(15)	1.494(7)
C(6)-C(10)	1.482(8)	C(6)-C(13)	1.373(8)
C(6)-C(16)	1.367(8)	C(7)-C(9)	1.369(8)
C(8)-C(11)	1.354(8)	C(8)-C(14)	1.394(8)
C(10)-C(12)	1.505(8)	C(11)-C(13)	1.389(9)
C(12)-C(15)	1.499(8)	C(14)-C(16)	1.377(8)
C(20)-C(21)	1.498(8)		

C(1)-O(1)-C(10)	117.2(5)	C(3)-O(2)-C(4)	113.7(6)
C(14)-O(3)-C(19)	117.8(6)	C(6)-O(4)-C(17)	118.5(6)
C(7)-O(5)-C(18)	116.1(6)	C(12)-O(6)-C(21)	117.9(6)
O(1)-C(1)-C(9)	120.9(7)	O(1)-C(1)-C(5)	115.1(6)
C(5)-C(1)-C(9)	124.0(6)	Br(1)-C(2)-C(4)	120.2(5)
Br(1)-C(2)-C(7)	119.8(6)	C(4)-C(2)-C(7)	120.0(5)
O(2)-C(4)-C(2)	119.1(6)	O(2)-C(4)-C(5)	120.6(6)
C(2)-C(4)-C(5)	120.2(6)	C(1)-C(5)-C(4)	117.1(6)
C(1)-C(5)-C(15)	122.4(6)	C(4)-C(5)-C(15)	120.5(6)
C(10)-C(6)-C(13)	118.8(7)	C(10)-C(6)-C(16)	123.5(6)
C(13)-C(6)-C(16)	117.5(7)	O(5)-C(7)-C(2)	116.0(6)
O(5)-C(7)-C(9)	122.7(6)	C(2)-C(7)-C(9)	121.2(6)
C(7)-C(8)-C(11)	123.9(7)	O(3)-C(8)-C(14)	116.3(7)
C(11)-C(8)-C(14)	119.7(7)	C(1)-C(9)-C(7)	117.3(6)
O(1)-C(10)-C(6)	108.6(5)	O(1)-C(10)-C(12)	108.6(5)
C(6)-C(10)-C(12)	115.8(5)	C(8)-C(11)-C(13)	120.7(7)
O(6)-C(12)-C(10)	105.7(6)	O(6)-C(12)-C(15)	113.2(6)
C(10)-C(12)-C(15)	111.7(6)	C(6)-C(13)-C(11)	120.4(7)
O(3)-C(14)-C(8)	116.4(6)	O(3)-C(14)-C(16)	125.4(7)
C(8)-C(14)-C(16)	119.1(7)	C(5)-C(15)-C(12)	109.4(5)
C(6)-C(16)-C(14)	122.4(6)	O(6)-C(21)-O(7)	123.3(7)
O(6)-C(21)-C(20)	112.8(8)	O(7)-C(21)-C(20)	123.9(8)

Table 59. Crystal data and details of the crystallographic analysis for OR02.

Formula	$C_{33}H_{30}O_7$
M	396.4
Space group	C2/c
a/angstrom	15.507(3)
b	8.936(1)
c	14.266(4)
α/degrees	90.00
β	109.66(2)
γ°	90.00
V/Å³	1861.7(7)
Z	8
D_c	1.41
F(000)	832
μ(Mo-Kα)/cm^{-1}	9.64
Scan range/°	3 to 25
Scan type	$\omega/2\theta$
h	-18 to 8
k	0 to 10
l	0 to 16
I(obs)	1274
Cut-off criterion	$I > 3\sigma(I)$
Number of parameters	174
Maximum δ/σ	0.005
Residual electron density/eÅ^{-3}	0.29
R	0.0507
R_w	0.0442
Weighting scheme	$W = 1/\sigma^2(F)$

Table 63. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR02.

	x/a	y/b	z/c	U(eq)
O(1)	5000	10728(3)	2500	51(1)
O(2)	6460(1)	10383(2)	3466(2)	58(1)
O(3)	4068(1)	2844(2)	3659(1)	47(1)
O(4)	5438(1)	1618(2)	4903(2)	49(1)
C(1)	3740(2)	9830(3)	3015(2)	46(1)
C(2)	5444(2)	8256(3)	2897(2)	41(1)
C(3)	5945(2)	7298(3)	3604(2)	43(1)
C(4)	5793(2)	5808(3)	3911(2)	40(1)
C(5)	4949(2)	5051(3)	3563(2)	39(1)
C(6)	4854(2)	3664(3)	3928(2)	39(1)
C(7)	5608(2)	2978(3)	4656(2)	42(1)
C(8)	6433(2)	3716(3)	4994(2)	43(1)
C(9)	6518(2)	5117(3)	4636(2)	43(1)
C(10)	3908(2)	9394(5)	2855(3)	56(1)
C(11)	6168(3)	892(5)	5726(3)	57(1)

Table 67. Bond lengths (Å) and angles (°) of OR02.

O(4)-C(7)	1.348(3)	O(4)-C(11)	1.435(4)
C(1)-C(2)	1.472(4)	C(2)-C(3)	1.351(4)
C(2)-C(2)	1.460(5)	C(3)-C(4)	1.445(4)
C(4)-C(5)	1.407(4)	C(4)-C(9)	1.390(4)
C(5)-C(6)	1.372(4)	C(6)-C(7)	1.415(4)
C(7)-C(8)	1.375(4)	C(8)-C(9)	1.375(4)
C(6)-O(3)-C(10)	117.4(2)	C(7)-O(4)-C(11)	117.6(2)
O(1)-C(1)-O(2)	120.2(3)	O(1)-C(1)-C(2)	108.8(2)
O(2)-C(1)-C(2)	131.1(3)	C(1)-O(1)-C(1)	109.5(3)
C(1)-C(2)-C(3)	116.2(3)	C(2)-C(3)-C(4)	134.3(3)
C(3)-C(4)-C(5)	124.9(3)	C(3)-C(4)-C(9)	117.5(3)
C(5)-C(4)-C(9)	118.2(3)	C(4)-C(5)-C(6)	120.4(3)
O(3)-C(6)-C(5)	125.4(3)	O(3)-C(6)-C(7)	114.5(2)
C(5)-C(6)-C(7)	120.1(3)	O(4)-C(7)-C(6)	115.2(2)
O(4)-C(7)-C(8)	125.4(3)	C(6)-C(7)-C(8)	119.4(3)

C(7)-C(8)-C(9)	120.0(3)	C(4)-C(9)-C(8)	121.8(3)

Table 60. Crystal data and details of the crystallographic analysis for OR08.

Formula	$C_{22}H_{18}O_7$
M	394.4
Space group	$P2_1/c$
a/angstrom	13.546(6)
b	9.141(8)
c	17.180(1)
α /degrees	90.00
β	101.05(6)
γ	90.00
V/A^3	1214.4(1.3)
Z	4
D_c	1.41
F(000)	8.24
μ(Mo-Kα)/cm^{-1}	0.05
Scan range/^{o}	3 to 25
Scan type	$\omega/2\theta$
h	-10 to 10
k	0 to 7
l	0 to 18
I(obs)	545
Cut-off criterion	$F > 3\sigma(F)$
Number of parameters	117
Maximum Δ/σ	0.005
Residual electron density/eA^{-3}	0.64
R	0.1492
R_w	-
Weighting scheme	unit weights

Table 64. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($\AA^2 \times 10^3$) for non-hydrogen atoms of OR08.

	x/a	y/b	z/c	U(eq)
O(1)	3871(18)	14069(33)	15618(16)	35(7)*
O(2)	4943(22)	13885(42)	16776(19)	65(10)*
O(3)	9435(19)	13093(34)	17642(16)	37(8)*
O(4)	8767(21)	11541(39)	16808(18)	58(9)*
O(5)	3101(19)	13810(37)	14346(16)	9(6)*
O(6)	7671(21)	8730(41)	13342(17)	56(9)*
O(7)	8684(23)	8662(47)	14720(18)	65(10)*
C(1)	4761(28)	13658(57)	16078(25)	37(11)*
C(2)	5334(41)	12713(77)	15641(33)	86(18)*
C(3)	6335(31)	11882(56)	15741(26)	44(13)*
C(4)	6869(33)	11972(61)	16598(27)	52(14)*
C(5)	7845(28)	12624(53)	16762(24)	41(11)*
C(6)	8481(24)	12389(47)	17426(19)	21(9)*
C(7)	8051(26)	11587(50)	18083(23)	29(11)*
C(8)	7098(23)	10869(44)	17957(19)	18(9)*
C(9)	6538(27)	11143(56)	17191(21)	32(10)*
C(10)	9885(45)	13768(92)	17046(35)	114(24)*
C(11)	8436(27)	10560(50)	19397(23)	34(11)*
C(12)	3740(30)	13527(63)	14849(26)	46(13)*
C(13)	4759(23)	12627(46)	14808(20)	20(8)*
C(14)	5114(24)	11993(43)	14257(20)	18(9)*
C(15)	6058(30)	11117(60)	14260(25)	43(12)*
C(16)	6368(26)	10299(53)	13689(26)	39(12)*
C(17)	7249(28)	9517(53)	13818(24)	36(11)*
C(18)	7842(32)	9487(60)	14675(26)	48(13)*
C(19)	7546(30)	10175(57)	15298(24)	43(13)*
C(20)	6606(22)	11099(47)	15133(17)	10(8)*
C(21)	7128(33)	8565(64)	12496(26)	59(15)*
C(22)	9332(37)	8332(69)	15545(30)	74(16)*

* Isotropic temperature factor.

Table 68. Bond lengths (Å) and angles (°) of OR08.

O(1)-O(1)	1.35(4)	O(1)-C(12)	1.37(4)
O(2)-O(1)	1.19(4)	O(3)-C(16)	1.40(4)
O(3)-C(10)	1.40(6)	O(4)-C(7)	1.45(4)
O(4)-C(11)	1.43(4)	O(5)-C(12)	1.12(4)
O(6)-C(17)	1.20(4)	O(6)-C(21)	1.50(5)
O(7)-C(18)	1.51(5)	O(7)-C(22)	1.54(5)
C(1)-C(2)	1.41(6)	C(2)-C(3)	1.50(6)
C(2)-C(19)	1.49(6)	C(3)-C(4)	1.51(6)
C(3)-C(20)	1.34(5)	C(4)-C(5)	1.40(5)
C(4)-C(9)	1.37(5)	C(5)-C(6)	1.31(5)
C(6)-C(7)	1.49(5)	C(7)-C(8)	1.40(4)
C(8)-C(9)	1.40(5)	C(12)-C(13)	1.58(5)
C(13)-C(14)	1.28(5)	C(14)-C(15)	1.46(5)
C(15)-C(16)	1.32(5)	C(15)-C(20)	1.54(5)
C(16)-C(17)	1.51(5)	C(17)-C(18)	1.54(5)
C(18)-C(19)	1.54(5)	C(19)-C(20)	1.46(5)
C(1)-O(1)-C(12)	115(3)	C(6)-O(3)-C(10)	116(3)
C(7)-O(4)-C(11)	113(3)	C(17)-O(6)-C(21)	118(4)
C(18)-O(7)-C(22)	119(4)	O(1)-C(1)-O(2)	123(4)
O(1)-C(1)-C(2)	110(4)	O(2)-C(1)-C(2)	126(4)
C(1)-C(2)-C(3)	140(5)	C(1)-C(2)-C(19)	107(5)
C(3)-C(2)-C(19)	113(5)	C(2)-C(3)-C(4)	110(5)
C(2)-C(3)-C(20)	120(5)	C(4)-C(3)-C(20)	129(4)
C(3)-C(4)-C(5)	118(4)	C(3)-C(4)-C(9)	123(4)
C(5)-C(4)-C(9)	118(4)	C(4)-C(5)-C(6)	124(4)
C(3)-C(6)-C(5)	126(4)	C(3)-C(6)-C(7)	117(3)
C(5)-C(6)-C(7)	116(3)	O(4)-C(7)-C(6)	112(3)
O(4)-C(7)-C(8)	123(4)	C(6)-C(7)-C(8)	125(3)
C(7)-C(8)-C(9)	112(3)	C(4)-C(9)-C(8)	126(4)
O(5)-C(12)-O(6)	128(4)	O(1)-C(12)-C(13)	104(3)
O(5)-C(12)-C(13)	128(4)	C(12)-C(13)-C(12)	104(4)
C(13)-C(14)-C(15)	123(4)	C(12)-C(13)-C(14)	133(4)
C(13)-C(14)-C(15)	129(4)	C(14)-C(15)-C(16)	129(4)
C(14)-C(15)-C(20)	107(4)	C(16)-C(15)-C(20)	124(4)
C(15)-C(16)-C(17)	121(4)	O(6)-C(17)-C(16)	130(4)
O(6)-C(17)-C(18)	113(4)	C(16)-C(17)-C(18)	117(4)
O(7)-C(18)-C(17)	111(4)	O(7)-C(18)-C(19)	124(4)
C(17)-C(18)-C(19)	125(4)	C(18)-C(19)-C(20)	116(4)
C(3)-C(20)-C(18)	127(4)	C(3)-C(20)-C(19)	117(4)
C(15)-C(20)-C(19)	116(4)		

Table 61. Crystal data and details of the crystallographic analysis for OR06.

Formula	$C_{22}H_{22}O_7$
Mr	398.4
Space group	$P2_12_12$
a/angstrom	4.971(2)
b	27.090(1)
c	7.268(3)
α/degrees	90.00
β	90.00
γ	90.00
V/Å³	959.2(6)
Z	2
Dc	1.37
F(000)	416
μ(Mo-Kα)/cm^{-1}	0.62
Scan range/σ	2 to 25
Scan type	$\omega/2\theta$
h	0 to 5
k	0 to 32
l	0 to 8
I(obs)	1001
Cut-off criterion	$F^2 > 1\sigma(F)$
Number of parameters	152
Maximum Δ/σ	0.05
Residual electron density/eÅ^{-3}	0.24
R	0.1949
R_w	0.0712
Weighting scheme	$N = 1/\sigma^2(F)$

Table 65. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2 \times 10^3$) for non-hydrogen atoms of OR08.

	x/a	y/b	z/c	U(eq)
O(1)	-5000	0	6837(15)	156(5)
O(2)	-2357(26)	645(4)	6520(11)	196(5)
O(3)	4755(12)	1920(2)	1106(8)	64(2)
O(4)	1868(13)	1945(2)	4086(9)	71(2)
C(1)	-3656(38)	354(5)	5781(16)	111(5)
C(2)	-4112(17)	201(3)	3843(12)	48(3)
C(3)	-2764(17)	390(3)	2346(13)	42(2)
C(4)	-870(22)	787(3)	2101(12)	56(3)
C(5)	875(23)	795(4)	516(13)	59(3)
C(6)	2800(17)	1141(3)	119(13)	44(2)
C(7)	3043(20)	1533(3)	1379(14)	53(3)
C(11)	6394(18)	1931(4)	-542(14)	64(3)
C(8)	1458(21)	1584(4)	2969(13)	54(3)
C(10)	190(24)	1993(4)	5656(15)	95(4)
C(5)	-391(17)	1162(3)	3332(14)	53(3)

Table 69. Bond lengths (Å) and angles (°) of OR08.

O(1)-C(1)	1.392(13)	O(2)-C(1)	1.144(13)
O(3)-C(7)	1.352(9)	O(3)-C(11)	1.440(9)
O(4)-C(8)	1.347(10)	O(4)-C(10)	1.415(9)
C(1)-C(2)	1.485(13)	C(2)-C(3)	1.365(10)
C(2)-C(2)	1.39(2)	C(3)-C(4)	1.435(11)
C(3)-C(9)	1.432(11)	C(4)-C(5)	1.413(11)
C(9)-C(8)	1.356(11)	C(8)-C(7)	1.405(11)
C(7)-C(6)	1.395(11)	C(6)-C(5)	1.382(10)
C(7)-O(3)-C(11)	118.6(7)	C(8)-O(4)-C(10)	117.8(8)
O(1)-C(1)-O(2)	118.5(11)	O(1)-C(1)-C(2)	105.1(11)
O(2)-C(1)-C(2)	136.0(13)	C(1)-O(1)-C(1)	113.1(13)
C(1)-C(2)-C(3)	125.5(9)	C(2)-C(3)-C(4)	123.4(8)
C(3)-C(4)-C(9)	119.8(8)	C(3)-C(4)-C(5)	126.6(9)
C(9)-C(4)-C(5)	113.5(9)	C(4)-C(9)-C(8)	126.3(9)
C(9)-C(8)-C(7)	116.3(8)	O(3)-C(7)-C(8)	123.0(9)
O(3)-C(7)-C(6)	115.3(9)	C(8)-C(7)-C(6)	121.7(9)
O(4)-C(6)-C(7)	116.7(9)	O(4)-C(6)-C(5)	124.1(9)
C(7)-C(6)-C(5)	119.2(10)	C(4)-C(5)-C(6)	122.8(9)

Table 62. Crystal data and details of the crystallographic analysis for OR41.

Formula	$C_{24}H_{28}NO_7$
M	247.29
Space group	$P2_1/c$
a/Angstrom	7.454(1)
b	16.402(2)
c	9.988(3)
α /degrees	90.00
β	96.02(2)
γ	90.00
V/Å^3	1214.4(4)
z	4
D_c	1.35
F(000)	528
μ(Mo-Kα)/cm^{-1}	0.56
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-10 to 10
k	0 to 22
l	0 to 14
I(obs)	2814
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	209
Maximum Δ/σ	0.03
Residual electron density/eÅ^{-3}	0.39
R	0.0716
R_w	-
Weighting scheme	unit weights

Table 66. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times10^3$) for non-hydrogen atoms of OR41.

	x/a	y/b	z/c	U(eq)
O(1)	-450(1)	-3126(6)	6966(3)	64(1)
O(2)	-202(2)	-1588(7)	7947(2)	84(2)
O(3)	1747(1)	-3660(6)	5416(2)	63(1)
O(4)	2306(2)	-2617(6)	6342(2)	64(1)
O(2')	-538(2)	-4426(6)	5831(3)	74(1)
O(3')	1224(1)	2184(5)	5324(2)	57(1)
O(4')	1364(1)	1764(6)	3885(2)	61(1)
C(1)	-142(2)	-2172(9)	7319(4)	57(2)
C(2)	1(2)	-2067(7)	6807(4)	41(2)
C(3)	598(2)	-1775(6)	7150(3)	40(2)
C(4)	1033(2)	-1945(7)	6914(3)	39(2)
C(5)	1345(2)	-1372(7)	7748(3)	40(2)
C(6)	1774(2)	-1525(8)	7241(3)	51(2)
C(7)	1900(2)	-2295(8)	6570(3)	48(2)
C(8)	1585(2)	-2907(7)	6059(3)	45(2)
C(9)	1164(2)	-2765(7)	6230(3)	39(2)
C(10)	1466(2)	-39(8)	4777(3)	60(2)
C(11)	2634(2)	-1689(10)	6824(4)	78(2)
C(1')	-328(2)	-3540(8)	6217(4)	57(2)
C(2')	77(2)	-2666(7)	6051(3)	45(2)
C(3')	170(2)	-2270(7)	5303(3)	44(2)
C(4')	468(2)	-1260(7)	4945(3)	42(2)
C(5')	538(2)	-1398(7)	4150(3)	50(2)
C(6')	826(2)	-416(7)	3777(3)	52(2)
C(7')	1062(2)	746(8)	4185(3)	52(2)
C(8')	987(2)	973(7)	4988(3)	43(2)
C(9')	692(2)	-26(6)	5340(3)	40(1)
C(10')	1147(2)	2530(9)	6111(3)	67(2)
C(11')	1466(2)	1564(11)	3092(3)	81(2)
C(12')	2391(3)	-171(12)	4131(5)	91(3)
C(2")	2225(3)	477(11)	485( )	95(3)
N	2519(4)	-671(12)	38( )	143(3)

Table 70. Bond lengths (Å) and angles (°) of OR41.

O(1)-C(1)	1.381(8)	O(1')-C(1')	1.401(7)
O(2)-C(1)	1.205(7)	O(3')-C(8')	1.371(7)
O(3)-C(10)	1.442(8)	O(4')-C(7')	1.358(7)
O(4)-C(11)	1.425(7)	O(2')-C(1')	1.180(7)
O(3')-C(6')	1.355(6)	O(5')-C(10')	1.421(6)
O(4')-C(7')	1.358(7)	O(4')-C(11')	1.446(6)
C(1)-C(2)	1.484(8)	C(2')-C(3')	1.325(7)
C(2)-C(2')	1.485(8)	C(3')-C(4')	1.441(8)
C(4)-C(5)	1.407(8)	C(4')-C(9')	1.420(7)
C(5)-C(6)	1.383(8)	C(6')-C(7')	1.380(8)
C(7)-C(8)	1.422(8)	C(8')-C(9')	1.371(8)
C(2')-C(3')	1.486(8)	C(2')-C(3')	1.357(7)
C(3')-C(4')	1.437(8)	C(4')-C(5')	1.407(7)
C(4')-C(9')	1.400(7)	C(5')-C(6')	1.379(8)
C(6')-C(7')	1.394(7)	C(8')-C(9')	1.432(7)
C(8')-C(9')	1.374(7)	C(1")-C(2")	1.452(11)
C(1")-N	1.119(10)		
C(7')-O(4')-C(11')	118.0(5)	C(8')-O(3')-C(10')	117.4(5)
O(1')-C(1')-O(2')	117.7(5)	O(1')-C(1')-C(2')	120.5(6)
O(1)-C(1)-C(2)	109.9(6)	O(2')-C(1')-C(2')	130.5(7)
O(2)-C(1)-C(2)	115.9(6)	C(1')-C(2')-C(2')	104.6(5)
C(2')-C(2')-C(2')	138.2(5)	C(2')-C(3')-C(4')	133.5(5)
C(3)-C(4)-C(5)	118.6(5)	C(3')-C(4')-C(9')	124.1(5)
C(3)-C(4)-C(9)	117.7(6)	C(5')-C(4')-C(9')	121.4(5)
C(5)-C(6)-C(7)	120.5(6)	C(4')-C(7')-C(6')	125.5(6)
O(4)-C(7)-C(8)	115.9(5)	C(6')-C(7')-C(8')	116.7(6)
C(6)-C(7)-C(7)	119.6(6)	O(3')-C(8')-C(9')	125.3(5)
C(7)-C(8)-C(9)	121.1(6)	C(4')-C(9')-C(8')	120.3(5)
O(1')-C(1')-O(2')	121.8(7)	O(1')-C(1')-C(2')	108.0(6)
C(2')-C(1')-C(2')	130.9(7)	C(2')-C(2')-C(1')	105.2(6)
C(2')-C(2')-C(3')	136.4(6)	C(1')-C(2')-C(3')	117.1(6)
C(2')-C(3')-C(4')	133.9(5)	C(3')-C(4')-C(5')	116.9(5)
C(3')-C(4')-C(9')	123.2(5)	C(5')-C(4')-C(9')	117.9(5)
C(4')-C(5')-C(6')	121.6(6)	C(5')-C(6')-C(7')	119.9(5)
C(4')-C(7')-C(6')	128.4(5)	C(4')-C(9')-C(8')	114.7(5)
C(6')-C(7')-C(8')	115.6(6)	O(3')-C(8')-C(7')	114.8(6)
O(3')-C(8')-C(9')	126.3(4)	C(7')-C(8')-C(9')	118.9(5)
C(4')-C(9')-C(8')	121.6(5)	C(2")-C(1")-N	180.0(1)

Table 72. Crystal data and details of the crystallographic analysis for OR37.

Formula	$C_{20}H_{14}O_m$
M	330.34
Space group	$P2_1/n$
a/angstrom	4.859(5)
b	11.761(2)
c	29.499(9)
α /degrees	90.00
β	91.07(5)
γ	90.00
U/Å³	1686.1(1.8)
Z	4
D_c	1.38
F(000)	704
μ(Mo-Kα)/cm^{-1}	0.59
Scan range/θ	3 to 30
Scan type	$\omega/2\theta$
h	-5 to 5
k	0 to 16
l	0 to 41
I(obs)	1926
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	227
Maximum Δ/σ	0.09
Residual electron density/eÅ^{-3}	0.52
R	0.1246
R_w	-
Weighting scheme	unit weights

Table 74. Fractional coordinates ($x10^4$) and equivalent isotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR37.

	x/a	y/b	z/c	U(eq)
O(1)	2589(20)	8327(6)	992(3)	61(2)
O(2)	-863(20)	9172(7)	572(3)	71(2)
O(3)	6105(22)	7863(7)	1473(3)	73(3)
O(4)	13537(18)	9668(6)	3218(3)	56(2)
O(5)	-7272(19)	13941(7)	197(3)	60(3)
C(1)	1071(27)	9279(10)	853(4)	51(3)
C(2)	1974(23)	10241(9)	1143(3)	39(2)
C(3)	859(23)	11284(9)	1154(3)	39(2)
C(4)	-1298(24)	11908(9)	885(3)	42(2)
C(5)	-2457(26)	12886(9)	1070(4)	50(3)
C(6)	-4464(26)	13539(9)	843(4)	52(3)
C(7)	-5342(26)	13206(9)	401(4)	47(3)
C(8)	-4279(30)	12255(10)	212(4)	61(3)
C(9)	-2269(26)	11583(10)	442(3)	51(3)
C(10)	-7952(31)	13740(11)	-278(4)	69(4)
C(11)	4585(26)	8595(9)	1331(4)	49(3)
C(12)	4227(22)	9775(8)	1452(3)	35(2)
C(13)	5615(21)	10399(9)	1776(3)	38(2)
C(14)	7760(23)	10132(9)	2133(3)	38(2)
C(15)	8874(23)	11071(9)	2365(3)	42(2)
C(16)	10744(24)	10958(9)	2724(3)	45(3)
C(17)	11640(23)	9899(9)	2862(3)	42(2)
C(18)	10638(25)	8926(10)	2631(4)	[illegible]
C(19)	8670(24)	9069(9)	2272(4)	[illegible]
C(20)	14528(30)	10617(10)	3479(4)	[illegible]

Table 76. Bond lengths (Å) and angles (°) of OR37.

O(1)-C(1)	1.374(12)	O(1)-C(11)	1.469(12)
O(2)-C(1)	1.218(12)	O(3)-C(1)	1.195(13)
O(4)-C(17)	1.386(11)	O(4)-C(20)	1.429(13)
O(5)-C(7)	1.378(11)	O(5)-C(10)	1.449(12)
C(1)-C(2)	1.476(14)	C(2)-C(3)	1.332(13)
C(2)-C(12)	1.482(13)	C(3)-C(4)	1.469(13)
C(4)-C(5)	1.389(14)	C(4)-C(9)	1.422(13)
C(5)-C(6)	1.379(13)	C(6)-C(7)	1.415(13)
C(7)-C(8)	1.35(2)	C(8)-C(9)	1.393(14)
C(11)-C(12)	1.443(14)	C(12)-C(13)	1.360(13)
C(13)-C(14)	1.472(12)	C(14)-C(15)	1.393(14)
C(14)-C(19)	1.380(14)	C(15)-C(16)	1.396(13)
C(16)-C(17)	1.359(14)	C(17)-C(18)	1.41(2)
C(18)-C(19)	1.397(13)		
C(1)-O(1)-C(11)	110.5(8)	C(17)-O(4)-C(20)	116.8(8)
C(7)-O(5)-C(10)	116.6(9)	O(1)-C(1)-O(2)	110.4(10)
O(1)-C(1)-C(2)	108.8(8)	O(2)-C(1)-C(2)	132.9(11)
O(2)-C(1)-O(3)	123.6(9)	C(1)-C(2)-C(12)	105.3(8)
O(3)-C(2)-C(12)	126.8(9)	C(2)-C(3)-C(4)	135.2(9)
C(3)-C(4)-C(5)	117.7(8)	C(3)-C(4)-C(9)	124.4(10)
C(3)-C(4)-C(9)	117.8(9)	C(4)-C(5)-C(6)	122.5(9)
C(5)-C(6)-C(7)	118.4(10)	O(5)-C(7)-C(6)	113.8(9)
O(5)-C(7)-C(8)	125.6(9)	C(6)-C(7)-C(8)	120.6(9)
C(7)-C(8)-C(9)	121.3(9)	C(4)-C(9)-C(8)	119.4(10)
O(1)-C(11)-C(9)	118.1(10)	O(1)-C(11)-C(12)	108.5(9)
C(9)-C(11)-C(12)	133.4(10)	C(2)-C(12)-C(11)	106.8(8)
C(11)-C(12)-C(13)	123.6(9)	C(11)-C(12)-C(13)	129.6(9)
C(12)-C(13)-C(14)	134.1(10)	C(13)-C(14)-C(15)	115.1(9)
C(13)-C(14)-C(19)	127.3(9)	C(15)-C(14)-C(19)	117.6(8)
C(14)-C(15)-C(16)	122.1(9)	C(15)-C(16)-C(17)	119.1(10)
O(4)-C(17)-C(16)	124.9(9)	O(4)-C(17)-C(18)	114.1(9)
C(16)-C(17)-C(18)	120.9(9)	C(17)-C(18)-C(19)	118.5(10)
C(14)-C(19)-C(18)	121.8(10)		

Table 73. Crystal data and details of the crystallographic analysis for OR38.

Formula	$C_{20}H_{14}O_6$
M	336.34
Space group	$P2_1/c$
a/angstrom	10.732(2)
b	18.984(3)
c	8.494(1)
α /degrees	90.0
β	104.12(9)
γ	90.00
V/Å^3	1674.5(5)
Z	4
D_c	1.33
F(000)	704
μ(Mo-Kα)/cm^{-1}	0.57
Scan range/°	3 to 30
Scan type	$\omega/2\theta$
h	-7 to 7
k	0 to 17
l	0 to 9
I(obs)	2388
Cut-off criterion	$F > 4\sigma(F)$
Number of parameters	227
Maximum Δ/σ	0.005
Residual electron density/eÅ^{-3}	0.18
R	0.0475
R_w	-
Weighting scheme	unit weights

Table 75. Fractional coordinates ($\times10^4$) and equivalent isotropic temperature factors (Å^2, $\times10^3$) for non-hydrogen atoms of OR38.

	x/a	y/b	z/c	U(eq)
O(1)	5999(2)	3859(1)	968(3)	79(1)
O(2)	4401(2)	8276(1)	1532(3)	64(1)
O(3)	3182(3)	7418(2)	2143(4)	147(1)
O(4)	8630(3)	4391(1)	492(3)	114(1)
O(5)	11247(2)	5560(1)	4305(3)	75(1)
O(6)	4179(4)	7575(2)	1660(5)	92(1)
C(1)	5911(3)	7151(2)	1759(4)	64(1)
C(2)	6305(3)	7656(1)	1663(3)	54(1)
C(3)	5642(3)	8346(2)	1335(3)	64(1)
C(4)	5165(3)	6442(2)	1700(4)	70(1)
C(5)	6002(3)	5910(1)	1334(3)	60(1)
C(6)	6863(3)	6037(1)	372(3)	64(1)
C(7)	7674(4)	5529(2)	39(3)	76(1)
C(8)	7658(4)	4858(2)	806(4)	78(1)
C(9)	6795(4)	4713(2)	1723(4)	74(1)
C(10)	5913(3)	5237(2)	1958(4)	71(1)
C(11)	8567(5)	3729(2)	1300(5)	119(1)
C(12)	7601(3)	7661(1)	1988(3)	54(1)
C(13)	8542(3)	7421(1)	2916(3)	49(1)
C(14)	9739(3)	7152(1)	2250(3)	58(1)
C(15)	10663(3)	6642(1)	2770(3)	61(1)
C(16)	10406(3)	6091(1)	3744(3)	56(1)
C(17)	9224(3)	6061(1)	4166(3)	55(1)
C(18)	8323(3)	6563(1)	3612(3)	51(1)
C(19)	8533(3)	7112(1)	2648(3)	50(1)
C(20)	12425(3)	5534(2)	3819(4)	77(1)

Table 77. Bond lengths (Å) and angles (°) of OR38.

O(1)-C(4)	1.186(3)	O(2)-C(1)	1.396(4)
O(2)-C(3)	1.390(3)	O(3)-C(1)	1.186(4)
O(4)-C(9)	1.364(4)	O(4)-C(12)	1.421(4)
O(5)-C(17)	1.389(3)	O(5)-C(20)	1.426(3)
C(1)-C(2)	1.474(4)	C(2)-C(3)	1.457(4)

C(2)-C(5)	1.355(4)	C(3)-C(4)	1.464(4)
C(3)-C(19)	1.349(3)	C(5)-C(6)	1.436(4)
C(6)-C(7)	1.399(4)	C(6)-C(11)	1.406(4)
C(7)-C(8)	1.367(4)	C(8)-C(9)	1.402(4)
C(9)-C(10)	1.361(4)	C(10)-C(11)	1.372(4)
C(10)-C(14)	1.449(3)	C(14)-C(15)	1.398(3)
C(14)-C(19)	1.412(3)	C(15)-C(16)	1.376(4)
C(16)-C(17)	1.399(3)	C(17)-C(18)	1.391(4)
C(18)-C(19)	1.367(3)		
C(1)-O(2)-C(4)	109.7(2)	C(9)-O(4)-C(12)	118.2(3)
C(17)-C(5)-C(20)	118.5(2)	O(2)-C(1)-O(3)	119.4(3)
O(2)-C(1)-C(2)	108.9(3)	O(3)-C(1)-C(2)	131.7(4)
C(1)-C(2)-C(3)	105.6(3)	C(1)-C(2)-C(5)	117.5(3)
C(3)-C(2)-C(5)	136.5(3)	C(2)-C(3)-C(4)	105.8(2)
C(2)-C(3)-C(19)	136.8(3)	C(4)-C(3)-C(19)	116.9(2)
O(1)-C(4)-O(2)	119.9(3)	O(1)-C(4)-C(3)	131.6(3)
O(2)-C(4)-C(3)	108.5(2)	C(2)-C(5)-C(6)	130.0(3)
C(5)-C(6)-C(7)	123.1(3)	C(5)-C(6)-C(11)	119.1(3)
C(7)-C(6)-C(11)	117.8(3)	C(6)-C(7)-C(8)	121.4(3)
C(7)-C(8)-C(9)	119.3(3)	O(4)-C(9)-C(8)	114.8(3)
O(4)-C(9)-C(10)	124.8(3)	C(8)-C(9)-C(10)	120.4(3)
C(9)-C(10)-C(11)	119.6(3)	C(6)-C(11)-C(10)	121.2(3)
C(3)-C(19)-C(14)	131.1(2)	C(19)-C(14)-C(15)	119.0(2)
C(13)-C(14)-C(19)	123.3(2)	C(15)-C(14)-C(19)	117.7(2)
C(14)-C(15)-C(16)	121.9(2)	C(15)-C(16)-C(17)	118.9(3)
C(5)-C(17)-C(16)	123.8(3)	C(5)-C(17)-C(18)	115.6(2)
C(16)-C(17)-C(18)	120.6(3)	C(17)-C(16)-C(19)	119.9(2)
C(14)-C(19)-C(18)	120.9(2)		

Anisotropic temperature factors ($Å^2$, $x10^3$)
for non-hydrogen atoms of OR26.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
S	93(1)	43(1)	35(1)	-4(1)	-7(1)	7(1)
N	58(1)	43(1)	28(1)	0(1)	-6(1)	0(1)
C(1)	47(1)	41(1)	34(1)	0(1)	3(1)	1(1)
C(2)	49(2)	47(1)	34(1)	-2(1)	5(1)	-4(1)
C(3)	65(2)	47(2)	56(2)	-4(1)	14(2)	-7(1)
C(4)	84(3)	49(2)	74(2)	19(2)	13(2)	9(2)
C(5)	76(2)	65(2)	65(2)	18(2)	-12(2)	11(2)
C(6)	61(2)	55(2)	51(2)	5(1)	-14(2)	2(2)
C(7)	78(3)	67(2)	44(2)	2(2)	-14(2)	-22(2)
C(8)	62(2)	47(1)	30(1)	-2(1)	-4(1)	3(1)

Fractional coordinates ($x10^3$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR26.

	x/a	y/b	z/c	U
H	373(2)	368(2)	161(4)	73(12)
H(3)	451(2)	696(2)	141(4)	74(10)
H(4)	348(2)	745(3)	-92(4)	84(11)
H(5)	265(2)	632(2)	-235(5)	93(13)
H(6)	269(2)	463(2)	-160(4)	57(9)
H(71)	501(2)	575(3)	305(5)	88(12)
H(72)	439(2)	501(3)	340(4)	67(14)
H(73)	502(2)	457(3)	254(5)	89(12)
H(8)	340(3)	335(3)	-156(5)	114(14)

Anisotropic temperature factors ($Å^2$, $x10^3$)
for non-hydrogen atoms of OR09.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O	42(2)	39(1)	133(3)	1(2)	10(2)	1(1)
N	29(2)	44(2)	70(3)	2(2)	3(2)	-4(2)
C(1)	38(2)	44(2)	53(3)	-2(2)	14(2)	-1(2)
C(2)	40(2)	50(3)	50(4)	1(2)	11(2)	3(2)
C(3)	53(3)	46(3)	72(4)	10(3)	15(3)	12(2)
C(4)	65(3)	43(3)	97(5)	-6(3)	24(3)	-10(2)
C(5)	49(3)	59(3)	86(4)	-8(3)	3(3)	-12(2)
C(6)	39(2)	51(3)	72(4)	5(3)	-3(2)	-1(2)
C(7)	45(2)	64(3)	71(4)	7(4)	-8(3)	1(3)
C(8)	35(3)	57(3)	90(4)	-2(3)	11(3)	3(2)

Fractional coordinates ($x10^3$; and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR09.

	x/a	y/b	z/c	U
H	39(4)	101(2)	-12(5)	4(1)
H(3)	10(4)	439(2)	-147(4)	6(1)
H(4)	310(4)	497(3)	12(5)	7(1)
H(5)	523(5)	383(2)	176(5)	7(1)
H(6)	440(4)	210(2)	176(4)	5(1)
H(71)	-221(5)	229(3)	-123(6)	9(2)
H(72)	-117(5)	200(3)	-271(5)	8(2)
H(73)	-191(5)	310(3)	-261(5)	8(2)
H(8)	421(4)	75(2)	-13(5)	6(1)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR24.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	56(1)	36(1)	52(1)	-6(1)	4(1)	-12(1)
O(2)	58(1)	32(1)	42(1)	2(1)	13(1)	2(1)
O(3)	56(1)	29(1)	46(1)	4(1)	3(1)	6(1)
N	38(1)	26(1)	35(1)	-1(1)	6(1)	0(1)
C(1)	35(1)	33(1)	36(1)	-6(1)	9(1)	-3(1)
C(2)	34(1)	26(1)	34(1)	-2(1)	6(1)	1(1)
C(3)	37(1)	42(2)	46(2)	-10(1)	8(1)	5(1)
C(4)	32(2)	58(2)	62(2)	-8(2)	4(1)	8(1)
C(5)	32(1)	52(2)	54(2)	-3(2)	-3(1)	-2(1)
C(6)	40(2)	37(1)	36(1)	-3(1)	-1(1)	-1(1)
C(7)	31(1)	27(1)	34(1)	-2(1)	3(1)	0(1)
C(8)	31(1)	34(1)	33(1)	1( )	11(1)	3(1)
C(9)	42(2)	39(2)	41(2)	5( )	14(1)	8(1)
C(10)	42(2)	57(2)	41(2)	12(1)	13(1)	17(1)
C(11)	31(1)	70(2)	37(2)	3(2)	6(1)	10(1)
C(12)	31(1)	54(2)	39(2)	-4(1)	6(1)	-1(1)
C(13)	29(1)	38(1)	34(1)	-1(1)	7(1)	1(1)
C(14)	46(2)	95(3)	45(2)	-1(2)	-5(2)	12(2)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR24.

	x/a	y/b	z/c	U
H(32)	-3009(4)	1468(2)	2768(3)	51(2)
H(51)	-5624(4)	944(2)	-463(4)	51(2)
H(61)	-2810(4)	1397(2)	-1180(3)	51(2)
H(62)	-2638(4)	350(2)	-785(3)	51(2)
H(12)	2580(4)	2000(2)	4733(3)	51(2)
H	-826(46)	-57(23)	1156(34)	51(2)
H(1)	519(47)	1584(22)	-714(35)	51(2)
H(2)	-726(48)	2999(23)	894(35)	51(2)
H(52)	-4586(46)	522(22)	1041(35)	51(2)
H(31)	-3094(46)	2503(22)	2584(34)	51(2)
H(41)	-4728(47)	2287(22)	361(35)	51(2)
H(42)	-5787(48)	1843(22)	1508(34)	51(2)
H(9)	-727(46)	-690(23)	3076(35)	51(2)
H(10)	2513(46)	-554(22)	5144(35)	51(2)
H(141)	4197(47)	245(23)	6836(36)	51(2)
H(142)	4670(51)	1185(23)	6500(37)	51(2)
H(143)	3367(49)	1109(23)	7114(37)	51(2)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR22.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Cl(1)	62(1)	60(1)	53(1)	-14(1)	7(1)	2(1)
Cl(2)	82(1)	80(1)	86(1)	40(1)	-5(1)	-4(1)
Cl(3)	48(1)	62(1)	49(1)	9(1)	7(1)	-1(1)
Cl(4)	85(1)	52(1)	73(1)	-18(1)	-3(1)	-2(1)
N	47(1)	40(1)	39(1)	4(1)	9(1)	0(1)
O(1)	67(1)	64(1)	50(1)	21(1)	10(1)	12(1)
O(2)	81(1)	80(1)	106(2)	-21(1)	-47(1)	9(1)
C(1)	50(1)	45(1)	41(1)	0(1)	6(1)	1(1)
C(2)	45(1)	44(1)	57(1)	6(1)	7(1)	3(1)
C(3)	41(1)	55(1)	55(1)	19(1)	4(1)	1(1)
C(4)	34(1)	65(1)	41(1)	9(1)	6(1)	2(1)
C(5)	38(1)	47(1)	49(1)	-1(1)	8(1)	2(1)
C(6)	39(1)	42(1)	38(1)	5(1)	6(1)	2(1)
C(7)	52(1)	49(1)	54(1)	7(1)	14(1)	-2(1)
C(8)	70(2)	68(2)	58(2)	9(1)	25(2)	6(2)
C(9)	54(1)	42(1)	37(1)	-2(1)	8(1)	7(1)
C(10)	52(1)	42(1)	39(1)	-3(1)	-1(1)	-1(1)
C(11)	52(1)	56(1)	51(1)	1(1)	-7(1)	2(1)
C(12)	45(1)	50(1)	46(1)	5(1)	7(1)	5(1)
C(13)	51(1)	50(1)	62(2)	-2(1)	1(1)	6(1)
C(14)	65(2)	60(2)	65(2)	-10(1)	14(1)	-5(1)
C(15)	92(2)	49(2)	68(2)	-3(1)	32(2)	7(2)
C(16)	82(2)	67(2)	72(2)	14(2)	21(2)	36(2)
C(17)	55(1)	70(2)	52(1)	6(1)	4(1)	12(1)
C(18)	85(2)	44(2)	86(3)	3(2)	12(2)	6(2)
C(19)	52(2)	96(2)	40(1)	8(1)	2(1)	7(2)

Fractional coordinates ($x10^3$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR22.

	x/a	y/b	z/c	U
H(1)	210(2)	-117(2)	170(1)	48(6)
H(71)	142(2)	-330(2)	185(1)	58(6)
H(72)	52(2)	-249(2)	130(1)	65(7)
H(81)	98(2)	-148(3)	257(1)	61(8)
H(82)	-25(2)	-189(3)	231(1)	72(8)
H(83)	23(2)	-51(3)	207(1)	63(7)
H(13)	431(2)	-387(2)	86(1)	52(7)
H(14)	434(2)	-608(3)	49(1)	69(8)
H(15)	591(2)	-763(3)	86(1)	65(8)
H(16)	733(2)	-690(3)	162(1)	79(9)
H(17)	735(2)	-457(2)	198(1)	49(6)
H(181)	81(2)	411(3)	99(1)	127(10)
H(182)	164(4)	369(4)	156(2)	140(20)
H(183)	204(3)	433(3)	97(1)	104(11)
H(191)	155(2)	108(3)	-99(1)	97(11)
H(192)	75(3)	-20(3)	-89(1)	101(11)
H(193)	208(3)	-41(3)	-89(1)	106(13)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR15.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O	61(4)	65(4)	56(4)	15(4)	-12(3)	21(4)
N	44(4)	41(4)	36(4)	-2(4)	-7(3)	12(4)
C(1)	56(6)	38(6)	40(5)	-3(5)	7(5)	1(5)
C(2)	49(6)	37(5)	49(7)	12(6)	2(5)	-13(5)
C(3)	39(5)	76(8)	56(7)	-11(6)	-10(5)	1(6)
C(4)	68(7)	71(7)	42(5)	-10(6)	-8(5)	-13(7)
C(5)	79(8)	56(7)	50(6)	-19(5)	36(5)	-11(5)
C(6)	42(6)	55(6)	45(5)	6(4)	6(5)	12(6)
C(7)	26(5)	48(5)	29(5)	-10(5)	-12(4)	8(5)
C(8)	51(6)	47(6)	47(5)	4(4)	-2(4)	0(5)
C(9)	80(7)	52(6)	58(6)	-16(6)	-9(6)	18(7)
C(10)	51(6)	76(8)	56(6)	-13(6)	-8(5)	15(7)
C(11)	60(6)	71(8)	83(7)	-4(8)	26(5)	6(6)
C(12)	47(5)	50(6)	56(6)	7(5)	10(5)	-7(5)
C(13)	47(6)	62(7)	52(6)	7(6)	11(5)	-3(6)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR15.

	x/a	y/b	z/c	U
H	3849(7)	6284(11)	8934(5)	88(8)
H(3)	566(10)	5209(15)	5066(9)	88(8)
H(4)	2207(12)	2646(15)	4048(8)	88(8)
H(5)	4988(11)	1283(15)	5117(8)	88(8)
H(6)	6066(10)	2342(16)	7305(7)	88(8)
H(8)	2996(11)	8653(14)	6499(7)	88(8)
H(9)	1240(12)	11183(14)	7438(8)	88(8)
H(10)	-1381(11)	10593(17)	8942(9)	88(8)
H(11)	-2264(11)	7572(18)	9455(8)	88(8)
H(12)	-558(12)	5010(14)	8555(8)	88(8)
H(13)	5662(11)	4923(12)	10497(9)	88(8)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR12.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Cl(2)	33(1)	57(1)	61(1)	9(1)	10(1)	6(1)
Cl(1)	54(1)	45(1)	69(1)	-5(1)	3(1)	7(1)
Cl(4)	60(1)	85(1)	60(1)	25(1)	27(1)	45(1)
Cl(1)	41(1)	134(1)	85(1)	53(1)	19(1)	43(1)
N	42(2)	44(2)	37(2)	8(2)	2(2)	20(2)
C(12)	31(2)	41(2)	44(2)	18(2)	0(2)	14(2)
C(3)	29(2)	39(2)	35(2)	12(2)	3(2)	15(2)
C(4)	28(2)	38(2)	36(2)	11(2)	2(2)	14(2)
C(5)	33(2)	47(2)	36(2)	17(2)	6(2)	20(2)
C(15)	44(2)	61(3)	43(2)	26(2)	13(2)	31(2)
C(1)	37(2)	39(2)	38(2)	6(2)	-1(2)	13(2)
C(10)	49(3)	40(3)	53(3)	8(2)	-7(2)	16(2)
C(2)	29(2)	43(2)	37(2)	11(2)	4(2)	13(2)
C(13)	31(2)	47(3)	58(3)	25(2)	1(2)	13(2)
C(16)	40(3)	50(3)	87(4)	24(3)	0(3)	5(2)
C(14)	34(2)	64(3)	53(3)	35(2)	9(2)	21(2)
C(6)	33(2)	39(2)	37(2)	12(2)	-1(2)	16(2)
C(8)	65(3)	55(3)	48(3)	10(2)	8(3)	36(3)
C(7)	41(3)	50(3)	41(3)	13(2)	2(2)	22(2)
C(11)	36(2)	38(2)	45(2)	14(2)	-4(2)	14(2)
C(9)	76(4)	45(3)	49(3)	2(2)	0(3)	32(3)
C(17)	37(3)	86(4)	71(4)	44(3)	16(2)	22(3)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR12.

	x/a	y/b	z/c	U
H(5)	-2917(5)	-4448(6)	-196(7)	123(21)
H(7)	-4140(5)	-3889(6)	965(7)	122(21)
H(6)	-3134(5)	-4745(6)	1743(7)	140(28)
H(8)	-4203(56)	-2827(69)	3537(66)	62(20)
H(2)	2662(42)	-3072(45)	-2486(48)	46(11)
H(1)	3142(53)	-628(58)	-479(60)	43(16)
H(4)	-1173(44)	-4638(49)	-1220(48)	54(11)
H(9)	-4112(71)	-1151(80)	4676(82)	94(25)
H(3)	453(40)	-4889(22)	-2546(34)	29(11)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR11.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Br(1)	77(1)	67(1)	84(1)	6(1)	-3(1)	-21(1)
O(1)	60(3)	42(2)	46(3)	0(3)	9(3)	-2(2)
O(2)	76(3)	48(3)	57(3)	-14(3)	8(3)	-6(3)
O(3)	69(3)	55(3)	118(5)	-5(3)	53(4)	-1(3)
O(4)	65(4)	75(3)	58(3)	-5(3)	15(3)	-25(3)
O(5)	79(4)	64(3)	54(3)	1(3)	16(3)	-3(3)
O(6)	60(3)	58(3)	47(3)	3(3)	1(3)	11(3)
O(7)	129(5)	100(5)	50(4)	0(3)	-1(3)	44(4)
C(1)	40(4)	37(4)	57(6)	10(4)	-2(5)	8(3)
C(2)	46(4)	34(3)	62(4)	14(5)	-14(5)	3(3)
C(3)	103(7)	47(5)	99(7)	-11(6)	6(6)	29(6)
C(4)	34(4)	45(4)	45(5)	10(4)	-6(4)	9(4)
C(5)	48(4)	35(4)	39(4)	10(4)	-4(4)	3(4)
C(6)	46(5)	48(4)	45(4)	-1(4)	6(4)	9(4)
C(7)	40(4)	49(5)	47(5)	11(4)	6(4)	7(4)
C(8)	58(5)	54(5)	49(5)	2(4)	9(4)	-6(5)
C(9)	40(4)	48(4)	41(5)	5(4)	0(4)	9(4)
C(10)	47(4)	39(4)	38(4)	-3(4)	-6(4)	3(4)
C(11)	53(6)	74(6)	65(7)	-1(5)	22(5)	-8(5)
C(12)	37(4)	56(5)	58(6)	8(4)	25(5)	17(4)
C(13)	60(6)	57(5)	74(6)	-19(5)	18(5)	10(5)
C(14)	54(5)	48(5)	53(5)	0(4)	8(4)	-2(4)
C(15)	55(4)	46(4)	44(5)	1(4)	0(4)	6(4)
C(16)	38(5)	62(5)	70(6)	2(4)	9(5)	-12(5)
C(17)	78(8)	103(8)	74(7)	5(7)	20(7)	-29(6)
C(18)	111(10)	102(8)	77(7)	1(7)	45(8)	-9(7)
C(19)	92(7)	91(6)	146(10)	-28(9)	65(9)	15(6)
C(20)	95(5)	84(6)	105(7)	38(8)	10(8)	42(6)
C(21)	74(6)	57(5)	81(8)	3(5)	0(6)	0(4)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR11.

	x/a	y/b	z/c	U
H(161)	1362(5)	-3108(6)	-5612(9)	93(29)
H(162)	1558(5)	-3054(6)	-3720(9)	84(22)
H(163)	2036(5)	-2250(6)	-4945(9)	149(40)
H(2)	2911(4)	650(4)	-3458(7)	21(14)
H(14)	4550(4)	3187(6)	-4436(9)	104(20)
H(41)	1556(4)	-311(4)	-6175(7)	82(23)
H(42)	2350(4)	-729(4)	-5213(7)	45(17)
H(191)	4709(5)	5923(7)	-4551(11)	120(30)
H(192)	4657(5)	4832(7)	-5667(11)	117(30)
H(193)	5013(5)	4740(7)	-3871(11)	64(32)
H(171)	-557(6)	-34(7)	1328(11)	100(27)
H(172)	-260(6)	913(7)	93(11)	132(33)
H(173)	394(6)	290(7)	1219(11)	109(34)
H(181)	2122(5)	5574(7)	-1811(12)	282(52)
H(182)	1613(5)	4460(7)	-1424(12)	215(44)
H(183)	1469(5)	5110(7)	-3090(12)	691(90)
H(211)	752(4)	2864(7)	-8365(11)	85(21)
H(212)	1271(4)	3354(7)	-6884(11)	348(62)
H(213)	585(4)	2420(7)	-6572(11)	92(33)
H(15)	3629(23)	1632(30)	-4401(50)	16(13)
H(11)	2097(22)	3013(33)	-3152(49)	10(17)
H(3)	2594(21)	901(29)	-5850(42)	0(13)
H(9)	985(21)	1009(26)	-589(44)	20(11)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR08.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	247(18)	157(11)	65(8)	0	0	-159(12)
O(2)	293(15)	227(12)	67(6)	33(7)	-65(8)	-207(11)
O(3)	69(5)	48(4)	75(4)	-1(5)	15(4)	-8(4)
O(4)	87(5)	53(5)	73(5)	-24(4)	21(5)	-9(4)
C(1)	146(14)	124(13)	64(10)	18(9)	-27(10)	-44(11)
C(2)	47(7)	56(7)	41(5)	-9(5)	-8(6)	-1(5)
C(3)	11(6)	50(6)	66(7)	3(5)	-7(5)	1(5)
C(4)	88(9)	47(7)	33(6)	0(6)	6(6)	17(6)
C(9)	92(9)	40(7)	44(6)	-4(5)	-11(7)	-1(6)
C(8)	22(5)	51(6)	59(7)	10(6)	2(5)	-5(5)
C(7)	57(7)	34(5)	69(7)	10(6)	0(7)	-11(5)
C(11)	47(7)	75(7)	71(7)	4(7)	17(6)	5(7)
C(6)	57(8)	61(8)	43(7)	4(6)	9(6)	15(6)
C(10)	162(12)	71(8)	53(7)	-19(7)	44(9)	-12(8)
C(5)	51(7)	46(6)	61(7)	8(6)	-2(7)	-1(6)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR08.

	x/a	y/b	z/c	U
H(9)	657(23)	496(4)	-455(13)	178(15)
H(111)	7165(18)	2291(4)	-173(14)	178(15)
H(112)	4933(18)	1967(4)	-1652(14)	178(15)
H(113)	8077(18)	1700(4)	-979(14)	178(15)
H(101)	758(24)	2290(4)	6569(13)	178(15)
H(102)	292(24)	1647(4)	6388(13)	178(15)
H(103)	-1877(24)	2051(4)	5164(13)	178(15)
H(5)	-1524(17)	1193(3)	4608(14)	178(15)
H(3)	-4010(214)	192(32)	413(123)	178(15)
H(8)	4121(195)	1306(33)	-747(139)	178(15)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR07.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Cl(4)	65(1)	42(1)	79(1)	9(1)	13(1)	10(1)
Cl(3)	56(1)	62(1)	83(1)	20(1)	13(1)	22(1)
Cl(2)	48(1)	75(1)	68(1)	-17(1)	6(1)	-9(1)
Cl(1)	117(1)	39(1)	127(1)	-8(1)	39(1)	-20(1)
Cl(5)	128(1)	132(1)	31(1)	-5(1)	20(1)	17(1)
C(10)	51(3)	48(3)	37(3)	-4(3)	9(2)	-6(3)
C(7)	30(2)	54(3)	48(3)	14(3)	10(2)	-2(2)
(0)	106(3)	66(3)	133(4)	53(3)	48(3)	1(2)
C(6)	31(2)	39(3)	44(3)	-3(2)	6(2)	-2(2)
C(4)	56(3)	36(3)	73(3)	10(2)	18(3)	5(2)
C(19)	40(3)	44(3)	47(3)	2(2)	11(2)	6(2)
C(4)	27(2)	40(3)	30(2)	0(2)	4(2)	-2(2)
C(9)	33(2)	48(3)	39(3)	-5(2)	6(2)	1(2)
N	53(2)	63(3)	57(3)	26(2)	24(2)	9(2)
C(3)	39(2)	35(2)	52(3)	-3(2)	12(2)	-8(2)
C(11)	67(5)	84(5)	88(5)	49(4)	25(4)	3(4)
C(5)	28(2)	44(3)	29(2)	3(2)	8(2)	1(2)
C(18)	46(3)	51(3)	68(4)	7(3)	21(3)	3(3)
C(14)	48(3)	37(3)	43(3)	-2(2)	15(2)	2(2)
C(1)	62(3)	62(4)	73(4)	29(3)	18(3)	-2(3)
C(15)	68(4)	50(3)	65(3)	-15(3)	20(3)	-6(3)
C(8)	46(3)	70(4)	28(2)	5(3)	10(2)	-1(2)
C(16)	96(5)	73(4)	50(4)	-14(3)	28(3)	9(4)
C(17)	70(4)	72(4)	68(4)	5(3)	41(3)	13(3)
C(12)	67(4)	106(5)	87(4)	-31(4)	51(3)	3(4)
C(13)	65(4)	225(9)	98(5)	-13(6)	35(4)	26(5)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR07.

	x/a	y/b	z/c	U
H(4)	4535(2)	-3158(6)	-436(6)	362(53)
H(5)	4740(2)	-4262(6)	988(6)	45(61)
H(1)	5643(3)	-3616(10)	1058(7)	122(24)
H(2)	5343(3)	-4009(10)	2698(7)	363(50)
H(3)	5319(3)	-2444(10)	2098(7)	426(89)
H(10)	3647(20)	-333(38)	6574(40)	59(13)
H(12)	2523(16)	-1591(32)	4410(38)	32(10)
H(11)	4058(18)	-1550(36)	6642(39)	35(12)
H(9)	4428(19)	-254(35)	6668(38)	45(12)
H(16)	3511(20)	-4315(37)	7078(43)	61(14)
H(14)	1877(22)	-2466(38)	8147(46)	68(14)
H(6)	4198(33)	[illegible]	495(74)	148(28)
H(13)	1852(20)	[illegible]	6083(40)	41(13)
H(7)	4508(34)	[illegible]	1560(77)	136(32)
H(8)	3746(35)	[illegible]	1204(76)	144(35)
H(15)	2780(22)	[illegible]	8697(50)	74(16)

Anisotropic temperature factors ($Å^2$, x10^3)
for non-hydrogen atoms of OR05.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Br(1)	45(1)	50(1)	56(1)	-2(2)	1(1)	-1(1)
Br(2)	49(1)	36(1)	43(1)	0(2)	11(1)	-2(2)
Br(3)	49(1)	74(2)	60(1)	4(2)	-6(1)	-5(2)

Fractional coordinates (x10^4) and common isotropic
temperature factors($Å^2$, x10^3) for hydrogen atoms
of OR05.

	x/a	y/b	z/c	U
H(1)	-835(8)	351(13)	-793(12)	97(12)
H(21)	-1181(9)	2211(18)	2024(14)	97(12)
H(22)	-723(9)	3411(18)	1266(14)	97(12)
H(31)	-2132(9)	3585(18)	-40(14)	97(12)
H(32)	-2564(9)	2289(18)	649(14)	97(12)
H(41)	-1983(10)	2276(16)	-1526(16)	97(12)
H(42)	-2463(10)	1001(16)	-872(16)	97(12)
H(1')	753(8)	1545(12)	3489(11)	97(12)
H(21')	3110(9)	2278(15)	4752(14)	97(12)
H(22')	2607(9)	1054(15)	5434(14)	97(12)
H(31')	1765(8)	2671(16)	5921(14)	97(12)
H(32')	2129(8)	3819(16)	5021(14)	97(12)
H(41')	756(9)	3581(18)	3733(15)	97(12)
H(42')	481(9)	2219(18)	4505(15)	97(12)
H(1")	4406(7)	5845(13)	253(12)	97(12)
H(21")	6166(10)	6006(17)	3381(16)	97(12)
H(22")	6442(10)	7353(17)	2593(16)	97(12)
H(31")	5267(10)	8508(22)	2897(16)	97(12)
H(32")	4936(10)	7107(22)	3571(16)	97(12)
H(41")	4195(8)	8107(18)	1269(14)	97(12)
H(42")	3815(8)	6779(18)	1978(14)	97(12)

Anisotropic temperature factors ($Å^2$, x10^3)
for non-hydrogen atoms of OR10.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O	69(4)	50(4)	69(4)	-9(3)	23(3)	-23(4)
N	50(4)	55(5)	49(4)	3(4)	16(4)	1(4)
C(1)	42(5)	56(6)	45(5)	-5(5)	10(4)	-2(5)
C(2)	58(6)	58(6)	50(5)	-4(5)	7(5)	-1(5)
C(3)	54(6)	81(7)	70(7)	1(6)	-1(5)	-7(6)
C(4)	49(6)	100(9)	87(8)	-11(7)	23(6)	-16(6)
C(5)	64(7)	107(8)	73(7)	-9(7)	37(6)	3(7)
C(6)	53(6)	82(7)	50(5)	-6(5)	20(4)	-2(5)
C(7)	75(7)	131(10)	64(6)	-45(7)	3(6)	6(7)
C(8)	52(6)	67(6)	40(5)	9(5)	14(4)	-7(6)

Fractional coordinates (x10^4) and common isotropic
temperature factors($Å^2$, x10^3) for hydrogen atoms
of OR10.

	x/a	Y/b	z/c	U
H	4374(5)	-841(9)	2069(9)	187(18)
H(3)	612(7)	-993(12)	704(13)	187(18)
H(4)	68(7)	450(13)	3332(14)	187(18)
H(5)	1404(8)	1814(13)	5710(13)	187(18)
H(6)	3317(7)	1770(11)	5398(12)	187(18)
H(71)	3403(7)	-1112(13)	-240(12)	187(18)
H(72)	2134(7)	-657(13)	-1577(12)	187(18)
H(73)	2340(7)	-2474(13)	-279(12)	187(18)
H(8)	5617(7)	1230(12)	2766(11)	187(18)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR03.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	56(2)	57(2)	66(2)	-12(2)	8(2)	-11(2)
O(2)	100(4)	126(5)	198(6)	36(4)	7(4)	-54(4)
O(3)	45(2)	141(5)	263(7)	-39(5)	9(3)	-6(3)
N	75(4)	111(5)	105(4)	3(4)	-9(3)	-41(4)
C(1)	47(3)	65(3)	75(3)	-1(3)	-5(2)	-10(2)
C(2)	69(3)	54(3)	61(3)	-5(2)	-2(2)	-9(2)
C(3)	85(4)	57(3)	63(3)	-6(3)	-4(3)	-11(3)
C(4)	67(3)	55(3)	108(5)	8(3)	15(3)	-4(3)
C(5)	65(3)	86(4)	69(3)	-10(3)	-2(3)	-29(3)
C(6)	66(3)	93(4)	72(3)	23(3)	-26(3)	-23(3)
C(7)	72(4)	60(4)	156(7)	-5(4)	1(4)	17(3)
O(1')	69(2)	65(2)	54(2)	-3(2)	-20(2)	-16(2)
O(2')	91(3)	54(2)	96(3)	-21(2)	3(2)	7(2)
O(3')	102(3)	89(3)	44(2)	-10(2)	-6(3)	-20(2)
N'	59(2)	60(3)	47(2)	-8(2)	11(2)	-22(2)
C(1')	47(2)	44(2)	38(2)	-3(2)	3(2)	-12(2)
C(2')	46(2)	51(3)	49(2)	-1(2)	-6(2)	-4(2)
C(3')	52(2)	47(3)	44(2)	2(2)	-8(2)	-12(2)
C(4')	52(3)	43(2)	52(3)	-6(2)	0(2)	-1(2)
C(5')	66(3)	60(3)	50(3)	-8(3)	16(	18(3)
C(6')	68(3)	41(2)	42(2)	4(2)	1	14(2)
C(7')	49(2)	51(3)	50(3)	0(2)	-	-3(2)
O(1")	71(2)	60(2)	67(2)	-22(2)	-	1(2)
O(2")	73(3)	72(3)	132(4)	23(3)	-21(2)	-26(2)
O(3")	66(2)	127(4)	80(3)	-15(3)	-26(2)	-22(2)
N"	44(2)	64(3)	47(3)	-9(2)	-8(2)	-8(2)
C(1")	36(2)	48(2)	48(2)	-2(2)	-2(2)	2(2)
C(2")	43(2)	49(3)	43(2)	2(2)	2(2)	3(2)
C(3")	53(3)	43(2)	56(3)	-6(2)	-3(2)	6(2)
C(4")	59(3)	59(3)	57(3)	7(3)	0(2)	-17(2)
C(5")	39(2)	74(4)	68(3)	-17(3)	9(2)	-2(2)
C(6")	42(2)	49(3)	61(3)	-10(2)	-10(2)	9(2)
C(7")	63(3)	70(3)	46(3)	8(3)	-6(2)	-7(3)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR03.

	x/a	y/b	z/c	U
H(2)	1406(4)	826(8)	3775(2)	91(3)
H(3)	1460(5)	4140(8)	3404(2)	91(3)
H(4)	2032(5)	7180(9)	4040(2)	91(3)
H(51)	3177(5)	4286(9)	4452(2)	91(3)
H(52)	2112(5)	5430(9)	4792(2)	91(3)
H(6)	1810(4)	1738(10)	4275(2)	91(3)
H(71)	11(5)	6316(10)	3864(3)	91(3)
H(72)	-141(44)	6146(83)	4260(17)	91(3)
H(2')	4037(4)	10043(7)	4073(1)	91(3)
H(3')	4488(4)	6336(7)	4156(1)	91(3)
H(4')	6718(42)	5605(82)	3883(16)	91(3)
H(51')	6491(42)	8742(81)	3462(18)	91(3)
H(52')	7577(45)	9074(83)	3663(16)	91(3)
H(6')	6152(44)	11949(84)	3935(17)	91(3)
H(71')	6386(44)	6915(84)	4656(17)	91(3)
H(72')	7502(45)	8074(81)	4457(16)	91(3)
H(2")	1697(4)	7409(7)	3090(1)	91(3)
H(3")	1196(4)	10951(7)	2779(2)	91(3)
H(4")	3494(43)	10868(83)	2176(17)	91(3)
H(51")	4713(4)	7908(8)	2565(2)	91(3)
H(52")	4250(43)	7297(83)	2071(17)	91(3)
H(6")	3448(4)	4827(7)	2599(2)	91(3)
H(71")	2289(42)	8393(80)	1681(17)	91(3)
H(72")	1382(44)	9897(82)	1979(16)	91(3)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR02.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	58(2)	39(2)	56(2)	-5(10)	13(2)	-28(7)
O(2)	52(1)	50(1)	71(2)	-4(1)	6(1)	-14(1)
O(3)	36(1)	50(1)	56(1)	5(1)	4(1)	-4(1)
O(4)	45(1)	47(1)	56(1)	13(1)	5(1)	0(1)
C(1)	47(2)	42(2)	49(2)	2(2)	15(2)	-1(2)
C(2)	39(2)	36(2)	49(2)	-1(1)	12(1)	0(1)
C(3)	38(2)	41(2)	49(2)	-3(2)	8(2)	-2(1)
C(4)	40(2)	39(2)	41(2)	-2(1)	9(1)	2(.)
C(5)	35(1)	42(2)	39(3)	0(1)	7(1)	4(1)
C(6)	34(2)	42(2)	40(2)	-4(1)	9(1)	-1(1)
C(7)	41(2)	38(2)	47(2)	2(1)	12(1)	3(1)
C(8)	38(2)	47(2)	43(2)	5(2)	4(1)	4(1)
C(9)	37(2)	45(2)	48(2)	-2(2)	6(1)	-1(2)
C(10)	40(2)	58(2)	71(3)	5(2)	-3(2)	-4(2)
C(11)	48(2)	61(2)	63(3)	22(2)	7(2)	5(2)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR02.

	x/a	y/b	z/c	U
H(3)	6539(18)	7817(28)	4035(19)	45(8)
H(5)	4377(16)	5562(26)	3132(18)	37(7)
H(8)	6929(19)	3250(32)	5536(21)	57(9)
H(9)	7095(17)	5668(30)	4900(20)	19(8)
H(101)	2864(20)	2543(35)	2728(23)	75(11)
H(102)	3170(21)	4459(39)	3029(24)	81(12)
H(103)	3509(23)	3549(40)	2279(26)	88(14)
H(111)	5905(21)	-272(39)	5827(23)	83(11)
H(112)	6652(21)	801(35)	5811(22)	61(11)
H(113)	6387(23)	1668(41)	6294(25)	93(13)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR04.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Br	121(2)	81(1)	65(1)	29(2)	22(1)	5(2)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR04.

	x/a	y/b	z/c	U
H(11)	4922(13)	2172(17)	3446(10)	164(17)
H(12)	5503(13)	4136(17)	3486(10)	164(17)
H(13)	5146(13)	2997(17)	2448(10)	164(17)
H(5)	3690(14)	64(19)	5630(12)	164(17)
H(6)	3597(13)	-2375(22)	4728(11)	164(17)
H(7)	3079(13)	-2345(22)	3089(11)	164(17)
H(8)	2790(14)	332(19)	2235(11)	164(17)
H(91)	1415(13)	3622(18)	3298(10)	164(17)
H(92)	1870(13)	5260(18)	2758(10)	164(17)
H(11)	-244(19)	2553(22)	2353(15)	164(17)
H(12)	-1408(17)	1459(22)	836(13)	164(17)
H(14)	851(16)	3302(19)	-464(14)	164(17)
H(15)	2146(17)	3994(24)	1097(14)	164(17)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR01.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	67(3)	74(4)	71(3)	-13(3)	3(3)	17(3)
O(2)	68(3)	89(4)	68(3)	-16(3)	6(3)	12(3)
N	64(4)	57(4)	63(4)	-8(3)	11(3)	4(3)
F(1)	77(3)	176(5)	124(4)	-34(4)	29(3)	-24(3)
F(2)	188(5)	96(4)	85(3)	13(3)	38(3)	46(3)
B	61(6)	89(8)	86(8)	17(6)	21(6)	2(6)
C(1)	83(6)	86(7)	87(6)	-11(5)	29(5)	-10(5)
C(2)	90(7)	150(10)	106(7)	-8(8)	38(6)	-26(8)
C(3)	133(10)	73(7)	133(9)	4(6)	11(8)	-39(7)
C(4)	102(7)	70(7)	133(8)	-4(6)	6(6)	0(6)
C(5)	72(5)	72(6)	95(6)	-18(5)	10(4)	-4(5)
C(6)	54(4)	64(5)	56(5)	-11(4)	10(4)	1(4)
C(7)	95(5)	50(5)	57(5)	-8(4)	15(4)	-9(4)
C(8)	63(5)	52(5)	72(5)	-17(4)	14(4)	2(4)
C(9)	103(7)	64(6)	69(5)	4(5)	-10(5)	7(5)
C(10)	121(8)	79(7)	98(7)	3(6)	-23(7)	8(7)
C(11)	92(7)	85(8)	123(9)	-27(6)	-9(6)	8(6)
C(12)	96(7)	80(7)	132(9)	-16(6)	12(6)	4(6)
C(13)	82(6)	75(6)	103(7)	-1(5)	21(6)	11(5)
C(14)	149(8)	82(6)	222(10)	-17(7)	-33(7)	-9(6)
C(15)	286(19)	211(10)	226(15)	28(10)	3(14)	-41(11)
C(16)	58(5)	60(5)	65(5)	1(4)	13(4)	2(4)
C(17)	65(5)	61(5)	61(5)	-9(4)	15(4)	1(4)
C(18)	63(5)	51(5)	65(5)	3(4)	11(4)	-4(4)
C(19)	61(4)	63(6)	66(5)	-11(5)	22(4)	-10(4)
C(20)	110(7)	74(6)	83(6)	-19(5)	25(5)	3(5)
C(21)	144(9)	93(7)	104(8)	-37(7)	20(7)	11(7)
C(22)	119(8)	105(8)	89(7)	-36(7)	36(6)	-29(7)
C(23)	90(6)	95(7)	73(6)	1(6)	17(5)	-29(6)
C(24)	73(5)	77(6)	54(5)	-6(5)	12(4)	-19(4)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR01.

	x/a	y/b	z/c	U
H(1)	11623(55)	1796(31)	3858(51)	184(6)
H(2)	12366(24)	3284(51)	3670(50)	184(6)
H(3)	10927(11)	4696(7)	3091(7)	184(6)
H(4)	8890(9)	4413(7)	4372(7)	184(6)
H(5)	8229(7)	2811(6)	4597(5)	184(6)
H(7A)	8762(7)	1267(4)	4801(5)	184(6)
H(7B)	10287(7)	677(4)	4684(5)	184(6)
H(9)	8355(8)	2150(5)	2058(5)	184(6)
H(10)	6169(11)	2646(8)	1308(6)	184(6)
H(11)	4314(9)	1910(7)	1751(7)	184(6)
H(12)	4561(9)	676(6)	2941(7)	184(6)
H(14A)	6009(9)	195(6)	4509(6)	184(6)
H(14B)	7639(9)	71(6)	4466(6)	184(6)
H(15A)	5772(13)	-1338(6)	4256(8)	184(6)
H(15B)	7319(13)	-1109(6)	3172(8)	184(6)
H(15C)	5689(13)	-985(6)	3215(8)	184(6)
H(17)	8113(6)	-9(5)	1672(5)	184(6)
H(20)	10364(7)	-2418(6)	1088(6)	184(6)
H(21)	9632(9)	-3147(7)	-515(7)	184(6)
H(22)	8297(9)	-2278(7)	-1834(7)	184(6)
H(23)	7687(7)	-708(7)	-1567(6)	184(6)
H(24)	8318(7)	16(5)	53(5)	184(6)

Bond lengths (Å) involving hydrogen atoms for OR15.

N-H	1.080(0)	C(3)-H(3)	1.080(0)
C(4)-H(4)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(8)-H(8)	1.080(0)
C(9)-H(9)	1.080(0)	C(10)-H(10)	1.080(0)
C(11)-H(11)	1.080(0)	C(12)-H(12)	1.080(0)
C(13)-H(13)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR22.

C(1)-H(1)	.91(2)	C(7)-H(71)	.98(2)
C(7)-H(72)	1.05(2)	C(8)-H(81)	.89(2)
C(8)-H(82)	.89(2)	C(8)-H(83)	.98(2)
C(13)-H(13)	.87(2)	C(14)-H(14)	.90(2)
C(15)-H(15)	.84(2)	C(16)-H(16)	.84(2)
C(17)-H(17)	.91(2)	C(18)-H(181)	.97(2)
C(18)-H(182)	.87(4)	C(18)-H(183)	.88(3)
C(19)-H(191)	.97(3)	C(19)-H(192)	.92(3)
C(19)-H(193)	.82(3)		

Bond lengths (Å) involving hydrogen atoms for OR24.

O(2)-H(1)	.93(4)	O(3)-H(2)	.93(4)
N-H	.95(4)	C(3)-H(32)	1.080(0)
C(3)-H(31)	1.05(4)	C(4)-H(41)	1.00(3)
C(4)-H(42)	1.03(3)	C(5)-H(51)	1.080(0)
C(5)-H(52)	1.08(3)	C(6)-H(61)	1.080(0)
C(6)-H(62)	1.080(0)	C(9)-H(9)	1.01(4)
C(10)-H(10)	1.02(3)	C(12)-H(12)	1.080(0)
C(14)-H(141)	1.02(4)	C(14)-H(142)	.89(4)
C(14)-H(143)	.92(4)		

Bond lengths (Å) involving hydrogen atoms for OR26.

N-H	.72(3)	C(1)-H	1.87(3)
C(7)-H(72)	1.85(4)	C(3)-H(3)	1.09(3)
C(4)-H(4)	.95(4)	C(5)-H(5)	.97(4)
C(6)-H(6)	1.01(3)	C(7)-H(71)	.99(4)
C(7)-H(72)	.67(3)	C(7)-H(73)	1.07(4)
C(8)-H	1.77(3)	C(8)-H(8)	1.03(4)
H(71)-H(72)	1.43(5)	H(72)-H(73)	1.32(5)

Bond lengths (Å) involving hydrogen atoms for OR34.

C(3)-H(31)	1.09(7)	C(3)-H(32)	1.09(6)
C(5)-H(5)	1.080(0)	C(7)-H(7)	.92(6)
C(10)-H(10)	1.080(0)	C(11)-H(11)	1.080(0)
C(12)-H(121)	1.080(0)	C(12)-H(122)	1.080(0)
C(12)-H(123)	1.080(0)	C(13)-H(131)	1.080(0)
C(13)-H(132)	1.080(0)	C(13)-H(133)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR37.

C(3)-H(3)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(8)-H(8)	1.080(0)
C(9)-H(9)	1.080(0)	C(10)-H(101)	1.080(0)
C(10)-H(102)	1.080(0)	C(10)-H(103)	1.080(0)
C(13)-H(13)	1.080(0)	C(15)-H(15)	1.080(0)
C(16)-H(16)	1.080(0)	C(18)-H(18)	1.080(0)
C(19)-H(19)	1.080(0)	C(20)-H(201)	1.080(0)
C(20)-H(202)	1.080(0)	C(20)-H(203)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR07.

C(10)-H(10)	.98(4)	C(10)-H(11)	.92(4)
C(10)-H(9)	1.00(4)	C(11)-H(6)	1.01(7)
C(11)-H(7)	.82(7)	C(11)-H(8)	.82(7)
C(12)-H(4)	1.080(0)	C(12)-H(5)	1.080(0)
H(5)-C(13)	1.476(9)	C(13)-H(1)	1.080(0)
C(13)-H(2)	1.080(0)	C(13)-H(3)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR08.

C(9)-H(9)	1.080(0)	C(8)-H(8)	1.01(9)
C(11)-H(111)	1.080(0)	C(11)-H(112)	1.080(0)
C(11)-H(113)	1.080(0)	C(10)-H(101)	1.080(0)
C(10)-H(102)	1.080(0)	C(10)-H(103)	1.080(0)
C(5)-H(5)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR09.

N-H	.79(3)	C(3)-H(3)	.99(3)
C(4)-H(4)	.99(3)	C(5)-H(5)	1.00(3)
C(6)-H(6)	1.01(3)	C(7)-H(71)	1.01(4)
C(7)-H(72)	.99(4)	C(7)-H(73)	.95(4)
C(8)-H(8)	1.11(3)		

Bond lengths (Å) involving hydrogen atoms for OR10.

N-H	1.080(0)	C(3)-H(3)	1.080(0)
C(4)-H(4)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(7)-H(71)	1.080(0)
C(7)-H(72)	1.080(0)	C(7)-H(73)	1.080(0)
C(8)-H(8)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR11.

C(3)-H(161)	1.080(0)	C(3)-H(162)	1.080(0)
C(3)-H(163)	1.080(0)	C(9)-H(9)	1.26(4)
C(10)-H(2)	1.080(0)	C(11)-H(14)	1.080(0)
C(12)-H(3)	.90(4)	C(13)-H(15)	.94(4)
C(15)-H(41)	1.080(0)	C(15)-H(42)	1.080(0)
C(16)-H(11)	.79(4)	C(17)-H(191)	1.080(0)
C(17)-H(192)	1.080(0)	C(17)-H(193)	1.080(0)
C(18)-H(171)	1.080(0)	C(18)-H(172)	1.080(0)
C(18)-H(173)	1.080(0)	C(19)-H(181)	1.080(0)
C(19)-H(182)	1.080(0)	C(19)-H(183)	1.080(0)
C(20)-H(211)	1.080(0)	C(20)-H(212)	1.080(0)
C(20)-H(213)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR12.

C(10)-H(4)	.92(4)	C(16)-H(5)	1.080(0)
C(16)-H(7)	1.080(0)	C(16)-H(6)	1.030(0)
C(8)-H(2)	.98(4)	C(7)-H(1)	.86(5)
C(9)-H(3)	.801(2)	C(17)-H(8)	.93(5)
C(17)-H(9)	.96(6)		

Bond lengths (Å) involving hydrogen atoms for OR01.

C(1)-H(1)	1.081(5)	C(2)-H(2)	1.081(5)
C(3)-H(3)	1.080(0)	C(4)-H(4)	1.080(0)
C(5)-H(5)	1.080(0)	C(7)-H(71)	1.080(0)
C(7)-H(72)	1.080(0)	C(9)-H(9)	1.080(0)
C(10)-H(10)	1.080(0)	C(11)-H(11)	1.080(0)
C(12)-H(12)	1.080(0)	C(14)-H(141)	1.080(0)
C(14)-H(142)	1.080(0)	C(15)-H(151)	1.080(0)
C(15)-H(152)	1.080(0)	C(15)-H(153)	1.080(0)
C(17)-H(17)	1.080(0)	C(20)-H(20)	1.080(0)
C(21)-H(21)	1.080(0)	C(22)-H(22)	1.080(0)
C(23)-H(23)	1.080(0)	C(24)-H(20)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR02.

C(3)-H(3)	1.03(3)	C(5)-H(5)	1.00(2)
C(8)-H(8)	.98(3)	C(9)-H(9)	.98(3)
C(10)-H(101)	1.00(3)	C(10)-H(102)	1.02(3)
C(10)-H(103)	.98(3)	C(11)-H(111)	1.14(3)
C(11)-H(112)	.91(3)	C(11)-H(113)	1.03(3)

Bond lengths (Å) involving hydrogen atoms for OR03.

C(2)-H(2)	1.080(0)	C(3)-H(3)	1.080(0)
C(4)-H(4)	1.080(0)	C(5)-H(51)	1.080(0)
C(5)-H(52)	1.080(0)	C(6)-H(6)	1.080(0)
C(7)-H(71)	1.080(0)	C(7)-H(72)	.87(5)
C(2')-H(2')	1.080(0)	C(3')-H(3')	1.080(0)
C(4')-H(4')	1.07(5)	C(5')-H(51')	.99(5)
C(5')-H(52')	.95(5)	C(6')-H(6')	.93(5)
C(7')-H(71')	.97(5)	C(7')-H(72')	.96(5)
C(2")-H(2")	1.080(0)	C(3")-H(3")	1.080(0)
C(4")-H(4")	1.00(5)	C(5")-H(51")	1.080(0)
C(5")-H(52")	.98(5)	C(6")-H(6")	1.080(0)
C(7")-H(71")	1.02(5)	C(7")-H(72")	1.08(5)

Bond lengths (Å) involving hydrogen atoms for OR04.

C(1)-H(11)	1.080(0)	C(1)-H(12)	1.080(0)
C(1)-H(13)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(7)-H(7)	1.080(0)
C(8)-H(8)	1.080(0)	C(9)-H(91)	1.080(0)
C(9)-H(92)	1.080(0)	C(11)-H(11)	1.080(0)
C(12)-H(12)	1.080(0)	C(14)-H(14)	1.080(0)
C(15)-H(15)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR05.

H(1)-H(1)	1.080(0)	C(2)-H(21)	1.080(0)
C(2)-H(22)	1.080(0)	C(3)-H(31)	1.080(0)
C(3)-H(32)	1.080(0)	C(4)-H(41)	1.080(0)
C(4)-H(42)	1.080(0)	N(1')-H(1')	1.080(0)
C(2')-H(21')	1.080(0)	C(2')-H(22')	1.080(0)
C(3')-H(31')	1.080(0)	C(3')-H(32')	1.080(0)
C(4')-H(41')	1.080(0)	C(4')-H(42')	1.080(0)
N(1")-H(1")	1.080(0)	C(2")-H(21")	1.080(0)
C(2")-H(22")	1.080(0)	C(3")-H(31")	1.080(0)
C(3")-H(32")	1.080(0)	C(4")-H(41")	1.080(0)
C(4")-H(42")	1.080(0)		

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR44.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
C(1)	45(3)	48(4)	47(4)	-2(3)	8(3)	7(3)
C(2)	56(4)	64(4)	56(5)	8(4)	2(4)	7(4)
C(3)	70(5)	49(4)	81(6)	-6(4)	15(5)	-4(4)
C(4)	58(4)	59(5)	70(6)	-17(4)	12(4)	-8(4)
C(5)	55(4)	91(6)	47(4)	-15(5)	-9(4)	-6(4)
C(6)	63(5)	90(6)	47(5)	10(5)	-14(4)	5(4)
C(7)	69(5)	67(5)	53(5)	16(4)	0(4)	6(4)
C(8)	48(4)	58(4)	41(4)	-1(3)	-1(3)	1(3)
C(9)	41(3)	67(4)	42(4)	-10(4)	13(3)	-1(3)
C(10)	38(3)	54(4)	31(3)	-2(3)	4(3)	-2(3)
C(11)	51(4)	57(4)	42(4)	4(3)	-9(3)	6(3)
C(12)	55(4)	65(5)	47(4)	5(4)	-2(4)	-1(4)
C(13)	[illegible]5)	74(5)	64(6)	11(5)	-19(4)	-8(4)
C(14)	[illegible]	69(5)	73(6)	-8(5)	-40(6)	10(5)
C(15)	[illegible]	70(5)	63(6)	-13(5)	-32(5)	15(5)
C(16)	[illegible]	78(5)	51(5)	-1(4)	3(4)	20(4)
C(17)	[illegible]4)	135(8)	66(6)	-12(6)	11(5)	-11(5)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR44.

	x/a	y/b	z/c	U
H(2)	2327(5)	2494(3)	3824(14)	85(7)
H(3)	1280(5)	1846(4)	2104(15)	85(7)
H(4)	420(5)	2315(4)	-243(15)	85(7)
H(5)	-9(5)	3251(4)	-1990(13)	85(7)
H(6)	128(5)	4357(4)	-2598(14)	85(7)
H(7)	1132(5)	5029(4)	-886(14)	85(7)
H(8)	2049(5)	4560(3)	1418(13)	85(7)
H(13)	4699(5)	4324(4)	3544(13)	85(7)
H(14)	4187(7)	4752(4)	6375(16)	85(7)
H(16)	1733(5)	3847(4)	5652(14)	85(7)
H(171)	4472(5)	3755(5)	1005(15)	85(7)
H(172)	3397(5)	3767(5)	175(15)	85(7)
H(173)	3802(5)	3093(5)	1289(15)	85(7)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR43.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
N	33(2)	38(2)	41(2)	2(2)	3(2)	1(2)
O(1)	49(2)	37(2)	118(4)	18(3)	-5(2)	5(2)
O(2)	37(2)	44(2)	46(2)	-8(2)	9(2)	2(2)
O(3)	48(2)	55(2)	78(3)	26(2)	5(2)	20(2)
C(1	60(4)	82(5)	96(5)	-9(4)	29(4)	-28(4)
C(4)	43(3)	59(4)	57(4)	-8(3)	14(3)	-13(3)
C(3)	44(3)	45(3)	51(4)	-1(3)	9(2)	-6(3)
C(4)	35(3)	37(3)	41(3)	-2(2)	4(2)	-3(2)
C(5)	41(3)	33(3)	54(3)	0(3)	-2(2)	-2(2)
C(6)	29(2)	33(2)	40(3)	2(2)	-2(2)	2(2)
C(7)	44(3)	42(3)	37(3)	1(2)	-6(2)	1(2)
C(8)	50(3)	43(3)	46(3)	-6(3)	-6(2)	-8(3)
C(9)	56(3)	40(3)	49(3)	-7(3)	7(3)	-6(3)
C(10)	51(3)	30(3)	50(3)	6(2)	11(2)	-1(2)
C(11)	52(3)	43(3)	33(3)	5(3)	10(2)	9(3)
C(12)	57(3)	43(3)	29(3)	-2(2)	4(2)	6(2)
C(13)	38(3)	54(3)	51(3)	-3(3)	4(2)	4(3)
C(14)	32(3)	75(4)	54(4)	-9(3)	12(2)	3(3)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR43.

	x/a	y/b	z/c	U
H(11)	10485(7)	5182(8)	6371(4)	80(5)
H(12)	9052(7)	6477(8)	6653(4)	80(5)
H(13)	9159(7)	6025(8)	5615(4)	80(5)
H(31)	6161(6)	5564(6)	5976(3)	80(5)
H(71)	1675(6)	1691(6)	5307(3)	80(5)
H(72)	3342(6)	741(6)	5025(3)	80(5)
H(81)	1117(6)	-992(6)	4998(3)	80(5)
H(82)	1001(6)	-633(6)	6045(3)	80(5)
H(91)	2212(6)	-3128(6)	5759(3)	80(5)
H(92)	3704(6)	-2096(6)	5343(3)	80(5)
H(101)	4110(6)	-3074(6)	6847(3)	80(5)
H(102)	2965(6)	-1452(6)	7055(3)	80(5)
H(131)	8293(5)	315(7)	6745(3)	80(5)
H(141)	9987(6)	2565(7)	6619(3)	80(5)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR42.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	22(1)	29(1)	36(1)	-9(1)	-2(1)	1(1)
O(2)	27(1)	47(1)	42(1)	-3(1)	6(1)	3(1)
O(3)	31(1)	65(2)	75(2)	-8(2)	15(1)	7(1)
O(4)	45(1)	58(2)	64(2)	2(2)	5(1)	22(1)
O(5)	49(2)	95(2)	153(4)	22(3)	9(2)	34(2)
O(6)	47(2)	56(2)	41(1)	-15(1)	3(1)	19(1)
C(1)	31(1)	44(2)	38(2)	5(1)	7(1)	-3(1)
C(2)	41(2)	56(2)	39(2)	12(2)	8(2)	2(2)
C(3)	40(2)	44(2)	38(2)	10(2)	-2(2)	5(2)
C(4)	29(1)	37(2)	35(2)	-1(1)	-1(1)	4(1)
C(5)	26(1)	28(1)	28(1)	-3(1)	0(1)	0(1)
C(6)	25(1)	34(2)	39(2)	0(1)	3(1)	-2(1)
C(7)	33(2)	48(2)	37(2)	8(2)	3(1)	-6(2)
C(8)	31(1)	43(2)	30(1)	-4(1)	1(1)	6(1)
C(9)	26(1)	29(1)	30(1)	-8(1)	-2(1)	1(1)
C(10)	25(1)	28(1)	32(1)	-3(1)	2(1)	-1(1)
C(11)	30(1)	35(2)	50(2)	-15(2)	-7(1)	-1(1)
C(12)	28(2)	48(2)	51(2)	-18(2)	-7(1)	1(2)
C(13)	23(1)	56(1)	36(2)	-9(1)	-1(1)	1(1)
C(14)	31(2)	43(2)	54(2)	0(2)	2(2)	5(1)
C(15)	36(2)	47(2)	89(3)	-9(2)	7(2)	7(2)
C(16)	31(2)	55(2)	45(2)	-5(2)	7(2)	-1(2)
C(17)	43(2)	63(2)	32(2)	6(2)	0(1)	10(2)
C(18)	41(2)	54(2)	40(2)	-7(2)	-10(2)	-1(2)
C(19)	28(1)	40(2)	45(2)	-3(2)	-1(1)	2(1)
C(20)	41(2)	37(1)	46(2)	-5(1)	-2(2)	2(1)

Fractional coordinates ($x10^4$) and common isotropic temperature factors ($Å^2$, $x10^3$) for hydrogen atoms of OR42.

	x/a	y/b	z/c	U
H(11)	2403(3)	11118(3)	2526(2)	56(2)
H(12)	2188(3)	12560(3)	2710(2)	56(2)
H(21)	873(4)	11075(3)	1425(2)	56(2)
H(22)	1302(4)	12502(3)	1349(2)	56(2)
H(31)	-758(4)	13000(3)	2166(2)	56(2)
H(32)	-1176(4)	12199(3)	1237(2)	56(2)
H(5)	236(3)	10054(3)	2783(2)	56(2)
H(6)	-1744(3)	9486(3)	3599(2)	56(2)
H(71)	-1395(3)	9998(3)	5143(2)	56(2)
H(72)	-318(3)	9076(3)	4567(2)	56(2)
H(111)	2672(3)	11998(3)	5060(2)	56(2)
H(112)	3157(3)	12159(3)	3957(2)	56(2)
H(121)	3984(3)	10379(3)	5237(2)	56(2)
H(122)	4962(3)	11000(3)	4404(2)	56(2)
H(141)	3285(3)	7948(3)	3536(3)	56(2)
H(142)	3872(3)	8493(3)	4939(3)	56(2)
H(161)	4536(3)	10650(3)	2880(2)	56(2)
H(162)	3713(3)	9412(3)	2545(2)	56(2)
H(171)	1718(4)	9114(3)	5336(2)	56(2)
H(172)	627(4)	9649(2)	6152(2)	56(2)
H(173)	2186(4)	10323(3)	5921(2)	56(2)
H(181)	-1450(4)	10112(3)	1401(2)	56(2)
H(182)	-2981(4)	10861(3)	1557(2)	56(2)
H(183)	-2528(4)	9784(3)	2288(2)	56(2)
H(201)	-366(4)	12530(3)	4343(2)	56(2)
H(202)	-334(4)	13077(3)	3772(2)	56(2)
H(203)	1127(4)	13127(3)	3944(2)	56(2)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR41.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	49(4)	72(3)	71(3)	23(2)	16(2)	-4(3)
O(2)	75(4)	129(5)	47(3)	5(3)	25(2)	8(3)
O(3)	43(3)	91(3)	56(2)	-22(2)	-3(2)	12(3)
O(4)	30(4)	99(4)	62(3)	-2(3)	-4(2)	1(3)
O(2')	62(4)	59(3)	101(4)	-1(3)	-6(3)	-15(3)
O(3')	73(4)	60(3)	39(2)	-2(2)	8(2)	-20(2)
O(4')	61(3)	82(3)	40(2)	8(2)	19(2)	-5(3)
C(1)	42(6)	75(5)	56(4)	23(3)	6(3)	3(4)
C(2)	29(5)	55(4)	39(3)	8(3)	4(3)	0(3)
C(3)	35(6)	46(3)	39(3)	12(2)	3(3)	0(3)
C(4)	34(5)	46(3)	37(3)	9(2)	-6(3)	-5(3)
C(5)	59(6)	57(4)	34(3)	1(3)	1(3)	-1(4)
C(6)	48(6)	69(4)	36(3)	-4(3)	-10(3)	-9(4)
C(7)	46(5)	53(4)	46(3)	8(3)	-14(3)	-3(4)
C(8)	45(6)	48(3)	43(3)	5(3)	-4(3)	5(3)
C(9)	32(5)	45(3)	39(3)	2(2)	-9(3)	-1(3)
C(10)	63(5)	75(4)	42(3)	-7(3)	-12(3)	7(4)
C(11)	35(6)	118(7)	81(5)	1(5)	-23(4)	-14(4)
C(1')	63(6)	38(3)	71(4)	13(3)	3(4)	-1(4)
C(2')	42(5)	38(3)	56(3)	7(3)	9(3)	0(3)
C(3')	47(5)	46(3)	40(3)	-2(3)	-5(3)	0(3)
C(4')	48(5)	43(3)	34(3)	-3(2)	-4(3)	0(3)
C(5')	68(5)	51(3)	31(3)	-1(3)	-2(3)	4(3)
C(6')	74(5)	57(4)	24(3)	1(3)	4(3)	6(4)
C(7')	67(5)	57(4)	33(3)	8(3)	8(3)	0(4)
C(8')	62(5)	41(3)	26(2)	1(2)	-4(3)	0(3)
C(9')	56(5)	37(3)	26(2)	7(2)	2(3)	0(3)
C(10')	92(6)	73(4)	36(3)	-15(3)	8(3)	-20(4)
C(11')	74(6)	127(7)	42(3)	23(4)	31(3)	7(5)
C(1")	88(9)	109(7)	77(5)	15(5)	-15(5)	1(6)
C(2")	85(7)	131(8)	70(5)	1(5)	-4(5)	-14(6)
N	188(9)	154(9)	88(5)	-6(6)	16(7)	17(7)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR41.

	x/a	y/b	z/c	U
H(3)	564(2)	-1312(6)	7729(3)	99(5)
H(5)	1258(2)	-799(7)	7954(3)	99(5)
H(6)	2008(2)	-1033(8)	7632(3)	99(5)
H(9)	930(2)	-3279(7)	5844(3)	99(5)
H(101)	1637(2)	-4539(8)	4317(3)	99(5)
H(102)	1342(2)	-2753(8)	4580(3)	99(5)
H(103)	1208(2)	-4738(8)	4956(3)	99(5)
H(111)	2937(2)	-2156(10)	6558(4)	99(5)
H(112)	2616(2)	-2485(10)	7379(4)	99(5)
H(113)	2598(2)	-549(10)	6890(4)	99(5)
H(3')	-15(2)	-3100(7)	4904(3)	99(5)
H(5')	357(2)	-2300(7)	3824(3)	99(5)
H(6')	874(2)	-550(7)	3167(3)	99(5)
H(9')	639(2)	146(6)	5951(3)	99(5)
H(101)	1356(2)	3514(9)	6302(3)	99(5)
H(102)	1203(2)	1411(9)	6446(3)	99(5)
H(103)	823(2)	2927(9)	6184(3)	99(5)
H(111)	1710(2)	2449(11)	2925(3)	99(5)
H(112)	1188(2)	1790(11)	2740(3)	99(5)
H(113)	1578(2)	303(11)	2982(3)	99(5)
H(21")	1552(5)	199(11)	4929(4)	99(5)
H(22")	2229(3)	1799(11)	4839(4)	99(5)
H(23")	2396(3)	-26(11)	5315(4)	99(5)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR38.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	95(2)	51(1)	93(2)	10(1)	31(1)	8(1)
O(2)	72(1)	73(1)	105(2)	6(1)	34(1)	13(1)
O(3)	86(2)	119(2)	237(4)	25(2)	88(2)	2(2)
O(4)	193(3)	64(1)	84(2)	6(1)	46(2)	31(2)
O(5)	70(1)	68(1)	88(1)	22(1)	19(1)	9(1)
C(1)	74(2)	84(2)	117(3)	10(2)	36(2)	-1(2)
C(2)	60(2)	63(2)	69(2)	7(1)	18(1)	-2(1)
C(3)	64(2)	48(1)	50(1)	1(1)	20(1)	0(1)
C(4)	72(2)	62(2)	58(2)	-1(1)	21(1)	6(2)
C(5)	66(2)	67(2)	77(2)	11(2)	10(2)	-13(2)
C(6)	72(2)	51(2)	57(2)	1(1)	-2(1)	-15(1)
C(7)	101(2)	47(1)	45(1)	-2(1)	9(1)	-12(2)
C(8)	124(3)	53(2)	52(2)	-4(1)	25(2)	-3(2)
C(9)	128(3)	54(2)	51(2)	-4(1)	8(2)	5(2)
C(10)	114(3)	48(2)	61(2)	5(1)	1(2)	-14(2)
C(11)	84(2)	60(2)	68(2)	9(2)	4(2)	-24(2)
C(12)	201(5)	71(2)	86(3)	12(2)	26(3)	40(3)
C(13)	72(2)	44(1)	50(1)	-1(1)	21(1)	-5(1)
C(14)	57(2)	42(1)	48(1)	-4(1)	14(1)	-8(1)
C(15)	61(2)	46(1)	66(2)	4(1)	22(1)	-8(1)
C(16)	57(2)	56(2)	69(2)	4(1)	22(1)	-5(1)
C(17)	61(2)	51(1)	56(2)	3(1)	10(1)	-1(1)
C(18)	62(2)	55(2)	48(1)	6(1)	13(1)	-9(1)
C(19)	56(2)	54(1)	43(1)	-2(1)	13(1)	-10(1)
C(20)	73(2)	75(2)	83(2)	3(2)	19(2)	18(2)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR38.

	x/a	y/b	z/c	U
H(5)	4283(3)	6247(2)	1932(4)	97(3)
H(7)	6887(3)	6552(1)	-158(3)	97(3)
H(8)	8311(4)	5627(2)	-680(3)	97(3)
H(10)	6777(4)	4197(2)	2254(4)	97(3)
H(11)	5260(3)	5104(2)	2633(4)	97(3)
H(121)	9387(5)	3420(2)	927(5)	97(3)
H(122)	7758(5)	3453(2)	988(5)	97(3)
H(123)	8960(5)	3812(2)	2599(5)	97(3)
H(13)	8004(3)	8153(1)	1714(3)	97(3)
H(15)	9945(3)	7587(1)	1537(3)	97(3)
H(16)	11572(3)	6669(1)	2444(3)	97(3)
H(18)	9069(3)	5643(1)	4945(3)	97(3)
H(19)	7416(3)	6531(1)	3939(3)	97(3)
H(201)	12983(3)	5085(2)	4380(4)	97(3)
H(202)	12966(3)	6011(2)	4199(4)	97(3)
H(203)	12219(3)	5486(2)	2512(4)	97(3)

Anisotropic temperature factors ($Å^2$, $x10^3$)
for non-hydrogen atoms of OR37.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	95(7)	37(4)	49(4)	-5(4)	-39(5)	5(4)
O(2)	98(7)	52(5)	64(6)	-6(4)	-56(5)	0(5)
O(3)	112(8)	45(5)	61(5)	-4(4)	-48(5)	23(5)
O(4)	76(6)	44(5)	50(5)	7(4)	-39(4)	0(4)
O(5)	85(6)	45(4)	49(5)	-1(4)	-38(4)	19(4)
C(1)	65(8)	46(7)	41(6)	5(5)	-12(6)	-3(6)
C(2)	50(7)	38(6)	28(5)	3(4)	-15(4)	0(5)
C(3)	47(6)	42(6)	29(5)	3(4)	-11(4)	6(5)
C(4)	52(7)	41(6)	33(5)	0(4)	-21(5)	7(5)
C(5)	76(9)	30(5)	44(6)	-7(5)	-32(6)	5(6)
C(6)	74(9)	33(6)	49(6)	-9(5)	-30(6)	15(6)
C(7)	62(8)	36(6)	44(6)	0(5)	-27(5)	10(5)
C(8)	98(10)	48(7)	37(6)	-7(5)	-37(6)	25(7)
C(9)	73(8)	51(7)	29(5)	-4(5)	-18(5)	17(6)
C(10)	102(11)	56(8)	50(7)	3(6)	-43(7)	20(8)
C(11)	63(8)	45(6)	39(6)	0(5)	-17(5)	-1(6)
C(12)	41(6)	31(5)	34(5)	6(4)	-9(4)	-1(4)
C(13)	36(6)	43(6)	36(5)	6(5)	-10(4)	2(5)
C(14)	46(6)	39(6)	30(5)	5(4)	-8(4)	4(5)
C(15)	49(7)	37(6)	40(6)	3(5)	-14(5)	7(5)
C(16)	53(7)	44(6)	38(5)	0(5)	-18(5)	5(5)
C(17)	47(7)	48(6)	31(5)	9(5)	-12(5)	2(5)
C(18)	63(8)	44(6)	49(7)	13(5)	-25(6)	-4(6)
C(19)	56(7)	41(6)	45(6)	7(5)	-22(5)	-5(6)
C(20)	90(10)	49(7)	50(8)	4(6)	-47(7)	-9(7)

Fractional coordinates ($x10^4$) and common isotropic
temperature factors($Å^2$, $x10^3$) for hydrogen atoms
of OR37.

	x/a	y/b	z/c	U
H(3)	1753(23)	11791(9)	1427(3)	68(9)
H(5)	-1751(26)	13144(9)	1405(4)	68(9)
H(6)	-5351(26)	14289(9)	999(4)	68(9)
H(8)	-5006(30)	12009(10)	-124(4)	68(9)
H(9)	-1437(26)	10822(10)	285(3)	68(9)
H(101)	-9477(31)	14368(11)	-397(4)	68(9)
H(102)	-8863(31)	12901(11)	-317(4)	68(9)
H(103)	-6017(31)	13799(11)	-472(4)	68(9)
H(13)	5009(21)	11285(9)	1771(3)	68(9)
H(15)	8192(23)	11912(9)	2263(3)	68(9)
H(16)	11724(24)	11702(9)	2891(3)	68(9)
H(18)	11373(25)	8089(10)	2728(4)	68(9)
H(19)	7639(24)	8327(9)	2098(4)	68(9)
H(201)	15983(30)	10324(10)	3744(4)	68(9)
H(202)	12719(30)	11037(10)	3630(4)	68(9)
H(203)	15631(30)	11206(10)	3262(4)	68(9)

Anisotropic temperature factors ($Å^2$, $x10^3$) for non-hydrogen atoms of OR34.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	61(2)	49(2)	44(2)	-11(2)	25(2)	-17(2)
O(2)	65(3)	62(3)	71(3)	-20(3)	40(2)	-21(3)
O(3)	70(3)	56(3)	59(3)	-24(2)	5(2)	-10(2)
O(4)	46(2)	43(2)	49(2)	-7(2)	9(2)	-8(2)
O(5)	74(3)	47(2)	47(2)	-16(2)	18(2)	-22(2)
C(1)	40(3)	38(3)	43(3)	1(2)	10(2)	-6(2)
C(2)	36(2)	40(3)	32(2)	-2(2)	6(2)	2(2)
C(3)	46(3)	39(3)	42(3)	-12(2)	13(2)	-4(2)
C(4)	44(3)	37(3)	40(3)	1(2)	2(2)	-2(2)
C(5)	40(3)	42(3)	32(2)	3(2)	9(2)	-7(2)
C(6)	38(2)	35(3)	32(2)	3(2)	5(2)	-6(2)
C(7)	40(2)	36(3)	34(2)	1(2)	9(2)	-9(2)
C(8)	39(2)	37(3)	35(2)	5(2)	11(2)	-8(2)
C(9)	47(3)	32(3)	36(2)	-1(2)	4(2)	-7(2)
C(10)	47(3)	41(3)	39(3)	3(2)	11(2)	-15(3)
C(11)	45(3)	45(3)	35(2)	1(2)	11(2)	-11(3)
C(12)	63(4)	56(4)	53(3)	-1(3)	28(3)	-19(3)
C(13)	80(4)	49(4)	53(3)	-16(3)	9(3)	-21(4)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR34.

	x/a	y/b	z/c	U
H(31)	3642(100)	-3960(109)	81(39)	64(6)
H(32)	5366(109)	-5706(101)	810(37)	64(6)
H(5)	5695(9)	-1766(10)	1145(3)	64(6)
H(7)	856(116)	-1125(107)	2834(39)	64(6)
H(10)	6961(10)	3736(10)	2672(3)	64(6)
H(11)	6910(9)	1071(11)	1648(3)	64(6)
H(121)	-1464(11)	-292(11)	4822(4)	64(6)
H(122)	475(11)	-1715(11)	4228(4)	64(6)
H(123)	-2134(11)	-572(11)	3683(4)	64(6)
H(131)	5430(13)	6486(12)	4506(4)	64(6)
H(132)	9458(13)	6243(12)	3564(4)	64(6)
H(133)	7556(13)	4914(12)	4077(4)	64(6)

Fractional coordinates ($x10^4$) and common isotropic temperature factors($Å^2$, $x10^3$) for hydrogen atoms of OR28.

	x/a	y/b	z/c	U
H(3)	9326(79)	5640(77)	7738(29)	69(3)
H(11)	1570(7	5622(6)	6611(2)	89(3)
H(12)	2998(5,	2911(6)	6379(2)	89(3)
H(21)	933(6)	4469(7)	5330(2)	89(3)
H(22)	3608(6)	3287(7)	5176(2)	89(3)
H(31)	1808(6)	6640(7)	4784(2)	89(3)
H(32)	772(6)	7819(7)	5655(2)	89(3)
H(5)	3212(5)	7694(5)	6668(2)	89(3)
H(61)	6228(6)	8229(6)	6643(2)	89(3)
H(62)	7852(6)	5530(6)	6477(2)	89(3)
H(111)	4939(5)	1705(5)	7270(2)	89(3)
H(112)	5846(5)	2417(5)	8138(2)	89(3)
H(121)	1402(5)	4550(5)	7551(2)	89(3)
H(122)	2304(5)	5285(5)	8416(2)	89(3)
H(14)	818(5)	1823(5)	7318(2)	89(3)
H(161)	3758(8)	3089(8)	9246(2)	89(3)
H(162)	4310(8)	516(8)	9033(2)	89(3)
H(163)	1812(8)	2584(8)	9383(2)	89(3)
H(171)	5709(6)	4704(6)	8810(2)	89(3)
H(172)	5490(6)	7182(6)	8927(2)	89(3)
H(173)	7936(6)	4962(6)	8768(2)	89(3)
H(181)	6266(7)	3953(7)	5061(2)	89(3)
H(182)	7017(7)	5889(7)	5218(2)	89(3)
H(183)	5157(7)	5932(7)	4524(2)	89(3)
H(191)	4591(8)	9472(6)	6757(3)	89(3)
H(192)	2040(8)	10162(6)	5950(3)	89(3)
H(193)	2799(8)	9487(6)	5039(3)	89(3)
H(201)	6615(5)	2356(5)	5635(2)	89(3)
H(202)	6797(5)	1106(5)	6360(2)	89(3)
H(203)	8170(5)	2489(5)	6417(2)	89(3)
H(11)	3306(5)	-2429(5)	1549(2)	89(3)
H(12)	4600(5)	-1181(5)	1335(2)	89(3)
H(21)	2774(6)	-830(6)	144(2)	89(3)
H(22)	4193(6)	-3440(6)	264(2)	89(3)
H(31)	707(6)	-2549(7)	-278(2)	89(3)
H(32)	1081(6)	-3359(7)	571(2)	89(3)
H(5)	-350(5)	-680(5)	1639(2)	89(3)
H(61)	-3851(5)	2402(6)	1649(2)	89(3)
H(62)	-2720(5)	3874(6)	1503(2)	89(3)
H(111)	4602(5)	1019(5)	2237(2)	89(3)
H(112)	3614(5)	2228(5)	3113(2)	89(3)
H(121)	5362(5)	-2337(5)	2499(2)	89(3)
H(122)	4272(5)	-1158(5)	3363(2)	89(3)
H(14)	8604(5)	-3034(5)	2296(2)	89(3)
H(161)	5589(6)	556(8)	4222(2)	89(3)
H(162)	7611(6)	897(8)	4036(2)	89(3)
H(163)	8171(6)	-1342(8)	4364(2)	89(3)
H(171)	1904(5)	2318(6)	3805(2)	89(3)
H(172)	-370(5)	2293(6)	3925(2)	89(3)
H(173)	-529(5)	4561(6)	3800(2)	89(3)
H(181)	-586(7)	1883(8)	74(2)	89(3)
H(182)	-3196(7)	2787(8)	262(2)	89(3)
H(183)	-2012(7)	771(8)	-471(2)	89(3)
H(191)	-4204(7)	514(9)	757(3)	89(3)
H(192)	-2321(7)	-1879(9)	917(3)	89(3)
H(193)	-3037(7)	-1381(9)	17(3)	89(3)
H(201)	1145(5)	2271(5)	635(2)	89(3)
H(202)	2781(5)	2605(5)	1357(2)	89(3)
H(203)	65(5)	4043(5)	1434(2)	89(3)

Anisotropic temperature factors ($Å^2$, $x10^3$)

for non-hydrogen atoms of OR28.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	48(1)	54(1)	69(2)	1(1)	-1(1)	-37(1)
O(2)	84(2)	75(2)	54(2)	19(1)	0(1)	-57(2)
O(3)	49(1)	56(2)	68(2)	14(1)	-4(1)	-36(1)
C(1)	44(2)	48(2)	50(2)	5(2)	-4(1)	-30(2)
C(2)	55(2)	71(3)	50(2)	4(2)	-9(2)	-39(2)
C(3)	53(2)	67(3)	49(2)	14(2)	-7(2)	-25(2)
C(4)	60(2)	45(2)	44(2)	7(2)	4(2)	-26(2)
C(5)	43(2)	39(1)	40(2)	2(1)	7(1)	-22(1)
C(6)	53(2)	50(2)	53(2)	4(2)	8(2)	-38(2)
C(7)	29(1)	35(2)	55(2)	0(1)	6(1)	-17(1)
C(8)	31(1)	38(2)	46(2)	3(1)	5(1)	-15(1)
C(9)	27(1)	32(1)	44(2)	3(1)	6(1)	-15(1)
C(10)	33(1)	30(1)	43(2)	1(1)	3(1)	-17(1)
C(11)	32(1)	33(1)	52(2)	7(1)	6(1)	-16(1)
C(12)	36(2)	39(2)	64(2)	9(2)	11(1)	-20(1)
C(13)	33(1)	40(2)	50(2)	8(1)	8(1)	-19(1)
C(14)	34(1)	41(2)	46(2)	11(1)	4(1)	-20(1)
C(15)	32(1)	38(2)	46(2)	4(1)	9(1)	-17(1)
C(16)	95(3)	89(3)	56(2)	23(2)	-16(2)	-68(3)
C(17)	49(2)	62(2)	43(2)	2(2)	0(2)	-32(2)
C(18)	78(3)	68(3)	47(2)	6(2)	15(2)	-33(2)
C(19)	98(4)	50(2)	70(3)	19(2)	4(3)	-33(2)
C(20)	47(2)	36(2)	48(2)	-1(1)	11(2)	-12(1)
O(1')	28(1)	53(1)	60(2)	1(1)	15(1)	-9(1)
O(2')	33(1)	85(2)	52(2)	1(1)	-2(1)	-23(1)
O(3')	28(1)	55(1)	64(2)	-4(1)	8(1)	-14(1)
C(1')	29(1)	46(2)	53(2)	3(2)	12(1)	-13(1)
C(2')	43(2)	59(2)	52(2)	-3(2)	18(2)	-17(2)
C(3')	59(2)	66(2)	51(2)	-10(2)	10(2)	-33(2)
C(4')	41(2)	73(2)	45(2)	-1(2)	6(2)	-32(2)
C(5')	27(1)	50(2)	40(2)	2(1)	5(1)	-19(1)
C(6')	23(1)	62(2)	47(2)	7(2)	2(1)	-14(1)
C(7')	25(1)	36(1)	51(2)	6(1)	7(1)	-10(1)
C(8')	25(1)	37(1)	44(2)	7(1)	2(1)	-10(1)
C(9')	22(1)	32(1)	47(2)	9(1)	2(1)	-10(1)
C(10')	21(1)	40(2)	43(2)	7(1)	4(1)	-11(1)
C(11')	24(1)	39(2)	56(2)	13(1)	-2(1)	-13(1)
C(12')	24(1)	40(2)	64(2)	15(2)	-2(1)	-12(1)
C(13')	25(1)	38(2)	50(2)	11(1)	-2(1)	-10(1)
C(14')	27(1)	39(2)	47(2)	6(1)	-3(1)	-12(1)
C(15')	27(1)	39(2)	47(2)	13(1)	0(1)	-13(1)
C(16')	38(2)	91(3)	56(2)	-10(2)	6(2)	-18(2)
C(17')	38(2)	53(2)	44(2)	4(2)	4(1)	-14(2)
C(18')	51(2)	96(3)	48(2)	12(2)	-5(2)	-31(2)
C(19')	67(3)	121(4)	64(3)	-14(3)	8(2)	-67(3)
C(20')	40(2)	49(2)	56(2)	13(2)	4(1)	-24(1)

Bond lengths (Å) involving hydrogen atoms for OR42.

C(1)-H(11)	1.080(0)	C(1)-H(12)	1.080(0)
C(2)-H(21)	1.080(0)	C(2)-H(22)	1.080(0)
C(3)-H(31)	1.080(0)	C(3)-H(32)	1.080(0)
C(5)-H(5)	1.080(0)	C(6)-H(6)	1.080(0)
C(7)-H(71)	1.080(0)	C(7)-H(72)	1.080(0)
C(11)-H(111)	1.080(0)	C(11)-H(112)	1.080(0)
C(12)-H(121)	1.080(0)	C(12)-H(122)	1.080(0)
C(14)-H(141)	1.080(0)	C(14)-H(142)	1.080(0)
C(16)-H(161)	1.080(0)	C(16)-H(162)	1.080(0)
C(17)-H(171)	1.080(0)	C(17)-H(172)	1.080(0)
C(17)-H(173)	1.080(0)	C(18)-H(181)	1.080(0)
C(18)-H(182)	1.080(0)	C(18)-H(183)	1.080(0)
C(20)-H(201)	1.080(0)	C(20)-H(202)	1.080(0)
C(20)-H(203)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR43.

C(1)-H(11)	1.080(0)	C(1)-H(12)	1.080(0)
C(1)-H(13)	1.080(0)	C(3)-H(31)	1.080(0)
C(7)-H(71)	1.080(0)	C(7)-H(72)	1.080(0)
C(8)-H(81)	1.080(0)	C(8)-H(82)	1.080(0)
C(9)-H(91)	1.080(0)	C(9)-H(92)	1.080(0)
C(10)-H(101)	1.080(0)	C(10)-H(102)	1.080(0)
C(13)-H(131)	1.080(0)	C(14)-H(141)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR44.

C(2)-H(2)	1.080(0)	C(3)-H(3)	1.080(0)
C(4)-H(4)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(7)-H(7)	1.080(0)
C(8)-H(8)	1.080(0)	C(13)-H(13)	1.080(0)
C(14)-H(14)	1.080(0)	C(16)-H(16)	1.080(0)
C(17)-H(171)	1.080(0)	C(17)-H(172)	1.080(0)
C(17)-H(173)	1.080(0)		

Bond lengths (Å) involving hydrogen atoms for OR28.

O(3)-H(3)	.78(7)	C(1)-H(11)	1.080(4)
C(1)-H(12)	1.080(7)	C(2)-H(21)	1.080(7)
C(2)-H(22)	1.080(5)	C(3)-H(31)	1.080(6)
C(3)-H(32)	1.080(5)	C(5)-H(5)	1.080(4)
C(6)-H(61)	1.080(7)	C(6)-H(62)	1.080(5)
C(11)-H(111)	1.080(5)	C(11)-H(112)	1.080(5)
C(12)-H(121)	1.080(5)	C(12)-H(122)	1.080(5)
C(14)-H(14)	1.080(5)	C(16)-H(161)	1.080(9)
C(16)-H(162)	1.080(6)	C(16)-H(163)	1.080(8)
C(17)-H(171)	1.080(8)	C(17)-H(172)	1.080(5)
C(17)-H(173)	1.080(5)	C(18)-H(181)	1.080(7)
C(18)-H(182)	1.080(8)	C(18)-H(183)	1.080(6)
C(19)-H(191)	1.080(9)	C(19)-H(192)	1.080(7)
C(19)-H(193)	1.080(7)	C(20)-H(201)	1.080(5)
C(20)-H(202)	1.080(6)	C(20)-H(203)	1.080(6)
C(1')-H(11)	1.080(6)	C(1')-H(12)	1.080(6)
C(2')-H(21)	1.080(7)	C(2')-H(22)	1.080(5)
C(3')-H(31)	1.080(6)	C(3')-H(32)	1.080(8)
C(5')-H(5)	1.080(6)	C(6')-H(61)	..080(6)
C(6')-H(62)	1.080(7)	C(11')-H(111)	1.080(6)
C(11')-H(112)	1.080(5)	C(12')-H(121)	1.080(5)
C(12')-H(122)	1.080(6)	C(14')-H(14)	1.080(5)
C(16')-H(161)	1.080(6)	C(16')-H(162)	1.080(9)
C(16')-H(163)	1.080(6)	C(17')-H(171)	1.080(5)
C(17')-H(172)	1.080(7)	C(17')-H(173)	1.080(5)
C(18')-H(181)	1.080(9)	C(18')-H(182)	1.080(5)
C(18')-H(183)	1.080(6)	C(19')-H(191)	1.080(6)
C(19')-H(192)	1.080(9)	C(19')-H(193)	1.080(7)
C(20'-H(201)	1.080(5)	C(20')-H(202)	1.080(6)
C(20')-H(203)	1.080(4)		

Bond lengths (Å) involving hydrogen atoms for OR38.

C(5)-H(5)	1.080(0)	C(7)-H(7)	1.080(0)
C(8)-H(8)	1.080(0)	C(10)-H(10)	1.080(0)
C(11)-H(11)	1.080(0)	C(12)-H(121)	1.080(0)
C(12)-H(122)	1.080(0)	C(12)-H(123)	1.080(0)
C(13)-H(13)	1.080(0)	C(15)-H(15)	1.080(0)
C(16)-H(16)	1.080(0)	C(18)-H(18)	1.080(0)
C(19)-H(19)	1.080(0)	C(20)-H(201)	1.080(0)
C(20)-H(202)	1.080(0)	C(20)-H(203)	1.080(0)

Bond lengths (Å) involving hydrogen atoms for OR41.

C(3)-H(3)	1.080(0)	C(5)-H(5)	1.080(0)
C(6)-H(6)	1.080(0)	C(9)-H(9)	1.080(0)
C(10)-H(101)	1.080(0)	C(10)-H(102)	1.080(0)
C(10)-H(103)	1.080(0)	C(11)-H(111)	1.080(0)
C(11)-H(112)	1.080(0)	C(11)-H(113)	1.080(0)
C(3')-H(3')	1.080(0)	C(5')-H(5')	1.080(0)
C(6')-H(6')	1.080(0)	C(9')-H(9')	1.080(0)
C(10')-H(101)	1.080(0)	C(10')-H(102)	1.080(0)
C(10')-H(103)	1.080(0)	C(11')-H(111)	1.080(0)
C(11')-H(112)	1.080(0)	C(11')-H(113)	1.080(0)
C(2")-H(21")	.975(0)	C(2")-H(22")	1.054(0)
C(2")-H(23")	1.054(0)		

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-8	3	10	6	6	-2	6	10	4	2	2	13	10	4	5	1	3	11	6	-3	-3	6	11	17	-15
-7	3	10	7	7	0	6	10	4	-4	-7	0	11	3	2	2	3	11	13	12	-2	6	11	3	-3
-6	3	10	3	-1	3	6	10	3	2	-1	0	11	11	12	3	3	11	10	-10	1	6	11	11	11
-4	3	10	4	5	-8	7	10	6	-6	1	0	11	18	-18	4	3	11	13	13	2	6	11	7	-6
-1	3	10	10	11	-5	7	10	7	7	3	0	11	21	-22	5	3	11	4	-3	3	6	11	14	17
0	3	10	6	-6	-3	7	10	8	7	5	0	11	7	6	7	3	11	4	0	4	6	11	4	4
3	3	10	7	7	-2	7	10	6	6	-8	1	11	4	3	-10	4	11	4	4	-7	7	11	5	-5
4	3	10	9	10	-1	7	10	5	-4	-5	1	11	3	-4	-7	4	11	9	-8	-2	7	11	9	-9
5	3	10	5	-5	0	7	10	3	2	-4	1	11	6	-6	-6	4	11	8	-9	0	7	11	6	5
8	3	10	2	0	3	7	10	7	7	-3	1	11	3	-3	-5	4	11	6	-8	1	7	11	4	-4
-10	4	10	4	0	6	7	10	3	3	-2	1	11	9	8	-4	4	11	9	-10	2	7	11	8	9
-9	4	10	4	-4	-8	8	10	4	-2	-1	1	11	3	3	-3	4	11	18	-17	3	7	11	4	3
-8	4	10	5	6	-7	8	10	4	2	1	1	11	3	2	-1	4	11	12	-12	-9	8	11	4	-3
-7	4	10	5	5	-5	8	10	5	7	3	1	11	10	9	0	4	11	6	6	-5	8	11	7	7
-6	4	10	3	0	-4	8	10	9	9	5	1	11	3	-2	2	4	11	6	-4	-4	8	11	3	0
-4	4	10	2	0	-3	8	10	12	11	6	1	11	5	5	3	4	11	17	17	-1	8	11	3	-3
-3	4	10	4	-4	-1	8	10	6	6	-9	2	11	5	4	4	4	11	9	-8	-2	8	11	10	-9
-2	4	10	8	10	0	8	10	3	-1	-6	2	11	3	-4	5	4	11	3	1	-1	8	11	4	-5
0	4	10	4	3	3	8	10	3	2	-5	2	11	3	-1	6	4	11	3	-4	1	8	11	5	2
1	4	10	4	3	-8	9	10	4	2	-4	2	11	7	-9	-10	5	11	4	4	-7	9	11	4	3
3	4	10	8	-7	-5	9	10	5	6	-3	2	11	11	-12	-9	5	11	4	5	-5	9	11	6	5
7	4	10	5	3	-2	9	10	4	-2	-2	2	11	3	1	-7	5	11	4	3	-2	9	11	6	-6
-8	5	10	11	-11	-3	9	10	4	-2	-1	2	11	6	6	-5	5	11	11	-10	-1	9	11	6	-6
-6	5	10	9	-9	4	9	10	4	1	1	2	11	9	10	-4	5	11	20	-19	0	9	11	3	2
-5	5	10	16	-16	-5	10	10	6	-6	3	2	11	10	8	-3	5	11	8	-8	-6	10	11	4	1
-4	5	10	3	-3	-2	10	10	4	-5	4	2	11	9	-9	-2	5	11	19	-19	-5	10	11	4	-1
-3	5	10	10	10	-1	10	10	4	-3	5	2	11	6	6	0	5	11	11	-12	1	10	11	3	-3
-2	5	10	4	-3	1	10	10	3	-1	-10	3	11	4	3	1	5	11	8	-7	0	11	11	4	0
0	5	10	4	3	-2	11	10	2	-1	-6	3	11	9	-8	2	5	11	13	14	3	11	11	4	3
-9	6	10	7	-8	1	11	10	3	-2	-5	3	11	18	-19	3	5	11	4	-3	-2	12	11	4	2
-8	6	10	3	-2	3	11	10	4	-6	-4	3	11	7	-7	4	5	11	3	3	-10	0	12	4	3
-7	6	10	12	-12	-2	12	10	4	4	-3	3	11	5	-4	-7	6	11	4	-1	-6	0	12	11	10
-5	6	10	6	-7	2	12	10	4	-1	-2	3	11	5	-6	-6	6	11	3	-4	-2	0	12	9	8
-4	6	10	9	8	-4	13	10	4	-1	-1	3	11	11	12	-5	6	11	6	-7	0	0	12	5	-6

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22N02F2B, OR01. PAGE 11

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	12	8	6	6	-6	2	9	8	5	2	4	9	3	-3	5	7	9	3	-4	-5	13	9	6	6
1	12	8	3	4	-5	2	9	10	-10	3	4	9	11	-13	-9	8	9	3	-3	-1	13	9	4	-4
4	12	8	4	2	-4	2	9	16	-17	4	4	9	8	10	-8	8	9	4	-4	-4	14	9	4	4
-5	13	8	4	-3	-1	2	9	3	6	-11	5	9	3	-2	-5	8	9	14	12	-10	0	10	4	-5
-2	13	8	4	-4	0	2	9	6	6	-5	5	9	9	10	-4	8	9	3	6	-8	0	10	9	9
0	13	8	4	-2	1	2	9	16	16	-4	5	9	15	15	-3	8	9	15	16	-4	0	10	3	-6
2	13	8	5	-4	2	2	9	6	-5	-3	5	9	17	17	-2	8	9	16	17	-2	0	10	23	-22
2	14	8	4	-1	3	2	9	6	-5	-2	5	9	3	1	-1	8	9	9	10	0	0	10	20	20
-3	15	8	4	-3	4	2	9	17	18	-1	5	9	8	-7	1	8	9	3	-2	2	0	10	7	-8
-1	15	8	4	2	5	2	9	9	-8	0	5	9	2	-1	3	8	9	3	2	4	0	10	4	-7
-11	0	9	2	2	6	2	9	7	9	1	5	9	3	-4	4	8	9	10	-11	-7	1	10	4	4
-7	0	9	5	5	7	2	9	4	-3	2	5	9	6	-7	-6	9	9	4	4	-6	1	10	12	-12
-5	0	9	11	-13	9	2	9	4	3	3	5	9	9	8	-2	9	9	8	7	-5	1	10	3	5
-3	0	9	7	8	-8	3	9	10	-9	4	5	9	6	7	-1	9	9	11	10	-4	1	10	10	-9
-1	0	9	18	19	-7	3	9	7	-7	-9	6	9	4	-5	0	9	9	6	-7	-3	1	10	4	-4
1	0	9	3	0	-6	3	9	5	-5	-7	6	9	5	-7	1	9	9	3	0	-1	1	10	3	1
3	0	9	27	-27	-5	3	9	5	7	-5	6	9	4	-3	3	9	9	4	4	0	1	10	19	-20
5	0	9	21	-20	-4	3	9	5	5	-4	6	9	13	15	4	9	9	6	-5	1	1	10	3	-3
7	0	9	6	-7	-3	3	9	5	-7	-3	6	9	19	20	4	9	9	4	-5	2	1	10	16	-17
-10	1	9	4	4	-2	3	9	12	11	-2	6	9	26	26	-6	10	9	4	3	3	1	10	13	-14
-9	1	9	4	4	1	3	9	7	-7	-1	6	9	10	9	-3	10	9	6	-5	5	1	10	3	-7
-7	1	9	3	3	3	3	9	9	9	0	6	9	3	-2	-1	10	9	9	9	6	1	10	4	4
-6	1	9	4	4	4	3	9	13	-13	2	6	9	9	7	0	10	9	8	7	7	1	10	3	1
-5	1	9	6	-6	5	3	9	6	6	3	6	9	8	-8	1	10	9	7	8	-10	2	10	4	-3
-4	1	9	16	-16	6	3	9	6	-5	4	6	9	5	-5	5	10	9	4	-4	-8	2	10	4	3
-3	1	9	15	-16	7	3	9	5	-3	7	6	9	3	-2	-6	11	9	6	-7	-7	2	10	3	-3
-1	1	9	5	-5	-11	4	9	4	2	-9	7	9	4	-5	-4	11	9	4	-5	-5	2	10	13	-13
0	1	9	18	19	-9	4	9	4	-4	-7	7	9	7	-7	-1	11	9	11	10	-4	2	10	7	6
1	1	9	12	11	-8	4	9	4	-4	-6	7	9	6	-5	0	11	9	8	9	-2	2	10	5	6
2	1	9	4	-4	-6	4	9	6	5	-4	7	9	10	11	4	11	9	3	-2	-1	2	10	18	-18
3	1	9	18	18	-3	4	9	11	11	-3	7	9	25	26	5	11	9	4	3	0	2	10	11	11
4	1	9	19	-20	-2	4	9	9	-7	-2	7	9	31	33	-4	12	9	4	4	1	2	10	10	-10
5	1	9	18	18	-1	4	9	5	-6	2	7	9	4	-2	-2	12	9	4	-2	2	2	10	12	-11
6	1	9	11	-11	0	4	9	20	-18	3	7	9	14	-14	2	12	9	6	-4	5	2	10	8	7

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22NO2F2B, OR01.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	11	7	12	-11	-12	1	8	4	0	-3	3	8	8	8	-9	6	8	6	7	-2	8	8	7	-7
0	11	7	8	-9	-7	1	8	6	-5	-2	3	8	9	9	-8	6	8	4	2	-1	8	8	16	-17
1	11	7	10	9	-6	1	8	3	1	1	3	8	10	-9	-6	6	8	6	5	1	8	8	4	-5
3	11	7	7	7	-5	1	8	4	-3	3	3	8	10	10	-5	6	8	10	-11	3	8	8	3	1
4	11	7	4	-3	-4	1	8	12	11	4	3	8	8	-7	-4	6	8	16	-17	5	8	8	10	-9
5	11	7	6	6	-3	1	8	32	-34	5	3	8	15	15	-3	6	8	4	-3	-6	9	8	4	6
6	11	7	3	3	-1	1	8	25	-23	6	3	8	10	10	-2	6	8	15	-14	-5	9	8	5	-4
7	11	7	4	2	0	1	8	13	-13	-10	4	8	4	-3	-1	6	8	10	10	-4	9	8	4	2
-7	12	7	4	5	1	1	8	10	-8	-9	4	8	4	6	0	6	8	8	9	-3	9	8	7	-9
-6	12	7	6	6	2	1	8	19	-19	-8	4	8	9	-10	1	6	8	13	-13	-2	9	8	14	-13
-4	12	7	4	-2	5	1	8	15	14	-7	4	8	3	-1	2	6	8	6	6	-1	9	8	21	-21
-2	12	7	6	-8	6	1	8	6	5	-6	4	8	10	-12	3	6	8	4	-3	0	9	8	17	-16
0	12	7	4	-1	7	1	8	11	11	-4	4	8	9	-8	4	6	8	7	8	2	9	8	3	3
1	12	7	7	-7	9	1	8	3	0	-3	4	8	8	9	5	6	8	4	4	3	9	8	3	-3
5	12	7	3	4	-10	2	8	4	5	-2	4	8	16	17	6	6	8	4	3	4	9	8	3	-3
-7	13	7	4	-2	-7	2	8	3	0	-1	4	8	32	31	7	6	8	4	-4	6	9	8	4	-4
-6	13	7	5	6	-6	2	8	3	1	0	4	8	12	-13	-9	7	8	4	4	-6	10	8	4	4
-5	13	7	3	2	-4	2	8	4	-3	1	4	8	10	-9	-8	7	8	7	6	-5	10	8	4	4
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8	0	8	6	5	-4	3	8	6	4	8	5	8	3	-2	-4	8	8	3	2	-2	12	8	9	10

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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3	12	6	4	-2	1	1	7	16	15	2	3	7	11	-11	7	5	7	6	5	-7	8	7	6	3
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2	13	6	4	4	6	1	7	10	10	7	3	7	4	4	-7	6	7	6	-6	-2	8	7	17	-20
3	13	6	3	-3	7	1	7	13	13	9	3	7	4	-3	-6	6	7	3	2	-1	8	7	14	-14
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3	0	7	42	41	4	2	7	14	14	7	4	7	3	1	-6	7	7	7	-8	4	9	7	6	8
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7	0	7	12	11	6	2	7	13	13	-7	5	7	5	5	-4	7	7	11	11	-6	10	7	3	-3
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11	1	7	4	3	9	2	7	5	-3	-5	5	7	4	2	-2	7	7	15	-13	-3	10	7	6	-6
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-2	1	7	20	-20	-4	3	7	8	8	3	5	7	3	-4	9	7	7	9	-3	-3	11	7	19	-12
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-2	0	6	24	25	2	2	6	28	28	-1	4	6	20	-21	2	6	6	15	15	0	9	6	3	1
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4	0	6	33	-32	6	2	6	14	-15	3	4	6	19	18	6	6	6	9	-10	5	9	6	8	-8
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-1	2	5	9	8	-2	4	5	11	-11	-1	6	5	62	-63	-10	9	5	3	3	-1	12	5	10	11
0	2	5	18	-18	-1	4	5	7	-9	0	6	5	21	20	-7	9	5	7	-0	0	12	5	10	9
1	2	5	12	-11	0	4	5	4	-2	2	6	5	25	-27	-5	9	5	10	9	1	12	5	4	-3
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3	2	5	4	-3	2	4	5	16	-15	4	6	5	22	-20	-3	9	5	5	5	3	12	5	9	-8
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6	2	5	7	-6	5	4	5	4	4	-9	7	5	3	-1	0	9	5	3	-5	6	12	5	2	-3
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9	2	5	3	-1	7	4	5	6	-7	-6	7	5	4	2	3	9	5	3	3	-1	13	5	11	11
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3	3	5	6	6	-1	5	5	34	32	8	7	5	9	-8	2	10	5	4	4	0	15	5	4	4
4	3	5	8	-8	0	5	5	31	28	-7	8	5	7	-7	3	10	5	16	17	4	15	5	3	2
5	3	5	16	-17	1	5	5	43	-41	-6	8	5	13	14	5	10	5	7	7	-10	0	6	4	5
7	3	5	8	-7	2	5	5	9	10	-5	8	5	14	-14	-6	11	5	4	-5	-6	0	6	11	8

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22N2O2F2B, OR01.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-5	4	4	12	-11	-4	6	4	38	38	0	8	4	15	-15	0	11	4	10	11	-2	16	4	4	3
-4	4	4	18	19	-2	6	4	15	14	1	8	4	11	-12	1	11	4	6	6	1	16	4	6	-7
-3	4	4	18	-20	-1	6	4	23	-22	2	8	4	3	0	2	11	4	14	17	2	16	4	7	-7
-1	4	4	18	-18	1	6	4	10	-11	3	8	4	18	-18	4	11	4	3	1	-3	16	4	4	-1
0	4	4	12	13	2	6	4	8	-7	4	8	4	9	-9	-6	12	4	4	6	-11	0	5	10	10
1	4	4	30	-33	4	6	4	3	2	5	8	4	6	-6	-3	12	4	5	6	-9	0	5	9	10
2	4	4	24	-24	5	6	4	10	10	6	8	4	5	-5	-1	12	4	22	-21	-7	0	5	13	-14
3	4	4	18	-17	6	6	4	7	9	8	8	4	3	3	0	12	4	3	-2	-5	0	5	56	-53
4	4	4	28	-28	7	6	4	8	7	-8	9	4	7	6	3	12	4	3	4	-3	0	5	28	-27
6	4	4	11	12	8	6	4	18	18	-7	9	4	4	-2	5	12	4	3	3	-1	0	5	6	7
7	4	4	14	13	10	6	4	7	6	-6	9	4	4	4	-7	13	4	4	5	1	0	5	82	86
8	4	4	9	9	-10	7	4	4	0	-5	9	4	10	-10	-6	13	4	6	5	3	0	5	20	20
9	4	4	7	-8	-7	7	4	9	-9	-4	9	4	8	-8	-5	13	4	8	10	5	0	5	15	14
-9	5	4	5	-4	-6	7	4	4	-9	-3	9	4	6	7	-2	13	4	11	-12	7	0	5	3	1
-8	5	4	6	7	-5	7	4	5	3	-1	9	4	9	-9	-1	13	4	3	-5	9	0	5	7	6
-7	5	4	12	-11	-4	7	4	7	9	0	9	4	3	3	0	13	4	16	-16	-12	1	5	5	5
-6	5	4	2	-2	-3	7	4	27	30	2	9	4	9	-9	1	13	4	9	-8	-11	1	5	2	-2
-5	5	4	27	28	-2	7	4	30	-30	3	9	4	3	4	2	13	4	4	-4	-10	1	5	20	19
-3	5	4	12	12	-1	7	4	17	16	4	9	4	5	4	3	13	4	11	-10	-7	1	5	7	-7
-2	5	4	40	-41	0	7	4	15	15	5	9	4	8	9	4	13	4	4	2	-5	1	5	6	-7
-1	5	4	46	46	1	7	4	23	-22	-7	10	4	6	8	5	13	4	6	-6	-4	1	5	25	-28
0	5	4	36	-32	2	7	4	23	-22	-5	10	4	3	5	6	13	4	4	3	-3	1	5	9	-7
1	5	4	56	-57	3	7	4	9	-9	-3	10	4	8	8	-4	14	4	4	0	-1	1	5	24	-23
2	5	4	33	-31	4	7	4	4	3	-2	10	4	6	-9	-1	14	4	9	-8	0	1	5	31	-33
3	5	4	17	-16	7	7	4	6	7	0	10	4	3	-4	0	14	4	6	-7	1	1	5	13	-12
4	5	4	8	6	8	7	4	12	12	1	10	4	4	6	1	14	4	8	-9	2	1	5	22	22
5	5	4	8	8	9	7	4	10	10	2	10	4	3	4	2	14	4	7	-6	3	1	5	8	8
7	5	4	14	15	-10	8	4	3	0	3	10	4	17	18	4	14	4	6	-8	4	1	5	19	-20
8	5	4	7	7	-8	8	4	4	0	6	10	4	5	-7	-3	15	4	4	2	5	1	5	3	1
-9	6	4	4	4	-5	8	4	23	-22	8	10	4	4	4	0	15	4	6	-6	6	1	5	8	9
-8	6	4	18	-19	-4	8	4	18	20	-7	11	4	4	3	1	15	4	4	-4	7	1	5	3	3
-7	6	4	15	15	-3	8	4	15	-15	-5	11	4	4	2	2	15	4	7	-6	8	1	5	5	8
-6	6	4	16	-15	-2	8	4	5	7	-3	11	4	8	-8	3	15	4	4	-4	10	1	5	3	2

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22NO2F2B, OR01.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
10	5	3	6	5	-1	8	3	7	7	2	11	3	6	6	-6	0	4	20	-17	-1	2	4	34	-37
-8	6	3	7	-8	0	8	3	3	0	3	11	3	6	-7	-4	0	4	53	55	0	2	4	12	12
-5	6	3	11	12	1	8	3	8	8	5	11	3	7	7	-2	0	4	25	24	1	2	4	22	-23
-4	6	3	25	-25	5	8	3	9	10	6	11	3	4	-5	0	0	4	71	-73	2	2	4	10	-10
-3	6	3	32	30	6	8	3	9	10	-7	12	3	3	0	2	0	4	14	-13	4	2	4	17	-16
-1	6	3	26	27	8	8	3	9	9	-6	12	3	3	-3	4	0	4	27	27	5	2	4	30	31
0	6	3	16	-19	10	8	3	4	2	-5	12	3	3	4	6	0	4	17	-17	6	2	4	12	12
1	6	3	20	20	-10	9	3	3	-1	-4	12	3	11	11	8	0	4	5	-5	7	2	4	10	8
2	6	3	4	4	-9	9	3	3	-3	-3	12	3	10	12	10	0	4	5	3	9	2	4	8	-8
3	6	3	7	6	-6	9	3	8	-11	-2	12	3	6	7	-11	1	4	4	-4	-11	3	4	4	-3
4	6	3	14	16	-5	9	3	3	0	-1	12	3	4	3	-8	1	4	5	6	-10	3	4	8	-8
5	6	3	13	11	-3	9	3	7	-6	0	12	3	13	13	-7	1	4	3	1	-9	3	4	12	-9
6	6	3	4	-3	-2	9	3	15	15	2	12	3	9	9	-6	1	4	16	14	-8	3	4	12	-13
9	6	3	7	-6	0	9	3	8	7	3	12	3	9	-9	-5	1	4	6	-9	-6	3	4	24	-24
-9	7	3	3	1	1	9	3	15	15	4	12	3	5	6	-4	1	4	24	24	-5	3	4	31	28
-7	7	3	3	-4	3	9	3	13	-14	5	12	3	15	-16	-3	1	4	35	-37	-4	3	4	3	0
-5	7	3	6	-6	4	9	3	26	28	7	12	3	4	-6	-2	1	4	19	19	-3	3	4	7	-8
-4	7	3	9	9	6	9	3	3	-2	-4	13	3	6	-6	-1	1	4	26	25	-2	3	4	13	12
-2	7	3	16	15	-9	10	3	3	2	0	13	3	4	3	0	1	4	69	71	-1	3	4	49	50
-1	7	3	25	24	-8	10	3	4	-4	1	13	3	5	-3	1	1	4	26	-24	0	3	4	67	-68
1	7	3	25	28	-4	10	3	3	4	4	13	3	14	-13	2	1	4	46	44	1	3	4	10	-11
5	7	3	26	24	-3	10	3	5	-6	-5	14	3	6	-7	3	1	4	9	9	2	3	4	33	-34
8	7	3	17	18	-2	10	3	4	-5	-4	14	3	4	-2	4	1	4	5	5	3	3	4	26	-24
9	7	3	4	4	-1	10	3	5	-4	-1	14	3	4	-7	5	1	4	7	-6	4	3	4	20	19
6	7	3	3	-4	2	10	3	3	0	0	14	3	5	-5	6	1	4	6	-5	5	3	4	8	-7
7	7	3	3	-4	3	10	3	17	16	1	14	3	4	4	-11	2	4	3	-2	6	3	4	10	10
8	7	3	7	-7	4	10	3	10	-10	2	14	3	3	2	-9	2	4	14	-13	7	3	4	17	18
9	7	3	3	-3	5	10	3	3	1	3	14	3	7	-6	-8	2	4	19	17	8	3	4	9	-9
-10	8	3	3	3	7	10	3	5	-4	-3	15	3	4	-4	-7	2	4	3	4	9	3	4	6	5
-9	8	3	5	-5	-8	11	3	3	-1	3	15	3	5	3	-6	2	4	7	-6	10	3	4	9	-8
-7	8	3	13	-12	-4	11	3	5	-4	-3	16	3	4	-4	-5	2	4	11	-10	-11	4	4	3	-2
-6	8	3	8	9	-3	11	3	8	9	0	17	3	3	0	-4	2	4	27	-25	-10	4	4	7	-7
-4	8	3	14	-13	0	11	3	4	-5	-10	0	4	15	-12	-3	2	4	29	28	-9	4	4	10	-12
-3	8	3	8	7	1	11	3	3	-2	-8	0	4	21	19	-2	2	4	8	9	-8	4	4	17	-19

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22NO2F2B, OR01.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	9	2	3	-2	-4	12	2	4	-6	-11	0	3	5	-6	-4	2	3	36	36	-9	4	3	15	-16
-2	9	2	6	-7	-3	12	2	10	-8	-7	0	3	4	6	-3	2	3	17	17	-8	4	3	7	-6
0	9	2	12	-11	-2	12	2	18	-20	-5	0	3	51	-49	-2	2	3	17	-18	-7	4	3	11	-10
1	9	2	5	-5	-1	12	2	4	3	-3	0	3	17	-15	-1	2	3	71	-73	-6	4	3	4	-4
2	9	2	8	7	0	12	2	13	-13	-1	0	3	100	101	0	2	3	82	77	-5	4	3	7	9
3	9	2	17	18	3	12	2	6	-2	1	0	3	13	10	1	2	3	28	29	-4	4	3	9	-10
5	9	2	4	5	5	12	2	17	-17	5	0	3	11	-10	2	2	3	49	51	-3	4	3	86	86
6	9	2	8	-9	6	12	2	4	4	7	0	3	6	6	3	2	3	11	-11	-2	4	3	46	47
7	9	2	2	2	7	12	2	4	-4	9	0	3	8	-8	4	2	3	15	15	-1	4	3	7	9
8	9	2	4	-8	-4	13	2	4	-6	11	0	3	5	-5	5	2	3	26	-26	0	4	3	27	-28
9	9	2	4	-5	-2	13	2	4	2	-11	1	3	4	-3	6	2	3	9	-8	1	4	3	50	51
-10	10	2	3	2	-1	13	2	4	-1	-10	1	3	12	-10	7	2	3	12	-13	2	4	3	4	-3
-5	10	2	10	8	0	13	2	6	8	-9	1	3	4	-4	8	2	3	3	2	3	4	3	25	-26
-3	10	2	4	-3	1	13	2	3	-2	-8	1	3	6	-5	9	2	3	4	-3	4	4	3	19	19
-2	10	2	11	12	4	13	2	11	-12	-7	1	3	7	-8	11	2	3	3	-3	5	4	3	21	-19
0	10	2	9	-9	6	13	2	4	-4	-5	1	3	33	33	-8	3	3	4	4	6	4	3	15	15
1	10	2	11	10	-6	14	2	5	-6	-4	1	3	46	-47	-7	3	3	4	-5	8	4	3	3	-3
2	10	2	6	-5	-4	14	2	5	7	-3	1	3	47	47	-6	3	3	12	11	-11	5	3	4	-4
4	10	2	10	10	-2	14	2	5	-5	-2	1	3	22	-23	-5	3	3	8	-7	-9	5	3	9	-9
5	10	2	7	-7	-1	14	2	4	3	-1	1	3	95	-98	-4	3	3	35	35	-8	5	3	16	-17
6	10	2	18	18	1	14	2	8	8	0	1	3	93	97	-2	3	3	15	16	-7	5	3	6	-7
7	10	2	5	-5	3	14	2	4	-5	1	1	3	116	-120	-1	3	3	75	-73	-6	5	3	11	-12
-5	11	2	6	-6	4	14	2	3	2	2	1	3	48	-48	0	3	3	68	63	-5	5	3	5	-5
-4	11	2	3	2	-5	15	2	4	-5	3	1	3	31	-31	1	3	3	32	32	-4	5	3	60	60
-2	11	2	6	6	-3	15	2	6	-5	4	1	3	22	22	2	3	3	19	-17	-3	5	3	23	24
0	11	2	8	9	-1	15	2	4	-2	5	1	3	5	4	4	3	3	23	-23	-2	5	3	75	75
1	11	2	21	-22	0	15	2	3	5	6	1	3	7	-7	5	3	3	7	6	-1	5	3	19	-17
2	11	2	20	21	1	15	2	4	3	10	1	3	9	-8	6	3	3	18	-17	0	5	3	23	-24
4	11	2	6	7	2	15	2	4	4	-11	2	3	3	0	8	3	3	3	-4	1	5	3	5	-2
5	11	2	14	15	3	15	2	4	1	-10	2	3	10	-11	9	3	3	12	11	2	5	3	9	-8
6	11	2	16	-16	-3	16	2	4	2	-9	2	3	9	-11	10	3	3	8	-7	3	5	3	6	4
7	11	2	3	3	1	16	2	3	5	-8	2	3	9	-8	11	3	3	3	1	4	5	3	11	-11
8	11	2	7	-6	2	16	2	6	7	-7	2	3	11	11	-12	4	3	6	-5	5	5	3	22	20
-5	12	2	8	9	1	17	2	4	3	-6	2	3	3	-2	-10	4	3	3	-4	6	5	3	5	-6

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	15	1	5	5	8	1	2	4	5	0	3	2	54	52	-2	5	2	38	39	-8	7	2	11	11
0	15	1	3	0	10	1	2	5	5	1	3	2	55	-53	-1	5	2	49	-49	-6	7	2	17	19
5	15	1	4	3	11	1	2	6	-5	2	3	2	25	23	0	5	2	33	38	-5	7	2	3	-2
-1	16	1	4	2	-10	2	2	5	4	3	3	2	29	-30	1	5	2	35	36	-4	7	2	14	-13
-1	17	1	3	2	-9	2	2	18	18	4	3	2	3	-4	2	5	2	9	6	-3	7	2	11	-9
0	17	1	4	-1	-8	2	2	5	-6	5	3	2	10	10	3	5	2	29	29	-2	7	2	37	-37
-12	0	2	4	1	-7	2	2	18	-19	7	3	2	8	-10	4	5	2	22	-22	-1	7	2	34	-31
-10	0	2	10	9	-6	2	2	12	14	-10	4	2	10	9	5	5	2	18	20	0	7	2	34	-29
-8	0	2	23	-21	-5	2	2	5	5	-9	4	2	9	11	6	5	2	10	10	2	7	2	6	4
-6	0	2	27	29	-4	2	2	29	-29	-8	4	2	18	19	7	5	2	8	-9	3	7	2	15	17
-4	0	2	10	-10	-3	2	2	87	88	-7	4	2	7	-6	8	5	2	3	-3	4	7	2	5	-5
-2	0	2	13	12	-2	2	2	103	-106	-6	4	2	8	11	10	5	2	3	3	5	7	2	3	-2
0	0	2	69	-80	-1	2	2	110	114	-5	4	2	33	-34	11	5	2	3	-1	6	7	2	6	8
2	0	2	87	88	0	2	2	105	-103	-4	4	2	14	-14	-10	6	2	4	4	7	7	2	3	-5
4	0	2	29	-29	1	2	2	81	-79	-3	4	2	16	-14	-9	6	2	9	9	8	7	2	17	
6	0	2	19	18	2	2	2	3	2	-2	4	2	13	-12	-8	6	2	10	11	9	7	2		
10	0	2	5	-5	3	2	2	28	26	-1	4	2	41	36	-7	6	2	4	5	-8	8	2		
-9	1	2	3	-2	4	2	2	7	6	0	4	2	9	10	-6	6	2	16	18	-6	8	2		
-8	1	2	5	-4	5	2	2	19	-17	1	4	2	4	-4	-5	6	2	7	7	-5	8	2		7
-7	1	2	12	10	6	2	2	20	19	2	4	2	11	9	-4	6	2	12	-14	-4	8	2	8	-2
-6	1	2	27	-26	7	2	2	3	-4	3	4	2	28	-25	-3	6	2	6	4	-3	8	2	21	-20
-5	1	2	2	2	8	2	2	9	8	4	4	2	18	18	-2	6	2	39	-38	-2	8	2	17	-17
-4	1	2	13	14	10	2	2	7	-6	5	4	2	3	-5	-1	6	2	43	41	-1	8	2	26	-26
-3	1	2	5	3	-11	3	2	4	2	6	4	2	5	4	0	6	2	20	-17	0	8	2	14	14
-2	1	2	102	107	-10	3	2	4	-3	7	4	2	5	-7	1	6	2	7	6	1	8	2	9	9
-1	1	2	7	10	-9	3	2	7	7	9	4	2	3	3	2	6	2	18	17	2	8	2	22	24
0	1	2	58	56	-8	3	2	9	-9	-10	5	2	5	7	3	6	2	5	8	3	8	2	27	29
1	1	2	53	-54	-7	3	2	9	6	-9	5	2	18	19	4	6	2	10	-10	4	8	2	7	7
2	1	2	64	64	-6	3	2	42	-41	-8	5	2	11	11	5	6	2	18	17	7	8	2	17	-18
3	1	2	3	-5	-5	3	2	9	-9	-7	5	2	24	25	7	6	2	4	3	8	8	2	11	-10
4	1	2	15	14	-4	3	2	14	-14	-6	5	2	9	8	8	6	2	6	-6	9	8	2	10	-11
5	1	2	52	52	-3	3	2	38	-37	-5	5	2		-10	11	6	2	3	2	-7	9	2	8	9
6	1	2	9	-9	-2	3	2	92	96	-4	5	2		7	-11	7	2	4	2	-5	9	2	7	8
7	1	2	7	6	-1	3	2	79	-85	-3	5	2		-22	-9	7	2	3	4	-4	9	2	3	-1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
9	2	1	4	5	3	4	1	8	10	3	6	1	4	4	2	8	1	22	-21	-6	11	1	9	10
11	2	1	6	6	4	4	1	7	8	4	6	1	19	20	3	8	1	12	-11	-4	11	1	7	7
-11	3	1	4	3	5	4	1	32	-32	5	6	1	3	3	4	8	1	8	-7	-3	11	1	4	2
-9	3	1	3	2	7	4	1	4	4	7	6	1	3	-2	5	8	1	6	-5	-2	11	1	3	4
-8	3	1	6	5	8	4	1	13	13	8	6	1	6	-6	6	8	1	6	6	-1	11	1	7	-4
-7	3	1	14	14	9	4	1	6	-5	10	6	1	3	-5	8	8	1	6	-8	0	11	1	10	10
-6	3	1	6	5	10	4	1	3	2	-10	7	1	3	3	-10	9	1	3	-2	1	11	1	12	-12
-5	3	1	15	-17	11	4	1	3	0	-8	7	1	6	5	-8	9	1	9	-7	3	11	1	5	-6
-3	3	1	11	12	-7	5	1	9	9	-7	7	1	3	-4	-7	9	1	6	6	5	11	1	19	-18
-2	3	1	41	-44	-5	5	1	2	-3	-6	7	1	11	12	-6	9	1	5	-6	6	11	1	4	-5
-1	3	1	87	89	-3	5	1	15	-16	-5	7	1	7	-8	-5	9	1	3	1	7	11	1	4	-4
0	3	1	92	-77	-2	5	1	20	-22	-4	7	1	13	11	-3	9	1	16	-16	9	11	1	3	2
1	3	1	16	-17	-1	5	1	9	-3	-3	7	1	4	-5	-2	9	1	13	14	-8	12	1	5	8
2	3	1	19	22	0	5	1	3	-1	-2	7	1	19	-16	-1	9	1	15	-12	-4	12	1	8	-8
4	3	1	9	8	1	5	1	22	-24	-1	7	1	13	-7	1	9	1	12	-14	-2	12	1	2	4
5	3	1	7	7	2	5	1	12	-12	0	7	1	43	-42	2	9	1	5	-5	-1	12	1	5	4
6	3	1	10	10	3	5	1	4	-7	1	7	1	9	8	3	9	1	10	-11	0	12	1	5	-6
7	3	1	10	9	4	5	1	23	-22	2	7	1	8	6	4	9	1	8	-8	3	12	1	7	-6
8	3	1	3	4	5	5	1	3	1	3	7	1	7	5	5	9	1	11	-11	4	12	1	5	-6
9	3	1	7	-7	6	5	1	6	6	5	7	1	6	8	6	9	1	6	-6	6	12	1	4	-4
10	3	1	7	7	7	5	1	10	9	6	7	1	7	-8	7	9	1	3	-4	-6	13	1	3	3
11	3	1	6	5	8	5	1	10	10	7	7	1	10	11	8	9	1	4	-4	-4	13	1	3	3
12	3	1	4	2	9	5	1	9	-8	8	7	1	3	1	-9	10	1	4	-3	-1	13	1	11	-7
-11	4	1	6	-6	11	5	1	4	-1	11	7	1	3	0	-7	10	1	8	8	0	13	1	10	-11
-7	4	1	5	-4	-10	6	1	6	5	-8	8	1	6	6	-5	10	1	3	4	1	13	1	4	3
-6	4	1	8	-8	-9	6	1	9	9	-7	8	1	6	-4	-3	10	1	5	-4	2	13	1	7	-6
-5	4	1	43	42	-8	6	1	11	12	-6	8	1	3	-4	-2	10	1	11	-12	3	13	1	12	12
-4	4	1	23	23	-7	6	1	3	3	-5	8	1	3	-2	-1	10	1	14	-14	4	13	1	4	5
-3	4	1	23	-23	-6	6	1	7	-6	-4	8	1	8	-7	1	10	1	13	12	-5	14	1	7	8
-2	4	1	10	9	-4	6	1	23	23	-3	8	1	3	2	2	10	1	11	-11	-2	14	1	4	-7
-1	4	1	23	24	-2	6	1	34	-35	-2	8	1	28	-29	3	10	1	8	-9	0	14	1	4	-3
0	4	1	55	52	-1	6	1	7	-7	-1	8	1	8	-7	4	10	1	4	-3	2	14	1	5	-2
1	4	1	14	7	0	6	1	24	-27	0	8	1	13	11	5	10	1	2	4	3	14	1	3	4
2	4	1	19	-14	1	6	1	22	-24	1	8	1	25	-25	6	10	1	10	-11	4	14	1	3	-2

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C24H22NO2FPB, CHOI.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	94	95	1	4	0	11	14	0	8	0	24	26	0	12	0	4	2	-4	1	1	28	-27
4	0	0	39	-38	2	4	0	4	4	1	8	0	8	8	4	12	0	7	-5	-3	1	1	69	70
6	0	0	24	-24	3	4	0	20	20	2	8	0	17	14	5	12	0	10	11	-2	1	1	85	90
10	0	0	7	-8	5	4	0	6	4	3	8	0	8	-8	6	12	0	3	-1	-1	1	1	50	54
12	0	0	4	3	6	4	0	8	-9	4	8	0	3	3	9	12	0	3	4	0	1	1	4	-2
1	1	0	91	98	7	4	0	8	-7	5	8	0	3	3	1	13	0	9	7	1	1	1	42	-44
2	1	0	44	-44	8	4	0	3	4	6	8	0	18	-19	2	13	0	5	-7	2	1	1	12	-13
3	1	0	4	-3	9	4	0	4	-4	7	8	0	10	9	4	13	0	5	-5	3	1	1	21	-22
4	1	0	4	-4	10	4	0	5	4	8	8	0	14	-15	5	13	0	5	-7	4	1	1	11	12
5	1	0	35	-35	1	5	0	2	5	9	8	0	4	4	7	13	0	3	1	5	1	1	16	-15
7	1	0	3	-4	2	5	0	35	-38	1	9	0	25	22	3	14	0	6	5	6	1	1	15	15
8	1	0	11	-13	3	5	0	50	51	2	9	0	3	-2	4	14	0	8	-7	7	1	1	6	-6
11	1	0	3	2	5	5	0	9	9	3	9	0	4	6	5	14	0	4	1	8	1	1	10	-9
0	2	0	66	59	8	5	0	8	7	4	9	0	8	-7	6	14	0	8	7	9	1	1	6	-6
1	2	0	77	77	9	5	0	7	-7	6	9	0	12	12	1	15	0	4	5	10	1	1	9	9
2	2	0	32	-34	0	6	0	45	-42	7	9	0	8	8	2	16	0	4	-3	-12	2	1	3	-1
3	2	0	5	6	1	6	0	16	-9	8	9	0	18	18	-11	0	1	7	-7	-10	2	1	7	6
5	2	0	11	9	2	6	0	26	-27	9	9	0	4	2	-9	0	1	14	-14	-9	2	1	7	7
6	2	0	16	-17	3	6	0	34	-34	0	10	0	44	44	-7	0	1	14	-12	-8	2	1	9	8
7	2	0	15	-13	4	6	0	13	14	1	10	0	6	-4	-5	0	1	8	-8	-6	2	1	3	-2
8	2	0	10	-9	6	6	0	4	-5	2	10	0	11	10	-3	0	1	43	-43	-5	2	1	28	26
9	2	0	8	7	7	6	0	9	10	3	10	0	12	-14	-1	0	1	45	-51	-4	2	1	33	-31
10	2	0	5	-6	8	6	0	8	-4	5	10	0	4	3	1	0	1	17	-18	-3	2	1	82	-81
1	3	0	31	35	9	6	0	6	6	6	10	0	12	-13	3	0	1	13	-13	-2	2	1	103	103
2	3	0	55	55	10	6	0	3	-4	7	10	0	6	6	5	0	1	59	58	-1	2	1	31	30
3	3	0	36	36	2	7	0	18	-20	8	10	0	5	-4	7	0	1	23	22	0	2	1	9	-6
4	3	0	14	12	3	7	0	11	-10	9	10	0	6	5	9	0	1	7	7	1	2	1	21	19
5	3	0	29	-28	4	7	0	11	11	1	11	0	4	-7	11	0	1	4	4	2	2	1	71	-74
6	3	0	24	-23	5	7	0	12	-11	2	11	0	3	0	-11	1	1	4	0	3	2	1	9	-9
7	3	0	7	-6	6	7	0	5	5	3	11	0	4	-4	-10	1	1	9	-9	4	2	1	24	-22
8	3	0	9	-10	7	7	0	18	-19	5	11	0	9	-10	-8	1	1	11	10	5	2	1	17	-18
9	3	0	3	-4	8	7	0	4	6	6	11	0	7	8	-7	1	1	22	22	6	2	1	3	-4
11	3	0	4	1	9	7	0	11	-11	7	11	0	4	-5	-6	1	1	12	-12	7	2	1	19	18
0	4	0	26	-29	10	7	0	5	-4	8	11	0	4	3	-5	1	1	25	24	8	2	1	14	-14

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C7H9NO3, OR03.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-8	2	4	18	18	4	3	4	24	25	-2	5	4	24	-27	-11	1	5	11	-10	1	2	5	9	-6
-7	2	4	19	-18	5	3	4	9	-11	-1	5	4	13	14	-10	1	5	15	17	2	2	5	36	36
-5	2	4	64	-61	6	3	4	35	-32	0	5	4	15	-15	-9	1	5	5	5	3	2	5	35	-33
-4	2	4	9	9	7	3	4	7	7	1	5	4	3	0	-8	1	5	11	14	4	2	5	2	1
-3	2	4	17	17	8	3	4	8	8	2	5	4	7	-7	-7	1	5	21	21	5	2	5	20	21
-2	2	4	26	-21	9	3	4	5	-4	3	5	4	18	-17	-6	1	5	44	-44	6	2	5	11	-10
-1	2	4	14	-13	10	3	4	8	8	4	5	4	2	1	-5	1	5	22	-21	7	2	5	3	-3
0	2	4	18	18	12	3	4	5	4	5	5	4	8	6	-4	1	5	13	-13	8	2	5	4	4
1	2	4	31	35	-11	4	4	3	-4	6	5	4	14	-13	-3	1	5	96	-95	10	2	5	15	-14
2	2	4	13	17	-10	4	4	3	3	7	5	4	5	-5	-2	1	5	40	-38	11	2	5	3	5
3	2	4	32	31	-9	4	4	6	5	8	5	4	8	-8	-1	1	5	76	-79	-10	3	5	7	6
4	2	4	2	-3	-8	4	4	19	-18	9	5	4	9	9	0	1	5	82	-82	-9	3	5	9	-10
5	2	4	24	-23	-7	4	4	8	7	-9	6	4	2	-2	1	1	5	56	-55	-8	3	5	10	10
6	2	4	10	-10	-6	4	4	11	12	-8	6	4	6	6	2	1	5	60	57	-7	3	5	11	-9
7	2	4	13	14	-5	4	4	7	8	-7	6	4	4	3	3	1	5	3	4	-6	3	5	12	13
8	2	4	13	-13	-4	4	4	14	14	-6	6	4	2	-2	4	1	5	37	34	-5	3	5	5	7
9	2	4	3	3	-3	4	4	4	2	-2	6	4	3	-5	5	1	5	15	16	-4	3	5	12	12
10	2	4	4	-4	-2	4	4	38	-39	0	6	4	3	1	6	1	5	17	17	-3	3	5	26	-24
12	2	4	4	4	-1	4	4	25	25	1	6	4	10	-10	7	1	5	9	-11	-2	3	5	25	23
13	2	4	3	-2	1	4	4	7	-7	3	6	4	13	-12	8	1	5	6	6	-1	3	5	36	-39
-11	3	4	3	3	2	4	4	14	-14	7	6	4	6	5	9	1	5	9	-9	0	3	5	9	8
-9	3	4	6	5	4	4	4	4	4	9	6	4	5	-5	10	1	5	8	8	1	3	5	13	-10
-8	3	4	15	16	6	4	4	9	-9	-7	7	4	5	4	11	1	5	5	-5	2	3	5	2	-2
-7	3	4	18	-17	7	4	4	4	4	-4	7	4	3	3	12	1	5	6	-5	3	3	5	16	17
-6	3	4	9	10	8	4	4	4	-2	-3	7	4	3	-4	14	1	5	5	5	4	3	5	23	-24
-5	3	4	13	12	9	4	4	5	6	-2	7	4	3	-2	-11	2	5	6	-6	5	3	5	17	17
-4	3	4	27	-24	-11	5	4	3	4	0	7	4	3	-3	-10	2	5	16	-17	7	3	5	3	5
-3	3	4	49	47	-10	5	4	3	-1	1	7	4	5	-5	-9	2	5	13	12	9	3	5	15	-15
-2	3	4	17	16	-8	5	4	9	9	2	7	4	6	6	-8	2	5	17	17	10	3	5	10	10
-1	3	4	49	-53	-7	5	4	10	9	4	7	4	7	6	-7	2	5	7	-5	-12	4	5	4	4
0	3	4	54	-59	-6	5	4	8	-8	5	7	4	4	5	-6	2	5	15	-13	-11	4	5	5	-4
1	3	4	22	-19	-5	5	4	6	4	6	7	4	9	-7	-4	2	5	12	12	-7	4	5	6	-5
2	3	4	42	-40	-4	5	4	6	5	1	8	4	3	-4	-3	2	5	2	1	-6	4	5	7	7
3	3	4	6	-7	-3	5	4	10	-9	-13	1	5	5	3	-2	2	5	67	67	-5	4	5	16	16

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C7H9NO3, OR03.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	8	2	4	5	-5	2	3	32	-28	9	3	3	6	5	-4	6	3	5	4	9	0	4	13	13
1	8	2	3	2	-4	2	3	22	23	10	3	3	4	-4	-3	6	3	13	-14	10	0	4	16	15
-14	1	3	3	2	-3	2	3	16	15	-11	4	3	5	-4	-2	6	3	9	9	11	0	4	18	20
-13	1	3	6	-6	-2	2	3	73	-72	-10	4	3	6	6	0	6	3	9	-9	12	0	4	3	3
-12	1	3	9	10	-1	2	3	11	8	-8	4	3	8	8	3	6	3	5	-6	13	0	4	6	5
-11	1	3	4	-3	0	2	3	9	10	-6	4	3	5	-7	6	6	3	7	7	-13	1	4	7	6
-10	1	3	15	-16	1	2	3	34	-37	-5	4	3	22	-22	7	6	3	12	-10	-11	1	4	14	-13
-9	1	3	4	4	2	2	3	9	-7	-4	4	3	7	7	9	6	3	7	6	-10	1	4	14	-13
-8	1	3	7	-8	3	2	3	2	-3	-3	4	3	19	-17	-5	7	3	4	-4	-9	1	4	4	4
-7	1	3	14	-14	4	2	3	24	21	-2	4	3	7	-7	-1	7	3	3	-3	-8	1	4	7	-8
-6	1	3	40	-37	5	2	3	5	-4	-1	4	3	7	-7	0	7	3	4	-4	-7	1	4	15	15
-5	1	3	17	-17	6	2	3	24	24	0	4	3	16	-15	3	7	3	4	3	-6	1	4	35	-32
-4	1	3	24	21	7	2	3	18	19	1	4	3	32	-32	6	7	3	3	4	-5	1	4	36	37
-3	1	3	22	21	8	2	3	2	2	2	4	3	23	23	-2	8	3	4	4	-4	1	4	27	-24
-2	1	3	65	63	9	2	3	3	2	3	4	3	10	11	2	8	3	6	5	-3	1	4	44	-44
-1	1	3	73	-74	12	2	3	4	2	4	4	3	10	-10	-14	0	4	4	-3	-2	1	4	82	79
0	1	3	3	2	-13	3	3	2	0	5	4	3	10	11	-13	0	4	6	6	-1	1	4	75	-74
1	1	3	125	125	-10	3	3	10	9	7	4	3	19	-19	-12	0	4	6	-5	0	1	4	45	44
2	1	3	32	-30	-8	3	3	10	-9	8	4	3	29	29	-11	0	4	6	-4	1	1	4	159	158
3	1	3	13	-15	-7	3	3	10	-11	9	4	3	5	4	-10	0	4	7	0	2	1	4	16	-17
4	1	3	9	8	-5	3	3	11	11	-7	5	3	4	4	-8	0	4	17	-16	3	1	4	70	-66
5	1	3	3	-2	-4	3	3	13	12	-5	5	3	16	-15	-7	0	4	25	-26	4	1	4	8	-9
6	1	3	31	27	-3	3	3	21	22	-4	5	3	22	-23	-6	0	4	63	-62	5	1	4	23	-24
8	1	3	18	19	-2	3	3	14	-17	-3	5	3	11	-12	-5	0	4	8	-5	6	1	4	14	-12
9	1	3	9	9	-1	3	3	48	-53	-2	5	3	8	-6	-4	0	4	16	-15	7	1	4	28	29
14	1	3	2	-4	0	3	3	46	49	0	5	3	9	10	-3	0	4	70	73	8	1	4	8	-6
-13	2	3	4	-3	1	3	3	13	14	2	5	3	16	-18	-2	0	4	187	-185	9	1	4	12	-12
-12	2	3	4	-3	2	3	3	18	-14	4	5	3	20	21	-1	0	4	60	52	10	1	4	9	-10
-11	2	3	3	-4	3	3	3	11	12	5	5	3	19	16	1	0	4	7	3	11	1	4	10	12
-10	2	3	8	6	4	3	3	16	-16	6	5	3	9	8	2	0	4	74	68	12	1	4	2	-4
-9	2	3	8	8	5	3	3	12	-9	7	5	3	5	-6	3	0	4	14	-11	13	1	4	5	5
-8	2	3	21	-20	6	3	3	3	-2	9	5	3	4	-2	4	0	4	20	-19	14	1	4	3	2
-7	2	3	4	5	7	3	3	12	-11	10	5	3	5	4	5	0	4	19	18	-10	2	4	10	-10
-6	2	3	43	43	8	3	3	9	8	-6	6	3	7	-5	6	0	4	25	-27	-9	2	4	11	13

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	5	1	14	-13	-3	0	2	47	51	8	1	2	15	-14	-7	3	2	14	-12	11	4	2	4	-3
5	5	1	10	7	-2	0	2	31	22	9	1	2	10	-10	-6	3	2	30	-27	-11	5	2	5	5
6	5	1	9	9	2	0	2	147	153	10	1	2	7	-8	-5	3	2	32	32	-9	5	2	5	4
9	5	1	4	2	3	0	2	93	-91	11	1	2	14	-14	-4	3	2	8	9	-7	5	2	7	-7
-4	6	1	16	13	4	0	2	72	-74	12	1	2	3	-3	-3	3	2	8	-9	-6	5	2	7	-7
-2	6	1	3	3	5	0	2	37	36	13	1	2	5	5	-2	3	2	9	8	-4	5	2	22	-22
-1	6	1	11	-12	6	0	2	23	-22	-12	2	2	12	-12	-1	3	2	56	-61	-3	5	2	8	7
0	6	1	16	-15	7	0	2	34	35	-11	2	2	3	3	0	3	2	24	26	-2	5	2	7	-6
2	6	1	7	-6	8	0	2	20	20	-10	2	2	21	21	2	3	2	21	-20	-1	5	2	17	18
3	6	1	8	7	9	0	2	7	-6	-9	2	2	7	-7	3	3	2	37	38	0	5	2	11	-12
4	6	1	6	5	10	0	2	7	-7	-8	2	2	13	14	4	3	2	4	4	1	5	2	5	-5
5	6	1	8	-7	11	0	2	4	-6	-7	2	2	18	-18	5	3	2	20	-18	4	5	2	6	-7
6	6	1	9	-7	12	0	2	7	7	-6	2	2	12	-14	6	3	2	14	15	6	5	2	12	-13
7	6	1	11	10	-14	1	2	3	-4	-5	2	2	78	74	7	3	2	6	-7	7	5	2	15	15
8	6	1	9	8	-13	1	2	4	3	-4	2	2	12	12	8	3	2	6	-5	9	5	2	6	6
-6	7	1	7	5	-11	1	2	7	8	-3	2	2	11	12	9	3	2	5	5	10	5	2	6	-5
-4	7	1	3	-2	-10	1	2	9	9	-2	2	2	9	9	10	3	2	4	4	-7	6	2	4	-4
-1	7	1	4	5	-9	1	2	11	-12	-1	2	2	38	-40	-10	4	2	6	-6	-6	6	2	7	-6
0	7	1	6	-4	-8	1	2	11	-9	0	2	2	48	-50	-9	4	2	7	-7	-5	6	2	7	7
1	7	1	6	5	-7	1	2	26	25	1	2	2	10	-7	-6	4	2	7	7	-4	6	2	9	-9
2	7	1	11	-10	-6	1	2	10	11	2	2	2	17	-18	-5	4	2	13	-14	-2	6	2	15	15
4	7	1	3	4	-5	1	2	5	3	3	2	2	56	-54	-4	4	2	15	15	-1	6	2	5	4
5	7	1	11	-9	-4	1	2	48	-45	4	2	2	20	18	-3	4	2	3	-4	0	6	2	5	5
-2	8	1	3	-2	-3	1	2	72	-74	5	2	2	32	29	-2	4	2	4	5	2	6	2	4	2
1	8	1	4	-4	-2	1	2	20	-16	7	2	2	3	-1	-1	4	2	15	16	4	6	2	7	8
3	8	1	4	-3	-1	1	2	165	159	8	2	2	6	-5	0	4	2	5	3	5	6	2	10	10
-11	0	2	10	-9	0	1	2	48	51	9	2	2	3	-2	1	4	2	12	13	6	6	2	10	-7
-10	0	2	29	-31	1	1	2	50	47	10	2	2	8	9	2	4	2	15	14	8	6	2	5	-5
-9	0	2	17	-18	2	1	2	39	-40	11	2	2	8	-9	3	4	2	16	-15	-7	7	2	6	-5
-8	0	2	17	16	3	1	2	36	32	12	2	2	3	-4	4	4	2	5	-5	-5	7	2	3	-2
-7	0	2	22	-20	4	1	2	7	-12	13	2	2	3	3	5	4	2	11	-11	-3	7	2	6	5
-6	0	2	62	61	5	1	2	15	16	-11	3	2	3	2	6	4	2	3	4	1	7	2	3	-4
-5	0	2	21	27	6	1	2	25	-22	-10	3	2	11	10	8	4	2	11	-11	3	7	2	5	4
-4	0	2	4	4	7	1	2	13	12	-9	3	2	6	6	10	4	2	8	8	4	7	2	7	-6

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C7H9NO3, OR03.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	25	-26	13	2	0	2	-1	2	6	0	3	3	-11	2	1	3	1	3	3	1	38	35
3	0	0	42	39	1	3	0	3	0	3	6	0	7	8	-9	2	1	2	4	4	3	1	27	-26
4	0	0	65	-62	2	3	0	3	1	4	6	0	5	4	-7	2	1	24	-23	5	3	1	6	6
5	0	0	8	12	3	3	0	10	10	6	6	0	6	-4	-6	2	1	26	28	6	3	1	21	21
6	0	0	35	37	4	3	0	31	29	7	6	0	6	7	-5	2	1	9	-8	7	3	1	22	-22
7	0	0	38	-39	5	3	0	20	-18	1	7	0	5	4	-4	2	1	53	49	8	3	1	4	3
8	0	0	3"	-39	6	3	0	6	4	2	7	0	7	5	-3	2	1	16	15	9	3	1	13	13
9	0	0	19	-20	7	3	0	4	-4	3	7	0	4	4	-2	2	1	67	-65	-8	4	1	8	-9
10	0	0	7	7	8	3	0	13	12	6	7	0	6	-6	-1	2	1	24	23	-6	4	1	13	-12
11	0	0	14	-14	9	3	0	13	12	7	7	0	4	2	0	2	1	11	12	-5	4	1	2	-2
12	0	0	3	3	11	3	0	2	1	2	8	0	6	-6	1	2	1	20	-21	-4	4	1	6	-7
13	0	0	4	4	13	3	0	3	1	3	8	0	3	2	2	2	1	12	-11	-3	4	1	17	-13
14	0	0	5	-5	0	4	0	34	34	-12	1	1	4	-5	3	2	1	19	-20	-2	4	1	15	-12
1	1	0	48	46	1	4	0	6	-6	-9	1	1	24	23	4	2	1	28	-27	-1	4	1	2	0
2	1	0	8	-11	2	4	0	21	21	-8	1	1	24	-24	5	2	1	13	-12	0	4	1	4	4
3	1	0	57	52	3	4	0	21	-20	-7	1	1	11	12	6	2	1	26	-24	1	4	1	22	-23
4	1	0	105	-99	5	4	0	5	4	-6	1	1	3	-3	7	2	1	3	3	3	4	1	17	-18
5	1	0	56	55	6	4	0	5	-5	-5	1	1	22	-20	8	2	1	7	-7	4	4	1	26	25
6	1	0	12	13	7	4	0	7	-10	-4	1	1	10	10	9	2	1	6	6	5	4	1	8	-6
8	1	0	5	4	8	4	0	12	12	-3	1	1	147	-144	10	2	1	4	4	6	4	1	15	16
9	1	0	17	-17	9	4	0	5	-5	-2	1	1	109	-106	11	2	1	11	-13	8	4	1	6	6
10	1	0	12	-12	10	4	0	5	-4	-1	1	1	2	[illegible]	12	2	1	5	6	10	4	1	3	-3
11	1	0	5	5	11	4	0	6	-4	0	1	1	[illegible]	[illegible]	13	2	1	3	3	11	4	1	5	5
0	2	0	4	4	12	4	0	3	3	1	1	1	[illegible]	[illegible]	11	3	1	4	-3	-11	5	1	3	3
1	2	0	45	46	1	5	0	7	10	2	1	1	1	1	10	3	1	4	5	-10	5	1	5	-6
2	2	0	8	-7	2	5	0	11	10	3	1	1	15	[illegible]	-8	3	1	2	1	-9	5	1	6	5
3	2	0	88	85	3	5	0	5	4	4	1	1	82	81	-7	3	1	19	17	-7	5	1	4	-3
4	2	0	21	19	4	5	0	9	9	5	1	1	50	49	-6	3	1	26	-26	-6	5	1	5	-8
5	2	0	25	-24	5	5	0	11	11	6	1	1	30	30	-4	3	1	12	13	-4	5	1	13	13
6	2	0	16	-16	8	5	0	3	1	7	1	1	28	30	-3	3	1	20	-21	-3	5	1	2	-1
7	2	0	18	-20	9	5	0	5	-5	9	1	1	5	-6	-2	3	1	58	-61	0	5	1	6	6
9	2	0	5	-5	11	5	0	4	3	10	1	1	10	-11	-1	3	1	53	54	1	5	1	27	-28
10	2	0	5	3	0	6	0	5	-5	11	1	1	10	10	0	3	1	50	-51	2	5	1	14	17
12	2	0	4	4	1	6	0	4	-4	12	1	1	5	-6	1	3	1	12	-9	3	5	1	4	5

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-9	3	13	7	7	-8	6	13	7	-7	1	1	14	5	-8	-4	4	14	15	15	-7	3	15	12	-11
-7	3	13	5	6	-4	6	13	5	-5	-14	2	14	6	8	-2	4	14	12	13	-5	3	15	5	-5
-5	3	13	5	5	-2	6	13	11	-12	-10	2	14	4	-5	2	4	14	6	10	-3	3	15	10	9
-3	3	13	24	23	-14	0	14	9	-10	-6	2	14	9	9	-9	5	14	21	20	-1	3	15	10	10
-1	3	13	15	19	-12	0	14	8	-9	-4	2	14	17	17	-5	5	14	10	-9	-4	4	15	10	10
1	3	13	6	-9	-10	0	14	4	-4	0	2	14	6	-8	-11	1	15	5	3	-2	4	15	12	12
3	3	13	6	-11	-6	0	14	3	4	-13	3	14	3	-6	-9	1	15	8	7	-6	0	16	13	-12
-12	4	13	6	-9	-4	0	14	5	-4	-9	3	14	7	7	-7	1	15	5	4	-4	0	16	5	-5
-10	4	13	12	-12	0	0	14	10	15	-5	3	14	8	8	-5	1	15	7	-7	-11	1	16	13	-12
-8	4	13	3	-3	4	0	14	4	-11	-3	3	14	29	28	-3	1	15	13	-12	-9	1	16	5	-5
-6	4	13	13	12	-11	1	14	10	10	-1	3	14	11	13	-1	1	15	5	-6	-6	2	16	5	-5
-4	4	13	11	11	-9	1	14	18	18	3	3	14	12	25	-6	2	15	3	-5	-4	2	16	4	4
2	4	13	3	-5	-7	1	14	10	11	-12	4	14	9	11	-4	2	15	6	-6	-2	2	16	5	5
-5	5	13	7	-7	-5	1	14	4	4	-10	4	14	17	18	0	2	15	4	4	-7	3	16	6	-5
-3	5	13	7	-8	-1	1	14	7	-9	-8	4	14	5	6	-9	3	15	9	-9					

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
10	2	9	8	-7	2	8	9	9	-10	7	3	10	11	-10	4	2	11	26	26	4	0	12	5	13
-17	3	9	6	-6	-16	0	10	4	4	9	3	10	3	-3	6	2	11	30	30	6	0	12	5	13
-15	3	9	4	-4	-14	0	10	7	8	-16	4	10	4	4	-15	3	11	1	-11	-11	1	12	3	4
-13	3	9	6	-6	-10	0	10	25	-23	-14	4	10	9	-8	-13	3	11	1	-26	-9	1	12	3	-3
-11	3	9	3	-1	-8	0	10	36	-34	-10	4	10	5	5	-11	3	11	2	-10	-7	1	12	13	-13
-9	3	9	16	16	-6	0	10	19	-20	-8	4	10	21	-22	-9	3	11	19	20	-1	1	12	14	-21
-7	3	9	16	16	-4	0	10	8	-8	-6	4	10	19	-18	-7	3	11	19	21	-14	2	12	7	-11
-5	3	9	7	-7	-2	0	10	7	-5	-4	4	10	14	14	-3	3	11	11	-10	-12	2	12	4	-6
-3	3	9	11	-11	0	0	10	9	9	-2	4	10	15	-15	-1	3	11	13	-14	-10	2	12	8	8
-1	3	9	4	4	2	0	10	28	26	0	4	10	47	-46	1	3	11	4	-6	-8	2	12	7	7
1	3	9	15	15	4	0	10	24	22	2	4	10	15	-16	3	3	11	9	9	-6	2	12	8	-7
3	3	9	8	8	6	0	10	5	-5	4	4	10	4	-4	5	3	11	11	12	-4	2	12	10	-11
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-8	4	9	5	5	-11	1	10	17	17	-1	5	10	45	-46	-4	4	11	12	13	1	3	12	6	8
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2	4	9	24	-22	-3	1	10	22	-21	-8	6	10	15	-15	-9	5	11	8	-8	-2	4	12	4	-4
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6	4	9	6	4	3	1	10	20	-18	-2	6	10	15	15	-5	5	11	4	-5	2	4	12	5	-7
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-15	5	9	3	4	-14	2	10	11	-10	2	6	10	6	7	-1	5	11	19	-18	-13	5	12	4	-5
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-7	5	9	12	12	-8	2	10	23	-22	-3	7	10	5	-5	-10	6	11	6	-6	-5	5	12	7	7
-5	5	9	4	3	-6	2	10	43	-41	-1	7	10	7	-7	-8	6	11	13	-14	-3	5	12	9	9
-3	5	9	18	-18	-4	2	10	6	-7	1	7	10	7	-6	-6	6	11	11	-12	-1	5	12	8	-8
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-10	6	9	7	6	4	2	10	22	-20	-17	1	11	6	-5	4	6	11	9	-9	0	6	12	9	-9
-6	6	9	7	-6	6	2	10	47	-45	-13	1	11	8	8	-9	7	11	5	5	-7	7	12	3	-3
-2	6	9	5	5	8	2	10	18	-17	-11	1	11	8	9	-7	7	11	6	5	-3	7	12	8	8
2	6	9	5	4	-15	3	10	14	-13	-7	1	11	5	-5	-5	7	11	6	5	-1	7	12	9	9
6	6	9	11	-11	-13	3	10	28	-26	-3	1	11	19	21	-1	7	11	10	-10	-15	1	13	4	5
-9	7	9	10	-10	-11	3	10	18	-17	-1	1	11	22	23	1	7	11	5	-5	-9	1	13	12	11
-7	7	9	12	-11	-9	3	10	20	-21	1	1	11	10	-11	-4	8	11	12	-12	-7	1	13	5	5
-3	7	9	4	2	-7	3	10	31	-30	3	1	11	21	-21	-12	0	12	6	-6	-3	1	13	18	20
1	7	9	14	13	-5	3	10	8	-7	7	1	11	7	7	-10	0	12	12	12	-1	1	13	12	15
3	7	9	4	4	-3	3	10	2	-5	-16	2	11	5	6	-8	0	12	35	34	-14	2	13	8	13
5	7	9	12	-11	-1	3	10	12	-11	-14	2	11	8	9	-6	0	12	16	15	-8	2	13	7	7
-8	8	9	4	4	1	3	10	14	14	-10	2	11	8	-8	-4	0	12	7	-7	-6	2	13	2	-2
-6	8	9	3	-2	3	3	10	11	10	-2	2	11	18	18	-2	0	12	19	-23	-2	2	13	5	-6
0	8	9	15	-13	5	3	10	17	-16	0	2	11	16	16	0	0	12	11	-16	0	2	13	9	-13

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-11	7	6	9	-9	-15	3	7	9	8	-5	7	7	14	16	-12	2	8	6	5	-6	6	8	17	17
-9	7	6	11	-11	-13	3	7	6	6	-3	7	7	28	27	-10	2	8	19	-17	-4	6	8	18	17
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-5	7	6	6	7	-9	3	7	12	-12	1	7	7	18	-17	-6	2	8	65	65	0	6	8	4	-4
-1	7	6	9	-10	-7	3	7	29	-28	3	7	7	17	-17	-4	2	8	70	70	2	6	8	8	8
1	7	6	18	-18	-5	3	7	36	-35	-10	8	7	5	-4	-2	2	8	19	-18	4	6	8	6	-5
3	7	6	20	-19	-3	3	7	7	7	-8	8	7	13	-12	0	2	8	31	-30	6	6	8	5	6
5	7	6	8	-8	-1	3	7	12	11	-6	8	7	13	-12	2	2	8	6	5	8	6	8	14	13
7	7	6	7	6	1	3	7	32	-32	-4	8	7	7	-7	4	2	8	5	6	-11	7	8	3	1
9	7	6	5	4	3	3	7	43	-42	-2	8	7	10	-9	6	2	8	10	10	-7	7	8	17	17
-6	8	6	8	8	5	3	7	13	-14	0	8	7	15	-14	8	2	8	22	21	-5	7	8	29	28
-4	8	6	3	2	7	3	7	6	-5	2	8	7	5	-4	10	2	8	6	6	-3	7	8	7	7
0	8	6	11	11	11	3	7	8	7	4	8	7	4	4	12	2	8	5	-6	-1	7	8	10	-9
2	8	6	9	8	-16	4	7	13	-13	-7	9	7	4	5	-17	3	8	9	9	1	7	8	7	6
4	8	6	8	-8	-12	4	7	28	27	-5	9	7	4	-3	-7	3	8	5	6	3	7	8	15	14
-9	9	6	6	-7	-10	4	7	34	32	1	9	7	19	-18	-5	3	8	24	22	5	7	8	11	10
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-5	9	6	7	-7	-4	4	7	51	47	-18	0	8	6	-5	-1	3	8	15	14	-10	8	8	4	-4
-3	9	6	9	-9	-2	4	7	31	30	-16	0	8	10	-10	1	3	8	7	-7	-6	8	8	7	6
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1	9	6	20	19	2	4	7	5	-4	-12	0	8	27	25	5	3	8	3	2	0	8	8	5	5
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-9	1	7	22	22	10	4	7	7	7	-4	0	8	49	50	-12	4	8	20	19	1	9	8	17	16
-7	1	7	47	47	-15	5	7	6	5	-2	0	8	11	10	-10	4	8	30	28	-13	1	9	14	-12
-5	1	7	19	-19	-13	5	7	5	-5	0	0	8	72	70	-8	4	8	5	4	-11	1	9	10	-9
-3	1	7	24	-23	-7	5	7	8	-7	2	0	8	79	76	-2	4	8	19	-18	-7	1	9	15	-15
-1	1	7	8	7	-5	5	7	16	-15	4	0	8	19	-17	0	4	8	13	-13	-5	1	9	21	-20
1	1	7	9	-9	-3	5	7	26	-26	6	0	8	8	-8	4	4	8	10	-10	-3	1	9	19	18
5	1	7	20	-19	-1	5	7	14	-14	8	0	8	36	35	6	4	8	14	-14	1	1	9	40	39
7	1	7	62	-61	1	5	7	20	19	10	0	8	13	13	8	4	8	7	6	3	1	9	19	16
9	1	7	43	-42	3	5	7	25	25	12	0	8	5	4	10	4	8	11	10	5	1	9	5	-6
11	1	7	4	4	5	5	7	12	13	-15	1	8	8	8	-13	5	8	16	15	7	1	9	7	-6
13	1	7	15	13	7	5	7	8	8	-13	1	8	10	10	-11	5	8	34	33	9	1	9	12	-10
-18	2	7	10	9	-14	6	7	5	-4	-11	1	8	19	18	-9	5	8	15	15	11	1	9	6	-5
-16	2	7	10	10	-10	6	7	5	5	-9	1	8	13	13	-7	5	8	14	-14	-14	2	9	8	-8
-14	2	7	10	9	-6	6	7	12	11	-7	1	8	30	-28	-5	5	8	12	-12	-12	2	9	10	-10
-12	2	7	7	7	-4	6	7	11	11	-5	1	8	39	-37	-3	5	8	9	-9	-10	2	9	11	10
-8	2	7	22	-21	-2	6	7	15	-14	-3	1	8	3	-3	-1	5	8	10	-10	-8	2	9	8	-8
-6	2	7	66	-66	0	6	7	16	14	-1	1	8	28	28	1	5	8	21	21	-6	2	9	54	-52
-4	2	7	58	-56	2	6	7	bb	b3	1	1	8	30	26	3	5	8	35	33	-4	2	9	29	-28
2	2	7	12	11	4	6	7	19	19	3	1	8	8	-10	5	5	8	5	5	-2	2	9	11	-11
4	2	7	40	38	6	6	7	16	-15	5	1	8	14	-13	9	5	8	4	4	0	2	9	27	-25
6	2	7	10	11	8	6	7	5	-4	7	1	8	12	11	-14	6	8	15	-14	2	2	9	5	-6
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12	2	7	6	5	-9	7	7	4	4	-16	2	8	13	13	-10	6	8	5	4	6	2	9	10	9
-17	3	7	9	-8	-7	7	7	12	12	-14	2	8	8	7	-8	6	8	4	3	8	2	9	9	-9

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C22H2004, OR02.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-6	4	4	4	-4	0	8	4	12	-12	1	3	5	3	-4	-1	7	5	14	-14	0	2	6	5	-4
-4	4	4	27	27	2	8	4	22	-21	3	3	5	35	36	1	7	5	3	3	2	2	6	35	-35
-2	4	4	14	-15	4	8	4	32	-31	5	3	5	10	10	3	7	5	17	16	4	2	6	28	-28
0	4	4	60	-61	6	8	4	10	-10	7	3	5	20	-20	-10	8	5	6	6	6	2	6	25	24
2	4	4	16	-16	8	8	4	6	5	9	3	5	6	-5	-8	8	5	7	8	8	2	6	14	15
4	4	4	14	14	-9	9	4	9	-10	11	3	5	17	17	-6	8	5	12	12	10	2	6	11	-11
6	4	4	8	-5	-7	9	4	4	3	13	3	5	5	6	-2	8	5	16	-16	12	2	6	5	4
10	4	4	14	15	-5	9	4	5	5	-12	4	5	3	-5	2	8	5	14	13	-17	3	6	21	21
-13	5	4	14	14	3	9	4	6	4	-10	4	5	3	-5	4	8	5	6	6	-13	3	6	9	-8
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-9	5	4	22	-22	-4	10	4	3	-5	-6	4	5	4	-4	-9	9	5	23	22	-9	3	6	27	-26
-7	5	4	18	-17	-2	10	4	6	-7	-4	4	5	31	-32	-5	9	5	10	-10	-7	3	6	28	-29
-3	5	4	28	-27	-17	1	5	13	13	-2	4	5	7	-7	-1	9	5	4	-6	-5	3	6	83	83
-1	5	4	30	-29	-15	1	5	19	20	0	4	5	6	8	3	9	5	5	5	-3	3	6	75	73
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7	5	4	23	24	-7	1	5	16	-16	6	4	5	9	-10	-14	0	6	9	9	3	3	6	32	33
9	5	4	9	-10	-5	1	5	43	46	8	4	5	10	-10	-12	0	6	49	-49	7	3	6	21	21
11	5	4	18	-18	-3	1	5	85	86	10	4	5	12	13	-8	0	6	36	-35	9	3	6	22	22
13	5	4	4	4	-1	1	5	31	31	-15	5	5	3	-3	-6	0	6	45	-45	-12	4	6	31	31
-14	6	4	11	-11	1	1	5	10	-10	-13	5	5	4	-4	0	0	6	111	-114	-10	4	6	48	46
-12	6	4	10	-10	3	1	5	38	-37	-11	5	5	5	5	-2	0	6	14	-12	-8	4	6	22	22
-10	6	4	25	-26	5	1	5	13	-14	-9	5	5	6	-6	0	0	6	65	65	-6	4	6	7	-7
-8	6	4	36	-36	7	1	5	3	-4	-7	5	5	23	-23	2	0	6	29	28	-2	4	6	6	-5
-6	6	4	10	-11	9	1	5	14	-14	-5	5	5	3	4	4	0	6	32	31	0	4	6	34	34
-4	6	4	8	-8	13	1	5	4	4	-3	5	5	34	35	6	0	6	28	29	2	4	6	41	40
-2	6	4	3	1	-18	2	5	7	7	3	5	5	37	36	10	0	6	19	-18	8	4	6	5	5
0	6	4	36	37	-16	2	5	20	19	5	5	5	16	15	12	0	6	10	-9	12	4	6	5	6
2	6	4	14	15	-14	2	5	6	6	7	5	5	5	-5	14	0	6	6	7	-15	5	6	5	-8
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6	6	4	43	-43	-10	2	5	12	-12	11	5	5	15	15	-9	1	6	5	-5	-11	5	6	26	26
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-13	7	4	6	6	-2	2	5	38	37	-8	6	5	20	-21	-3	1	6	20	20	-5	5	6	4	4
-11	7	4	21	19	0	2	5	28	28	-6	6	5	17	17	-1	1	6	7	-6	-1	5	6	13	-13
-7	7	4	8	-8	2	2	5	54	-52	-4	6	5	23	23	1	1	6	12	13	1	5	6	9	9
-5	7	4	16	-16	4	2	5	65	-62	-2	6	5	10	10	3	1	6	7	-6	3	5	6	24	25
-3	7	4	23	-22	6	2	5	12	12	0	6	5	16	16	5	1	6	27	-25	5	5	6	4	-4
-1	7	4	4	1	10	2	5	19	-19	4	6	5	20	-21	7	1	6	16	16	7	5	6	11	-12
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3	7	4	35	-35	-17	3	5	6	7	8	6	5	13	12	11	1	6	15	14	-10	6	6	3	-2
5	7	4	32	-32	-15	3	5	9	9	10	6	5	3	5	-16	2	6	4	-4	-8	6	6	4	3
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-8	8	4	13	-13	-7	3	5	9	8	-9	7	5	4	4	-8	2	6	5	4	0	6	6	33	33
-6	8	4	4	-3	-5	3	5	26	25	-7	7	5	11	-11	-6	2	6	51	51	2	6	6	48	46
-4	8	4	17	-18	-3	3	5	41	43	-5	7	5	5	5	-4	2	6	37	37	4	6	6	32	31
-2	8	4	21	-23	-1	3	5	13	-12	-3	7	5	6	5	-2	2	6	15	-16	6	6	6	9	9

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-8	4	2	26	-27	6	8	2	11	11	-3	3	3	[illegible]	-33	-7	7	3	4	3	-9	1	4	36	-36
-6	4	2	30	30	8	8	2	4	4	-1	3	3	7[illegible]	-80	-5	7	3	16	16	-7	1	4	29	30
-4	4	2	22	22	10	8	2	13	-12	1	3	3	24	-22	-3	7	3	6	7	-5	1	4	10	-8
-2	4	2	67	-66	-9	9	2	18	-16	3	3	3	33	32	-1	7	3	6	6	-3	1	4	293	-262
0	4	2	41	-41	-5	9	2	5	5	5	3	3	23	22	1	7	3	26	25	-1	1	4	204	-177
2	4	2	13	13	-3	9	2	9	-10	7	3	3	9	-9	5	7	3	43	-43	1	1	4	26	26
4	4	2	11	-10	1	9	2	11	11	9	3	3	15	-16	7	7	3	24	-24	3	1	4	65	-63
6	4	2	3	3	3	9	2	6	-6	11	3	3	8	-8	9	7	3	5	4	5	1	4	43	-43
8	4	2	13	12	5	9	2	8	-7	15	3	3	3	-2	11	7	3	5	-5	7	1	4	30	30
10	4	2	2	2	-6	10	2	6	-5	-14	4	3	22	-23	-10	8	3	13	13	9	1	4	21	-20
-15	5	2	6	5	-4	10	2	8	-7	-12	4	3	19	-20	-8	8	3	11	12	11	1	4	45	-44
-11	5	2	13	-14	2	10	2	8	-6	-10	4	3	20	21	-6	8	3	19	-19	13	1	4	4	-5
-9	5	2	29	-31	4	10	2	5	-5	-8	4	3	51	53	-4	8	3	24	-26	-16	2	4	14	-14
-7	5	2	31	-31	-17	1	3	11	-11	-6	4	3	49	50	-2	8	3	18	-19	-14	2	4	12	-12
-5	5	2	18	-18	-13	1	3	3	4	-4	4	3	20	20	0	8	3	22	-22	-12	2	4	7	8
-3	5	2	23	23	-11	1	3	17	-16	-2	4	3	13	-14	2	8	3	12	12	-10	2	4	23	24
-1	5	2	38	39	-9	1	3	19	-19	2	4	3	8	8	4	8	3	38	38	-6	2	4	50	-52
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9	5	2	6	-5	3	1	3	58	-58	-13	5	3	13	13	1	9	3	14	13	4	2	4	47	-45
11	5	2	6	-8	5	1	3	59	-56	-9	5	3	56	-58	3	9	3	9	9	6	2	4	25	-24
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0	6	2	6	5	-12	2	3	6	-6	7	5	3	29	30	-14	0	4	12	-13	-11	3	4	8	-8
2	6	2	13	15	-10	2	3	15	14	9	5	3	23	24	-12	0	4	33	-34	-9	3	4	14	-15
4	6	2	9	9	-8	2	3	39	41	11	5	3	5	-7	-10	0	4	55	-54	-7	3	4	5	-6
6	6	2	5	5	-6	2	3	42	-43	13	5	3	10	-12	-8	0	4	56	-57	-5	3	4	7	7
8	6	2	13	14	-4	2	3	20	-20	-10	6	3	7	-6	-6	0	4	37	37	-3	3	4	6	5
12	6	2	10	-10	-2	2	3	99	102	-8	6	3	24	-23	-4	0	4	88	90	-1	3	4	23	22
-13	7	2	9	8	0	2	3	10	10	-6	6	3	16	-15	-2	0	4	4	-5	1	3	4	59	59
-7	7	2	15	15	2	2	3	20	-19	-4	6	3	14	13	0	0	4	12	-12	3	3	4	21	21
-3	7	2	12	-10	4	2	3	56	56	-2	6	3	18	16	2	0	4	48	-47	5	3	4	34	-32
1	7	2	13	-13	6	2	3	59	58	0	6	3	8	-8	4	0	4	13	13	7	3	4	20	-19
3	7	2	37	-37	8	2	3	44	43	2	6	3	18	-20	6	0	4	56	55	9	3	4	3	-1
5	7	2	29	-30	10	2	3	30	31	4	6	3	16	-16	8	0	4	30	-28	11	3	4	13	-13
9	7	2	4	4	14	2	3	13	-12	6	6	3	6	-6	10	0	4	80	-80	13	3	4	10	-10
11	7	2	4	-5	-17	3	3	5	3	8	6	3	4	4	12	0	4	36	-36	-16	4	4	8	7
-8	8	2	19	20	-13	3	3	7	-7	10	6	3	4	6	14	0	4	4	3	-14	4	4	5	4
-6	8	2	29	28	-11	3	3	9	-8	12	6	3	3	-2	-17	1	4	9	-9	-12	4	4	9	-9
-4	8	2	4	3	-9	3	3	3	4	-13	7	3	7	-6	-15	1	4	10	-10	-10	4	4	19	-20
0	8	2	9	9	-7	3	3	26	25	-11	7	3	3	-3	-13	1	4	6	6	-8	4	4	19	-20

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C22H2004, OR02.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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6	0	0	27	26	8	6	0	15	-16	12	2	1	15	16	6	6	1	59	-59	-15	1	2	22	22
10	0	0	17	-18	10	6	0	33	-33	14	2	1	6	-6	8	6	1	21	-21	-13	1	2	32	33
12	0	0	31	32	12	6	0	4	-4	16	2	1	4	-5	12	6	1	6	-6	-11	1	2	6	-6
14	0	0	21	21	14	6	0	3	0	-13	3	1	5	4	-13	7	1	5	5	-9	1	2	18	17
16	0	0	10	-10	1	7	0	8	-8	-11	3	1	7	7	-7	7	1	17	17	-7	1	2	60	61
3	1	0	11	-12	3	7	0	7	-8	-9	3	1	17	16	-5	7	1	9	10	-5	1	2	9	-9
5	1	0	44	44	5	7	0	45	45	7	3	1	16	16	-3	7	1	16	-16	-3	1	2	17	-17
7	1	0	13	13	7	7	0	62	63	-3	3	1	40	39	1	7	1	3	-4	-1	1	2	95	84
9	1	0	8	7	9	7	0	16	17	-1	3	1	50	51	3	7	1	37	-37	1	1	2	43	49
11	1	0	30	31	13	7	0	7	7	1	3	1	34	-34	5	7	1	26	-25	3	1	2	14	-16
13	1	0	25	25	0	8	0	3	2	3	3	1	23	-23	7	7	1	3	4	7	1	2	11	-12
17	1	0	4	-4	2	8	0	3	-4	5	3	1	92	91	9	7	1	4	5	9	1	2	27	-29
0	2	0	29	31	6	8	0	14	15	7	3	1	74	74	11	7	1	3	4	11	1	2	8	-7
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6	2	0	83	82	1	9	0	32	34	-12	4	1	8	8	-4	8	1	15	15	-14	2	2	23	-24
8	2	0	47	47	3	9	0	11	11	-10	4	1	13	13	-2	8	1	12	13	-12	2	2	15	14
10	2	0	9	-9	5	9	0	3	0	-8	4	1	5	-5	0	8	1	6	8	-10	2	2	18	19
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16	2	0	11	11	2	10	0	5	5	-2	4	1	66	-64	6	8	1	11	10	-4	2	2	26	-27
1	3	0	31	-32	-15	1	1	18	-18	0	4	1	98	-99	8	8	1	16	14	-2	2	2	153	-136
3	3	0	84	83	-13	1	1	22	-23	2	4	1	8	-9	10	8	1	5	5	0	2	2	25	-26
5	3	0	50	50	-11	1	1	8	8	4	4	1	15	-15	-9	9	1	4	-4	2	2	2	5	-5
7	3	0	6	6	-9	1	1	48	48	6	4	1	35	-36	-3	9	1	6	-7	4	2	2	52	-51
9	3	0	5	-4	-7	1	1	71	71	10	4	1	8	7	3	9	1	4	-4	6	2	2	12	-12
11	3	0	26	-26	-5	1	1	38	38	12	4	1	22	-25	5	9	1	7	7	8	2	2	11	11
13	3	0	23	-23	-3	1	1	20	-20	14	4	1	10	-11	7	9	1	5	5	10	2	2	14	-15
15	3	0	3	-3	3	1	1	35	-36	-11	5	1	10	-10	-6	10	1	5	-3	12	2	2	12	-13
0	4	0	22	23	5	1	1	29	27	-9	5	1	11	11	-4	10	1	8	7	-15	3	2	9	-9
4	4	0	32	-32	7	1	1	15	-16	-7	5	1	13	14	-2	10	1	8	7	-13	3	2	6	-7
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3	4	18	5	-4	4	1	19	9	-8	5	3	19	9	-10	-5	0	20	17	-19	-5	2	20	13	15
4	4	18	7	7	5	1	19	4	3	6	3	19	3	-1	-4	0	20	7	7	-4	2	20	9	-9
7	4	18	4	-3	6	1	19	11	13	7	3	19	5	-6	-2	0	20	2	-1	-3	2	20	10	9
8	4	18	4	-4	7	1	19	13	-14	8	3	19	4	-4	-1	0	20	6	-7	-2	2	20	6	-7
10	4	18	9	9	10	1	19	7	-7	10	3	19	6	-6	0	0	20	11	11	-1	2	20	17	15
-8	5	18	4	2	11	1	19	2	2	-8	4	19	5	-5	1	0	20	34	-33	0	2	20	6	-9
-7	5	18	5	4	-10	2	19	3	-4	-7	4	19	6	5	2	0	20	16	16	1	2	20	3	-3
-6	5	18	6	-6	-8	2	19	7	-7	-4	4	19	7	6	3	0	20	18	-16	2	2	20	5	4
-3	5	18	3	-2	-6	2	19	3	2	-3	4	19	5	3	4	0	20	14	-16	3	2	20	12	-12
-2	5	18	9	8	-3	2	19	12	12	-2	4	19	11	-13	5	0	20	9	-9	4	2	20	21	23
-1	5	18	12	-13	-2	2	19	17	-16	-1	4	19	13	14	6	0	20	15	14	5	2	20	14	16
0	5	18	5	6	-1	2	19	5	-6	1	4	19	11	-11	8	0	20	7	7	6	2	20	5	-5
6	5	18	5	5	0	2	19	5	2	2	4	19	7	-9	9	0	20	8	9	8	2	20	9	-9
7	5	18	7	-7	1	2	19	10	-12	3	4	19	2	3	-10	1	20	5	-5	10	2	20	9	10
8	5	18	5	-4	3	2	19	11	11	4	4	19	5	-4	-8	1	20	4	-2	11	2	20	6	-6
9	5	18	8	-7	4	2	19	14	-14	5	4	19	6	4	-7	1	20	6	-5	-11	3	20	4	-3

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C7H9NO3, OR03.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	5	16	14	-15	9	1	17	4	3	9	3	17	12	12	-1	6	17	5	4	4	1	18	22	23
5	5	16	3	-4	10	1	17	6	6	10	3	17	4	-4	2	6	17	6	5	5	1	18	15	16
7	5	16	7	-6	-12	2	17	4	5	-11	4	17	2	1	3	6	17	7	-7	6	1	18	10	10
8	5	16	7	7	-8	2	17	11	-11	-10	4	17	4	4	5	6	17	5	-4	7	1	18	4	-4
-7	6	16	7	-6	-7	2	17	3	3	-9	4	17	3	1	6	6	17	3	-1	8	1	18	9	-10
	6	16	6	-5	-6	2	17	14	15	-7	4	17	4	-3	-2	7	17	4	4	9	1	18	16	-16
-3	7	16	6	5	-5	2	17	8	7	-5	4	17	5	-4	-1	7	17	5	-3	11	1	18	5	4
-2	7	16	5	-4	-4	2	17	28	-29	-4	4	17	3	-2	-3	7	17	3	-4	-11	2	18	3	2
-1	7	16	5	5	-3	2	17	5	5	-3	4	17	4	-3	-11	0	18	3	4	-10	2	18	4	3
0	7	16	6	-5	-2	2	17	7	7	-1	4	17	6	6	-9	0	18	5	-4	-7	2	18	13	14
2	7	16	5	4	-1	2	17	18	17	0	4	17	5	-7	-8	0	18	24	26	-6	2	18	19	-18
3	7	16	3	2	0	2	17	28	28	2	4	17	6	6	-7	0	18	16	-16	-5	2	18	10	11
4	7	16	6	-5	1	2	17	3	-4	4	4	17	17	-16	-6	0	18	8	-10	-4	2	18	4	6
-12	1	17	4	4	2	2	17	15	-17	5	4	17	5	5	-5	0	18	13	13	-3	2	18	8	6
-11	1	17	3	-4	3	2	17	3	-2	6	4	17	19	-19	-4	0	18	21	-22	-2	2	18	19	-20
-10	1	17	5	4	5	2	17	7	-7	7	4	17	2	4	-3	0	18	6	7	0	2	18	7	-7
-9	1	17	3	3	7	2	17	18	-19	8	4	17	8	-6	-1	0	18	20	17	1	2	18	3	1
-8	1	17	6	-4	9	2	17	3	-3	-9	5	17	7	-6	0	0	18	13	11	2	2	18	8	7
-7	1	17	8	-7	11	2	17	6	6	-7	5	17	5	5	2	0	18	36	-32	3	2	18	9	10
-6	1	17	9	10	12	2	17	3	-3	-5	5	17	7	-7	3	0	18	37	-37	4	2	18	8	-6
-5	1	17	26	-25	-11	3	17	5	5	-3	5	17	8	-6	7	0	18	17	-15	5	2	18	5	-5
-4	1	17	6	-5	-9	3	17	8	7	-1	5	17	6	6	8	0	18	7	6	6	2	18	34	-36
-3	1	17	7	-6	-8	3	17	11	-12	0	5	17	9	-10	12	0	18	12	11	7	2	18	16	11
-2	1	17	4	-2	-6	3	17	7	8	1	5	17	5	4	-9	1	18	5	-4	8	2	18	3	-4
-1	1	17	18	-18	-5	3	17	17	-17	2	5	17	5	-5	-7	1	18	10	-10	-11	3	18	5	5
0	1	17	34	-34	-4	3	17	26	26	3	5	17	5	3	-6	1	18	4	-4	-7	3	18	4	5
1	1	17	3	-4	-2	3	17	12	-11	6	5	17	4	5	-5	1	18	7	8	-6	3	18	17	17
2	1	17	8	-8	0	3	17	6	7	7	5	17	7	-7	-4	1	18	14	-14	-5	3	18	7	7
3	1	17	17	-17	1	3	17	23	-21	8	5	17	8	6	-3	1	18	10	9	-4	3	18	11	-13
4	1	17	20	20	2	3	17	8	8	9	5	17	6	5	-2	1	18	4	2	-3	3	18	20	-23
5	1	17	5	5	3	3	17	19	-20	-5	6	17	7	7	0	1	18	23	22	-2	3	18	18	18
6	1	17	4	3	6	3	17	12	11	-4	6	17	10	-9	1	1	18	4	-5	0	3	18	12	-11
7	1	17	9	8	7	3	17	7	-7	-3	6	17	3	2	2	1	18	30	29	1	3	18	26	25
8	1	17	20	22	8	3	17	6	-6	-2	6	17	8	7	3	1	18	16	-14	2	3	18	14	-15

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	2	15	4	-6	-3	4	15	4	-5	1	7	15	4	-4	2	1	16	11	11	-2	3	16	3	4
2	2	15	14	-12	-2	4	15	5	3	2	7	15	4	-3	3	1	16	11	10	-1	3	16	16	17
3	2	15	28	28	-1	4	15	15	-17	3	7	15	3	4	4	1	15	39	40	0	3	16	8	-9
4	2	15	9	-9	0	4	15	11	11	-11	0	16	7	-9	5	1	15	22	-21	2	3	16	18	-20
5	2	15	14	-15	1	4	15	24	-23	-8	0	16	25	-22	6	1	16	6	6	3	3	16	20	-21
6	2	15	25	25	2	4	15	4	-3	-7	0	16	21	-19	7	1	16	22	-25	4	3	16	13	-14
7	2	15	5	-6	3	4	15	13	14	-6	0	16	4	4	8	1	16	10	10	5	3	16	11	11
9	2	15	3	-4	4	4	15	5	3	-5	0	16	4	-6	9	1	16	5	5	6	3	16	7	-6
11	2	15	6	-6	5	4	15	7	9	-4	0	16	39	-40	12	1	16	3	-3	7	3	16	22	24
-11	3	15	5	5	9	4	15	8	7	-3	0	16	25	25	-12	2	16	5	-3	10	3	16	9	8
-10	3	15	7	-7	-9	5	15	4	4	-2	0	16	28	25	-9	2	16	6	6	11	3	16	6	6
-9	3	15	7	6	-6	5	15	8	6	-1	0	16	9	9	-8	2	16	5	-6	-11	4	16	3	-3
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-3	3	15	7	7	-1	5	15	6	5	4	0	16	6	7	-1	2	16	15	-14	-5	4	16	11	12
-2	3	15	3	2	0	5	15	8	-7	5	0	16	7	-7	0	2	16	23	23	-4	4	16	14	-14
-1	3	15	13	-13	1	5	15	11	13	6	0	16	26	25	1	2	16	22	-22	-3	4	16	3	4
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1	3	15	17	-17	6	5	15	4	4	8	0	16	13	-12	3	2	16	11	9	0	4	16	13	-14
2	3	15	19	19	7	5	15	10	8	9	0	16	5	-7	4	2	16	4	3	3	4	16	13	12
3	3	15	8	-9	-8	6	15	6	6	10	0	16	7	-8	5	2	16	14	14	4	4	16	14	-16
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5	3	15	28	-29	-6	6	15	3	-3	-10	1	16	6	6	9	2	16	4	4	9	4	16	7	-6
6	3	15	10	-10	-5	6	15	5	5	-9	1	16	6	-6	12	2	16	12	-12	10	4	16	4	-5
7	3	15	5	-5	-3	6	15	3	3	-8	1	16	5	-4	-12	3	16	4	-4	-10	5	16	3	-2
8	3	15	4	4	-2	6	15	8	6	-7	1	16	3	-2	-11	3	16	3	1	-6	5	16	4	9
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-10	4	15	3	-3	3	6	15	3	-2	-4	1	16	3	-3	-9	3	16	3	-2	-4	5	16	7	8
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-4	4	15	5	-5	0	7	15	6	6	1	1	16	23	-25	-3	3	16	2	-4	0	5	16	11	-13

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-9	5	13	3	3	2	0	14	31	-31	-9	2	14	5	-6	-10	4	14	4	4	-5	7	14	4	3
-6	5	13	5	4	3	0	14	16	17	-8	2	14	4	-5	-8	4	14	6	-6	-4	7	14	6	-5
-3	5	13	20	21	4	0	14	18	17	-7	2	14	7	9	-7	4	14	7	8	-2	7	14	3	-2
-1	5	13	11	13	5	0	14	29	26	-6	2	14	16	-14	-6	4	14	21	-22	1	7	14	7	6
1	5	13	6	-8	7	0	14	8	9	-5	2	14	11	-11	-5	4	14	8	-8	2	7	14	5	-5
2	5	13	4	5	8	0	14	19	-20	-4	2	14	4	5	-2	4	14	3	-2	3	7	14	6	-6
3	5	13	11	-10	9	0	14	23	25	-3	2	14	24	-24	-1	4	14	9	9	-12	1	15	6	-5
4	5	13	3	-2	10	0	14	4	4	-1	2	14	22	-20	0	4	14	3	-5	-11	1	15	2	3
9	5	13	3	4	11	0	14	4	4	0	2	14	2	2	1	4	14	14	13	-10	1	15	4	5
10	5	13	3	-2	-13	1	14	3	3	1	2	14	18	-17	2	4	14	4	2	-9	1	15	6	-4
-8	6	13	9	-8	-11	1	14	5	5	2	2	14	7	6	3	4	14	3	3	-8	1	15	9	10
-6	6	13	4	-4	-10	1	14	7	7	3	2	14	6	3	5	4	14	15	15	-7	1	15	3	-4
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-4	6	13	6	5	-8	1	14	18	17	5	2	14	7	-7	-9	5	14	8	8	-5	1	15	19	18
-2	6	13	8	9	-7	1	14	13	15	6	2	14	9	10	-5	5	14	6	-5	-4	1	15	24	24
-1	6	13	4	-4	-6	1	14	9	-7	8	2	14	40	39	-4	5	14	12	-13	-3	1	15	42	-38
1	6	13	3	-4	-5	1	14	13	15	10	2	14	5	-5	-3	5	14	10	9	-2	1	15	4	-3
2	6	13	6	-6	-3	1	14	13	13	11	2	14	3	5	-1	5	14	14	-15	-1	1	15	32	32
4	6	13	5	5	-2	1	14	31	-28	-11	3	14	4	5	0	5	14	11	-11	0	1	15	5	-4
5	6	13	6	-6	-1	1	14	24	-23	-8	3	14	9	10	1	5	14	11	11	1	1	15	6	-7
8	6	13	6	-4	0	1	14	24	-26	-7	3	14	8	6	3	5	14	3	2	3	1	15	31	-30
-6	7	13	6	5	1	1	14	2	-1	-6	3	14	3	-3	4	5	14	3	4	4	1	15	9	9
-3	7	13	3	-2	2	1	14	34	35	-5	3	14	7	-10	6	5	14	7	-6	5	1	15	7	-7
5	7	13	4	-4	3	1	14	30	29	-4	3	14	10	-8	7	5	14	4	5	6	1	15	21	-23
-13	0	14	3	2	4	1	14	30	-28	-1	3	14	13	15	8	5	14	5	-5	9	1	15	5	-7
-11	0	14	6	6	5	1	14	5	-7	0	3	14	30	-32	10	5	14	8	6	10	1	15	6	6
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-7	0	14	8	-7	8	1	14	10	-11	3	3	14	3	-4	-3	6	14	4	-3	-10	2	15	3	2
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-5	0	14	27	-26	12	1	14	4	6	5	3	14	11	-10	-1	6	14	4	4	-7	2	15	11	11
-2	0	14	43	-44	13	1	14	7	-7	7	3	14	6	6	0	6	14	4	4	-6	2	15	21	20
-1	0	14	18	18	-13	2	14	5	-4	8	3	14	9	-10	2	6	14	8	7	-3	2	15	29	30
1	0	14	35	35	-10	2	14	13	14	11	3	14	3	-4	8	6	14	6	6	-1	2	15	6	-6

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
9	1	12	23	24	-1	3	12	18	-17	-4	5	12	7	7	1	1	13	29	-27	-8	3	13	8	-6
10	1	12	7	-6	0	3	12	9	9	-3	5	12	8	7	2	1	13	43	42	-7	3	13	22	-21
11	1	12	7	-6	1	3	12	16	17	-2	5	12	8	-8	3	1	13	34	35	-6	3	13	20	21
12	1	12	8	-7	3	3	12	8	-10	2	5	12	7	-6	4	1	13	13	-16	-5	3	13	4	5
-11	2	12	4	-3	4	3	12	5	-6	3	5	12	15	16	5	1	13	12	13	-4	3	13	12	-12
-10	2	12	5	-6	5	3	12	9	10	4	5	12	7	-8	6	1	13	19	19	-3	3	13	8	9
-9	2	12	3	4	7	3	12	9	8	5	5	12	7	7	7	1	13	12	12	-2	3	13	4	-5
-8	2	12	5	5	8	3	12	9	10	6	5	12	3	4	8	1	13	6	6	-1	3	13	6	-9
-7	2	1	8	-10	9	3	12	16	-16	7	5	12	5	5	10	1	13	5	-7	0	3	13	9	9
-6	2	1	17	16	10	3	12	7	6	9	5	12	10	9	11	1	13	7	-7	1	3	13	7	7
-5	2	12	6	-6	11	3	12	4	3	10	5	12	4	-4	-13	2	13	4	-4	2	3	13	11	-10
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-3	2	12	28	-25	-10	4	12	3	4	-3	6	12	3	-1	-11	2	13	3	5	8	3	13	8	-7
-2	2	12	44	44	-9	4	12	6	6	-2	6	12	5	-3	-9	2	13	3	3	9	3	13	9	8
-1	2	12	20	-21	-8	4	12	9	-8	1	6	12	3	3	-6	2	13	3	-4	10	3	13	3	2
0	2	12	14	16	-6	4	12	2	4	4	6	12	2	-3	-7	2	13	3	-2	11	3	13	4	-4
1	2	12	2	0	-5	4	12	11	11	6	6	12	9	8	-6	2	13	17	-19	-11	4	13	5	-4
2	2	12	27	-28	-4	4	12	8	-8	8	6	12	3	4	-5	2	13	10	-10	-9	4	13	3	4
3	2	12	17	16	-3	4	12	12	-14	-6	7	12	4	-4	-4	2	13	10	10	-8	4	13	3	-2
4	2	12	7	6	-2	4	12	8	8	1	7	12	9	-10	-3	2	13	33	-32	-7	4	13	15	-14
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OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C7H9NO3, OR03. PAGE 8

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-10	5	4	39	42	-2	8	4	38	-44	-16	2	5	20	9	17	4	5	20	-1	-6	8	5	17	-17

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
11	8	2	20	19	16	1	3	24	20	2	4	3	47	49	1	7	3	17	17	-10	0	4	54	-57
12	8	2	26	-28	17	1	3	46	-40	3	4	3	9	11	2	7	3	24	32	-9	0	4	65	64
13	8	2	22	-17	-13	2	3	21	-22	4	4	3	25	-32	3	7	3	100	105	-8	0	4	30	-29
-9	9	2	27	-31	-11	2	3	11	-1	5	4	3	29	-24	5	7	3	51	-55	-7	0	4	32	32
-8	9	2	28	34	-10	2	3	32	30	-16	5	3	45	-42	6	7	3	15	-13	-6	0	4	35	-38
-7	9	2	21	7	-9	2	3	24	-28	-10	5	3	69	86	7	7	3	26	-21	-5	0	4	111	114
-3	9	2	49	54	-7	2	3	9	15	-9	5	3	66	65	9	7	3	45	-44	-4	0	4	184	181
-2	9	2	25	-22	-6	2	3	27	-23	-8	5	3	39	-31	10	7	3	14	-9	-3	0	4	194	-196
-1	9	2	47	-50	-4	2	3	122	-115	-6	5	3	13	-8	11	7	3	39	40	-2	0	4	90	-85
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5	9	2	55	63	1	2	3	26	-32	-4	5	3	120	-109	-10	8	3	31	32	0	0	4	101	-99
10	9	2	18	18	2	2	3	35	-27	-3	5	3	158	-145	-4	8	3	18	-27	1	0	4	71	-70
11	9	2	21	-23	4	2	3	36	-37	-2	5	3	59	55	-3	8	3	35	36	2	0	4	299	-315
-9	10	2	30	-31	5	2	3	23	24	-1	5	3	58	60	-2	8	3	36	37	3	0	4	102	100
-8	10	2	25	-27	6	2	3	29	-27	0	5	3	29	33	-1	8	3	21	-22	4	0	4	169	168
-6	10	2	21	11	10	2	3	20	20	1	5	3	36	38	2	8	3	25	28	5	0	4	15	15
-3	10	2	26	31	-16	3	3	65	-67	2	5	3	47	51	4	8	3	38	-46	6	0	4	75	75
-2	10	2	55	59	-11	3	3	37	-34	3	5	3	159	165	5	8	3	18	28	7	0	4	19	-17
4	10	2	43	-47	-10	3	3	97	99	5	5	3	72	-78	-16	9	3	24	-15	8	0	4	107	94
-9	11	2	24	-24	-9	3	3	75	78	7	5	3	34	-35	-15	9	3	17	-10	10	0	4	146	-144
5	11	2	21	27	-8	3	3	26	-22	9	5	3	67	-64	-10	9	3	41	44	11	0	4	29	-26
6	11	2	18	-13	-6	3	3	75	-66	11	5	3	71	73	-9	9	3	26	25	12	0	4	27	-26
12	11	2	24	-6	-5	3	3	51	47	15	5	3	15	14	-4	9	3	45	-48	16	0	4	56	49
-8	12	2	22	10	-4	3	3	77	-67	17	5	3	23	-23	-3	9	3	56	-58	-11	1	4	14	-12
-3	17	2	24	16	-3	3	3	126	-120	-15	6	3	16	19	-2	9	3	18	14	-8	1	4	12	16
-16	1	3	60	-55	-2	3	3	48	50	-12	6	3	20	-13	2	9	3	26	28	-4	1	4	21	-19
-11	1	3	20	-21	-1	3	3	26	26	-11	6	3	22	24	3	9	3	45	45	-3	1	4	10	-11
-10	1	3	140	143	0	3	3	20	28	-10	6	3	30	32	5	9	3	37	-40	1	1	4	37	-34
-9	1	3	103	106	1	3	3	42	42	-9	6	3	26	-27	6	9	3	21	-24	2	1	4	29	26
-8	1	3	51	-53	2	3	3	40	40	-4	6	3	38	-29	9	9	3	27	-28	3	1	4	16	14
-6	1	3	63	-68	3	3	3	163	191	-3	6	3	29	30	14	9	3	23	1	5	1	4	24	-29
-4	1	3	148	-158	4	3	3	24	-24	-2	6	3	65	67	-4	10	3	13	-37	8	1	4	16	17
-3	1	3	275	-301	5	3	3	101	-106	0	6	3	32	30	-2	10	3	15	26	10	1	4	24	24
-2	1	3	108	108	7	3	3	50	-55	2	6	3	25	29	2	10	3	14	23	-17	2	4	57	52
-1	1	3	57	55	9	3	3	88	-b:	4	6	3	43	-46	4	10	3	29	-33	-11	2	4	112	-121
0	1	3	118	114	10	3	3	21	-24	5	6	3	27	31	6	10	3	21	-7	-10	2	4	57	-56
1	1	3	38	35	11	3	3	89	87	10	6	3	32	35	10	10	3	22	24	-9	2	4	82	85
2	1	3	14	14	12	3	3	38	39	13	6	3	20	1	12	10	3	23	-9	-8	2	4	16	-19
3	1	3	192	198	13	3	3	30	32	-16	7	3	34	-31	-9	11	3	22	21	-7	2	4	44	47
4	1	3	53	-51	14	3	3	17	15	-10	7	3	77	73	-4	11	3	20	-33	-6	2	4	26	-27
5	1	3	120	-118	17	3	3	33	-31	-9	7	3	41	38	-3	11	3	30	-26	-5	2	4	106	118
6	1	3	38	-34	-11	4	3	17	-19	-8	7	3	21	-16	0	11	3	20	25	-4	2	4	146	149
7	1	3	75	-80	-9	4	3	33	-31	-7	7	3	28	-25	5	11	3	16	-34	-3	2	4	137	-138
8	1	3	11	-13	-5	4	3	34	27	-4	7	3	72	-69	8	12	3	23	13	-2	2	4	46	-45
9	1	3	81	-76	-4	4	3	42	-48	-3	7	3	102	-99	-17	0	4	60	58	-1	2	4	26	20
10	1	3	31	-32	-3	4	3	16	16	-2	7	3	34	34	-13	0	4	17	-17	0	2	4	76	-77
11	1	3	92	93	-2	4	3	25	-18	-1	7	3	43	44	-12	0	4	33	29	1	2	4	31	-33
13	1	3	20	18	1	4	3	16	-18	0	7	3	17	21	-11	0	4	114	-125	2	2	4	191	-196

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
14	5	1	29	29	-7	9	1	60	61	-6	1	2	32	-27	-3	3	2	38	-27	4	5	2	16	-20
17	5	1	16	7	-2	9	1	32	-38	-9	1	2	71	-75	-2	3	2	75	-64	5	5	2	70	79
-10	6	1	18	-7	-1	9	1	67	-69	-2	1	2	44	-42	-1	3	2	118	-116	6	5	2	32	-35
-8	6	1	30	35	1	9	1	36	41	-1	1	2	76	-76	0	3	2	63	61	8	5	2	17	-13
-7	6	1	34	-30	5	9	1	52	58	0	1	2	68	68	1	3	2	18	-19	10	5	2	18	13
-3	6	1	29	29	7	9	1	26	-29	1	1	2	21	-19	2	3	2	17	13	11	5	2	38	-36
-1	6	1	31	27	-12	10	1	23	6	5	1	2	32	-31	3	3	2	61	-57	13	5	2	22	26
0	6	1	54	53	-6	10	1	25	-24	4	1	2	30	-29	5	3	2	50	54	18	5	2	20	10
1	6	1	24	-25	0	10	1	34	35	5	1	2	20	21	6	3	2	34	-35	-15	6	2	44	41
3	6	1	24	-21	6	10	1	31	-38	6	1	2	25	-21	7	3	2	24	-33	-14	6	2	17	27
4	6	1	34	38	-8	11	1	21	24	7	1	2	13	-4	10	3	2	25	24	-12	6	2	18	19
5	6	1	23	29	-2	11	1	20	-22	8	1	2	25	-20	11	3	2	37	-36	-9	6	2	103	-98
6	6	1	37	-45	-1	11	1	39	-40	13	1	2	18	-9	-19	4	2	23	-10	-8	6	2	96	-94
11	6	1	16	-12	5	11	1	32	37	16	1	2	20	-2	-17	4	2	15	-12	-5	6	2	22	17
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-13	7	1	24	-22	-21	0	2	32	-22	-15	2	2	74	72	-13	4	2	22	-25	-1	6	2	40	-38
-9	7	1	36	-32	-15	0	2	106	102	-14	2	2	26	28	-11	4	2	24	-24	0	6	2	43	-43
-8	7	1	59	59	-14	0	2	19	20	-12	2	2	32	31	-10	4	2	35	41	1	6	2	27	-30
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-3	7	1	33	30	-10	0	2	50	53	-9	2	2	130	-137	-8	4	2	127	-119	4	6	2	94	-100
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3	7	1	17	17	-6	0	2	36	-41	-3	2	2	149	131	-3	4	2	80	68	10	6	2	37	36
4	7	1	22	25	-5	0	2	62	63	-2	2	2	395	468	-2	4	2	218	204	11	6	2	34	30
5	7	1	101	104	-4	0	2	50	-50	-1	2	2	135	-131	-1	4	2	72	-64	12	6	2	52	-52
6	7	1	22	21	-3	0	2	59	60	0	2	2	83	-81	0	4	2	58	-54	-5	7	2	24	-24
7	7	1	44	-42	-2	0	2	195	219	1	2	2	72	-71	2	4	2	59	-61	-6	7	2	50	51
10	7	1	19	-23	-1	0	2	71	-67	2	2	2	69	-70	4	4	2	181	-190	-3	7	2	59	59
11	7	1	46	-48	0	0	2	19	-17	3	2	2	13	-9	6	4	2	94	97	-2	7	2	30	-31
12	7	1	34	-37	1	0	2	137	136	4	2	2	299	-323	8	4	2	32	31	-1	7	2	29	-30
13	7	1	29	30	2	0	2	86	-81	5	2	2	29	-30	10	4	2	75	75	0	7	2	45	47
14	7	1	23	23	4	0	2	240	-248	6	2	2	151	156	11	4	2	69	70	1	7	2	12	14
-10	8	1	16	-10	5	0	2	19	-17	7	2	2	32	32	12	4	2	71	-71	3	7	2	20	-23
-9	8	1	13	17	6	0	2	146	149	8	2	2	92	91	17	4	2	33	-29	5	7	2	60	68
-8	8	1	35	33	7	0	2	75	-71	10	2	2	83	73	18	4	2	22	18	11	7	2	31	-33
-7	8	1	25	-24	8	0	2	48	46	11	2	2	45	40	-10	5	2	25	-25	-15	8	2	23	32
-6	8	1	27	-24	9	0	2	21	-18	12	2	2	99	-99	-9	5	2	60	-56	-10	8	2	20	19
-2	8	1	49	-48	10	0	2	109	102	13	2	2	24	-22	-8	5	2	43	44	-9	8	2	32	-28
0	8	1	41	46	11	0	2	78	76	14	2	2	19	-12	-7	5	2	18	14	-8	8	2	51	-51
4	8	1	29	32	12	0	2	107	-97	17	2	2	35	-26	-6	5	2	47	-44	-3	8	2	44	41
5	8	1	16	22	13	0	2	35	-34	18	2	2	33	30	-4	5	2	15	-11	-2	8	2	87	87
6	8	1	27	-26	14	0	2	38	-33	-14	3	2	29	-27	-3	5	2	141	126	0	8	2	18	-18
12	8	1	25	25	17	0	2	41	-36	-12	3	2	15	-20	-1	5	2	57	-54	3	8	2	15	-21
-14	9	1	28	-26	18	0	2	30	24	-9	3	2	22	-27	0	5	2	25	26	4	8	2	84	-90
13	9	1	21	-20	-11	1	2	14	-11	-8	3	2	36	38	2	5	2	22	18	6	8	2	42	48
-8	9	1	51	55	-8	1	2	14	13	-5	3	2	24	-23	3	5	2	29	-36	10	8	2	33	35

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C12H22N3O3BR3, OR05.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	12	-11	19	2	0	37	-30	9	6	0	14	-19	-5	1	1	46	43	-3	3	1	20	14
3	0	0	108	-109	1	3	0	87	-80	12	6	0	36	36	-4	1	1	66	-67	-2	3	1	118	-98
4	0	0	54	-54	2	3	0	56	53	13	6	0	49	48	-3	1	1	61	67	-1	3	1	335	-338
5	0	0	77	76	3	3	0	13	10	14	6	0	30	-27	-2	1	1	61	-56	0	3	1	14	-4
6	0	0	279	-284	4	3	0	59	58	19	6	0	24	-19	-1	1	1	213	-222	1	3	1	31	31
7	0	0	132	-135	5	3	0	41	-40	1	7	0	81	-83	0	1	1	24	22	2	3	1	26	22
8	0	0	117	118	6	3	0	59	-59	4	7	0	17	18	1	1	1	63	64	3	3	1	43	42
9	0	0	30	27	7	3	0	40	41	5	7	0	19	-22	2	1	1	32	26	4	3	1	11	14
10	0	0	96	93	8	3	0	16	-15	7	7	0	79	89	3	1	1	83	85	5	3	1	161	170
12	0	0	59	55	9	3	0	13	-1	12	7	0	26	29	4	1	1	14	15	6	3	1	49	51
13	0	0	75	70	10	3	0	27	-24	13	7	0	22	-22	5	1	1	219	230	7	3	1	86	-91
14	0	0	66	-63	12	3	0	15	8	0	8	0	121	125	6	1	1	49	51	9	3	1	44	-47
15	0	0	15	-13	14	3	0	19	-19	1	8	0	26	26	7	1	1	90	-90	11	3	1	42	-41
17	0	0	13	-9	16	3	0	15	-14	2	8	0	15	3	8	1	1	14	-1	12	3	1	51	-50
19	0	0	34	-27	0	4	0	269	259	3	8	0	12	-9	9	1	1	47	-49	13	3	1	60	59
20	0	0	20	16	2	4	0	72	-67	6	8	0	64	-66	10	1	1	17	-16	-16	4	1	20	13
21	0	0	26	20	4	4	0	65	-63	7	8	0	45	-49	11	1	1	80	-72	-8	4	1	46	48
1	1	0	58	-54	5	4	0	14	10	8	8	0	25	25	12	1	1	75	-71	-7	4	1	13	-21
2	1	0	24	23	6	4	0	182	-181	10	8	0	14	25	13	1	1	66	66	-6	4	1	13	-14
3	1	0	15	10	7	4	0	90	-93	12	8	0	18	23	14	1	1	16	16	-4	4	1	31	-24
4	1	0	12	11	8	4	0	76	76	13	8	0	32	25	15	1	1	19	17	-2	4	1	44	-35
5	1	0	10	-1	9	4	0	13	-5	14	8	0	15	-14	18	1	1	21	28	0	4	1	56	54
6	1	0	16	-18	10	4	0	40	39	17	8	0	17	-8	20	1	1	19	-17	1	4	1	23	-34
7	1	0	31	34	12	4	0	53	48	1	9	0	37	-43	-13	2	1	22	20	2	4	1	18	16
8	1	0	22	-19	13	4	0	66	62	2	9	0	13	19	-11	2	1	17	14	4	4	1	24	-18
9	1	0	12	-12	14	4	0	45	-45	6	9	0	24	-28	-8	2	1	31	28	6	4	1	60	-63
10	1	0	14	-7	16	4	0	27	-28	7	9	0	36	42	-5	2	1	32	-28	7	4	1	20	19
11	1	0	14	-2	16	4	0	20	-14	0	10	0	50	55	-4	2	1	31	-27	11	4	1	18	-22
13	1	0	22	-21	19	4	0	29	-24	5	10	0	20	-4	-3	2	1	30	26	12	4	1	22	23
14	1	0	13	-8	20	4	0	22	7	6	10	0	39	-37	-2	2	1	136	-119	19	4	1	22	-5
0	2	0	252	239	1	5	0	68	-68	8	10	0	14	19	-1	2	1	36	-34	-14	5	1	61	-63
1	2	0	15	-12	2	5	0	33	30	11	10	0	21	12	0	2	1	82	-81	-13	5	1	31	-36
2	2	0	47	-43	5	5	0	42	-37	12	10	0	18	20	1	2	1	55	52	-9	5	1	50	-49
3	2	0	106	103	6	5	0	43	-43	1	11	0	33	-38	2	2	1	15	15	-8	5	1	62	58
4	2	0	44	-43	7	5	0	44	46	6	11	0	17	-23	6	2	1	23	-27	-7	5	1	104	94
5	2	0	63	62	10	5	0	15	18	7	11	0	17	24	7	2	1	33	-27	-2	5	1	67	-63
6	2	0	216	-221	12	5	0	36	38	0	12	0	24	33	9	2	1	26	-24	-1	5	1	231	-221
7	2	0	83	-85	13	5	0	25	-24	6	12	0	19	-26	-20	3	1	19	20	0	5	1	15	-12
8	2	0	107	110	14	5	0	16	-15	8	12	0	20	9	-15	3	1	27	25	1	5	1	11	10
9	2	0	15	-16	0	6	0	171	160	2	14	0	21	-3	-14	3	1	57	-62	4	5	1	14	15
10	2	0	30	27	1	6	0	22	16	-20	1	1	35	24	-13	3	1	44	-43	5	5	1	160	168
11	2	0	12	-7	2	6	0	46	-44	-15	1	1	32	29	-11	3	1	30	-34	6	5	1	54	55
12	2	0	63	58	3	6	0	13	12	-14	1	1	76	-73	-10	3	1	22	-19	7	5	1	76	-75
13	2	0	105	104	4	6	0	32	-32	-13	1	1	53	-47	-9	3	1	43	-40	8	5	1	35	-36
14	2	0	46	-42	5	6	0	19	11	-10	1	1	42	40	-8	3	1	115	114	9	5	1	40	-44
15	2	0	21	-19	6	6	0	131	-129	-9	1	1	67	-69	-7	3	1	175	169	11	5	1	53	-53
16	2	0	19	-16	7	6	0	64	-65	-8	1	1	125	126	-6	3	1	31	-25	12	5	1	39	-37
17	2	0	16	-16	8	6	0	41	45	-7	1	1	166	169	-5	3	1	25	27	13	5	1	39	36

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C14H1303NBR, OR04.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	2	9	64	66	1	0	10	28	-29	-4	7	10	16	2	-6	0	12	24	20	3	2	13	20	-18
0	2	9	53	-53	2	0	10	32	38	0	7	10	14	-8	-5	0	12	29	-33	-9	3	13	16	-10
2	2	9	45	44	4	0	10	28	-25	-6	1	11	26	25	-3	0	12	29	28	-4	3	13	13	14
3	2	9	27	-31	-8	1	10	16	-15	-5	1	11	17	-17	-2	0	12	33	-28	-2	3	13	19	19
5	2	9	21	24	-5	1	10	20	-19	-3	1	11	26	30	-1	0	12	36	33	0	3	13	12	-5
6	2	9	19	-24	-4	1	10	36	29	-2	1	11	27	-28	0	0	12	29	21	2	3	13	19	-16
7	2	9	13	7	-2	1	10	12	-13	0	1	11	16	19	1	0	12	21	-28	-6	5	13	13	-10
-6	3	9	28	-28	-1	1	10	40	42	1	1	11	22	-22	2	0	12	24	26	-3	5	13	13	-12
-5	3	9	24	26	0	1	10	28	-26	2	1	11	12	10	5	0	12	23	21	0	5	13	13	-13
-3	3	9	61	-58	2	1	10	23	21	-8	2	11	18	-16	-7	1	12	19	15	-1	6	13	16	-15
-2	3	9	58	58	3	1	10	23	-21	-7	2	11	25	25	-5	1	12	11	-13	-8	0	14	21	-19
-1	3	9	10	-12	4	1	10	15	5	-5	2	11	22	-20	-4	1	12	34	31	-5	0	14	25	-24
0	3	9	47	-50	7	1	10	16	9	-4	2	11	45	41	-3	1	12	24	-23	-3	0	14	21	23
1	3	9	57	59	-10	2	10	15	7	-3	2	11	26	-27	-2	1	12	14	-7	-2	0	14	30	-37
3	3	9	22	-26	2	2	10	18	-18	-2	2	11	26	-23	0	1	12	44	-37	0	0	14	16	1C
4	3	9	37	40	-5	3	10	20	-18	-1	2	11	21	24	3	1	12	19	-21	1	0	14	25	-27
5	3	9	13	-13	-4	3	10	16	10	0	2	11	23	-24	4	1	12	17	16	-3	1	14	35	-26
6	3	9	13	-13	-2	3	10	24	-25	2	2	11	28	29	-1	2	12	25	-24	-2	1	14	21	-10
7	3	9	14	14	-1	3	10	20	21	3	2	11	13	-18	2	2	12	19	-16	0	1	14	22	-23
-6	4	9	22	22	0	3	10	13	5	-9	3	11	11	-14	-4	3	12	19	17	-5	4	14	21	-18
-4	4	9	22	-19	1	3	10	17	-20	-7	3	11	19	17	-1	3	12	18	16	-3	4	14	20	-17
-3	4	9	14	18	2	3	10	15	17	-6	3	11	38	-35	-8	4	12	16	10	-2	4	14	19	17
-1	4	9	22	-23	5	3	10	15	12	-5	3	11	21	19	-6	4	12	17	-18	0	4	14	14	-11
0	4	9	31	31	-7	4	10	11	6	-3	3	11	28	-29	-5	4	12	26	26	1	4	14	13	14
2	4	9	17	-16	-6	4	10	11	-11	-2	3	11	32	26	-2	4	12	31	31	-7	5	14	17	-14
5	4	9	12	-10	-3	4	10	13	-11	-1	3	11	16	16	-1	4	12	18	-18	0	5	14	14	15
-4	5	9	19	22	-2	4	10	27	24	0	3	11	20	-20	1	4	12	31	31	-5	1	15	16	-10
-1	5	9	18	20	0	4	10	13	-14	1	3	11	13	15	-4	5	12	24	-23	-4	2	15	15	14
0	5	9	20	-21	1	4	10	15	19	2	3	11	12	-6	-1	5	12	14	-15	-1	2	15	16	13
4	5	9	13	9	2	4	10	15	-17	6	3	11	11	2	0	5	12	17	18	0	2	15	15	-14
-1	6	9	18	-14	4	4	10	18	19	-7	4	11	18	-17	3	5	12	15	14	-5	3	15	21	17
3	6	9	11	9	-7	5	10	16	-13	-5	4	11	17	19	4	5	12	15	-9	-2	3	15	16	12
-2	7	9	15	-16	-4	5	10	23	-21	-2	4	11	14	-13	1	13	14	-2	1	3	15	13	10
1	7	9	16	-16	-1	5	10	24	-24	2	4	11	15	-9	-2	1	13	22	-17	-9	0	16	17	11
-4	8	9	17	14	0	5	10	23	24	-4	5	11	19	-14	-8	2	13	15	-14	-5	0	16	17	-16
0	8	9	14	-13	?	5	10	18	20	-1	6	11	19	-16	-7	2	13	20	17	-3	0	16	13	11
-10	0	10	13	-7	-8	6	10	15	1	0	6	11	12	8	-5	2	13	27	-26	1	0	16	13	-8
-6	0	10	21	26	-6	6	10	15	12	2	6	11	13	-12	-4	2	13	23	22	-7	1	16	16	13
-5	0	10	16	-27	-5	6	10	14	-14	-6	7	11	12	12	-3	2	13	19	-14	1	1	16	14	5
-4	0	10	18	19	-2	6	10	20	-17	1	7	11	12	-13	-1	2	13	24	27	2	1	16	15	0
-3	0	10	28	27	1	6	10	15	-13	-12	0	12	15	4	0	2	13	18	-21	-2	4	16	16	12
-2	0	10	55	-53	2	6	10	16	15	-9	0	12	17	16	2	2	13	13	14	-4	2	17	14	12
0	0	10	43	40																				

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C14H13O3NBR, OR04.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	6	5	20	19	0	3	6	19	23	2	1	7	38	38	2	5	7	12	10	0	3	8	21	-21
2	6	5	23	-21	2	3	6	29	29	3	1	7	26	25	-8	6	7	17	11	1	3	8	12	-12
5	6	5	18	-17	5	3	6	19	17	4	1	7	33	-34	-2	6	7	20	21	2	3	8	29	28
7	6	5	13	5	6	3	6	16	-11	5	1	7	22	24	-1	6	7	21	-20	3	3	8	13	-14
-3	7	5	22	21	-7	4	6	18	15	7	1	7	12	-15	1	6	7	13	14	5	3	8	14	10
0	7	5	17	17	-6	4	6	12	-11	10	1	7	14	-5	2	6	7	20	-18	-10	4	8	15	9
1	7	5	20	-18	-4	4	6	19	15	-14	2	7	16	-4	6	6	7	15	8	-6	4	8	30	-31
2	7	5	13	6	-3	4	6	37	-38	-10	2	7	15	20	-2	7	7	19	-17	-5	4	8	13	16
4	7	5	12	-15	-2	4	6	26	24	-9	2	7	10	10	0	7	7	17	16	-3	4	8	36	-36
-3	8	5	10	-5	-1	4	6	29	27	-8	2	7	32	-31	1	7	7	20	-20	-2	4	8	36	35
0	8	5	13	-12	0	4	6	28	-28	-5	2	7	62	-57	0	8	7	15	-9	0	4	8	20	-18
-7	0	6	29	-30	1	4	6	39	40	-4	2	7	47	47	-10	0	8	51	-47	1	4	8	26	27
-6	0	6	47	49	3	4	6	26	-27	-2	2	7	13	-17	-7	0	8	40	-40	2	4	8	16	-13
-5	0	6	34	-44	4	4	6	32	34	-1	2	7	55	53	-6	0	8	37	37	4	4	8	14	12
-3	0	6	93	117	5	4	6	13	5	0	2	7	30	-31	-5	0	8	24	19	5	4	8	14	-16
-1	0	6	51	-52	6	4	6	13	-8	1	2	7	14	-13	-4	0	8	20	-36	-7	5	8	19	-17
0	0	6	82	80	7	4	6	13	11	2	2	7	48	48	-3	0	8	82	79	-6	5	8	14	-10
1	0	6	25	-26	-8	5	6	18	18	3	2	7	26	-30	-2	0	8	24	-26	-5	5	8	20	19
2	0	6	39	35	-4	5	6	35	-34	4	2	7	16	13	0	0	8	29	29	-4	5	8	29	-25
3	0	6	71	60	-2	5	6	13	12	6	2	7	19	-22	1	0	8	42	-42	-2	5	8	17	16
4	0	6	19	-21	-1	5	6	44	-44	-10	3	7	17	20	2	0	8	19	19	-1	5	8	31	-32
10	0	6	15	-2	0	5	6	18	19	-9	3	7	21	-21	4	0	8	42	-39	0	5	8	20	19
-10	1	6	14	5	1	5	6	14	12	-7	3	7	25	24	5	0	8	23	24	2	5	8	24	-22
-8	1	6	30	-27	2	5	6	26	-27	-6	3	7	32	-31	-11	1	8	18	-14	3	5	8	13	15
-7	1	6	19	17	3	5	6	27	28	-5	3	7	15	11	-8	1	8	28	-27	6	5	8	14	11
-6	1	6	21	21	6	5	6	13	11	-4	3	7	31	33	-5	1	8	58	-52	8	5	8	11	2
-5	1	6	36	-43	-7	6	6	13	-9	-3	3	7	46	-44	-4	1	8	15	22	-5	6	8	10	6
-4	1	6	54	58	-6	6	6	16	17	-2	3	7	29	30	-3	1	8	25	-22	-2	7	8	10	10
-2	1	6	56	-52	-5	6	6	16	-15	0	3	7	12	-12	-2	1	8	15	-19	0	7	8	11	-2
-1	1	6	56	57	-3	6	6	23	21	1	3	7	35	37	-1	1	8	46	46	1	7	8	13	13
0	1	6	22	-22	-2	6	6	17	-16	2	3	7	32	-31	0	1	8	53	-53	0	8	8	12	12
1	1	6	74	-68	0	6	6	14	16	3	3	7	18	-19	2	1	8	32	35	-6	1	9	20	21
2	1	6	15	21	1	6	6	25	-23	4	3	7	33	33	3	1	8	31	-30	-3	1	9	44	37
3	1	6	49	-48	3	7	6	13	-2	5	3	7	29	-32	4	1	8	13	14	-2	1	9	35	-33
5	1	6	17	19	7	7	6	14	1	7	3	7	21	20	6	1	8	22	-23	-1	1	9	13	0
7	1	6	17	12	-4	8	6	15	-6	8	3	7	11	-14	-11	2	8	13	12	0	1	9	34	33
8	1	6	10	11	0	8	6	13	8	10	3	7	15	11	-8	2	8	23	25	1	1	9	30	-30
10	1	6	14	5	3	8	6	12	9	-8	4	7	16	18	-5	2	8	13	6	2	1	9	10	8
-6	2	6	18	-12	-12	1	7	15	6	-5	4	7	35	35	-4	2	8	27	-22	4	1	9	23	-24
-3	2	6	32	-25	-9	1	7	15	21	-4	4	7	15	-14	-3	2	8	21	-16	5	1	9	16	12
0	2	6	36	-34	-6	1	7	28	33	-1	4	7	28	-26	-1	2	8	20	-23	6	1	9	11	6
2	2	6	23	-21	-5	1	7	42	-43	0	4	7	12	13	0	2	8	14	12	7	1	9	15	-9
3	2	6	19	-19	-4	1	7	9	-13	2	4	7	16	-17	2	2	8	13	-16	-12	2	9	15	11
6	2	6	10	-11	-3	1	7	59	62	3	4	7	26	26	4	2	8	10	4	-8	2	9	33	-31
8	2	6	12	-2	-2	1	7	50	-51	4	4	7	19	-23	-7	3	8	22	18	-7	2	9	14	17
-4	3	6	10	9	-1	1	7	16	11	8	4	7	15	-3	-5	3	8	17	-19	-5	2	9	35	-34
-2	3	6	31	-32	0	1	7	16	20	0	5	7	11	-11	-4	3	8	27	26	-4	2	9	46	39
-1	3	6	29	33	1	1	7	62	-61	1	5	7	12	6	-1	3	8	36	35	-2	2	9	21	-23

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C14H13O3NSBr, OR04.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	7	2	10	11	6	3	3	17	-25	-2	0	4	37	-45	-6	4	4	28	-26	-6	2	5	22	21
1	7	2	10	-8	7	3	3	20	23	-1	0	4	28	-29	-4	4	4	34	33	-5	2	5	48	-50
7	7	2	15	1	8	3	3	23	-18	0	0	4	121	120	-3	4	4	32	-31	-4	2	5	36	41
0	8	2	11	12	9	3	3	14	-14	1	0	4	105	-175	-2	4	4	15	9	-3	2	5	21	-19
2	9	2	14	11	-5	4	3	17	18	3	0	4	145	136	0	4	4	29	-29	-2	2	5	72	-69
-9	1	3	17	22	-3	4	3	14	16	4	0	4	51	-55	1	4	4	48	48	-1	2	5	123	120
-6	1	3	55	55	-2	4	3	10	10	5	0	4	49	47	3	4	4	10	-17	0	2	5	33	-33
-5	1	3	57	-58	-1	4	3	48	-53	6	0	4	47	50	4	4	4	25	25	1	2	5	80	-79
-4	1	3	32	-29	0	4	3	26	26	7	0	4	48	-49	5	4	4	17	-16	2	2	5	46	49
-3	1	3	80	81	2	4	3	25	-23	10	0	4	13	-11	7	4	4	13	18	3	2	5	50	-49
-2	1	3	85	-87	3	4	3	47	51	-13	1	4	16	-6	8	4	4	10	-7	5	2	5	51	52
-1	1	3	53	52	5	4	3	30	-30	-9	1	4	53	30	-9	5	4	21	-17	6	2	5	27	-27
0	1	3	54	54	6	4	3	19	21	-8	1	4	26	-23	-8	5	4	15	18	10	2	5	15	10
1	1	3	105	-107	8	4	3	13	-12	-7	1	4	16	17	-6	5	4	15	-15	-7	3	5	28	28
2	1	3	9	18	-6	5	3	14	-12	-6	1	4	27	24	-5	5	4	32	28	-6	3	5	67	-69
3	1	3	39	38	-1	5	3	13	11	-5	1	4	14	-20	-4	5	4	16	-18	-5	3	5	43	44
4	1	3	35	-37	0	5	3	14	-17	-4	1	4	50	49	-2	5	4	42	42	-4	3	5	37	37
5	1	3	28	29	1	5	3	12	8	-3	1	4	17	16	-1	5	4	55	-51	-3	3	5	92	-88
7	1	3	11	-11	3	5	3	11	-13	-2	1	4	50	-53	0	5	4	14	11	-2	3	5	71	69
8	1	3	20	18	6	5	3	12	4	-1	1	4	143	140	1	5	4	19	19	-1	3	5	45	46
-9	2	3	26	26	7	5	3	12	11	0	1	4	57	-57	2	5	4	36	-35	0	3	5	77	-75
-8	2	3	28	-26	-5	6	3	21	19	1	1	4	41	-39	3	5	4	28	26	1	3	5	104	101
-6	2	3	8	9	-2	6	3	19	24	2	1	4	44	47	5	5	4	14	-18	3	3	5	41	-43
-5	2	3	61	-57	-1	6	3	25	-23	3	1	4	64	-64	6	5	4	29	27	4	3	5	33	32
-4	2	3	39	39	2	6	3	25	-25	4	1	4	18	-19	-3	6	4	16	17	5	3	5	25	-26
-3	2	3	11	8	3	6	3	13	13	5	1	4	44	44	0	6	4	15	13	6	3	5	19	-19
-2	2	3	66	-72	4	6	3	11	3	6	1	4	34	-35	1	6	4	26	-27	7	3	5	12	11
-1	2	3	99	99	5	6	3	15	-12	8	1	4	25	22	3	6	4	15	18	8	3	5	20	-21
0	2	3	12	-14	6	6	3	17	13	-4	2	4	16	-19	4	6	4	19	-16	-7	4	5	15	-18
1	2	3	22	-22	-6	7	3	15	11	-3	2	4	11	11	7	6	4	12	-11	-6	4	5	11	13
2	2	3	52	49	-5	7	3	11	-7	-1	2	4	15	-13	-1	7	4	14	1	-5	4	5	19	17
3	2	3	66	-66	-3	7	3	22	19	0	2	4	35	33	1	7	4	15	13	-4	4	5	27	-31
4	2	3	21	-22	-2	7	3	17	-17	2	2	4	36	-33	-6	8	4	13	7	-3	4	5	13	14
5	2	3	41	42	0	7	3	24	24	4	2	4	21	-19	-4	8	4	13	-11	-2	4	5	29	24
6	2	3	31	-31	1	7	3	24	-21	5	2	4	16	-20	-7	1	5	18	-20	-1	4	5	35	-32
7	2	3	11	15	3	7	3	13	12	9	2	4	11	7	-6	1	5	24	29	0	4	5	21	25
8	2	3	19	21	4	7	3	21	-21	-5	3	4	39	-39	-4	1	5	28	-29	1	4	5	22	22
-11	3	3	15	-8	4	8	3	13	-4	-4	3	4	19	23	-3	1	5	22	21	2	4	5	11	-13
-6	3	3	22	-23	-13	0	4	12	-15	-3	3	4	14	-12	-1	1	5	41	-38	9	4	5	13	2
-4	3	3	47	44	-11	0	4	35	37	-2	3	4	40	-42	0	1	5	33	37	-7	5	5	14	12
-3	3	3	53	-53	-10	0	4	34	-34	-1	3	4	48	48	1	1	5	40	-42	-4	5	5	23	21
-2	3	3	46	49	-9	0	4	14	-15	0	3	4	15	-15	3	1	5	15	19	-3	5	5	18	-18
-1	3	3	28	-21	-8	0	4	36	40	1	3	4	28	-28	4	1	5	21	-23	0	5	5	30	-31
0	3	3	57	-57	-7	0	4	49	-54	2	3	4	40	42	5	1	5	11	16	3	5	5	16	-14
1	3	3	86	89	-6	0	4	38	39	3	3	4	11	-11	6	1	5	12	15	4	5	5	24	25
3	3	3	96	-97	-5	0	4	26	27	5	3	4	12	13	8	1	5	14	14	7	5	5	14	10
4	3	3	67	64	-4	0	4	83	-92	6	3	4	24	-8	2	5	26	-27	-2	6	5	19	19
5	3	3	27	-29	-3	0	4	70	85	-7	4	4	17	17	-7	2	5	22	22	-1	6	5	25	-27

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C14H13O3NBR. OR04.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	56	-56	0	6	0	49	52	0	3	1	94	-94	-3	9	1	15	0	-6	3	2	10	10
3	0	0	207	202	1	6	0	25	-22	1	3	1	51	50	-11	0	2	14	3	-5	3	2	11	8
4	0	0	143	-141	2	6	0	16	-15	2	3	1	18	-13	-10	0	2	33	-37	-4	3	2	45	44
5	0	0	55	-58	3	6	0	26	23	3	3	1	31	-33	-9	0	2	24	31	-3	3	2	20	20
6	0	0	85	84	4	6	0	20	-23	4	3	1	50	50	-7	0	2	112	-107	-2	3	2	27	-27
7	0	0	76	-75	5	6	0	15	-20	5	3	1	34	-34	-6	0	2	34	34	-1	3	2	32	29
8	0	0	9	9	6	6	0	12	12	6	3	1	12	4	-5	0	2	30	32	2	3	2	45	42
9	0	0	10	13	7	6	0	11	-11	7	3	1	36	33	-4	0	2	194	-190	3	3	2	18	-17
1	1	0	98	-97	4	7	0	17	-16	8	3	1	29	-28	-3	0	2	161	158	4	3	2	18	-14
2	1	0	132	131	6	7	0	13	8	10	3	1	11	12	-2	0	2	110	116	5	3	2	30	31
3	1	0	8	-7	0	8	0	15	8	11	3	1	13	-14	-1	0	2	18	-25	6	3	2	10	-9
4	1	0	27	-29	3	8	0	13	10	-6	4	1	9	-5	0	0	2	86	87	9	3	2	16	-13
5	1	0	77	76	-9	1	1	19	-15	-5	4	1	41	40	2	0	2	107	-99	-10	4	2	14	10
7	1	0	10	-7	-8	1	1	9	12	-2	4	1	45	46	3	0	2	39	39	-7	4	2	31	30
9	1	0	14	-12	-5	1	1	45	41	0	4	1	31	-31	4	0	2	46	-41	-6	4	2	32	-27
10	1	0	13	9	-3	1	1	37	-37	2	4	1	47	-56	5	0	2	25	21	-4	4	2	46	49
0	2	0	113	-116	-1	1	1	22	-21	4	4	1	11	-15	7	0	2	18	-19	-3	4	2	59	-57
1	2	0	84	-90	0	1	1	34	35	6	4	1	13	13	8	0	2	22	21	-1	4	2	49	41
2	2	0	66	-68	2	1	1	11	9	10	4	1	11	-10	10	0	2	25	-29	0	4	2	55	-56
3	2	0	9	-8	3	1	1	28	30	-3	5	1	13	-11	-11	1	2	16	-18	1	4	2	40	41
4	2	0	19	-21	4	1	1	42	-41	-2	5	1	16	17	-9	1	2	13	14	2	4	2	42	42
5	2	0	32	29	5	1	1	11	5	-1	5	1	15	18	-8	1	2	38	-37	3	4	2	47	-47
6	2	0	9	8	6	1	1	19	21	1	5	1	11	12	-6	1	2	30	31	4	4	2	25	26
10	2	0	12	-4	7	1	1	30	-27	3	5	1	13	-16	-5	1	2	83	-79	5	4	2	13	-18
11	2	0	12	6	-9	2	1	18	17	4	5	1	13	14	-4	1	2	51	47	7	4	2	10	12
1	3	0	42	-40	-6	2	1	38	36	6	5	1	12	-12	-3	1	2	7	-5	9	4	2	13	-8
2	3	0	7	4	-5	2	1	80	-78	-6	6	1	14	-19	-2	1	2	131	-131	-6	5	2	20	-22
3	3	0	36	-38	-4	2	1	18	19	-5	6	1	20	15	-1	1	2	10	-9	-5	5	2	21	21
4	3	0	15	-16	-3	2	1	33	-33	-4	6	1	14	-17	0	1	2	14	15	-4	5	2	37	-39
5	3	0	17	15	-2	2	1	159	-162	-3	6	1	24	-25	1	1	2	51	-50	-2	5	2	28	28
6	3	0	19	-19	-1	2	1	63	56	-2	6	1	38	36	2	1	2	68	64	-1	5	2	31	-33
7	3	0	12	0	0	2	1	96	97	-1	6	1	23	-24	3	1	2	65	-60	1	5	2	21	23
9	3	0	14	-8	1	2	1	24	-17	0	6	1	17	-14	4	1	2	13	-17	2	5	2	39	-40
0	4	0	77	-80	2	2	1	147	149	1	6	1	38	37	5	1	2	29	31	3	5	2	19	20
1	4	0	50	51	3	2	1	45	-41	2	6	1	20	-16	6	1	2	34	-34	5	5	2	19	-21
2	4	0	41	46	4	2	1	33	-32	4	6	1	27	29	8	1	2	14	17	6	5	2	19	16
3	4	0	28	-22	5	2	1	53	53	5	6	1	21	-19	9	1	2	11	-17	7	6	2	22	-22
4	4	0	48	51	6	2	1	28	-30	6	6	1	14	7	-7	2	2	36	33	-6	6	2	16	16
6	4	0	28	-29	7	2	1	12	-7	-1	7	1	12	-9	-4	2	2	40	39	-4	6	2	30	-30
7	4	0	23	23	8	2	1	16	20	0	7	1	11	13	-3	2	2	56	-57	-3	6	2	27	30
10	4	0	12	4	9	2	1	21	-22	1	7	1	18	-19	-2	2	2	22	-24	-1	6	2	14	-9
1	5	0	51	49	10	2	1	15	15	3	7	1	9	13	-1	2	2	17	-18	0	6	2	20	22
2	5	0	44	-45	-7	3	1	29	30	4	7	1	15	-13	0	2	2	62	-61	1	6	2	14	-16
3	5	0	31	31	-6	3	1	39	-38	-1	8	1	16	9	2	2	2	38	37	3	6	2	12	5
4	5	0	26	30	-4	3	1	33	36	0	8	1	11	-9	3	2	2	17	-13	4	6	2	13	-18
5	5	0	33	-31	-3	3	1	50	-49	2	8	1	13	12	4	2	2	24	26	5	6	2	17	16
6	5	0	13	17	-2	3	1	42	44	3	8	1	15	-14	5	2	2	10	-14	-6	7	2	18	22
9	5	0	11	12	-1	3	1	30	29	7	8	1	15	3	6	2	2	8	-6	-5	7	2	21	-17

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C14H13O3NBER, 0804.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	56	-56	0	6	0	49	52	0	3	1	94	-94	-3	9	1	15	0	-6	3	2	10	10
3	0	0	207	202	1	6	0	25	-22	1	3	1	51	50	-11	0	2	14	3	-5	3	2	11	8
4	0	0	143	-141	2	6	0	16	-15	2	3	1	18	-13	-10	0	2	33	-37	-4	3	2	45	44
5	0	0	55	-50	3	6	0	26	23	3	3	1	31	-33	-9	0	2	24	31	-3	3	2	20	20
5	0	0	85	84	4	6	0	20	-23	4	3	1	50	50	-7	0	2	112	-107	-2	3	2	27	-27
7	0	0	76	-75	5	6	0	15	-20	5	3	1	34	-34	-6	0	2	34	34	-1	3	2	32	29
8	0	0	9	9	6	6	0	19	12	6	3	1	12	4	-5	0	2	30	32	2	3	2	45	42
9	0	0	10	13	7	6	0	11	-11	7	3	1	36	33	-4	0	2	154	-150	3	3	2	18	-17
1	1	0	98	-97	4	7	0	17	-16	8	3	1	29	-28	-3	0	2	161	158	4	3	2	18	-14
2	1	0	132	131	6	7	0	13	8	10	3	1	11	12	-2	0	2	110	116	5	3	2	30	31
3	1	0	8	-7	0	8	0	15	8	11	3	1	13	-14	-1	0	2	18	-25	6	3	2	10	-9
4	1	0	27	-29	3	8	0	13	10	-6	4	1	9	-5	0	0	2	88	87	9	3	2	16	-13
5	1	0	77	76	-9	1	1	13	-15	-5	4	1	41	40	2	0	2	107	-99	-10	4	2	14	10
7	1	0	10	-7	-8	1	1	9	12	-2	4	1	45	46	3	0	2	39	39	-7	4	2	31	30
9	1	0	14	-12	-5	1	1	45	41	0	4	1	31	-31	4	0	2	46	-41	-6	4	2	32	-27
10	1	0	13	9	-3	1	1	37	-37	2	4	1	47	-56	5	0	2	25	21	-4	4	2	46	49
0	2	0	113	-116	-1	1	1	22	-21	4	4	1	11	-15	7	0	2	18	-19	-3	4	2	59	-57
1	2	0	84	-90	0	1	1	34	35	6	4	1	13	13	8	0	2	22	21	-1	4	2	49	41
2	2	0	66	-66	2	1	1	11	9	10	4	1	11	-10	10	0	2	25	-29	0	4	2	55	-56
3	2	0	9	-9	3	1	1	28	30	-3	5	1	13	-11	-11	1	2	18	-18	1	4	2	40	41
4	2	0	13	-11	4	1	1	42	-41	-2	5	1	16	17	-9	1	2	13	14	2	4	2	42	42
5	2	0	32	29	5	1	1	11	5	-1	5	1	15	18	-8	1	2	38	-37	3	4	2	47	-47
6	2	0	9	8	6	1	1	19	21	1	5	1	11	12	-6	1	2	30	31	4	4	2	25	26
10	2	0	12	-4	7	1	1	30	-27	3	5	1	13	-16	-5	1	2	83	-79	6	4	2	13	-18
11	2	0	12	6	-9	2	1	18	17	4	5	1	13	14	-4	1	2	51	47	7	4	2	10	12
1	3	0	42	-40	-6	2	1	38	36	6	5	1	12	-12	-3	1	2	7	-5	9	4	2	13	-8
2	3	0	7	4	-5	2	1	80	-78	-6	6	1	14	-19	-2	1	2	131	-131	-6	5	2	20	-22
3	3	0	36	-38	-4	2	1	18	19	-5	6	1	20	15	-1	1	2	10	-9	-5	5	2	21	21
4	3	0	15	-16	-3	2	1	33	-33	-4	6	1	14	-17	0	1	2	14	15	-4	5	2	37	-39
5	3	0	17	15	-2	2	1	159	-162	-3	6	1	24	-25	1	1	2	51	-50	-3	5	2	38	38
6	3	0	19	-19	-1	2	1	63	56	-2	6	1	38	36	2	1	2	68	64	-1	5	2	31	-33
7	3	0	12	0	0	2	1	96	97	-1	6	1	23	-24	3	1	2	65	-60	1	5	2	21	23
9	3	0	14	-8	1	2	1	24	-17	0	6	1	17	-14	4	1	2	13	-17	2	5	2	39	-40
0	4	0	77	-80	2	2	1	147	149	1	6	1	38	37	5	1	2	29	31	3	5	2	19	20
1	4	0	50	51	3	2	1	45	-41	2	6	1	20	-16	6	1	2	34	-34	5	5	2	19	-21
2	4	0	41	46	4	2	1	33	-32	4	6	1	27	29	8	1	2	14	17	6	5	2	19	16
3	4	0	28	-22	5	2	1	53	53	5	6	1	21	-19	9	1	2	11	-17	-7	6	2	22	-22
4	4	0	48	51	6	2	1	28	-30	6	6	1	14	7	-7	2	2	36	33	-6	6	2	16	16
6	4	0	28	-29	7	2	1	12	-7	-1	7	1	12	-9	-4	2	2	40	39	-4	6	2	30	-30
7	4	0	23	23	8	2	1	16	20	0	7	1	11	13	-3	2	2	86	-87	-3	6	2	27	30
10	4	0	12	4	9	2	1	21	-22	1	7	1	18	-19	-2	2	2	22	-24	-1	6	2	14	-9
1	5	0	51	49	10	2	1	15	15	3	7	1	9	13	-1	2	2	17	-10	0	6	2	20	22
2	5	0	44	-45	-7	3	1	29	30	4	7	1	15	-13	0	2	2	62	-61	1	6	2	14	-16
3	5	0	31	31	-6	3	1	39	-38	-1	8	1	15	9	2	2	2	38	37	3	6	2	12	5
4	5	0	26	30	-4	3	1	33	36	0	8	1	11	-9	3	2	2	17	-13	4	6	2	13	-18
5	5	0	33	-31	-3	3	1	50	-49	2	8	1	13	12	4	2	2	24	26	5	6	2	17	16
6	5	0	13	17	-2	3	1	42	44	3	8	1	15	-14	5	2	2	10	-14	-6	7	2	18	22
9	5	0	11	12	-1	3	1	30	29	7	8	1	15	3	6	2	2	8	-6	-5	7	2	21	-17

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-5	1	29	11	-11	3	0	30	3	5	-1	2	31	3	-4	-2	2	32	8	6	-2	1	34	7	6
-2	1	29	6	-7	4	0	30	5	5	0	2	31	9	10	2	2	32	5	4	-1	1	34	3	-2
0	1	29	4	5	8	0	30	4	4	2	2	31	5	-5	4	2	32	4	-4	0	1	34	3	-4
1	1	29	3	-2	-7	1	30	3	3	3	2	31	9	8	-6	3	32	6	4	1	1	34	4	4
2	1	29	4	5	-6	1	30	6	-6	4	2	31	5	2	-1	3	32	3	2	3	1	34	7	6
3	1	29	3	3	-4	1	30	9	9	-7	3	31	4	4	2	3	32	3	0	-3	2	34	4	-4
4	1	29	6	6	-2	1	30	6	-6	-2	3	31	7	6	3	3	32	6	-6	3	2	34	4	-3
5	1	29	4	3	-1	1	30	4	6	1	3	31	4	3	4	3	32	8	7	-2	3	34	2	0
-3	2	29	4	3	-8	2	30	3	-1	2	3	31	6	-6	-3	4	32	4	3	0	3	34	7	-7
-2	2	29	4	-4	-7	2	30	5	5	3	3	31	5	3	-2	1	33	10	-9	1	3	34	9	8
2	2	29	9	7	1	2	30	5	-5	4	3	31	5	-5	-1	1	33	6	5	3	3	34	8	-7
4	2	29	8	-8	-5	3	30	4	-2	5	3	31	4	-3	0	1	33	3	-5	-5	1	35	6	-5
5	2	29	4	4	-4	3	30	7	6	1	4	31	6	5	2	1	33	4	3	-4	1	35	5	5
1	3	29	5	5	-3	3	30	4	-3	2	4	31	10	-9	-6	2	33	3	2	-1	1	35	4	5
5	3	29	8	-6	-2	3	30	4	4	4	4	31	4	-3	-1	2	33	4	-5	-4	2	35	4	-3
-5	4	29	4	-4	2	3	30	9	8	-7	0	32	5	4	0	2	33	3	3	4	2	35	6	-4
-1	4	29	4	-4	3	3	30	11	-10	-6	0	32	6	-5	1	2	33	3	-2	-2	3	35	3	3
0	4	29	3	-4	1	4	30	7	-6	-5	0	32	3	-1	5	2	33	5	4	-4	0	36	5	-4
2	4	29	5	-4	2	4	30	10	8	-4	0	32	4	-3	3	3	33	9	7	0	0	36	9	8
3	4	29	4	3	-6	1	31	5	4	-3	0	32	3	-3	-5	0	34	7	-6	1	0	36	8	-7
0	5	29	4	4	-4	1	31	5	-3	-2	0	32	5	4	-5	0	34	13	13	2	0	36	11	-10
-7	0	30	4	-4	-3	1	31	3	3	0	0	32	5	3	-4	0	34	3	-2	3	0	36	8	6
-6	0	30	7	8	-1	1	31	9	9	4	0	32	6	6	-3	0	34	4	-5	-2	1	36	4	-4
-4	0	30	14	-13	0	1	31	7	-7	5	0	32	5	-5	-2	0	34	12	11	-1	1	36	3	-2
-3	0	30	6	-5	3	1	31	5	-4	7	0	32	3	-4	-1	0	34	12	-11	-2	2	36	5	-4
-1	0	30	9	-8	4	1	31	2	-3	-5	1	32	5	-4	0	0	34	3	-3	2	2	36	8	7
0	0	30	9	7	-7	2	31	3	0	1	1	32	4	4	4	0	34	8	-4	-3	1	37	6	-6
1	0	30	4	-5	-4	2	31	3	-4	4	1	32	4	4	-4	1	34	3	3	-2	1	37	3	3
2	0	30	11	-10	-3	2	31	6	7	-3	2	32	4	-3	-3	1	34	7	-7					

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	2	25	4	-4	5	0	26	5	5	3	3	26	9	9	6	2	27	6	-8	-5	1	28	7	-9
2	2	25	3	-2	8	0	26	3	3	4	3	26	8	7	-8	3	27	2	0	-3	1	28	4	4
4	2	25	5	-4	10	0	26	9	-9	5	3	26	5	5	0	3	27	8	-10	0	1	28	7	-8
5	2	25	12	11	-8	1	26	6	5	6	3	26	3	-2	2	3	27	5	-5	1	1	28	2	3
6	2	25	7	6	-7	1	26	11	-11	-5	4	26	3	-4	3	3	27	8	-7	3	1	28	7	6
7	2	25	8	-8	-6	1	26	3	3	-3	4	26	6	5	4	3	27	5	6	5	1	28	4	-4
-6	3	25	5	-5	-5	1	26	12	12	-2	4	26	10	-10	5	3	27	3	3	6	1	28	2	0
-5	3	25	7	-7	-4	1	26	12	12	0	4	26	4	4	-2	4	27	5	-5	9	1	28	4	-3
-3	3	25	6	7	-3	1	26	12	-12	1	4	26	5	-6	-1	4	27	3	2	-8	2	28	4	-4
-2	3	25	3	-1	-2	1	26	3	2	3	4	26	9	8	2	4	27	4	-2	-7	2	28	4	3
2	3	25	5	5	-1	1	26	21	-20	-3	5	26	3	-1	3	4	27	8	-6	-5	2	28	8	9
4	3	25	3	-4	1	1	26	6	11	-1	5	26	3	-5	4	4	27	9	7	-4	2	28	9	-10
-3	4	25	3	4	2	1	26	10	-10	0	5	26	3	3	5	4	27	5	-6	-3	2	28	8	8
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2	5	25	5	-5	1	2	26	9	9	-2	1	27	5	-5	-10	0	28	3	-3	-3	3	28	4	-4
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-8	0	26	4	-5	6	2	26	9	9	3	1	27	5	-6	-2	0	28	32	32	-3	4	28	3	-3
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3	0	26	17	15	1	3	26	7	7	1	2	27	9	-10	-8	1	28	3	2	-4	5	28	3	-2
4	0	26	8	9	2	3	26	8	-7	4	2	27	11	-11	-6	1	28	12	12	-1	5	28	5	-5



H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-9	9	2	6	7	4	10	2	5	3	19	0	3	8	-8	13	1	3	5	4	9	2	3	4	-4
-8	9	2	4	-5	5	10	2	7	6	-21	1	3	20	18	15	1	3	19	17	10	2	3	3	-3
-7	9	2	33	-33	6	10	2	9	7	-20	1	3	7	-5	16	1	3	12	11	11	2	3	9	-9
-6	9	2	4	3	9	10	2	16	-17	-19	1	3	14	-15	18	1	3	10	11	12	2	3	26	26
-5	9	2	12	-10	-7	11	2	4	1	-17	1	3	7	9	-21	2	3	4	3	13	2	3	19	-20
-4	9	2	6	-2	-6	11	2	9	7	-16	1	3	15	15	-20	2	3	10	9	14	2	3	3	-6
-3	9	2	8	7	-4	11	2	18	19	-15	1	3	24	-22	-19	2	3	3	-3	15	2	3	15	15
-2	9	2	15	-14	-3	11	2	7	4	-14	1	3	4	-6	-18	2	3	4	5	16	2	3	9	10
-1	9	2	13	11	-2	11	2	13	11	-13	1	3	7	6	-17	2	3	21	21	17	2	3	6	4
0	9	2	3	2	-1	11	2	10	-10	-12	1	3	3	0	-16	2	3	44	45	18	2	3	8	-9
1	9	2	33	33	1	11	2	12	12	-11	1	3	3	1	-15	2	3	7	-6	19	2	3	11	12
2	9	2	7	5	2	11	2	15	-17	-10	1	3	4	-3	-14	2	3	23	-23	-20	3	3	9	-8
3	9	2	6	6	3	11	2	4	2	-9	1	3	35	37	-13	2	3	3	5	-19	3	3	6	3
5	9	2	26	-23	4	11	2	6	-4	-8	1	3	45	45	-12	2	3	14	-15	-18	3	3	4	2
6	9	2	17	19	-21	0	2	14	-15	-7	1	3	64	-62	-11	2	3	12	-11	-17	3	3	4	3
7	9	2	7	-8	-19	0	3	6	-5	-6	1	3	34	-36	-10	2	3	41	-42	-16	3	3	6	4
8	9	2	8	-8	-17	0	3	11	-12	-5	1	3	12	13	-9	2	3	24	-23	-15	3	3	18	19
9	9	2	14	-15	-15	0	3	36	32	-4	1	3	64	-64	-8	2	3	54	-53	-14	3	3	4	4
10	9	2	3	-5	-13	0	3	16	15	-3	1	3	79	-78	-7	2	3	44	44	-13	3	3	36	-36
11	9	2	10	10	-11	0	3	19	18	-2	1	3	52	53	-6	2	3	8	-7	-12	3	3	24	25
12	9	2	13	-14	-9	0	3	14	-18	-1	1	3	49	50	-5	2	3	8	-7	-11	3	3	7	-7
-11	10	2	5	4	-7	0	3	9	-10	0	1	3	56	54	-4	2	3	116	122	-10	3	3	7	-6
-10	10	2	3	-3	-5	0	3	92	-93	1	1	3	18	17	-3	2	3	32	-32	-9	3	3	14	-15
-9	10	2	9	8	-3	0	3	48	48	2	1	3	13	11	-2	2	3	52	53	-8	3	3	48	-48
-8	10	2	9	11	-1	0	3	82	81	3	1	3	43	43	-1	2	3	6	-7	-7	3	3	13	14
-7	10	2	12	-11	1	0	3	103	101	4	1	3	12	11	0	2	3	9	-1	-6	3	3	10	10
-6	10	2	20	-19	3	0	3	31	30	5	1	3	32	-33	1	2	3	53	-52	-5	3	3	15	16
-5	10	2	10	7	5	0	3	27	-29	6	1	3	28	-30	2	2	3	112	-114	-4	3	3	53	-56
-4	10	2	15	-14	7	0	3	89	-93	7	1	3	14	-14	3	2	3	76	76	-3	3	3	10	-11
-3	10	2	9	-10	9	0	3	19	19	8	1	3	39	37	4	2	3	38	-37	-2	3	3	60	59
-2	10	2	13	-14	11	0	3	30	-31	9	1	3	26	-28	5	2	3	22	-21	-1	3	3	21	21
-1	10	2	4	4	13	0	3	48	47	10	1	3	32	-31	6	2	3	6	5	0	3	3	9	-10
0	10	2	10	12	15	0	3	17	-18	11	1	3	31	31	7	2	3	17	16	1	3	3	54	51
3	10	2	4	6	17	0	3	5	5	12	1	3	29	-29	8	2	3	34	35	2	3	3	10	11

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C19WH16CL50, OR07.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
17	3	2	13	-14	14	4	2	15	-16	12	5	2	4	-4	11	6	2	9	-10	14	7	2	6	-7
19	3	2	10	-11	15	4	2	31	30	13	5	2	4	5	12	6	2	4	3	15	7	2	5	-6
-19	4	2	5	-7	16	4	2	4	1	14	5	2	8	-11	13	6	2	20	-20	-15	8	2	7	7
-18	4	2	22	-20	17	4	2	12	-10	15	5	2	19	-19	14	6	2	17	17	-14	8	2	8	7
-17	4	2	9	10	19	4	2	4	-6	16	5	2	10	-8	15	6	2	3	5	-13	8	2	4	-4
-16	4	2	7	8	-19	5	2	9	9	17	5	2	6	-6	16	6	2	20	-19	-12	8	2	3	0
-15	4	2	10	10	-18	5	2	4	3	18	5	2	9	-8	-17	7	2	3	2	-11	8	2	14	13
-14	4	2	25	-25	-16	5	2	4	-1	-18	6	2	7	-6	-16	7	2	8	-9	-10	8	2	14	-12
-13	4	2	10	9	-15	5	2	5	-6	-16	6	2	4	-2	-15	7	2	7	-7	-9	8	2	5	-7
-12	4	2	8	9	-14	5	2	26	27	-15	6	2	4	4	-13	7	2	19	-17	-8	8	2	7	7
-11	4	2	48	-47	-13	5	2	54	-56	-14	6	2	27	27	-12	7	2	18	17	-7	8	2	12	-11
-10	4	2	22	-22	-12	5	2	9	-9	-13	6	2	5	-3	-11	7	2	6	-6	-6	8	2	18	-19
-9	4	2	19	21	-11	5	2	9	9	-12	6	2	18	-18	-10	7	2	30	32	-5	8	2	10	11
-8	4	2	16	17	-10	5	2	13	13	-11	6	2	23	23	-9	7	2	18	19	-4	8	2	20	-19
-7	4	2	12	-13	-9	5	2	44	42	-10	6	2	10	10	-8	7	2	8	8	-3	8	2	9	10
-6	4	2	64	65	-8	5	2	61	-61	-9	6	2	9	10	-7	7	2	19	19	-2	8	2	7	-7
-5	4	2	91	92	-7	5	2	54	54	-8	6	2	34	-34	-6	7	2	19	-19	-1	8	2	10	10
-4	4	2	61	59	-6	5	2	12	13	-7	6	2	13	-14	-5	7	2	14	-12	0	8	2	1	-8
-3	4	2	98	-100	-5	5	2	3	6	-6	6	2	32	32	-4	7	2	16	-16	1	8	2	4	-9
-2	4	2	56	-55	-4	5	2	14	13	-5	6	2	5	7	-3	7	2	7	6	2	8	2	25	24
-1	4	2	34	-31	-3	5	2	72	-68	-3	6	2	36	-35	-2	7	2	49	-50	3	8	2	26	-26
0	4	2	46	-45	-2	5	2	20	19	-2	6	2	14	14	-1	7	2	26	-27	4	8	2	5	-4
1	4	2	12	-13	0	5	2	10	11	-1	6	2	5	-4	0	7	2	33	33	5	8	2	17	16
2	4	2	83	-81	1	5	2	20	18	0	6	2	27	-27	1	7	2	7	5	6	8	2	5	1
3	4	2	75	76	2	5	2	62	-61	1	6	2	6	-8	3	7	2	5	-5	8	8	2	4	5
4	4	2	31	32	3	5	2	22	-23	2	6	2	9	9	4	7	2	49	50	9	8	2	6	5
5	4	2	30	-29	4	5	2	25	-22	3	6	2	16	-16	6	7	2	9	8	10	8	2	8	9
6	4	2	21	21	5	5	2	11	-11	4	6	2	25	-26	7	7	2	27	27	11	8	2	7	5
7	4	2	5	6	6	5	2	9	8	5	6	2	61	62	8	7	2	30	-32	12	8	2	14	14
8	4	2	41	42	7	5	2	14	13	6	6	2	26	26	9	7	2	3	3	13	8	2	13	12
9	4	2	14	-44	8	5	2	28	30	7	6	2	10	-9	10	7	2	3	2	14	8	2	15	-14
11	4	2	27	27	9	5	2	19	18	8	6	2	8	-10	11	7	2	12	-13	-13	9	2	7	7
12	4	2	6	7	10	5	2	31	32	9	6	2	10	9	12	7	2	4	-2	-11	9	2	10	-10
13	4	2	4	3	11	5	2	9	12	10	6	2	7	6	13	7	2	8	-7	-10	9	2	5	7

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
13	9	1	12	-12	-20	0	2	6	-4	-7	1	2	22	22	-14	2	2	10	-11	-21	3	2	6	6
-10	10	1	4	-2	-18	0	2	9	8	-6	1	2	2	-1	-13	2	2	74	-73	-20	3	2	20	20
-9	10	1	4	-2	-16	0	2	31	31	-5	1	2	27	27	-12	2	2	17	16	-19	3	2	21	-20
-8	10	1	13	12	-14	0	2	38	38	-4	1	2	52	51	-11	2	2	25	-26	-18	3	2	8	-11
-6	10	1	6	-7	-12	0	2	13	-12	-3	1	2	15	-15	-10	2	2	24	-26	-15	3	2	32	-34
-5	10	1	14	-14	-10	0	2	15	16	-2	1	2	6	3	-9	2	2	41	-45	-14	3	2	6	-4
-4	10	1	7	8	-8	0	2	87	-88	-1	1	2	8	-9	-8	2	2	5	6	-13	3	2	34	33
-3	10	1	16	17	-6	0	2	19	19	0	1	2	99	96	-7	2	2	54	54	-11	3	2	23	22
-2	10	1	19	-16	-4	0	2	163	-170	1	1	2	48	-45	-6	2	2	23	22	-9	3	2	3	-6
-1	10	1	7	-5	-2	0	2	108	110	2	1	2	20	-22	-5	2	2	61	61	-8	3	2	45	-44
0	10	1	22	22	0	0	2	31	29	3	1	2	107	109	-4	2	2	11	-12	-7	3	2	25	-25
1	10	1	5	5	2	0	2	124	125	4	1	2	48	47	-3	2	2	95	96	-6	3	2	33	-32
2	10	1	8	-9	4	0	2	8	7	5	1	2	30	30	-2	2	2	20	-20	-5	3	2	8	8
3	10	1	4	-5	6	0	2	48	-50	6	1	2	44	-46	-1	2	2	12	-12	-4	3	2	84	87
4	10	1	6	5	8	0	2	66	-65	7	1	2	61	-60	0	2	2	19	19	-3	3	2	17	-17
5	10	1	6	6	10	0	2	12	-11	8	1	2	47	-47	1	2	2	130	130	-2	3	2	44	47
6	10	1	4	-3	12	0	2	15	-15	9	1	2	19	-17	2	2	2	23	-24	-1	3	2	49	49
7	10	1	12	12	14	0	2	36	39	10	1	2	22	-22	3	2	2	114	-117	0	3	2	37	38
8	10	1	4	-2	16	0	2	3	-2	11	1	2	19	-19	4	2	2	58	59	1	3	2	7	-7
9	10	1	8	-9	18	0	2	8	8	12	1	2	21	22	5	2	2	7	-8	2	3	2	31	-31
10	10	1	8	-7	20	0	2	16	-15	13	1	2	4	6	6	2	2	62	-60	3	3	2	10	13
-6	11	1	7	-7	-21	1	2	4	4	14	1	2	10	10	7	2	2	88	-88	4	3	2	92	-90
-5	11	1	12	11	-20	1	2	12	12	15	1	2	3	1	8	2	2	25	26	5	3	2	28	-29
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5	11	1	9	-9	-10	1	2	40	38	-17	2	2	10	10	18	2	2	13	12	14	3	2	14	13
6	11	1	9	7	-9	1	2	80	-82	-16	2	2	5	4	19	2	2	5	-10	15	3	2	10	10
7	11	1	4	-6	-8	1	2	8	-8	-15	2	2	37	36	20	2	2	11	12	16	3	2	8	10

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
7	4	1	14	13	2	5	1	9	-11	1	6	1	25	-24	0	7	1	33	-32	6	8	1	51	50
8	4	1	7	-5	3	5	1	42	-43	2	6	1	11	12	1	7	1	33	33	7	8	1	8	-6
9	4	1	56	58	4	5	1	11	12	3	6	1	18	-17	2	7	1	7	9	9	8	1	7	-7
0	4	1	35	-33	5	5	1	31	-29	5	6	1	57	-56	3	7	1	6	3	10	8	1	7	5
1	4	1	18	-18	6	5	1	46	47	6	6	1	41	41	4	7	1	5	7	11	8	1	10	-10
2	4	1	20	21	7	5	1	25	24	7	6	1	47	48	5	7	1	47	47	12	8	1	19	-18
3	4	1	9	-9	9	5	1	12	13	8	6	1	8	-7	6	7	1	11	12	13	8	1	3	3
4	4	1	3	1	10	5	1	8	7	9	6	1	6	7	8	7	1	18	21	14	8	1	8	-9
5	4	1	19	-19	11	5	1	32	32	10	6	1	3	3	9	7	1	29	-29	15	8	1	6	-10
6	4	1	29	29	12	5	1	3	3	11	6	1	51	51	10	7	1	13	-12	-14	9	1	13	11
7	4	1	4	-3	13	5	1	20	-18	12	6	1	15	-14	11	7	1	8	-8	-12	9	1	6	4
8	4	1	34	-22	14	5	1	3	5	13	6	1	7	-8	12	7	1	6	-7	-11	9	1	12	11
9	4	1	6	-4	15	5	1	14	-12	14	6	1	15	-15	14	7	1	8	-8	-10	9	1	8	-6
9	5	1	6	4	16	5	1	14	-13	15	6	1	4	4	16	7	1	4	-3	-9	9	1	26	26
8	5	1	10	-9	17	5	1	10	-10	16	6	1	4	-1	-15	8	1	10	10	-8	9	1	12	-12
7	5	1	4	-2	18	5	1	10	-9	17	6	1	16	-16	-14	8	1	27	26	-7	9	1	7	-7
6	5	1	4	-5	19	5	1	6	-1	18	6	1	7	8	-13	8	1	4	-3	-6	9	1	30	-30
5	5	1	9	9	-18	6	1	4	-4	-17	7	1	10	-9	-12	8	1	17	-16	-5	9	1	18	-19
4	5	1	5	-4	-17	6	1	6	-5	-16	7	1	15	-13	-11	8	1	15	-15	-4	9	1	3	-2
3	5	1	3	0	-16	6	1	12	13	-15	7	1	4	-2	-10	8	1	6	6	-3	9	1	19	-21
2	5	1	21	-23	-14	6	1	8	7	-14	7	1	13	-13	-9	8	1	8	-10	-2	9	1	14	14
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9	5	1	4	2	-11	6	1	6	-6	-11	7	1	30	30	-6	8	1	7	9	1	9	1	43	44
8	5	1	13	15	-10	6	1	23	22	-10	7	1	3	4	-4	8	1	15	16	2	9	1	36	36
7	5	1	27	-28	-9	6	1	21	19	-9	7	1	39	40	-3	8	1	23	23	3	9	1	17	-17
6	5	1	73	73	-8	6	1	3	4	-8	7	1	24	24	-2	8	1	13	-14	4	9	1	7	-8
5	5	1	28	28	-7	6	1	36	-36	-7	7	1	26	-26	-1	8	1	32	-33	5	9	1	4	-2
4	5	1	15	15	-6	6	1	16	-17	-6	7	1	5	-5	0	8	1	9	-10	6	9	1	23	-23
3	5	1	26	25	-5	6	1	10	9	-5	7	1	28	-28	1	8	1	21	20	7	9	1	4	-3
2	5	1	46	-46	-4	6	1	13	-14	-4	7	1	22	21	2	8	1	9	-9	8	9	1	18	-18
1	5	1	4	3	-2	6	1	34	-36	-3	7	1	44	-44	3	8	1	18	19	10	9	1	9	-7
0	5	1	89	-90	-1	6	1	37	37	-2	7	1	21	-21	4	8	1	20	-19	11	9	1	5	3
1	5	1	31	30	0	6	1	14	13	-1	7	1	15	-14	5	8	1	3	4	12	9	1	17	16

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
9	10	0	8	8	-13	1	1	8	-7	-18	2	1	38	36	16	2	1	15	-15	9	3	1	30	30
10	10	0	7	-3	-12	1	1	24	-22	-17	2	1	17	-17	17	2	1	4	-1	11	3	1	40	41
11	10	0	13	-12	-11	1	1	14	14	-16	2	1	15	16	18	2	1	5	5	12	3	1	9	7
2	11	0	10	-12	-10	1	1	18	17	-15	2	1	16	15	19	2	1	9	9	14	3	1	19	19
3	11	0	12	10	-9	1	1	11	-11	-14	2	1	7	6	20	2	1	10	-10	16	3	1	6	-5
4	11	0	7	-8	-8	1	1	27	-27	-13	2	1	18	-18	-21	3	1	10	9	17	3	1	4	-1
5	11	0	6	-6	-7	1	1	11	12	-12	2	1	60	-64	-19	3	1	15	14	18	3	1	15	-15
6	11	0	12	-10	-6	1	1	26	27	-11	2	1	41	41	-18	3	1	4	-2	20	3	1	14	-15
7	11	0	7	6	-5	1	1	8	-5	-10	2	1	12	-11	-17	3	1	12	-10	-20	4	1	21	20
-21	0	1	16	17	-4	1	1	69	-70	-9	2	1	49	-50	-16	3	1	5	-3	-18	4	1	11	-11
-19	0	1	37	-37	-3	1	1	37	-36	-8	2	1	46	-46	-15	3	1	5	3	-17	4	1	17	-15
-17	0	1	17	16	-2	1	1	18	-17	-7	2	1	6	-8	-14	3	1	14	-15	-16	4	1	19	-17
-15	0	1	4	4	1	1	1	71	-73	-6	2	1	53	56	-13	3	1	10	-10	-15	4	1	13	-12
-13	0	1	81	81	2	1	1	39	-37	-5	2	1	29	29	-12	3	1	23	23	-14	4	1	7	-5
-11	0	1	29	28	3	1	1	45	44	-4	2	1	46	48	-11	3	1	34	-35	-13	4	1	23	-22
-9	0	1	68	-67	4	1	1	45	45	-3	2	1	4	-2	-10	3	1	24	24	-12	4	1	5	2
-7	0	1	212	-210	5	1	1	22	22	-2	2	1	74	75	-9	3	1	19	17	-11	4	1	26	26
-5	0	1	4	5	6	1	1	48	46	-1	2	1	52	51	-8	3	1	18	-18	-10	4	1	5	3
-3	0	1	52	53	7	1	1	46	-45	0	2	1	74	-76	-7	3	1	21	-21	-9	4	1	18	19
3	0	1	149	-143	8	1	1	61	-61	1	2	1	53	-53	-6	3	1	22	-23	-8	4	1	8	9
5	0	1	23	24	9	1	1	53	-51	2	2	1	105	-105	-5	3	1	37	38	-7	4	1	26	27
7	0	1	25	23	10	1	1	18	-18	3	2	1	40	40	-4	3	1	19	18	-6	4	1	59	-59
9	0	1	57	-59	11	1	1	13	14	4	2	1	78	-77	-3	3	1	10	10	-5	4	1	9	9
11	0	1	28	27	12	1	1	16	-17	5	2	1	54	53	-2	3	1	33	33	-4	4	1	98	98
13	0	1	16	-19	13	1	1	26	28	6	2	1	35	35	-1	3	1	2	-3	-3	4	1	8	7
15	0	1	50	51	14	1	1	25	26	7	2	1	65	-65	0	3	1	62	63	-2	4	1	43	-44
19	0	1	13	12	15	1	1	25	26	8	2	1	31	-30	1	3	1	88	86	-1	4	1	19	-20
21	0	1	8	-8	16	1	1	19	20	9	2	1	19	19	2	3	1	66	-65	0	4	1	52	51
-21	1	1	9	11	18	1	1	4	1	10	2	1	66	65	3	3	1	74	-75	1	4	1	25	-26
-20	1	1	4	2	19	1	1	16	-16	11	2	1	20	-20	4	3	1	71	70	2	4	1	22	-25
-19	1	1	3	1	20	1	1	4	0	12	2	1	5	-5	5	3	1	41	-41	3	4	1	56	-56
-18	1	1	17	17	-21	2	1	11	-10	13	2	1	11	10	6	3	1	70	-71	4	4	1	3	4
-16	1	1	23	-22	-20	2	1	4	2	14	2	1	20	21	7	3	1	60	-59	5	4	1	37	36
-14	1	1	15	-14	-19	2	1	18	16	15	2	1	16	-16	8	3	1	3	2	6	4	1	3	3

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR C19HH16CL5O, OR07.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	0	129	-129	8	2	0	26	-25	5	4	0	12	14	0	6	0	36	36	0	8	0	46	-46
6	0	0	236	-240	10	2	0	47	46	6	4	0	49	49	1	6	0	59	57	1	8	0	16	-15
8	0	0	13	12	11	2	0	112	110	7	4	0	37	-39	2	6	0	14	-15	2	8	0	21	20
10	0	0	53	62	12	2	0	18	-18	8	4	0	25	24	3	6	0	5	-7	3	8	0	26	-27
12	0	0	27	28	13	2	0	6	6	9	4	0	22	-23	4	6	0	33	31	4	8	0	15	14
14	0	0	44	-44	14	2	0	2	4	10	4	0	33	31	5	6	0	23	21	5	8	0	23	-23
16	0	0	15	15	15	2	0	4	1	11	4	0	5	3	6	6	0	42	-43	6	8	0	9	11
18	0	0	12	-10	16	2	0	3	-5	12	4	0	27	-28	7	6	0	15	15	7	8	0	58	57
20	0	0	15	16	17	2	0	38	-38	13	4	0	28	28	8	6	0	16	17	8	8	0	19	-18
3	1	0	41	-40	18	2	0	4	8	14	4	0	4	5	9	6	0	15	-14	10	8	0	15	-15
4	1	0	9	-8	21	2	0	6	5	15	4	0	7	-7	10	6	0	15	-15	11	8	0	8	9
5	1	0	198	199	2	3	0	78	-78	16	4	0	19	-19	11	6	0	3	3	13	8	0	34	-35
6	1	0	67	66	3	3	0	18	-17	17	4	0	15	14	12	6	0	29	28	14	8	0	11	10
7	1	0	2	-3	4	3	0	57	-56	18	4	0	4	-1	13	6	0	19	-18	15	8	0	4	-6
8	1	0	4	-4	5	3	0	60	-57	19	4	0	27	-27	14	6	0	4	-2	1	9	0	17	15
9	1	0	70	-67	6	3	0	14	14	1	5	0	17	-16	15	6	0	16	-16	2	9	0	60	60
10	1	0	25	-26	7	3	0	15	-14	2	5	0	3	-1	16	6	0	28	-26	3	9	0	18	18
11	1	0	54	-53	8	3	0	14	13	3	5	0	25	-25	1	7	0	7	-8	4	9	0	17	17
12	1	0	16	-16	9	3	0	27	26	4	5	0	73	-72	2	7	0	61	61			0	28	-22
13	1	0	13	-13	10	3	0	6	-4	5	5	0	3	1	3	7	0	3	0			0	17	-16
14	1	0	15	21	11	3	0	22	23	6	5	0	42	41	4	7	0	21	21			0	13	-12
15	1	0	38	40	12	3	0	18	17	7	5	0	26	28	5	7	0	6	-6	8	9	0	29	-29
16	1	0	41	43	13	3	0	8	6	8	5	0	3	3	6	7	0	28	26	9	9	0	21	-21
17	1	0	17	20	14	3	0	3	2	9	5	0	41	41	7	7	0	19	18	10	9	0	4	-3
19	1	0	7	-8	15	3	0	15	12	10	5	0	19	19	8	7	0	31	-32	11	9	0	3	6
21	1	0	9	-9	16	3	0	18	16	11	5	0	4	2	9	7	0	33	31	12	9	0	14	14
0	2	0	16	-16	15	3	0	3	-3	12	5	0	5	6	10	7	0	28	-29	13	9	0	11	10
1	2	0	77	-77	19	3	0	6	-6	13	5	0	22	-23	11	7	0	19	-18	1	10	0	7	11
2	2	0	51	53	20	3	0	13	-15	14	5	0	14	-15	12	7	0	3	4	2	10	0	13	13
3	2	0	13	-12	0	4	0	105	-101	15	5	0	8	-8	13	7	0	15	-14	3	10	0	10	-7
4	2	0	104	-102	1	4	0	109	108	16	5	0	12	-12	14	7	0	4	-2	4	10	0	15	-17
5	2	0	109	-107	2	4	0	46	-45	17	5	0	19	-18	15	7	0	24	-24	5	10	0	8	7
6	2	0	28	29	3	4	0	19	19	18	5	0	11	-8	16	7	0	5	7	7	10	0	4	-7
7	2	0	34	-33	4	4	0	6	6	19	5	0	5	7	17	7	0	14	13	8	10	0	10	10

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	4	11	32	-35	7	3	12	16	-11	-5	0	14	33	33	2	3	14	21	8	-1	0	16	22	-8
3	4	11	25	29	-6	4	12	21	26	-3	0	14	34	40	7	3	14	21	7	0	0	16	21	25
4	6	11	34	34	2	4	12	20	14	-2	0	14	29	29	3	4	14	20	23	-4	1	16	38	38
-6	0	12	22	-20	-7	5	12	20	6	-1	0	14	48	-55	-3	1	15	17	-15	-2	1	16	15	-21
1	0	12	30	-25	-1	5	12	24	-23	3	0	14	29	33	-2	1	15	36	-34	-1	1	16	21	-22
6	0	12	34	33	-2	1	13	19	-25	4	0	14	31	-26	-1	1	15	24	17	-3	2	16	16	19
7	0	12	25	-24	-1	2	13	30	36	-9	1	14	20	17	0	1	15	21	23	4	2	16	22	-10
-8	2	12	20	-15	1	2	13	33	-33	-2	1	14	46	47	-7	2	15	25	-33	-3	1	17	17	-16
-2	2	12	20	16	5	2	13	28	27	0	2	14	20	5	1	2	15	23	-21	-5	3	17	21	16
0	2	12	31	-37	0	3	13	39	39	-4	3	14	20	-3	-7	5	15	23	-13	2	1	18	21	10
1	2	12	16	16	-7	4	13	20	-20	-1	3	14	17	-13	-5	6	15	23	8	-5	3	18	18	22
-1	3	12	34	29	5	5	13	21	6	0	3	14	19	-19	0	6	15	20	-9	-1	3	18	16	11
0	3	12	26	-24	-12	0	14	20	-7	1	3	14	23	21	-9	0	16	25	-34	-3	4	18	21	-6

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-5	3	4	22	-26	4	3	5	35	-35	-15	3	6	19	-9	-1	0	8	23	27	8	2	9	50	45
-3	3	4	32	30	5	3	5	28	-28	-9	3	6	24	-29	0	0	8	39	-36	-11	3	9	20	-5
-2	3	4	69	69	6	3	5	31	25	-5	3	6	18	-12	1	0	8	40	41	0	3	9	26	-24
0	3	4	12	-1	11	3	5	20	-14	-3	3	6	27	-29	2	0	8	27	-24	-6	4	9	23	-22
3	3	4	17	-21	-11	4	5	19	-19	2	3	6	37	-43	7	0	8	36	27	2	4	9	14	-9
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-11	4	4	14	-11	0	4	5	34	34	7	3	6	39	44	10	0	8	49	-46	1	5	9	20	-20
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-4	5	4	14	-20	-8	6	5	21	31	-7	6	6	31	31	3	1	8	30	28	-2	0	10	45	45
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0	5	4	15	-11	-3	6	5	16	-20	5	6	6	20	12	-11	2	8	26	23	1	0	10	33	-27
1	5	4	30	28	-9	0	6	16	15	-7	8	6	19	-30	-3	2	8	24	25	2	0	10	16	-18
2	5	4	19	17	-8	0	6	42	47	-6	1	7	19	25	1	2	8	28	-27	6	0	10	28	25
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2	1	5	37	41	2	0	6	85	-87	10	1	7	17	10	3	3	8	21	21	7	1	10	24	-20
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6	1	5	30	-27	4	0	6	82	69	-3	2	7	38	-27	8	3	8	38	-33	-1	3	10	30	-32
9	1	5	45	-42	6	0	6	19	-13	0	2	7	31	32	11	3	8	20	12	4	3	10	20	10
10	1	5	21	27	8	0	6	20	-22	1	2	7	29	25	-9	4	8	30	-28	-8	4	10	16	-13
11	1	5	31	32	9	0	6	68	66	2	2	7	44	-39	-3	4	8	18	20	-2	4	10	29	-31
-11	2	5	14	-23	-8	1	6	34	-37	7	2	7	35	-30	0	4	8	22	23	6	4	10	47	-46
-6	2	5	43	48	-5	1	6	64	66	8	2	7	13	-8	8	4	8	20	-11	1	5	10	24	-32
-5	2	5	46	-53	2	1	6	24	-16	12	2	7	19	-15	10	4	8	18	14	3	6	10	27	-34
-4	2	5	27	27	3	1	6	38	-41	-9	3	7	45	52	-1	5	8	14	11	4	6	10	22	-12
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-1	2	5	47	-44	8	1	6	32	-25	4	3	7	33	34	-9	1	9	19	11	-2	8	10	16	15
2	2	5	27	-25	9	1	6	61	-53	7	3	7	41	35	-6	1	9	25	-27	-10	1	11	20	7
3	2	5	20	-21	10	1	6	22	24	10	3	7	20	8	-5	1	9	19	12	-3	1	11	20	-21
6	2	5	47	44	-9	2	6	19	-12	-9	4	7	30	27	-4	1	9	21	21	1	1	11	19	-13
11	2	5	20	27	-4	2	6	36	-26	-1	4	7	16	14	-3	1	9	17	-20	3	1	11	15	13
-10	3	5	23	21	-3	2	6	18	14	2	4	7	32	-29	0	1	9	16	-17	-6	2	11	21	-24
-6	3	5	36	-36	-2	2	6	41	41	4	4	7	19	-13	1	1	9	44	42	-4	2	11	34	-32
-5	3	5	39	-37	2	2	6	20	11	-6	5	7	20	12	-11	2	9	33	-37	0	2	11	24	33
-3	3	5	28	24	4	2	6	32	25	-5	0	8	63	-69	-2	2	9	26	29	4	2	11	23	-18
-1	3	5	55	52	12	2	6	15	15	-4	0	8	41	-50	0	2	9	40	38	0	3	11	19	-4

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