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197

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THE DETERMINATION OF THE CRYSTAL STRUCTURE OF FOUR COMPOUNDS OF INDUSTRIAL IMPORTANCE

A Thesis submitted by

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in partial fulfilment of the requirements for the award of Doctor of Philosophy by the Council for National Academic Awards.

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ABSTRACT

of Thesis

THE DETERMINATION OF THE CRYSTAL STRUCTURE OF FOUR COMPOUNDS OF INDUSTRIAL IMPORTANCE

by

Anuradha Banerjee

This thesis describes the determination of the crystal structures of one organo-metallic and three organic compounds. Originally the research programme had envisaged working on substances which were obtained by the corrosive action of hydrocarbon oils on metallic lead, but after some considerable time had been spent on attempting to crystallise these rather intractable materials, the research was switched to deal with other compounds of industrial importance which were more readily available in the Polytechnic. Of the four crystalline compounds investigated, two were connected with the inhibition of the corrosion of steel, one was an additive used in the polymer industry, and one was a molecular complex which had exhibited some unusual physico-chemical properties.

X-ray intensity data were collected in several different ways. One crystal structure was solved from visually estimated photographic data, two sets of data were collected on our Stoe Stadi-2 diffractometer which uses Weissenberg geometry, and one set which comprised over 7000 intensities, the largest data-set ever worked on in this Department, was recorded on the CAD-4 diffractometer at Queen Mary College, University of London, by arrangement with Dr M Hursthouse.

A variety of techniques were used in working out the crystal structures. One was solved by the MULTAN direct methods computer program, two by the use of the NRC direct methods program using $\Sigma 2$ relationships, and one by "trial-and-error" because none of the other methods produced any meaningful results. Refinement of all four structures was carried out using the NRC suite of programs implemented on our Polytechnic computers (DEC-10, DEC-20, and VAX 11/750). Anisotropic thermal vibrations were allowed for, and hydrogen atom positions were calculated resulting in the conventional R-factors of 6.7, 6.1, 6.6 and 6.1% for the four structures.

No abnormalities were encountered in the results for three of the structures, but the picric acid - naphthalene complex turned out to be disordered with some of the picric acid molecules occupying an alternative orientation in the unit cell which resulted in displacement of the naphthalene molecules also, and the possibility of proton transfer within the picric acid molecule which was confirmed by differences in bond lengths and anisotropic temperature parameters.

Papers describing three of the crystal structures have already appeared in Acta Crystallographica C, and the fourth has been submitted (January 1985). Copies of these papers are shown in the Appendix.

INDEX

Chapter 1.

INTRODUCTION

The discovery of X-ray diffraction in 1912 also marked the birth of X-ray crystallography, whereby it became possible to use X-ray diffraction patterns to reveal the arrangement of atoms in a crystalline substance. This discovery can be considered as the gateway of modern technology which depends so much on the understanding of the relationship between atomic arrangement and the macro properties of materials.

Prior to the development of X-ray methods, the solid state was the least tractable of all the states of matter and the internal structure of a solid could be deduced only by argument from its physical properties or from its chemical properties in the liquid or gaseous form. X-ray analysis has removed the determination of crystal structure and molecular configuration from the sphere of speculation to that of precise measurement.

Since the development of X-ray crystallography some outstanding achievements have been made in the following fields.

1. Metals and Alloys.

The X-ray analysis of atomic arrangements in metals and alloys has helped to explain the structure dependent properties of metals. X-ray powder analysis affords a powerful way of investigating the phases formed when metals are alloyed in different proportions.

It is now possible for a theoretical physicist to state the laws of metallic combination in alloys with reference to atomic proportions.

It has now been established that some alloys exist in a disordered state where atoms of different kinds are distributed completely at

random between all the sites or some particular set of sites or an ordered state where an atom of each kind segregates to sites which form a regular pattern (super-lattice). Tammann 1919 (14) suggested the existence of this ordering to explain the changes in electrical resistivity with heat treatment, and the ordered alloy has a lower resistivity than the disordered alloy.

2. Proteins and Biological systems

The success of X-ray diffraction studies of biological molecules has played an important part in the advancement that took place in the field of molecular biology.

From single crystal X-ray analysis studies of amino acids and peptides, Pauling and Corey in 1951 (10) proposed the α-helix as the basic structure of a group of fibrous proteins which occur in hair, muscle, skin and blood. In 1957 Kendrew and his co-workers (9) solved the structure of myoglobin which was the first three-dimensional structure determination of a protein molecule. Later on the double helical structure of DNA was established by X-ray diffraction studies. X-ray crystallography has also given much valuable information about the structure of viruses.

3. Understanding of Bonding

The development of X-ray crystal structure analysis threw new light on the nature of interatomic forces. It provided the means of determining experimentally the atomic configuration of molecules whose form had long been the subject of speculation based on chemical valency theories.

A review of crystal structures of different types of solid emphasises the dominating role of the interatomic forces in determining the

structural arrangement and these forces can be divided into six types.

a. Vander Waals Bonds. These are in effect the forces which exist between non-bonded atoms in essentially saturated molecules. Crystals held together in this way are sometimes called molecular crystals. Examples are nitrogen, carbon tetrachloride, benzene etc. The molecules tend to pack together as closely as their sizes and shapes allow.

b. Ionic Bonds. This type of bond is well-known in the case of NaCl. In the NaCl crystal, the coulombic interaction between oppositely charged ions leads to a regular three-dimensional structure: each positively charged Na^+ ion is surrounded by six negatively charged Cl^- ions, and each Cl^- is surrounded by six Na^+. There are no discrete NaCl molecules. The ionic bond is spherically symmetrical and undirected. An ion will be surrounded by as many oppositely charged ions as can be accommodated so that the requirement of overall electrical neutrality is satisfied.

c) Co-valent Bonds. These bonds are the results of the sharing of electrons by atoms. When extended through three dimensions, they may lead to a variety of crystal structures, depending on the number of electrons available for bond formation.

A good example is the diamond structure. This structure can be based on two interpenetrating face-centred cubic lattices. Each atom on one lattice is surrounded tetrahedrally by four equidistant atoms on the other lattice. This arrangement constitutes a three-dimensional polymer of carbon atoms joined together by tetrahedrally oriented bonds. Thus the configuration of the carbon bonds in diamond

is similar to that in aliphatic compounds, such as ethane. Germanium, silicon, and grey tin also crystallize in the diamond structure.

A similar structure is found in compounds such as ZnS (in the zinc-blende structure), AgI, AlP, SiC. In all these structures each atom is surrounded by four unlike atoms oriented at the corners of a regular tetrahedron. In every case, the binding is primarily co-valent. The structure can occur whenever the number of outer-shell electrons is four times the number of atoms. It is not necessary for each atom to provide the same number of valence electrons.

d) Bonds of Intermediate type. In solids, as in individual molecules, such bonds can be considered to result from resonance between co-valent and ionic contributions. Alternatively we may consider the polarization of one ion by an oppositely charged ion. An ion is polarized when its electron distribution is distorted by the presence of an oppositely charged ion. The smaller an ion, the more intense is its electric field and the greater is its polarizing power. Usually, therefore the larger anions tend to be strongly polarized by the smaller cations. Even apart from the effect of size, cations are less polarizable than anions because their net positive charge tends to hold their electrons in place. The structure of an ion is also important. Alkali metal cations, such as K^+ , have less polarizing power than transition cations of similar radii such as Ag^+ , because their positive nuclei are more effectively shielded.

The effect of polarization appears in the structures of the silver

halides. AgF, AgCl, AgBr have the NaCl structure but as the anion becomes larger, it becomes more strongly polarized by the small Ag^+ ion. Finally in AgI the binding has little ionic character and the crystal has the ZnS structure. It has been confirmed spectroscopically that crystalline silver iodide is composed of atoms and not ions.

e) Hydrogen Bonds. Hydrogen bonds are important in many crystal structures, especially inorganic and organic acids, salt hydrates and ice, etc. In the structure of ordinary ice the co-ordination is similar to that in Wurtzite, the hexagonal form of zinc sulfide; Each oxygen is surrounded tetrahedrally by four other oxygens at a distance of 0.276 nm. The hydrogen bonds hold the oxygens together, leading to a very open structure.

f) Metallic Bonds. The metallic bond is closely related to the ordinary covalent electron-pair bond. Each atom in a metal forms co-valent bonds by sharing electrons with its nearest neighbours, but the number of orbitals available for bond formation exceeds the number of electron pairs available to fill them. As a result a co-valent bond resonates among the available interatomic positions. In the case of a crystal this resonance extends throughout the entire structure thereby producing greatly enhanced stability. The empty orbitals permit a ready flow of electrons under the influence of an applied electric field, leading to metallic conductivity.

In this thesis we are dealing with certain compounds of industrial importance which require a fuller understanding of the relationship between structure and properties (reactivity etc).

i) N-Nitroso-Diphenyl-Amine is used as a antiscorch agent in the rubber manufacturing industry.

ii) Cadmium 2,6-dimethyl-γ-Pyrone is used as a protective coating on steel (corrosion inhibitor)

iii) Picric Acid/Naphthalene has been used for the separation of compounds because of their clathrate structures.

iv) 2,6-dimethyl-γ-thiapyrone is similar to the organic part of (ii) but with interchange of sulphur and oxygen, and was being tested for corrosion resistance.

Historical

X-rays were discovered by Röntgen in 1895 (11) and in 1912 M von Laue (5) published his discovery of diffraction of X-rays by crystals in the proceedings of the Royal Bavarian Academy of Sciences.

Laue and his co-workers, Friedrich and Knipping (5) initially placed a crystal in the path of an X-ray beam and attempted to intercept regularly scattered beams thrown back by the crystal on a photographic plate. But there was no success. Then they tried placing the photographic plate behind the crystal to catch rays bent through a small angle. This time they were successful. The photographic plate revealed the scattered beams by spots showing

four-fold symmetry. It was the first experiment done by Laue using zincblende crystal. Laue came to a conclusion that these scattered spots were related to the atomic order in the crystal. Later on crystals of NaCl, KCl, KBr and KI led to a complete solution of their structures. Between 1915-1920 Bragg used diffraction to determine the atomic positions. He established that when a beam of monochromatic X-rays falls upon a crystal, in general there is no diffraction of the X-rays unless some set of crystal planes is set at just the correct angle.

The law of X-ray diffraction is

$$n\lambda = 2d \sin \theta \quad \text{(Bragg's equation)}$$

where θ is the angle at which the waves are reflected by the planes, d is the spacing of the planes, λ is the wavelength of the X-rays and n is a whole number 1, 2, 3 etc., which is a measure of the order of reflection. Around that time a great advance was made by the development of the powder method, independently by Debye and Sherrer in Zurich 1916 (4) and Hull in America 1917 (7). In this powder method, X-rays fall on a mass of very fine micro-crystalline particles, which are randomly oriented. Particles are so oriented that the above relation holds good for different sets of planes and X-rays are reflected through corresponding 2θ angles. All particles so oriented diffract their rays along a cone, making a halo which is intercepted by a photographic film. Each line on the film corresponds to diffraction by a different set of crystal planes in that particular crystal.

This method is widely used in industry for identifying substances such as valve filament coatings, paint constituents, fluorescent screens, products of corrosion, impurities in metals, boiler scale constituents, intermediate products of chemical reactions etc. A comprehensive index of crystalline substances of all kinds (Powder Diffraction File) was started in the United States in 1941 (1) and is being added to regularly by world-wide contributions and now contains over 35,000 diffraction patterns.

From the same time basic research on structure analysis was started using X-ray diffraction from single crystals. By 1939 about 50-100 structures had been solved. Since 1939 about 50,000 structures have been solved. These days with the development of automatic techniques about 200 crystal structures are published every month.

Equipment Development

In 1927 the rotation camera was developed by Schiebold and Polanyi (13) and very widely used at that time.

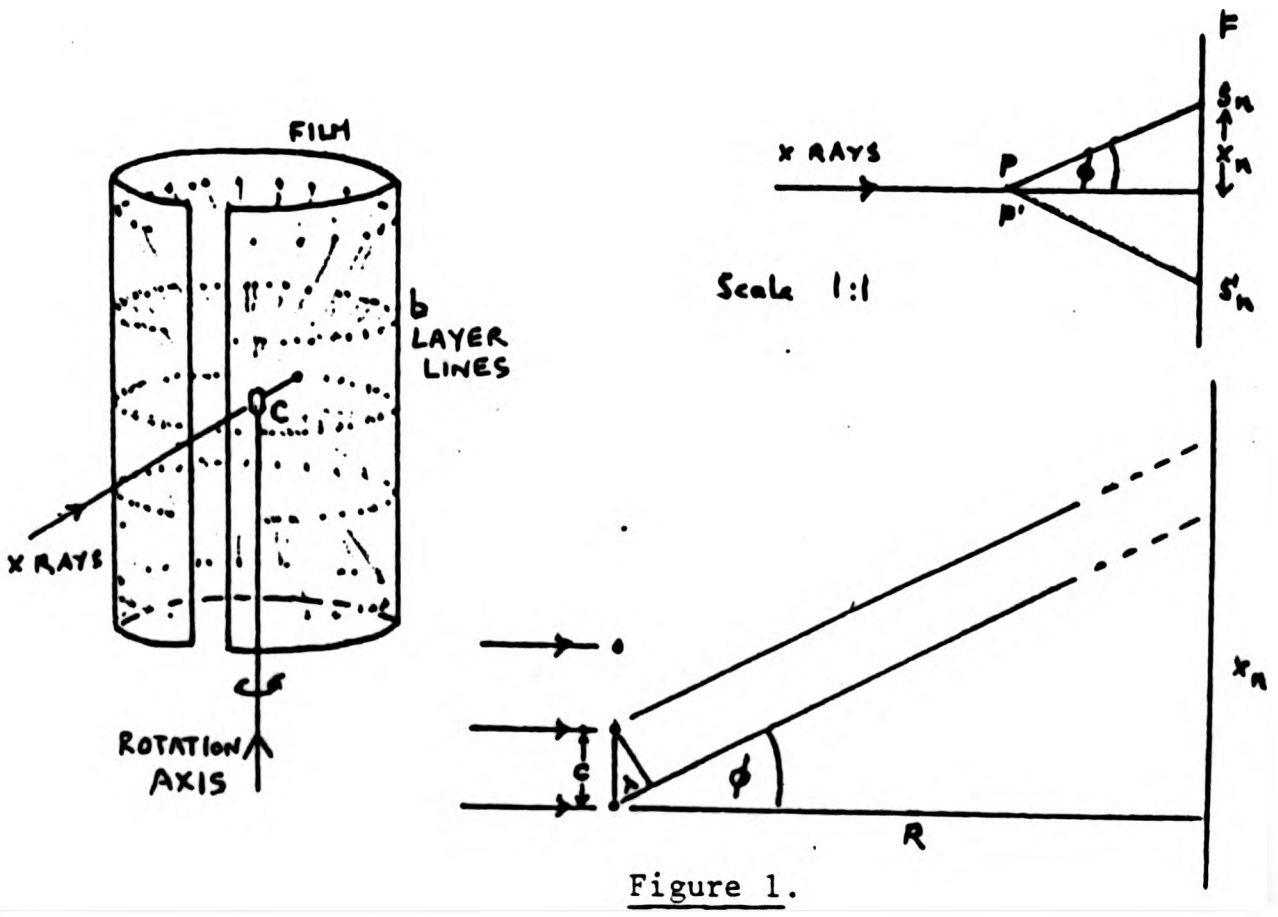

Figure 1.

A crystal is placed at ' c with one of its principal axes parallel to the vertical arrow in the figure about which it is rotated. Instead of reflecting X-rays from the face of a large crystal as in the spectrometer, a small single crystal, perhaps of about 0.1 mm^3 is used. The crystal is completely bathed in an incident beam of X-rays. The diffracted beams are recorded on a flat plate or on a cylindrical film. The resulting spots lie on a series of rows (layer lines). If the axis of rotation coincides with the c-axis of the crystal, for instance, there will be a series of layer lines as shown in figure (). All diffracted beams for which the path difference is ℓ wavelengths will make the same angle with the c-axis and so appear on the ℓ layer lines. The assignment of h and k values to each spot can be done but is less direct. The lengths of the other axes can be found by placing the crystal with each in turn parallel to the axes of rotation and a knowledge of these helps in the identification. A typical rotation photograph is shown in figure 2.

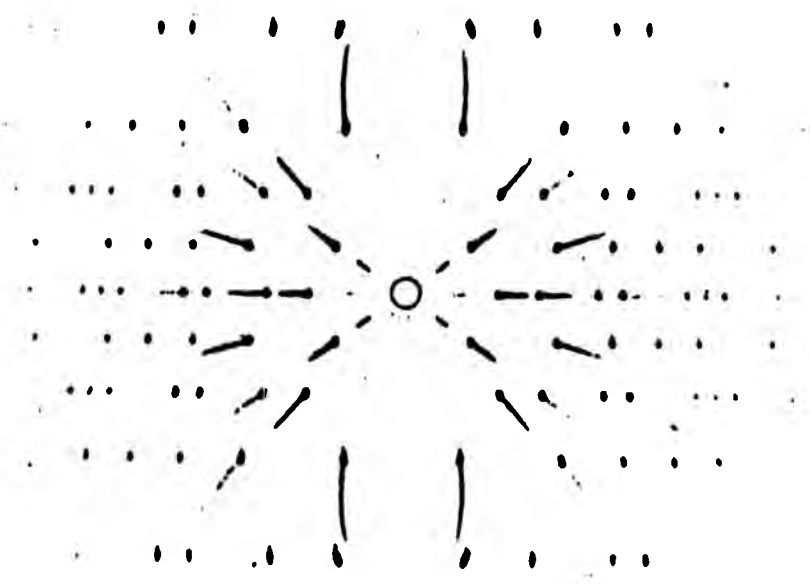

Figure 2.

In 1924 the Weissenberg Camera was developed. The Weissenberg (15) method is geometrically more nearly related to the rotation method and shares its ability to explore a large part of reciprocal space. To avoid the drawback of the rotation method - that too much information is compressed into each layer line - the layers are selected one at a time and photographed on a moving film, so that each two dimensional layer is recorded in two dimensions, although it is in a geometrically distorted form. The film forms a cylinder concentric with the crystal axis, as in a rotation/oscillation camera, and if it is kept stationary oscillation photographs can be recorded and used for checking crystal alignment. When the instrument is being used as a moving film camera, movable cylindrical metal screens are positioned so that only reflections from the selected layer can reach the film. The crystal oscillates through a predetermined angle, usually about 200°. The two motions are coupled and they reverse together; in most instruments the film moves 1 mm for each 2° of crystal rotation.

In the photograph the distance of any spot from the equator depends on $\xi(= 2 \sin \theta)$, while its horizontal distance along the film corresponds to the angular position of the crystal as the appropriate reciprocal lattice point of the crystal passes through the surface of the sphere of reflection. The axes are inclined to the equator because to bring successive lattice points into the sphere of reflection the reciprocal lattice has to rotate, and while it is doing so the film moves parallel to the axis of crystal rotation. Quantitative interpretation is usually done with the help of a Weissenberg chart which enables cartesian co-ordinates in reciprocal space to be read directly from the photograph.

Upper levels of the reciprocal lattice are photographed by moving the layer line screens to isolate the appropriate layers and at the same time the entire assembly is tilted so that the incoming X-ray beam lies in the same zone as the required level. The advantage of this equi-inclination method over other possible settings is that the form of curves is the same as for the zero layer, and allowing for spot shape the same chart can be used for interpreting the photographs, moreover it enables the maximum volume of reciprocal space to be examined.

For the upper layers the circular area of reciprocal lattice explored has a radius of $\sqrt{(1-(\xi/2)^2}$ and since this is spread over the same area of film as the zero layer, the scale of the upper layer is increased by $\frac{1}{\sqrt{(1-(\xi/2)^2)}}$. The effect is barely noticeable for small values of ξ, but increases rapidly with larger values.

The great disadvantage of the Weissenberg method is that it records a distorted representation of the reciprocal lattice. This can be avoided by precession (3) and de Jong-Bouman techniques (8). However these methods also have disadvantages. The distortion of the reciprocal lattice representation on a Weissenberg film results from the fact that the movement of the crystal, and therefore the movement of the reciprocal lattice is different from that of the film.

It follows that an undistorted representation can only be obtained if the crystal and the film movement are synchronised. This condition is realised in different ways in the de Jong-Bouman and the precession methods.

In the de Jong-Bouman technique, the film is rotated about an axis which is inclined by an angle ϕ_o against the primary beam direction. To prevent all but the zero layer cone from reaching the film, a screen having an annular opening of radius r_s is placed at a distance ds from the crystal with

$$ds = r_s/\tan \phi_o$$

On a de Jong-Bouman exposure of the zero layer, a spherical area of radius r_J is recorded. It holds that

$$\frac{r_{J/2}}{1/\lambda} = \sin \phi_o$$

$$\text{or} \quad r_J = (2/\lambda) \sin \phi_o$$

For a Weissenberg exposure, the corresponding area has a radius r_W equal to the diameter of the Ewald sphere, hence

$$r_W = 2/\lambda$$

Since $(\sin \phi_o) < 1$ for $\phi_o = \mu_o \neq 90^o$, the area covered by a de Jong-Bouman film is always smaller than for a Weissenberg film. In addition μ_o cannot be chosen too close to 90^o, since this would require a very large film. Usually $\mu_o = \phi_o = 45^o$ is chosen for which $\sin \phi_o = \sqrt{2}/2$ and

$$\frac{r_J}{r_W} = \frac{\sqrt{2}}{2}$$

since the corresponding areas F_J and F_W are related by square of r, it follows that

$$\frac{F_J}{F_W} = \frac{1}{2}$$

Under normal experimental conditions, the area recorded on a film of a de Jong-Bouman type has only half the size of the corresponding Weissenberg film. Hence the advantage of getting an undistorted lattice is off-set by a loss of information. For a crystal with small lattice constants it is therefore recommended that X-radiation of short wave-length (e. g. MoKα) be used for the de Jong-Bouman technique.

The question can arise: which method should be used for an actual problem? A general answer cannot be given. With modern techniques, the film methods are used only for space group determination and for examination of crystal quality. Therefore the information obtained from precession or de Jong-Bouman exposures is sufficient in most cases, even if CuK_α radiation is used. On the other hand, for an experienced crystallographer the distorted representation of a Weissenberg exposure causes no great problem, and this technique might be preferred since a Weissenberg instrument is less troublesome to operate. Because the mechanical parts of a Weissenberg camera are less complicated than the other two instruments, the problems of misalignment due to mechanical breakdown or mechanical wear are less frequent.

The most effective film technique, however, is to combine two of these methods. The Weissenberg and de Jong-Bouman technique need an axis orientation normal to the X-ray beam direction. This automatically implies the orientation of both the other axes in a plane containing the primary beam for all orthogonal crystal systems, also for a monoclinic crystal if the symmetry axis was chosen as a rotation axis. Therefore only a further azimuthal setting is necessary to orient the crystal for a precession photograph. Hence only one crystal

setting is necessary to obtain information about three mutually orthogonal reciprocal lattice planes, if a Weissenberg or de Jong-Bouman technique is combined with the precession technique. The resulting information is sufficient in most cases for a complete knowledge of all constants of the reciprocal lattice.

A camera designed to take both de Jong-Bouman and precession photographs has been developed by Wölfel (16). This instrument, named the 'Reciprocal Lattice Explorer' is a most convenient apparatus for combining different film techniques using only one crystal.

Methods

Up to the middle sixties, diffraction intensities were generally measured by film techniques, but since then nearly all single crystal investigations have been carried out using diffractometers. These instruments run automatically and measure intensities more precisely than film methods (with the exception of proteins with very large unit cell dimensions).

If film cameras are available it is recommended that these are used in the preliminary study of crystals, because the geometrical properties of the reciprocal lattice are more readily determined visually from photographs. Abnormal crystal properties, such as disorder, twinning, or crystal splitting, which influence the diffraction intensities are recognised far more easily on film than when using only a diffractometer.

While choosing a single crystal for film techniques one must consider three properties

1. The crystal must be a real single crystal; no more than one individual should be selected. A polarizing microscope should be used to observe whether the crystal is twinned or not.

2. The crystal should be as large as possible within the limitation given by the primary beam so that it is completely bathed in X-rays. A large crystal usually shortens the time of exposure unless there is a high absorption coefficient.

3. For crystal setting, it is useful to select a crystal with well-formed edges and faces, if possible. Needle shaped crystals are the most favourable for fast setting, but not so suitable for diffractometry because it is difficult to enclose all the crystal within the X-ray beam. Often it is found that the longest elongation of the crystal coincides with the shortest lattice dimension.

After the selection of a crystal, the proper techniques should be used to produce diffraction photographs of the reciprocal lattice. From these accurate cell dimensions can be worked out.

The next step is to survey the reciprocal lattice to see whether there are any systematic absences. Absences due to lattice centring occur throughout the whole of reciprocal space. A general survey to see whether there are any rules governing the permitted values of h, k, and ℓ will enable the lattice type to be determined. Absences that affect only certain layers or rows of points in the reciprocal lattice may also denote the presence of glide planes and screw axes.

For example, the presence of the a-glide perpendicular to c means that for any atom x, y, z there must be an identical atom $x + \frac{1}{2}, y, -z$. The structure factor summation must thus cover $N/2$ such pairs of atoms. The contribution from the rth pair is

$$f_r\{\cos 2\pi(hx_r + ky_r + \ell Z_r) + \cos 2\pi(h(x_r + \tfrac{1}{2}) + ky_r - \ell Z_r)\}$$

for hk0 reflections, $\ell = 0$ and the expression simplifies to -

$$f_r\{\cos 2\pi(hx_r + ky_r) + \cos 2\pi(hx_r + \frac{h}{2} + ky_r)\}$$

$$= f_r\{\cos 2\pi(hx_r + ky_r) + \cos(2\pi(hx_r + ky_r) + h\pi)\}$$

The two cosine terms are equal in magnitude but differ in phase by π, they therefore cancel when h is odd and reinforce when h is even, resulting in the structure factor F_{hk0} being zero when h is odd. so the hk0 reflections occur only when h is even.

Similar arguments can be applied to other translational symmetry elements, eg. screw axis. A 2_1 axis parallel to 'a' means that h00 reflections will occur only when h is even.

If, therefore, systematic absences are found that are additional to those due to any lattice centring, it can be safely concluded that translational symmetry elements are present. So the study of systematic absences should therefore begin with the most general reflections and work down to the less general.

Space group determination is occasionally complicated by phenomenon known as double reflection. If the beam reflected from a set of planes $(h_1k_1\ell_1)$ strikes another set of planes $(h_2k_2\ell_2)$ at the appropriate angle, it may be reflected again. This doubly reflected

beam will appear to arise from $(h^1k^1\ell^1)$ planes of the crystal where

$$h^1 = h_1 + h_2$$
$$k^1 = k_1 + k_2$$
$$\ell^1 = \ell_1 + \ell_2$$

The doubly reflected beam will often be rather weak but sometimes if $h_1k_1\ell_1$ and $h_2k_2\ell_2$ are both strong reflections and the true $h^1k^1\ell^1$ reflection is absent, a spurious reflection may result.

On film, double reflections may be distinguished by their appearance, because of the way they are formed. They are much sharper than the true reflections. They should disappear if the X-ray wavelength is changed or the crystal is remounted in a different orientation.

Density and Unit Cell Contents

The relationship between the volume of the unit cell V, measured in Å^3, and the density D, measured in g cm^{-3} is

$$D = \frac{1.66Z \times F}{V}$$

where F is the formula weight of the substance concerned and Z is the number of such formula units within the cell. Densities can be measured in various ways. The most common one is flotation in a liquid in which the crystal is not soluble. This uses a mixture of liquids whose proportions can be varied until the crystals neither sink nor float. The density of the liquid mixture is then determined by conventional means (Westphal balance).

Whichever method is used, errors tend to make the measured density too low. All methods may be affected by crystal imperfections.

Experience shows that the calculated density is often slightly higher than measured density.

Historically the first structure determination was done by the trial and error method. Although now seldom used in its straightforward form, many of the concepts are still an important part of more sophisticated methods. In this method an attempt is made to build a structure within the framework of the known unit cell and space group that satisfies the cell contents, explains the relative magnitude of a few key intensities and also makes the crystal chemically sensible. Trial structures deduced in this way are then checked by comparing the calculated structure factors or intensities with the observed values. Any parameters which are not fixed by space group restrictions are then adjusted to give the best possible agreement.

The calculation of electron densities from the Fourier summation of observed structure amplitudes was first used in 1929 (2) as a systematic way of improving the values of those atomic co-ordinates not fixed by the space group considerations. When a solution to a crystal structure is believed to be approximately correct, it must be refined to give the best possible fit between the observed and calculated data. Here the electron density map is used for adjusting co-ordinates. This is a relatively insensitive method and progress may be rather slow. One way round this is to use as Fourier coefficient $(F_o - F_c)$ instead of F_o . This produces a difference map, which consists essentially of the true electron density, with the electron density due to the assumed structure subtracted. If the assumed structure is correct the difference map should be featureless. Theoretically it should be zero, but in practice because of the random

errors in the observed data, this is only approximately true even for a fully refined structure.

In 1941 the least-squares method of refinement was developed (6). In this method the best values of the refined parameters are determined by choosing those that minimise $\Sigma_{hkl}(F_o - F_c)^2$. It is a very tedious procedure to apply by hand but with the use of high speed computers it has become a standard method of completing a structure determination.

Direct methods

The name Direct methods is derived from the fact that phases of the structures factors are derived from the magnitude of the F's using statistical theory. Extensive numerical calculations are necessary to apply this technique and the development of direct methods was only possible because of the rapid progress in computer technology made in the 1960's.

A large number of investigations on that subject have been initiated, of which one of the earlier important results was the Sayre Equation developed in 1952 (12) . It is one of the basic formulae of 'Direct Methods' and the equation is

$$E(h) = T\sum_{h^1} E(h^1)E(h - h^1)$$

From Sayre's equation two formulae are derived which are applied in actual phase determination.

In the centrosymmetric case, the E values have a sign of + or - . Sayre's equation can then be interpreted as follows. For the reflection h with $|E(h)|$ being sufficiently large it is likely that the sum of the

right side of the above equation will contain more terms $E(h^1)E(h - h^1)$ having the same sign as $E(h)$ itself, than terms of opposite sign. This is specially true for those terms of which $|E(h^1)|$ and $|E(h - h^1)|$ are large. So there exists more than 50% probability that for large E values -

$$S(h) = S(h^1)S(h - h^1)$$

where $S(h)$ denotes the sign of $E(h)$.

This equation remains valid if on the left side h is replaced by $-h$ (Since $S(h) = S(-h)$. Setting $-h = h_1$, $h^1 = h_2$ and $h - h^1 = h_3$, we get

$$S(h_1) = S(h_2)S(h_3)$$

or $$S(h_1)S(h_2)S(h_3) = 1$$

if the three reflections h_1, h_2, h_3 satisfy the equation

$$h_1 + h_2 + h_3 = 0$$

Reflection triplets for which the above equation holds are said to be related by a Σ_2 relation. These Σ_2 relations play an important role with all applications of direct methods.

The practical application of direct methods is possible if

1. The structure is not too large, say <100 atoms per molecule.
2. A starting set of known phases can be obtained.
3. This set can be used in a sufficient number of $\Sigma 2$ relations between reflections of large E-values for the determination of additional phases. Phase determination by direct methods is not a guaranteed success. However now powerful programs, such as Multan, have been developed and distributed

world wide. Experience in the last few years has shown that in spite of the fact that all formulae are approximations, direct methods are by far the most powerful general method of phase determination presently available. With a reasonably accurate intensity data set, a centrosymmetrical structure can be solved nearly without difficulty, if the number of atoms in the asymmetric unit is not larger than about 100.

Summaries

Ideal method for structure determination

a) Initial investigation using photographic method.

b) Crystal on a diffractometer.

c) Use of computer.

d) Direct method - use of Multan program

Difficulties - Not all crystals are reasonably ideal.

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Chapter 2.

N-Nitrosodiphenylamine ("Vulcatard")

Introduction

In the rubber manufacturing industry, certain additives are used during production to achieve faster processing. As a result the temperature rises quite rapidly during the mixing and often reaches 10 degrees below the vulcanizing temperature. Under such conditions marked and uncontrolled partial vulcanization of the rubber mix can take place depending on the partial accelerators in use. This phenomenon is known as pre-vulcanization or scorching and it gives finished rubber products with poor physical properties, which are unsatisfactory, and in case of tyres can result in blow outs. To avoid this 1-2% of retarders or anti-scorch agents are added to the rubber mix. These substances are certain organic acids or nitroso compounds which have been found to prevent to a large extent this undesirable pre-vulcanization reaction. N-Nitrosodiphenylamine is one of them. Its chemical formula is $C_{12}H_{10}N_2O$ and is marketed by Imperial Chemical Industries under the commercial name Vulcatard A. The molecule has two phenyl groups attached to the nitrosamine group.

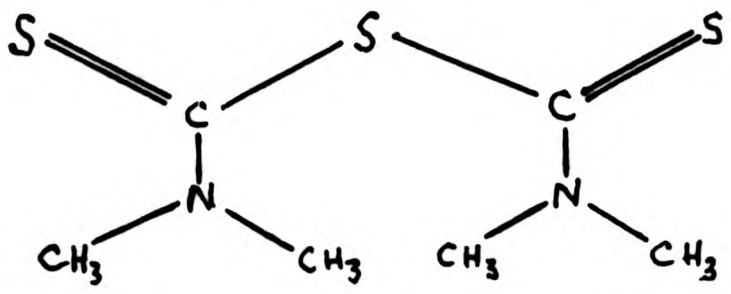
The compound was prepared in the following way:

> 40 g. of diphenylamine was added to 200 ml. alcohol and 30 ml. of hydrochloric acid was added to it. The solution was cooled, and to it 35 ml. of sodium nitrite solution (2:3) was added. The precipitate was filtered, and the residue was recrystallized from xylene at room temperature.

The Department was interested in determining the structures of some rubber chemicals. The structures which have already been done are:

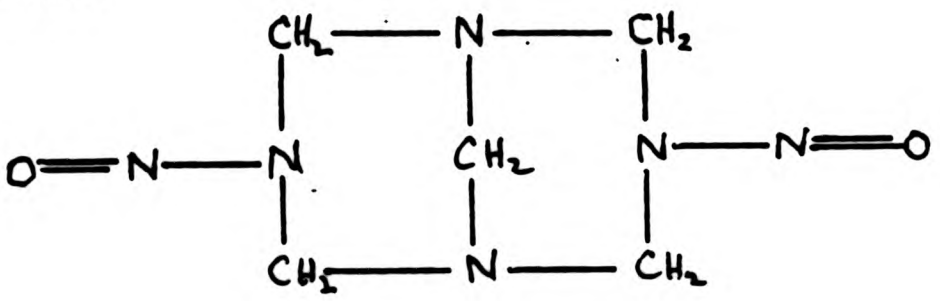
1. Vulcafor MS. This is used as a vulcanization accelerator.

Its chemical name is Tetramethyl thiuram mono-sulphide.



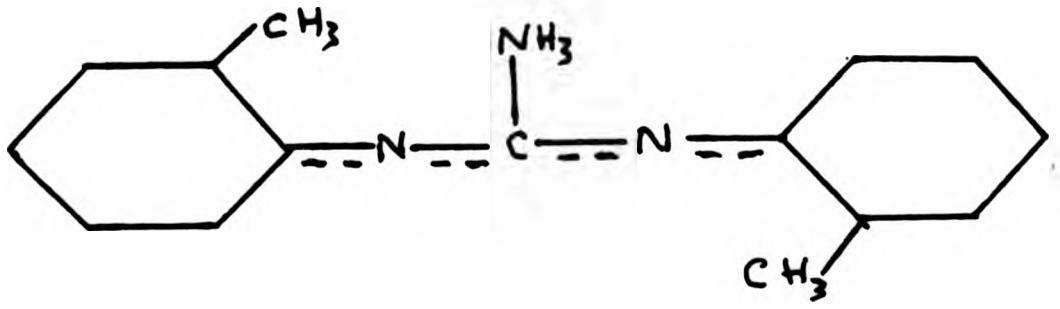
The structure was solved by Tasker in 1976 (7).

2. Vulcacel BN. Its chemical name is dinitrosopenta-methylene tetramine.



This is used as a blowing agent to produce cellular or sponge rubber. The structure was solved by Craft in 1975 (2).

3. Vulcafor DOTG. This is also an accelerator used in vulcanization. The chemical composition is di-ortho-tolyl guanidine.



The structure was solved by Gash in 1982 (3).

Vulcatard A was the last of this present series. Until now very few structures of nitroso compounds have been worked out. So we aimed to find the structure of the molecule and get more information about N-O bonding.

Experimental Measurements of N-Nitrosodiphenylamine

The analysis of a crystal structure may be summarized as follows:-

1. Preparation of crystals and measurement of the unit cell dimensions and the intensities of a large proportion of the diffracted beams from the crystal.

   These intensities depend on the number and type of atoms present and their relative positions in the unit cell.

2. The deduction by some method of a suggested atomic arrangement (trial structure). The intensities of the diffracted X-ray beams corresponding to this arrangement can then be calculated and compared with the observed values.

3. Modification of this suggested arrangement of scattering matter until the agreement between calculated and observed intensities is within the limits of error of observations.

Preparation of Crystals

Suitable crystals of N-Nitrosodiphenylamine were prepared in the following way:-

The compound was dissolved in absolute alcohol and warmed at $60^{o}C$ to make a saturated solution. Then the solution was filtered and transferred into a crystallising dish and allowed to evaporate very slowly. After 12 hours, brown platy crystals were formed and they were quite suitable in size for structure analysis by X-ray diffraction techniques.

Selection of Crystals

First an optical examination was made to select a small crystal of suitable dimensions and uniform in shape.

The crystal was examined very carefully under crossed-polars to make sure it was free from twinning. One extinction direction was along the long diagonal of the crystal.

The chosen size of the crystal was approximately 1 mm in length and 0.5 mm in cross-section, so that it produced enough intensity data and at the same time the effect of absorption became negligible.

The Space-Group and Unit-Cell Dimensions

The chosen crystal was mounted on a glass-fibre parallel to a direction of extinction, because in general a crystal extinguishes along one of the crystallographic axes of symmetry.

First the crystal was aligned by optical methods using the method of reflection. The correction on each arc was made from the position of the images in a telescope when rotated through 180^{o} . But the final alignment of the crystal was made using the method of double oscillations. When the crystal was perfectly aligned, a full 360^{o} rotation photograph was taken which produced sharp layer lines. But oscillation photographs showed no symmetry along that axis. The crystal was then transferred to a Weissenberg camera and a zero layer photograph was taken. A mirror symmetry was found along the axis 8.8Å and another axis was found at 90^{o} apart. Then the crystal was mounted along the 8.8Å axis which was the

short diagonal perpendicular to the length of the crystal. After the crystal was perfectly aligned a sharp rotation photograph was taken. An oscillation photograph showed a mirror symmetry across the zero layer. Similarly the crystal was transferred to the Weissenberg Camera and a zero and first layer photograph were taken. Then the crystal was mounted and aligned along the 3rd axis and a full rotation and a zero layer Weissenberg photograph were taken.

The space group was determined from the following systematic absences.

Reflection	Condition limiting possible reflection
$hk\ell$	$h + k = 2n$
$ho\ell$	$\ell = 2n\,(h = 2n)$

It shows that the crystal was monoclinic with lattice type C. centred with a glide plane along C perpendicular to b.

So the space group was C2/c or Cc . But later the N(z) test proved the crystal was centrosymmetric; C2/c .

The cell-parameters were measured accurately from the rotation photographs along the three crystallographic axes. The distance (x) of the layer lines from the equatorial layer line was measured accurately using a travelling microscope on the X-ray film.

The formula used in calculating the cell parameters from rotation photographs

$$\tan \phi = \frac{x}{R}$$

$$\phi = \tan^{-1} \frac{x}{R}$$

$$a = \frac{n\lambda}{\sin \phi}$$

where x is the distance of the layer line spacing from the equatorial line, and R is the radius of the camera. The camera radii for cylindrical and Weissenberg Cameras are 30 mm and 28.65 mm respectively. The wavelength (λ) for CuK_α radiation is 1.5418Å .

The information about the unit cell angles were taken from the three zero layer Weissenberg photographs. The photograph along b (unique axis) gave the a^* and c^* reciprocal axes. The angle between a^* and c^* which is β^* was measured very accurately. The angle β was calculated from the following expression

$$\beta = 180 - \beta^*$$
$$= 180 - 62.47$$
$$= 117.53^\circ (15)$$

The zero layer photograph along a-axis gave b^* and c^* reciprocal axes, and the angle between them α^* was measured to be 90°, so α was 90°. Similarly the angle between a^* and b^* axes γ^* was measured 90° and therefore $\gamma = 90^\circ$.

The layer line spacings were measured several times and the best values of the cell parameters obtained by a least squares fit to the 2θ values of certain reflections. The final values were a = 16.283(20), b = 8.827(10), c = 16.508(20)Å . Density, measured by flotation in NaI solution, = 1.25 g/cc confirming Z = 8 units per unit cell.

Intensity Measurements

Equi-inclination Weissenberg Photographs were taken for seven layers with a crystal mounted about b axis using nickel-filtered Cu-K_α radiation (λ = 1.5418Å) and the multiple film pack technique. and also the zero layer about c axis for correlation.

Packs of three films were used and exposed for 18 hours and 2 hours. Development and processing conditions were kept constant for all the films. In this way 6 films of recorded data, calibrated approximately as 18 hours, 6 hours, 2 hours, 2 hours, 40 minutes and 13 minutes were obtained. Considering the film factor is 3; this had been checked by a previous student on the particular batch of film (Ilford G).

The equi-inclination angle for each layer was obtained from the expression $\mu = \sin^{-1}(\xi/2)$ and the corresponding screen movement was calculated from the expression $s = r \tan \mu$ where r is the radius of the screen, ξ is the reciprocal spacing of the layer lines.

A scale was prepared for the visual estimation of the intensities, by choosing a well shaped intense spot, a separate one for each layer.

The oscillation range was set between 10° and 20°. Exposures were made using 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 14, 16, 18, 20, 22, 24, 26, 28 and 30 traverses of the camera, moving the film by 4 mm between each exposure. The intensities of the spots were marked as 1, 2, 3, 4 .... etc. Each spot was compared with the intensity scale and its estimated intensity was multiplied by necessary scale factor to bring all intensities to the same scale.

Structure Analysis

The intensity data were corrected by Lorentz(L) and Polarisation(P) factors.

The Lorentz factor arises due to the time difference between different crystal planes spent in reflecting positions. It is the length of

time the reciprocal lattice point remains in contact with the sphere of reflection and it depends upon the distance of the distance of the reciprocal point from the origin and the diffraction geometry.

In case of equi-inclination Weissenberg methods of data collection

$$L = \frac{\text{Sin } \theta}{\text{Sin } 2\theta(\text{Sin}^2\theta - \text{Sin}^2\mu)^{\frac{1}{2}}}$$

where θ is the Bragg angle, and μ is the equi-inclination setting angle.

The polarisation factor arises due to the polarisation of X-rays, when reflected by a crystal plane which results a reduction in the intensity of reflected beam.

It is expressed as $P = \frac{1}{2}(1 + \cos^2 2\theta)$ where θ is the Bragg angle. When $\theta = 0$ or 90° $P = 1$, that is there is no polarisation, but when $\theta = 45^\circ$ $P = \frac{1}{2}$, the reflected beam is completely polarised.

The observed structure amplitudes were computed from the expression

$$F_{rel} = (I/LP)^{\frac{1}{2}}$$

and no absorption correction was made since the absorption coefficient μ was only 0.5 mm^{-1} .

Originally the space-group was determined as A2/a . Attempts were made to determine the phases of structure amplitudes by Direct Methods using the symbolic addition program, N.R.C.4. But the program could not fix the origin. Later it was realised that this program excludes reflections with certain parities which is explained below in the summary of the procedure. Then

the space group was changed to C2/c and the program worked satisfactorily.

In this program statistical methods are used to determine the structure factors of a centrosymmetric structure (Hauptman & Karle (4) which involves Σ_2 relationships.

This procedure involved four basic operations which may be summarised as follows:-

a) Calculation of the overall isotropic temperature factor and scale and it is necessary to generate the structure factor normalisation curve. The temperature and scale factors were 2.99 and 1.32 respectively.

b) Calculation of the normalised structure factors (E) , their . statistics and the point-atom at rest sharpening function from the normalisation curve. It also sorts the reflections with strongest E's (E > 1.3) in descending orders of $|E|$. 197 reflections were listed with values of E's greater than 1.30.

c) Search for triplets of reflections with normalised structure factors greater than a specified minimum, which satisfy the Σ_2 relationship.

d) (i) Selection of the origin-defining reflections, assignment of the symbols and successive application of the Σ_2 relationship in order to determine the signs of the reflections with $|E|$'s greater than the specified optimum value.

At this stage the program selects these reflections excluding those with eee parity and sorted in descending order of assoc-

iated number of E_2E_3 pairs. Then a determinant of indices, taken to modulo 2, is evaluated for the first three reflections in the sorted list. If the result of +1 or -1, the corresponding E values are given positive signs. If the result is other than +1 or -1, the determinant is re-evaluated, with the fourth reflection replacing the third. This operation is .... 2, 3, 4 ; 2, 3, 5 .... $N\phi_{DR-2}$; $N\phi_{DR-1}$; $N\phi_{DR}$ until a non-zero result is obtained.

(ii) Estimation of the signs of normalised structure factors, whose amplitudes are less than the optimum value but greater than the minimum value by direct application of signs determined in stage d(i) to the sets of equivalent pairs (E_K , E_{n-K}) related to each E_h . The signed normalised structure factors are then sorted for input to the initial Fourier (E-map) calculation. using C2/c space group.

All these steps were calculated by five separate programmes namely SAP 1, 2, 3, 4(a) and 4(b) run sequentially. The signed normalised structure factors from SAP 4(b) were used for the Fourier calculation using the N.R.C.8 program.

The electron density map was computed for $\frac{1}{8}$ of the unit cell and the molecule was easily traced from the 15 peaks located in the electron density map.

Table 1 lists the co-ordinates of atoms as read from these peaks. A comparison of these co-ordinates with the final co-ordinate shows that this initial E-map was a very good representation of structure.

TABLE 1 E-MAP COORDINATES

Atom Type	x/a	y/b	z/c
C1	.30	.20	.34
C2	.22	.14	.36
C3	.15	.12	.30
C4	.125	.12	.215
C5	.18	.18	.18
C6	.28	.21	.26
C7	.47	.18	.41
C8	.545	.29	.415
C9	.62	.19	.42
C10	.62	.04	.385
C11	.545	.04	.375
C12	.47	.03	.375
N1	.39	.23	.41
N2	.415	.34	.475
O1	.34	.41	.48

Refinement

The structure factors may be expressed in the following way:

$$F(hk\ell) = \Sigma_r f_r\ (hk\ell)\ \exp\ 2\pi i(hx_r + ky_r + \ell z_r)$$

$$= A(hk\ell) + iB(hk\ell)$$

$$= |F|\ \exp\ i\alpha(hk\ell)$$

where Σ_r = sum over all atoms in the unit cell

f_r = atomic scattering factor corrected for atomic vibration

$(x,y,z)_r$ = Fractional atomic co-ordinates

α = Phase angle = $\tan^{-1}(\frac{B}{A})$

$|F|$ = Structure amplitude = $(A^2 + B^2)^{\frac{1}{2}}$

= $A \cos \alpha + B \sin \alpha$

The structure refinements were carried out using block diagonal least squares method where the atomic parameters were modified to give the best fit between a set of observed structure factors and the calculated structure factors. The parameters adjusted includes atomic co-ordinates, atomic vibration parameters, and the scale factor which was used to bring the observed structure factors to the absolute scale.

After several cycles of refinements using isotropic thermal parameters ($\exp - 8\pi^2 U \times \sin^2\theta/\lambda^2$) for non-hydrogen atoms, the positions of hydrogens bonded to carbon were calculated and then the anisotropic temperature factors

(exp $-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ell^2c^{*2}U_{33} + 2hka^*b^*U_{12} + 2h\ell a^*c^*U_{13} + 2k\ell b^*c^*U_{23}$) were used for the refinement of the non-hydrogen atomic parameters.

The procedure was repeated until no further improvement takes place as shown by the residual index

$$R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$$

The N.R.C - 10 program utilises information either from the data reduction programme N.R.C - 2 or from the previous run of structure factor least-square program. The atomic parameters could either be read from the previous program or supplied separately on the computer terminal.

The final value of R index obtained was 0.067 for 807 observed reflections. The final positional co-ordinates, thermal parameters, and their standard deviations are given in Table 2.

Results and Discussion

The interatomic distances and bond angles were calculated from the final parameters using a computer program.

This helped to compare the calculated bond lengths and angles with those values accepted as normal by previous researchers. (1) (5) (6).

The program produced the following information.

It scanned the given atoms for intramolecular distances and derived the equivalent positions and then scanned for the intermolecular distances. After that it prepared a summary of the co-ordination

around each of the given atoms and calculated the angle between the bonds and their standard deviation.

The inter-atomic distances and the bond angles are given in Figure 1 and in Tables 3 and 4. The average benzenoid C-C bond length is 1.398Å . The C-H length is 1.1Å. The N_1 - N_2 and N_2 - O_1 bond lengths are 1.344Å and 1.206Å respectively. There are no intra-molecular hydrogen bonds.

The least-square planes through the selected atoms are given in Table 5. The dihedral angle between two benzene rings is 101.198°. The molecular packing is given in Figure 2. All intermolecular contacts are larger than the sum of the Vander Waals radii.

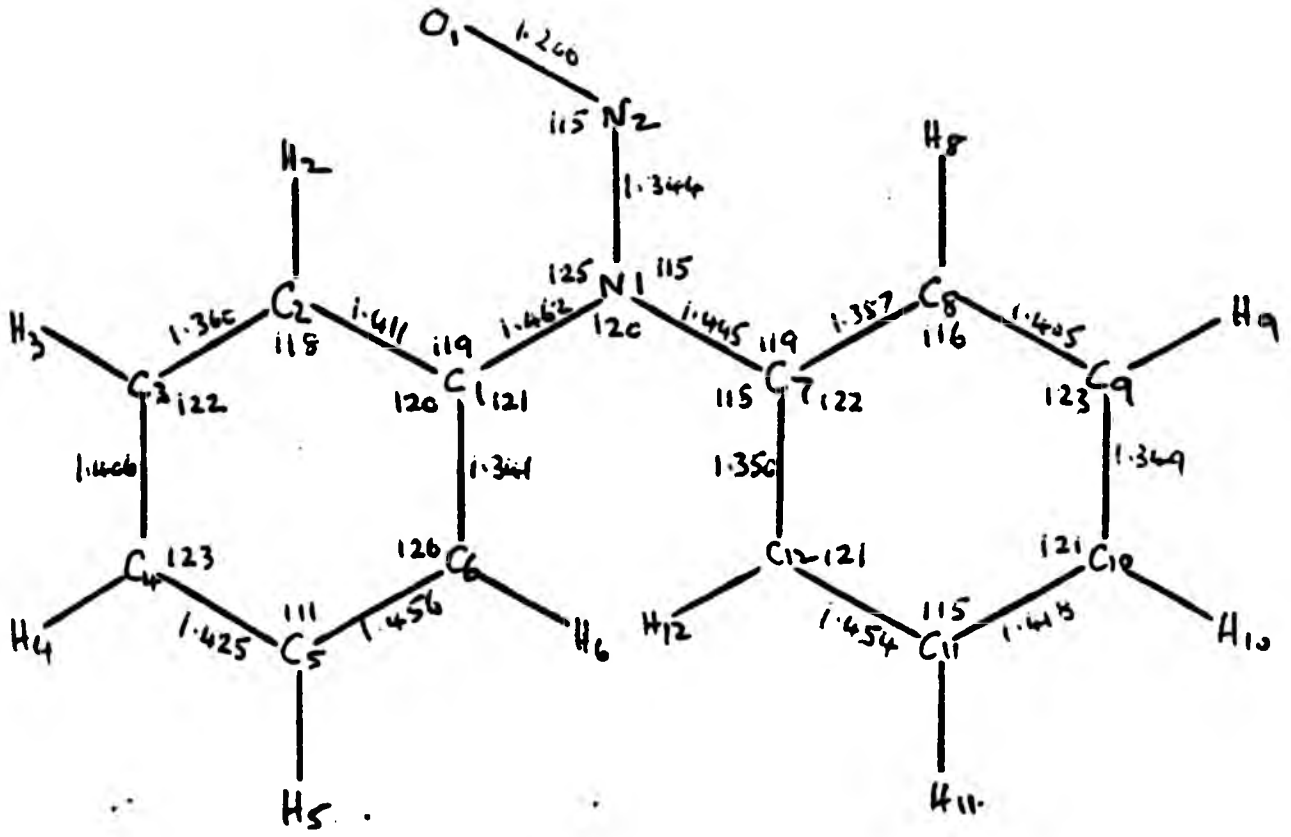


Figure 1.

Chemical formula showing numbering of atoms and bond lengths and inter-bond angles.

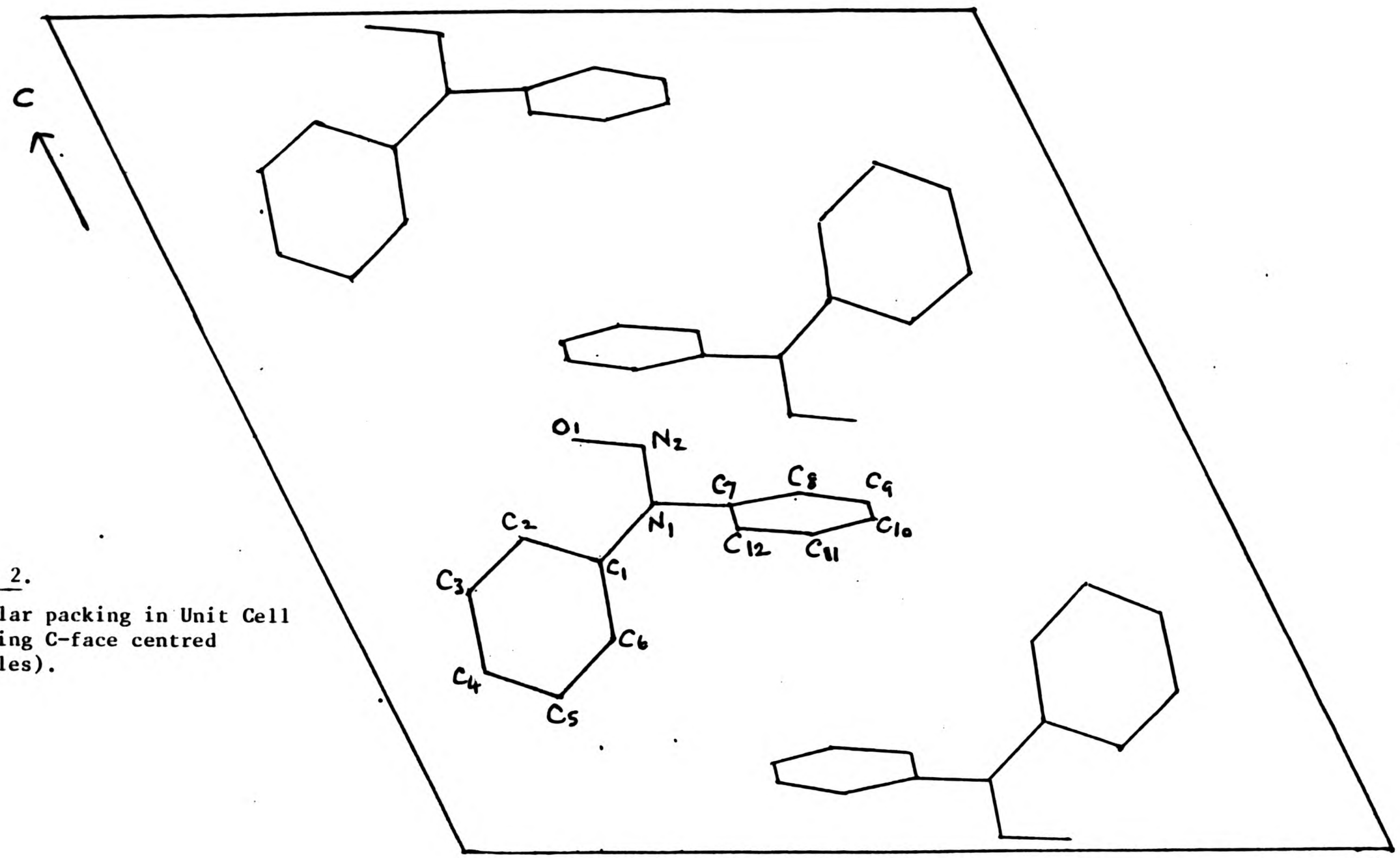


Figure 2.

Molecular packing in Unit Cell (omitting C-face centred molecules).

TABLE 2. ATOMIC PARAMETERS

VULCATARD

ATOM	NO.	ORIGINAL COORDINATES X/A	Y/B	Z/C	E.S.D.S SYG X/A	SYG Y/B	SYG Z/C
C	1	0.29865	0.19773	0.34240	0.00027	0.00041	0.00025
C	2	0.23660	0.13766	0.37184	0.00034	0.00038	0.00032
C	3	0.14928	0.14171	0.30710	0.00026	0.00041	0.00036
C	4	0.12333	0.11206	0.21333	0.00033	0.00051	0.00033
C	5	0.18141	0.17539	0.17880	0.00037	0.00054	0.00035
C	6	0.27245	0.21145	0.25289	0.00028	0.00049	0.00026
C	7	0.47202	0.17688	0.40703	0.00027	0.00052	0.00027
C	8	0.54328	0.27248	0.41593	0.00034	0.00054	0.00034
C	9	0.62456	0.20285	0.41529	0.00028	0.00057	0.00031
C	10	0.61993	0.05727	0.39021	0.00033	0.00057	0.00039
C	11	0.54487	-0.04050	0.37422	0.00036	0.00055	0.00035
C	12	0.46647	0.03290	0.37742	0.00031	0.00065	0.00032
N	1	0.39199	0.24008	0.41083	0.00023	0.00028	0.00022
N	2	0.41099	0.34418	0.47652	0.00031	0.00040	0.00026
O	1	0.34412	0.39730	0.47932	0.00025	0.00030	0.00024
H	2	0.25779	0.11926	0.44347	0.00000	0.00000	0.00000
H	3	0.09048	0.06939	0.32831	0.00000	0.00000	0.00000
H	4	0.05636	0.06852	0.16529	0.00000	0.00000	0.00000
H	5	0.16068	0.19411	0.10729	0.00000	0.00000	0.00000
H	6	0.32362	0.25310	0.23401	0.00000	0.00000	0.00000
H	8	0.53959	0.39359	0.42296	0.00000	0.00000	0.00000
H	9	0.68299	0.26854	0.43570	0.00000	0.00000	0.00000
H	10	0.67782	0.01427	0.38214	0.00000	0.00000	0.00000
H	11	0.54584	-0.16083	0.36073	0.00000	0.00000	0.00000
H	12	0.40252	-0.02904	0.35567	0.00000	0.00000	0.00000

TABLE 2 (continued)

Anisotropic Temperature Factors and their e.s.d's

	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
C(1)	0.00526 24	0.00118 60	0.00443 20	0.00459 52	0.00483 37	0.00094 54
C(2)	0.00800 31	0.00193 66	0.00691 26	-0.00477 62	0.01190 49	-0.00191 61
C(3)	0.00248 19	0.00099 61	0.00964 33	0.00095 73	0.00472 40	0.00167 51
C(4)	0.00549 31	0.01774 106	0.00479 25	0.00333 73	0.00394 44	0.00961 77
C(5)	0.00456 26	0.01151 87	0.00683 27	-0.01025 74	0.00334 45	-0.00150 66
C(6)	0.00574 27	0.00028 84	0.00522 23	0.00049 53	0.00472 41	-0.00263 57
C(7)	0.00404 24	0.01153 84	0.00503 25	-0.00952 62	0.00483 41	-0.00397 60
C(8)	0.00726 32	0.00009 90	0.00961 34	-0.00394 63	0.00742 55	-0.00156 64
C(9)	0.00390 25	0.01207 92	0.00802 31	-0.00229 71	0.00771 45	-0.00537 62
C(10)	0.00578 31	0.01042 89	0.00906 36	-0.00092 83	0.00974 55	0.00463 76
C(11)	0.00923 35	0.00918 84	0.00741 33	0.00361 77	0.01061 55	0.00609 77
C(12)	0.00422 28	0.02548 104	0.00464 25	-0.00123 80	0.00469 41	0.00139 74
N(1)	0.00509 18	0.00348 45	0.00413 15	-0.00690 46	0.00579 27	-0.00656 46
N(2)	0.00790 29	0.01441 69	0.00434 19	0.00583 57	0.00632 37	-0.00221 63
O(1)	0.00919 23	0.01336 57	0.00753 22	-0.00169 54	0.01059 38	0.00410 52

The temperature factor is given by the equation

$$T = \exp\left[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{23}kl + 2\beta_{31}lh + 2\beta_{12}hk)\right]$$

TABLE 3. BOND LENGTHS

VULCATARD

INTRAMOLECULAR DISTANCES

ATOM	N1	ATOM	N2	DISTANCE	E.S.D.	DIRECTION COSINES L	M	N
C	1	C	2	1.411	0.0072	-0.87500	-0.37568	0.30534
C	1	C	6	1.341	0.0054	0.19123	0.09033	-0.97738
C	1	N	1	1.462	0.0056	0.68224	0.25562	0.68498
C	2	C	3	1.360	0.0074	-0.68238	-0.22039	-0.69699
C	2	H	2	1.080	0.0047	-0.18658	-0.15038	0.97086
C	3	C	4	1.406	0.0072	0.20842	0.05244	-0.97663
C	3	H	3	1.080	0.0050	-0.91578	-0.28051	0.28750
C	4	C	5	1.425	0.0079	0.84865	0.39234	-0.35476
C	4	H	4	1.080	0.0053	-0.67032	-0.35587	-0.65118
C	5	C	6	1.456	0.0069	0.63005	0.21868	0.74513
C	5	H	5	1.080	0.0051	0.19267	0.15300	-0.96926
C	6	H	6	1.080	0.0048	0.90480	0.34030	-0.25589
C	7	C	8	1.387	0.0074	0.78780	0.60862	0.09397
C	7	C	12	1.350	0.0072	0.10044	-0.94160	-0.32117
C	7	N	1	1.445	0.0062	-0.92170	0.38599	0.03849
C	8	C	9	1.405	0.0078	0.89919	-0.43750	-0.00667
C	8	H	8	1.080	0.0048	-0.10530	0.98986	0.09529
C	9	C	10	1.349	0.0071	0.13429	-0.95282	-0.27222
C	9	H	9	1.080	0.0051	0.79702	0.53607	0.27664
C	10	C	11	1.418	0.0082	-0.77601	-0.60072	-0.16510
C	10	H	10	1.080	0.0060	0.92980	-0.35144	-0.10938
C	11	C	12	1.454	0.0083	-0.89468	0.44555	0.03221
C	11	H	11	1.080	0.0049	0.10994	-0.97697	-0.18286
C	12	H	12	1.080	0.0056	-0.81046	-0.50622	-0.29479
N	1	N	2	1.344	0.0047	-0.14276	0.68378	0.71558
N	2	O	1	1.206	0.0067	-0.92060	0.38084	0.03399

TABLE 4. INTER-BOND ANGLES

VULCATARD

	N1		N2		N3		ANGLE	E.S.D.
ANGLES INVOLVING ATOM C 1								
	C	2	C	1	C	6	119.98	0.399
	C	2	C	1	N	1	118.94	0.365
	C	6	C	1	N	1	121.06	0.369
ANGLES INVOLVING ATOM C 2								
	C	1	C	2	C	3	117.84	0.428
	C	1	C	2	H	2	121.08	0.427
	C	3	C	2	H	2	121.08	0.447
ANGLES INVOLVING ATOM C 3								
	C	2	C	3	C	4	121.80	0.448
	C	2	C	3	H	3	119.10	0.437
	C	4	C	3	H	3	119.10	0.435
ANGLES INVOLVING ATOM C 4								
	C	3	C	4	C	5	122.95	0.463
	C	3	C	4	H	4	118.53	0.457
	C	5	C	4	H	4	118.52	0.463
ANGLES INVOLVING ATOM C 5								
	C	4	C	5	C	6	118.86	0.438
	C	4	C	5	H	5	124.57	0.482
	C	6	C	5	H	5	124.57	0.466
ANGLES INVOLVING ATOM C 6								
	C	1	C	6	C	5	126.02	0.420
	C	1	C	6	H	6	116.99	0.399
	C	5	C	6	H	6	116.99	0.405
ANGLES INVOLVING ATOM C 7								
	C	8	C	7	C	12	121.61	0.456
	C	8	C	7	N	1	119.19	0.405
	C	12	C	7	N	1	117.93	0.411
ANGLES INVOLVING ATOM C 8								
	C	7	C	8	C	9	116.20	0.460
	C	7	C	8	H	8	121.90	0.477

TABLE 4 (continued)

VULCATARD

		N1		N2		N3		ANGLE	E.S.D.
		C	9	C	H	H	8	121.90	0.481
ANGLES INVOLVING ATOM	C 9								
		C	8	C	9	C	10	122.64	0.483
		C	8	C	9	H	9	110.60	0.453
		C	10	C	9	H	9	110.67	0.470
ANGLES INVOLVING ATOM	C 10								
		C	9	C	10	C	11	121.38	0.510
		C	9	C	10	H	10	119.31	0.511
		C	11	C	10	H	10	119.31	0.500
ANGLES INVOLVING ATOM	C 11								
		C	10	C	11	C	12	114.69	0.484
		C	10	C	11	H	11	122.66	0.500
		C	12	C	11	H	11	122.65	0.496
ANGLES INVOLVING ATOM	C 12								
		C	7	C	12	C	11	121.32	0.480
		C	7	C	12	H	12	119.34	0.482
		C	11	C	12	H	12	119.34	0.481
ANGLES INVOLVING ATOM	N 1								
		C	1	N	1	C	7	120.25	0.330
		C	1	N	1	N	2	124.58	0.343
		C	7	N	1	N	2	115.03	0.345
ANGLES INVOLVING ATOM	N 2								
		N	1	N	2	O	1	114.94	0.398
ANGLES INVOLVING ATOM	O 1	NONE							
ANGLES INVOLVING ATOM	H 2	NONE							
ANGLES INVOLVING ATOM	H 3	NONE							
ANGLES INVOLVING ATOM	H 4	NONE							
ANGLES INVOLVING ATOM	H 5	NONE							
ANGLES INVOLVING ATOM	H 6	NONE							
ANGLES INVOLVING ATOM	H 8	NONE							
ANGLES INVOLVING ATOM	H 9	NONE							
ANGLES INVOLVING ATOM	H 10	NONE							
ANGLES INVOLVING ATOM	H 11	NONE							
ANGLES INVOLVING ATOM	H 12	NONE							

TABLE 5. EQUATIONS OF MEAN PLANES

VULCAT&RD

PLANE 1 IS (0.3740)X + (-0.9271)Y + (-0.0199)Z -(-5.0119) = 0

CHI SQUARED = 173.0869

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	1	2.2304	1.7454	5.0123	-0.003	0.0038
	C	2	1.0193	1.2151	5.4433	0.017	0.0038
	C	3	0.7975	0.7155	6.4995	-0.033	0.0038
	C	4	0.3909	0.9091	3.1228	0.035	0.0047
	C	5	1.5037	1.5401	2.6174	-0.020	0.0049
	C	6	2.5046	1.8665	3.7020	0.005	0.0044
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.022007

OTHER ATOMS

	N	1	3.2491	2.1192	4.0140	0.003	0.0028

PLANE 2 IS (0.0400)X + (0.2463)Y + (-0.9684)Z -(-5.1359) = 0

CHI SQUARED = 584.7876

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	7	4.9802	1.9614	5.9594	-0.066	0.0040
	C	8	5.5726	2.4031	6.0817	0.058	0.0050
	C	9	6.9358	1.7906	6.0714	-0.033	0.0046
	C	10	7.1170	0.5035	5.7122	0.013	0.0057
	C	11	6.0167	-0.3573	5.4741	-0.016	0.0051
	C	12	4.7197	0.2904	5.5258	0.049	0.0047
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.043776

OTHER ATOMS

	N	1	3.2491	2.1192	4.0140	-0.036	0.0031

PLANE 3 IS (0.3044)X + (0.7335)Y + (-0.6077)Z -(-1.0097) = 0

CHI SQUARED = 73.3622

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	1	2.2304	1.7454	5.0123	0.007	0.0037
	C	7	4.9802	1.9614	5.9594	0.007	0.0044
	N	1	3.2491	2.1172	4.0140	-0.023	0.0029
	N	2	3.0363	3.0391	4.7736	0.008	0.0039
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.013309

OTHER ATOMS

	O	1	1.9460	3.3070	7.0166	-0.010	0.0032

PLANE 1	PLANE 2	DIHEDRAL ANGLE (DEGREES)
1	2	101.193
1	3	123.639
2	3	38.615

REPRODUCED
FROM THE
BEST
AVAILABLE
COPY

TABLE 5. EQUATIONS OF MEAN PLANES

VULCAFARO

PLANE 1 IS (0.3743)X + (-0.9271)Y + (-0.0199)Z -(-9.9719) = 0

CHI SQUARED = 173.0865

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	1	2.2504	1.7454	5.0123	-0.003	0.0038
	C	2	1.0193	1.2151	5.4433	0.017	0.0038
	C	3	0.7975	0.7135	6.4995	-0.033	0.0038
	C	4	0.3909	0.9891	3.1228	0.035	0.0047
	C	5	1.5837	1.5481	2.6174	-0.020	0.0049
	C	6	2.9046	1.8665	3.7020	0.009	0.0044
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.022807

OTHER ATOMS

	N	1	3.2491	2.1192	4.0140	0.003	0.0028

PLANE 2 IS (0.0400)X + (0.2463)Y + (-0.9684)Z -(-5.1355) = 0

CHI SQUARED = 584.7876

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	7	4.9802	1.9614	5.9594	-0.066	0.0040
	C	8	5.5726	2.4031	6.0817	0.050	0.0050
	C	9	6.9358	1.7906	6.0734	-0.033	0.0046
	C	10	7.1178	0.5035	5.7122	0.013	0.0057
	C	11	6.0167	-0.3575	5.4741	-0.016	0.0051
	C	12	4.7157	0.2904	5.5258	0.049	0.0047
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.043776

OTHER ATOMS

	N	1	3.2491	2.1192	4.0140	-0.036	0.0031

PLANE 3 IS (0.3044)X + (0.7335)Y + (-0.6077)Z -(-1.0097) = 0

CHI SQUARED = 73.3622

ATOMS IN PLANE	ATOM NO.		X	Y	Z	D	ESD(D)
	C	1	2.2504	1.7454	5.0123	0.007	0.0037
	C	7	4.9802	1.9614	5.9594	0.007	0.0044
	N	1	3.2491	2.1192	4.0140	-0.023	0.0029
	N	2	3.0563	3.0381	4.9736	0.008	0.0039
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.013309

OTHER ATOMS

	O	1	1.9460	3.5070	7.0166	-0.010	0.0032

PLANE 1	PLANE 2	DIHEDRAL ANGLE (DEGREES)
1	2	101.193
1	3	123.039
2	3	38.615

TABLE 6. INTERMOLECULAR DISTANCES

VULCATARD A.BANERJEE C.J.BROWN J.P.P.LEWIS

INTERMOLECULAR DISTANCES, WITH THE COORDINATES USED IN THE COMPUTATION

ATOM	N1	X/A	Y/B	Z/C	ATOM	N2	X/A	Y/B	Z/C	DISTANCE	S.D.
C	10	0.61993	0.05727	0.59021	C	2	0.76940	-0.12766	0.62016	3.920	0.0078
O	1	0.34412	0.39730	0.47932	C	2	0.26240	0.34234	0.42016	3.293	0.0066
C	11	0.34487	0.95950	0.37422	C	3	0.84928	0.40971	0.30710	3.968	0.0069
O	1	0.94412	0.39730	0.47932	C	3	0.39072	0.39629	0.69290	3.477	0.0064
C	4	0.27245	0.21145	0.25289	C	3	0.39072	0.40971	0.19290	3.970	0.0060
N	2	0.41099	0.34418	0.47652	C	4	0.62333	0.39794	0.71333	3.859	0.0065
C	4	0.27245	0.21145	0.25289	C	4	0.37667	0.41206	0.28667	3.943	0.0071
C	9	0.54328	0.27240	0.41599	C	4	0.37667	0.41206	0.28667	3.916	0.0073
C	11	0.34487	0.95950	0.37422	C	4	0.37667	0.41206	0.28667	3.817	0.0064
N	1	0.99199	0.24008	0.41003	C	4	0.37667	0.41206	0.28667	3.784	0.0061
N	2	0.41099	0.34418	0.47652	C	4	0.37667	0.41206	0.28667	3.943	0.0062
O	1	0.34412	0.39730	0.47932	C	5	0.60141	0.32461	0.67080	3.883	0.0073
C	9	0.54328	0.27240	0.41599	C	5	0.31859	0.47939	0.32120	3.717	0.0061
O	1	0.34412	0.39730	0.47932	C	5	0.31859	0.47939	0.32120	3.448	0.0059
C	12	0.46647	0.03290	0.37742	C	5	0.31859	-0.32461	0.32120	3.818	0.0078
C	9	0.62094	0.20205	0.41529	C	6	0.72755	0.21145	0.24711	3.901	0.0067
C	10	0.61993	0.05727	0.59021	C	6	0.72755	0.21145	0.24711	3.784	0.0074
C	11	0.34487	0.95950	0.37422	C	7	0.82798	0.02312	0.89297	3.938	0.0069
C	12	0.46647	0.03290	0.37742	C	7	0.82798	-0.17688	0.89297	3.718	0.0066
N	2	0.41099	0.34418	0.47652	C	8	0.49672	0.72752	0.89407	3.739	0.0060
O	1	0.34412	0.39730	0.47932	C	8	0.49672	0.72752	0.89407	3.492	0.0058
O	3	0.34412	0.39730	0.47932	C	9	0.37944	0.79711	0.39471	3.859	0.0057
C	9	0.62094	0.20205	0.41529	C	9	0.37944	0.29711	0.39471	3.922	0.0060
C	12	0.46647	0.03290	0.37742	C	10	0.89987	0.03727	0.10979	3.978	0.0074
O	1	0.34412	0.39730	0.47932	C	10	0.11993	0.53727	0.39021	3.838	0.0071
N	1	0.99199	0.24008	0.41003	C	11	0.49818	0.04050	0.62979	3.880	0.0060
N	2	0.41099	0.34418	0.47652	C	11	0.49818	0.04050	0.62979	3.484	0.0058
O	1	0.34412	0.39730	0.47932	C	11	0.49818	0.04050	0.62979	3.872	0.0058
C	11	0.34487	0.95950	0.37422	C	11	0.49818	0.99950	0.12979	3.883	0.0073
C	12	0.46647	0.03290	0.37742	C	12	0.53333	-0.03290	0.62258	3.718	0.0067
N	1	0.99199	0.24008	0.41003	C	12	0.53333	-0.03290	0.62258	3.884	0.0059
N	2	0.41099	0.34418	0.47652	N	2	0.50901	0.65982	0.02340	3.887	0.0069

TABLE 7. STRUCTURE FACTORS

PAGE 0 VULCATARD

UNIT CELL DIMENSIONS
A = 16.283
B = 8.827
C = 16.508
BETA = 117.53
SPACE GROUP C2/C
R-INDEX 0.0670
SCALE = 10.630
NUMBER OF REFLEXIONS USED IN THE REFINEMENT = 807
TOTAL NUMBER OF REFLEXIONS = 1067

PAGE 1 VULCAIANO

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 0. K= 0			-2	225	220	H= 14. K= 0			7	57	67	-15	0*	43
2	334	356	-4	227	-219	0	226	-210	8	87	-102	-16	0*	38
4	1280	-1260	-6	200	204	2	110	-122	9	208	-210	-17	94	100
6	377	-370	-8	320	-308	4	0*	-22	10	268	-279			
8	443	407	-10	129	-157	-2	0*	-24	11	226	196	H= 7. K= 1		
10	341	339	-12	92	89	-4	717	724	12	72	90	0	219	250
12	99	104	-14	0*	13	-6	226	213	13	90	-95	1	390	-368
14	101	-82	-16	159	-162	-8	226	-196	14	0*	-23	2	0*	5
16	63	-81	-18	0*	65	-10	102	-115	15	82	-70	3	222	240
18	88	-79				-12	228	220	-1	88	-97	4	236	218
			H= 8. K= 0			-14	222	202	-2	659	665	5	64	-54
H= 2. K= 0			0	338	324	-16	65	45	-3	855	849	6	67	66
0	106	90	2	0*	40	-18	78	91	-4	0*	-26	7	70	-49
2	890	864	4	0*	-85				-5	533	523	8	160	-143
4	695	-664	6	157	-130	H= 1. K= 1			-6	498	-472	9	0*	-8
6	981	-948	8	140	61	0	336	-312	-7	45	43	-1	102	199
8	226	-223	-2	71	-47	1	1203	1216	-8	0*	-47	-2	434	-414
10	150	113	-4	110	131	2	171	-158	-9	478	479	-3	554	-554
12	176	-178	-6	1015	-1031	3	1696	1668	-10	183	153	-4	203	208
14	97	-128	-8	173	168	4	945	956	-11	0*	90	-5	160	154
-2	1322	1279	-10	640	608	5	691	672	-12	230	239	-6	239	232
-4	1052	-1024	-12	66	66	6	146	170	-13	0*	6	-7	316	283
-6	91	120	-14	141	-82	7	72	62	-14	0*	24	-8	263	291
-8	471	471	-16	141	-80	8	305	-318	-15	0*	-15	-9	193	202
-10	644	641	-18	0*	10	9	85	-72	-16	0*	-5	-10	0*	12
-12	95	65				10	112	-105				-11	279	-234
-14	322	282	H= 10. K= 0			11	68	-36	H= 5. K= 1			-12	66	-88
-16	0*	22	0	109	-144	12	71	-62	0	216	-214	-13	0*	49
-18	0*	-46	2	0*	85	13	249	262	1	230	251	-14	0*	-39
			4	0*	4	14	0*	-20	2	450	475	-15	0*	24
H= 4. K= 0			6	0*	38	15	66	65	3	838	-797	-16	0*	0
0	879	-860	-2	168	-126	-1	229	193	4	117	94	-17	0*	-19
2	180	-178	-4	82	-116	-2	0*	24	5	177	-191	-18	0*	60
4	631	-645	-6	0*	-20	-3	1695	-1672	6	85	-104			
6	79	-86	-8	573	-553	-4	1788	1759	7	0*	-25	H= 9. K= 1		
8	456	435	-10	91	-110	-5	647	-625	8	96	-118	0	131	-150
10	389	-365	-12	154	164	-6	785	802	9	173	175	1	236	243
12	71	-54	-14	0*	10	-7	840	843	10	101	87	2	0*	71
-2	868	-854	-16	316	-291	-8	397	-356	11	123	-115	3	0*	-37
-4	392	-357	-18	126	-104	-9	177	179	12	0*	45	4	0*	-62
-6	184	-179				-10	192	196	13	89	-59	5	0*	10
-8	189	163	H= 12. K= 0			-11	65	-77	-1	119	147	6	0*	-27
-10	484	-377	0	352	-326	-12	194	181	-2	451	-444	7	0*	-49
-12	66	64	2	228	-251	-13	246	-234	-3	234	224	8	0*	37
-14	0*	-88	4	245	261	-14	72	84	-4	810	-780	-1	358	-365
-16	273	250	6	144	130	-15	221	-193	-5	284	-274	-2	302	-265
-18	0*	29	-2	438	457				-6	0*	-43	-3	242	-269
			-4	363	341	H= 3. K= 1			-7	113	120	-4	76	-88
H= 6. K= 0			-6	360	336	0	1255	-1242	-8	0*	32	-5	0*	-42
0	62	54	-8	189	-193	1	308	342	-9	53	-51	-6	0*	38
2	413	-427	-10	267	-268	2	442	-438	-10	181	-193	-7	250	275
4	178	171	-12	0*	-5	3	309	293	-11	87	66	-8	182	192
6	403	410	-14	142	-115	4	139	-155	-12	65	-69	-9	327	328
8	222	-215	-16	98	92	5	0*	-39	-13	97	75	-10	88	-86
10	161	137	-18	85	-81	6	235	-226	-14	71	-83	-11	205	218

PAGE 2 VULCAFARO

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 9. K= 1			-10	0*	47	H= 2. K= 2			-8	0*	-50	-2	114	127
-12	337	-312	-11	0*	11	0	666	631	-9	381	350	-3	356	345
-13	441	-416	-12	101	-114	1	1464	1420	-10	0*	-59	-4	275	303
-14	226	200	-13	0*	21	2	161	-166	-11	62	87	-5	759	731
-15	72	85	-14	0*	4	3	633	629	-12	132	-136	-6	401	404
-16	0*	61	-15	0*	69	4	225	-232	-13	195	204	-7	463	-457
-17	96	111	-16	94	85	5	45	-52	-14	0*	51	-8	79	-85
-18	0*	-41	-17	140	-152	6	71	84	-15	100	-85	-9	104	-103
-19	0*	55	-18	0*	-44	7	299	320	-16	0*	-66	-10	0*	-35
			-19	112	-133	8	264	264	-17	0*	78	-11	286	301
H= 11. K= 1						9	201	214	-18	0*	-14	-12	0*	2
0	95	110	H= 15. K= 1			10	212	-251				-13	0*	-35
1	69	-66	0	0*	40	11	0*	7	H= 6. K= 2			-14	158	119
2	0*	14	-1	155	186	12	0*	28	0	266	251	-15	0*	15
3	72	-61	-2	199	-193	13	99	-63	1	778	-782	-16	0*	-9
4	0*	14	-3	0*	-46	-1	0*	-36	2	0*	59	-17	0*	51
5	159	-136	-4	0*	58	-2	1561	-1514	3	240	239	-18	0*	80
6	0*	-65	-5	278	259	-3	985	955	4	81	-75			
7	92	78	-6	0*	-40	-4	73	-43	5	192	211	H= 10. K= 2		
8	84	90	-7	227	-202	-5	587	592	6	64	-78	0	284	-282
-1	93	72	-8	227	203	-6	550	545	7	214	-207	1	0*	-36
-2	143	114	-9	72	-73	-7	252	262	8	221	182	2	0*	-66
-3	63	-88	-10	72	73	-8	452	422	9	100	119	3	0*	-130
-4	279	-299	-11	159	175	-9	78	-100	10	0*	-25	4	158	136
-5	88	-121	-12	0*	-68	-10	267	-274	11	150	-128	-1	87	-100
-6	0*	-40	-13	155	169	-11	143	116	-1	646	-614	-2	0*	3
-7	140	-154	-14	67	99	-12	179	-175	-2	297	292	-3	59	-56
-8	64	-48	-15	0*	15	-13	0*	-77	-3	93	105	-4	131	125
-9	291	-274	-16	86	88	-14	100	-118	-4	598	579	-5	185	-158
-10	67	-42	-17	0*	-30	-15	0*	18	-5	138	-178	-6	59	-68
-11	97	-107	-18	0*	43	-16	146	141	-6	396	-356	-7	85	101
-12	99	-71	-19	60	-91	-17	59	-54	-7	153	135	-8	61	75
-13	319	289				-18	50	62	-8	163	-176	-9	89	-62
-14	227	-230	H= 0. K= 2						-9	245	-285	-10	0*	-24
-15	226	-209	0	1222	-1229	H= 4. K= 2			-10	0*	1	-11	67	-61
-16	0*	18	1	1897	-1877	0	0*	-84	-11	241	-246	-12	0*	-53
-17	0*	-67	2	108	-109	1	0*	-44	-12	147	134	-13	0*	40
-18	0*	-38	3	164	141	2	130	124	-13	97	100	-14	0*	-47
-19	0*	25	4	111	-101	3	317	-294	-14	100	-87	-15	158	154
			5	563	555	4	0*	42	-15	142	125	-16	154	112
H= 13. K= 1			6	597	614	5	183	184	-16	0*	3	-17	0*	58
0	227	261	7	426	414	6	57	-45	-17	0*	-32	-18	84	102
1	0*	14	8	295	307	7	0*	59	-18	84	-71			
2	100	109	9	454	460	8	92	-130				H= 12. K= 2		
3	138	-132	10	0*	37	9	374	-407	H= 8. K= 2			0	0*	45
4	66	-50	11	150	139	10	186	155	0	0*	12	1	0*	-69
-1	277	267	12	441	-441	11	0*	15	1	159	-363	2	100	127
-2	0*	5	13	0*	81	12	0*	40	2	266	-240	3	172	182
-3	496	468	14	0*	38	-1	588	-599	3	62	-93	-1	309	-297
-4	269	-306	15	0*	51	-2	766	754	4	92	-110	-2	68	-68
-5	309	-318	16	135	140	-3	370	368	5	96	108	-3	95	113
-6	218	215	17	0*	37	-4	833	-841	6	171	209	-4	94	-86
-7	346	-365	18	55	69	-5	512	513	7	100	85	-5	289	-240
-8	381	389				-6	210	-206	8	0*	-65	-6	94	-111
-9	157	-157				-7	145	138	-1	117	133	-7	0*	-42

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 12. K= 2			H= 1. K= 3			-11	90	93	-9	128	-155	-12	155	-151
-8	212	-221	0	736	-731	-12	0*	-3	-10	0*	-1	-13	0*	56
-9	96	-86	1	361	-351	-13	154	144	-11	90	93	-14	378	341
-10	154	133	2	384	-357	-14	98	-82	-12	66	-78	-15	151	112
-11	221	-199	3	48	62	-15	0*	30	-13	153	-113	-16	65	77
-12	223	215	4	0*	19	-16	0*	-66	-14	220	207	-17	0*	12
-13	159	-127	5	194	190				-15	0*	-8			
-14	100	-97	6	108	76	H= 5. K= 3			-16	0*	56	H= 13. K= 3		
-15	219	-228	7	334	342	0	206	188	-17	0*	-84	0	0*	-35
-16	210	-185	8	181	-197	1	98	120	-18	177	-166	1	0*	-8
-17	62	56	9	276	288	2	363	-391				2	67	-65
-18	0*	37	10	0*	-11	3	50	-63	H= 9. K= 3			3	91	100
			11	68	77	4	76	91	0	300	310	4	60	-64
H= 14. K= 2			12	311	308	5	258	-274	1	62	77	-1	0*	12
0	0*	75	13	69	-75	6	87	53	2	91	-119	-2	98	-105
1	0*	33	-1	1157	1139	7	0*	-29	3	0*	-101	-3	219	-226
-1	70	40	-2	181	216	8	214	208	4	97	-61	-4	154	135
-2	100	90	-3	518	535	9	69	-84	5	98	-107	-5	69	-75
-3	159	-166	-4	153	-154	10	0*	-5	6	0*	-65	-6	97	-98
-4	100	-109	-5	338	346	-1	0*	60	-1	411	407	-7	97	106
-5	274	296	-6	818	-829	-2	577	569	-2	80	53	-8	154	-156
-6	245	-244	-7	0*	-34	-3	579	-575	-3	125	-115	-9	98	124
-7	224	212	-8	291	-282	-4	488	464	-4	78	78	-10	98	-98
-8	0*	-79	-9	407	-388	-5	937	-956	-5	215	218	-11	98	-94
-9	0*	9	-10	0*	-58	-6	63	-45	-6	0*	-22	-12	155	-145
-10	275	275	-11	91	-73	-7	0*	-52	-7	58	55	-13	153	-129
-11	159	-197	-12	0*	93	-8	0*	-18	-8	84	-62	-14	95	-81
-12	223	215	-13	0*	-79	-9	551	566	-9	61	38	-15	289	-278
-13	98	-110	-14	0*	31	-10	102	-102	-10	0*	70	-16	136	149
-14	0*	-74	-15	146	128	-11	153	-156	-11	0*	-56	-17	56	68
-15	0*	35	-16	84	-69	-12	0*	52	-12	427	398			
-16	196	-185				-13	0*	-34	-13	154	135	H= 15. K= 3		
-17	57	-42	H= 3. K= 3			-14	0*	-14	-14	0*	-35	0	62	52
-18	51	-66	0	277	272	-15	0*	-12	-15	194	200	1	58	55
			1	416	435	-16	93	-113	-16	0*	64	-1	0*	30
H= 16. K= 2			2	168	161				-17	0*	-13	-2	0*	101
0	0*	-28	3	228	-246	H= 7. K= 3			-18	0*	7	-3	95	-108
-1	0*	2	4	145	123	0	0*	-57	-19	108	-115	-4	152	-165
-2	0*	-11	5	112	113	1	412	414				-5	68	89
-3	0*	24	6	172	188	2	217	228	H= 11. K= 3			-6	217	209
-4	0*	71	7	0*	97	3	0*	31	0	0*	-21	-7	154	132
-5	152	141	8	61	68	4	241	-258	1	0*	-10	-8	0*	-10
-6	97	-113	9	229	-216	5	65	-64	2	0*	-44	-9	153	152
-7	0*	-18	10	307	318	6	68	-83	3	0*	34	-10	215	-250
-8	0*	-8	11	70	-70	7	155	-166	-1	391	-401	-11	67	75
-9	0*	64	-1	411	-402	8	0*	60	-2	0*	6	-12	93	-108
-10	0*	58	-2	279	-252	9	192	199	-3	201	197	-13	144	-114
-11	150	149	-3	628	621	-1	379	371	-4	179	-187	-14	62	68
-12	93	84	-4	300	292	-2	417	423	-5	141	-154	-15	0*	29
-13	0*	8	-5	341	321	-3	364	-409	-6	0*	-16	-16	0*	50
-14	62	-71	-6	60	78	-4	67	72	-7	64	60	-17	85	-66
-15	58	78	-7	255	283	-5	215	-234	-8	204	204			
-16	0*	-74	-8	481	-500	-6	70	59	-9	147	-152	H= 0. K= 4		
			-9	0*	110	-7	73	-66	-10	0*	-52	0	128	-137
			-10	60	-51	-8	77	-86	-11	0*	-62	1	513	528

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H=	0. K=	4	11	65	85	6	67	-90	-2	150	-190	-7	0*	26
2	0*	-2	12	123	-104	7	149	-175	-1	67	-60	-8	175	-150
3	142	105	13	0*	59	8	0*	-62	-4	296	278	-9	187	182
4	706	-676	-1	713	-753	-1	347	340	-5	148	-136	-10	215	-207
5	60	-68	-2	430	-453	-2	0*	-32	-6	66	-71	-11	64	98
6	706	-701	-3	483	509	-3	149	121	-7	66	80			
7	52	-56	-4	713	732	-4	129	-158	-8	94	-106	H=	3. K=	5
8	79	82	-5	290	-313	-5	206	-180	-9	0*	-43	0	579	599
9	0*	41	-6	445	434	-6	0*	-3	-10	67	-86	1	347	-326
10	64	-88	-7	167	-201	-7	56	-50	-11	0*	-33	2	57	39
11	257	-293	-8	52	53	-8	0*	-26	-12	0*	-2	3	315	-345
12	213	189	-9	56	-61	-9	85	71	-13	94	114	4	344	334
13	115	130	-10	0*	-13	-10	62	65	-14	144	163	5	118	-130
			-11	0*	44	-11	223	226	-15	0*	60	6	57	38
H=	2. K=	4	-12	0*	-29	-12	257	267				7	134	127
0	519	527	-13	0*	31	-13	95	-82	H=	14. K=	4	8	177	-186
1	163	-187				-14	212	-219	0	139	-163	9	91	131
2	171	125	H=	6. K=	4	-15	147	-169	1	0*	-13	10	0*	-4
3	135	-117	0	300	-308	-16	88	-112	2	56	-66	11	62	-62
4	275	-278	1	71	91	-17	181	-203	-1	0*	60	12	59	65
5	107	96	2	129	146				-2	65	58	-1	316	338
6	235	263	3	56	55	H=	10. K=	4	-3	66	81	-2	100	-97
7	127	-112	4	59	57	0	144	179	-4	0*	-5	-3	186	-216
8	136	117	5	0*	-22	1	93	99	-5	67	-69	-4	204	178
9	248	-263	6	92	96	2	67	-70	-6	0*	67	-5	292	-322
10	258	278	7	0*	42	3	95	89	-7	150	-124	-6	101	131
11	213	205	8	0*	3	4	67	-54	-8	116	-113	-7	49	-46
12	66	-73	9	66	-76	5	93	-114	-9	116	-100	-8	120	124
13	0*	-4	10	168	-206	6	63	62	-10	148	-136	-9	0*	-14
-1	694	721	11	118	126	-1	89	-117	-11	66	76	-10	61	-63
-2	204	236	12	0*	-7	-2	87	82	-12	111	-135	-11	0*	-43
-3	525	-526	13	95	101	-3	136	137	-13	0*	67			
-4	697	-672	-1	46	46	-4	0*	-13	-14	190	-148	H=	5. K=	5
-5	39	49	-2	63	-70	-5	85	68				0	44	-36
-6	341	-342	-3	197	-195	-6	61	-70	H=	1. K=	5	1	148	-175
-7	154	180	-4	347	-367	-7	87	-99	0	675	-631	2	248	291
-8	75	-78	-5	0*	36	-8	62	93	1	35	39	3	205	-230
-9	181	173	-6	137	134	-9	349	338	2	0*	11	4	0*	-71
-10	61	-71	-7	569	-610	-10	291	272	3	115	102	5	0*	-23
-11	0*	35	-8	381	408	-11	297	307	4	59	89	6	62	-93
-12	211	-210	-9	255	-291	-12	150	142	5	46	48	7	205	295
-13	135	125	-10	191	194	-13	67	-44	6	102	81	8	249	-250
			-11	89	72	-14	0*	-13	7	149	-371	9	142	165
H=	4. K=	4	-12	294	-255	-15	144	-166	8	59	51	-1	164	-142
0	237	259	-13	67	71	-16	0*	-56	9	240	-202	-2	185	-173
1	57	68	-14	0*	-13				10	0*	-36	-3	227	235
2	62	-49	-15	207	-220	H=	12. K=	4	11	0*	35	-4	105	89
3	106	111				0	213	-207	12	89	-87	-5	45	41
4	443	456	H=	8. K=	4	1	0*	21	13	0*	17	-6	67	-70
5	349	-366	0	127	-133	2	66	-66	-1	433	-423	-7	72	-99
6	186	176	1	0*	-24	3	64	-72	-2	36	39	-8	0*	-4
7	279	-277	2	193	172	4	0*	-3	-3	242	-245	-9	266	-220
8	0*	35	3	179	159	5	0*	-70	-4	0*	-52	-10	521	510
9	212	168	4	185	201	6	51	58	-5	59	55	-11	140	-166
10	0*	-26	5	212	263	-1	67	74	-6	0*	-27			

PAGE 5 VULCATARD

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 7. K= 5			H= 11. K= 5											
0	76	-100	-1	00*	5									
1	56	65	-2	00*	58									
2	00*	43	-3	90	95									
3	135	-130	-4	240	-250									
4	139	100	-5	00*	17									
5	90	77	-6	91	-96									
6	144	-166	-7	64	-75									
7	221	220	-8	64	76									
-1	52	48	-9	207	-217									
-2	391	-367												
-3	193	-213												
-4	475	-504												
-5	51	54												
-6	74	74												
-7	297	335												
-8	219	221												
-9	83	101												
-10	273	-307												
-11	398	-420												
-12	00*	15												
-13	287	-319												
-14	63	-98												
H= 9. K= 5														
0	193	-186												
1	62	91												
2	63	73												
3	157	165												
-1	133	-141												
-2	143	176												
-3	82	-99												
-4	128	-122												
-5	223	226												
-6	260	263												
-7	418	435												
-8	85	87												
-9	00*	-32												
-10	89	-84												
-11	202	-207												
-12	322	-327												
H= 11. K= 5														
0	203	214												
1	00*	-5												
-1	64	87												
-2	349	-358												
-3	00*	4												
-4	282	310												
-5	89	88												
-6	63	71												
-7	00*	26												
-8	90	109												
-9	181	196												
-10	00*	8												

PAGE 5 VULCATARO

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 7. K= 5						H= 11. K= 5								
0	76	-100				-1	0*	5						
1	56	65				-2	0*	58						
2	0*	43				-3	90	95						
3	135	-138				-4	248	-258						
4	139	108				-5	0*	17						
5	90	77				-6	91	-96						
6	144	-166				-7	64	-75						
7	221	228				-8	64	76						
-1	52	48				-9	202	-217						
-2	391	-367												
-3	193	-213												
-4	475	-504												
-5	51	54												
-6	74	74												
-7	297	335												
-8	219	221												
-9	83	101												
-10	273	-307												
-11	398	-420												
-12	0*	15												
-13	287	-319												
-14	63	-98												
H= 9. K= 5														
0	193	-186												
1	62	91												
2	63	73												
3	157	165												
-1	133	-141												
-2	143	176												
-3	82	-99												
-4	128	-122												
-5	223	226												
-6	260	263												
-7	418	435												
-8	85	87												
-9	0*	-32												
-10	89	-84												
-11	202	-207												
-12	322	-327												
H= 11. K= 5														
0	203	214												
1	0*	-5												
-1	64	87												
-2	349	-358												
-3	0*	4												
-4	282	310												
-5	89	88												
-6	63	71												
-7	0*	26												
-8	90	109												
-9	181	196												
-10	0*	8												

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Chapter 3.

CADMIUM DINITRATO TRIS 2:6-DIMETHYL-γ-PYRONE

The metal complexes of 2,6-dimethyl-γ-pyrone are well known for their properties as corrosion inhibitors and have been used as a protective coating on steel.

Lewis (6) has already determined the structures of the zinc and copper complexes, details of which are given on page 82.

There are several other complexes of 2,6-dimethyl-γ-pyrone which are under investigation and cadmium 2,6-dimethyl-γ-pyrone complex is one of them.

Experimental Measurement of 2,6-dimethyl-γ-pyrone Cadmium Complex

1. Preparation of the Complex

The complex was prepared by Miss White, a student of the Chemistry Department, City of London Polytechnic.

2,6-dimethyl-γ-pyrone (2 equiv) and hydrated cadmium nitrate (1 equiv) were dissolved in the minimum volume of hot ethyl acetate in the presence of some 2,2'-dimethoxypropane which is added in order to absorb the water given off in the reaction. The product crystallised as the solution cooled in a desiccator, then it was filtered off, washed and dried.

Aqueous solutions of the complexes were analysed for metals with EDTA and appropriate indicators. Nitrate was estimated by reduction to ammonia. The analytical data are given in the table below.

TABLE

Complex $Cddmp_3(NO_3)_2$	Colour	m.pt	Cd	NO_3	C	H
FOUND	White	151-152°	17.8%	20.8%	41.5%	3.9%
CALCULATED	-	-	18.5%	20.4%	41.4%	4.0%

2. Preparation of Crystals

The compound was dissolved in ethyl acetate and warmed at 60°C to make a saturated solution. Then the solution was filtered and transferred into a crystallising dish and the temperature gradient

was maintained in such a way that white crystals of a suitable size were grown.

3. Selection of Crystals

Crystals were examined under the optical microscope. A careful examination was made under crossed polars to make sure that the crystal selected was free from twinning. One extinction direction was parallel to the needle axis.

Space-Group and Unit Cell Dimensions

The chosen crystal was mounted on a glass-fibre parallel to the direction of the needle axis. First the crystal was aligned by optical methods but the final adjustment was made using the method of double oscillation. The oscillation photographs showed mirror symmetry perpendicular to that axis which was 11.3Å. A full-rotation photograph was taken along this axis, then the crystal was transferred to a Stoe Weissenberg Camera, and zero and first layer photographs were taken. Similarly the crystal was mounted along the other two axes and rotation and zero layer Weissenberg photographs were taken .

The space group was determined from the following systematic absences.

Reflection	Conditions limiting possible reflections
hkℓ	No absences
hoℓ	$h + \ell = 2n$
oko	$k = 2n$

The crystal was therefore unambiguously space group $P2_1/n$

The cell parameters were measured as accurately as possible from the rotation photographs along the three chosen crystallographic axes.

Copper Kα radiation was used. The mathematical expression used was the same as in Chapter 2.

The final values were obtained from the diffractometer

a = 19.372Å (5)

b = 11.345Å (4)

c = 23.023Å (5)

β = 93.77° (5)

Density, measured by flotation in NaI solution, = 1.60 g/cc confirming Z = 8 mols per unit cell.

Intensity data Collection

Intensity data were collected at Queen Mary College, using their CAD-4 Kappa diffractometer.

In the Kappa Goniometer there is an axis called the kappa axis which intersects the omega axis at an angle less than 90° .

In this CAD-4 goniometer this angle is 50° . The phi axis intersects the kappa axis also at an angle of 50° . As a result of that by moving kappa axis, the phi axis describes a cone with top angle of 100° . The radius of the sphere of intersection of the 2 theta, omega, kappa and phi axes, the primary beam and secondary beam collimator is less than 10 microns.

With the kappa rotation, each reciprocal lattice vector can be brought into the equatorial plane. Rotating around omega brings this vector into the reflecting position. Rotating phi and correcting with kappa and omega, this vector remains in the reflecting position but makes an azimuthal or psi-rotation. Psi is defined to be zero when the reflection is measured in the bisecting position of the goniometer. Due to the possible full omega-rotation without any physical interference and obscuration, the range of the azimuthal rotation of each reflection is optimal and more complete than in an instrument using an Eulerian Cradle.

Since the angle between the omega and theta axes is larger than 45^{o} , a full azimuth rotation is always possible for a great number of reciprocal lattice vectors. The azimuthal scan is very important to make a good absorption correction only using intensity measurements. Moreover, the influence of multi-reflections can be eliminated. All axes are driven with special d.c motors with continuously variable slow speeds. This guarantees motion without vibrations even at the highest possible slow speeds.

While going to the next reflection, all axes are driven simultaneously at their maximum speed. The final correct position is always reached with a slow speed from the same direction to avoid even the smallest errors due to unavoidable back-lash.

Absolute encoders are of the voltage resolver type. The resolution of the angle readout of these encoders is 0.007^{o} . They maintain that accuracy over a long time.

The counter aperture unit has a continuously variable horizontal receiving aperture, a horizontal slit and two beam splitters. The apertures are computer controlled but manual control is also possible. The reproducibility of the positions of the different apertures is 10μ . The height of the aperture is determined by slits that can be inserted manually (1mm, 2mm, 3mm, 4mm, 6mm).

An incident beam balanced filter unit is provided as standard with the instrument and is simply fitted to the tube stand. One of the filter holders can be positioned and locked in order to balance the two absorption filters.

The balanced filters are also programmable or under manual control. A graphite monochromator is supplied as a standard and this requires considerable experience for correct adjustment. The tube shield is designed to accept fine focus as well as normal focus tubes. It is mounted parallel to the equatorial plane as an integral part of the goniometer and it makes the goniometer alignment independent of the table top movement.

A microscope which gives a comfortable 80 x magnification and has a cross-line and graduated reticule is fixed to the omega bracket. This microscope remains aligned during the operation of the diffractometer and does not give any physical interference. It enables the user of the diffractometer to make the necessary preliminary centering adjustments of the crystal. A second microscope is also mounted independently to observe any movement of the goniometer head.

All axes, the attenuation filter, the aperture disk, etc., can be controlled by a portable control unit which has the following functions - shutter switch and pilot lamp, balanced filter switch, attenuation filter switch, motor direction switch, speed range selector, motor speed continuous variation button, motor selector for ϕ, κ, Ω, 2θ and aperture disk, collision switch override buttons.

A Polaroid XR 57/1 cassette can be attached to the goniometer base allowing Laue or rotating crystal exposures to get information about crystal quality and symmetry.

Intensity data were collected using an automatic data collection program, and output in a condensed form. The SHELX program (7) was used to decode the data. The intensities were corrected for Lorentz and Polarisation and were reduced to structure factors for structure determination.

Structure Analysis

The S42A program which is generally used for data reduction, reduces the intensities onto one relative scale, computes $|F_o|$ and applies a sharpening function to $|F_o|^2$, so the program was modified to compute F^2 from given F , and sort data into suitable ascending orders of h, k and ℓ which is a requirement for the operation of the subsequent Fourier programs.

The structure was determined by Direct Methods. Here, as mentioned before, the statistical methods are used which involves the Σ_2 relationship

$$SE_h \sim S\Sigma_{kr} E_k E_{h-k}$$

where E_h is the normalized structure factor of reflection hkℓ. The whole operation was performed in five stages using five separate programs, parts of a Symbolic Addition Procedure (SAP) program.

The SAP 1 program uses the Wilson plot to estimate the overall temperature factor and scale factor. Here the theoretical and observed scattering powers are summed over equal ranges of $\sin^3\theta$ ie. over equal volumes of reciprocal space, and a linear least squares fit was calculated for the mean values,

$$\log e \frac{\left\langle \sum_M m.@. \sum_N nf^2(s) \right\rangle}{\left\langle \sum_M m.F_o^2 \right\rangle} \quad \text{and} \quad \langle \sin^2\theta \rangle \text{ over all space}$$

where m is the reflection multiplicity, @ is a factor to allow for systematic absences, S is $\sin\theta/\lambda$, N is the number of atom types in the full unit cell, M is the number of reflections in each range.

A_o and A_1 , the coefficients of the regression line, the temperature factor (B) and the scale(K') calculated from the following expressions

$$\log e \ (\langle m.@.\sum_N nf^2(s)\rangle / \langle m.|F_o|^2\rangle) = A_o + A_1\langle \sin^2\theta \rangle$$

$$B = A_1 . \lambda^2/2$$

$$K' = \exp(A_o/2)$$

The values of temperature and scale factor were 4.53 and 0.0521 respectively.

The SAP 2 program calculates the normalized structure factors. The normalized structure factors are calculated using the

expression

$$|E_{hk\ell}| = \frac{k(s)}{\sqrt{@.\Sigma nf2(s)}} \; |F_{hk\ell}|$$

where $s = \sin\theta/\lambda$ and $k(s) = \exp(A_o + A_1.s^2\lambda^2)$

$= K'^2 \exp(2B.s^2)$

The normalised structure factors are calculated for all reflections, observed and unobserved. A total of 908 reflections with $|E|$'s greater than 1.5 were sorted in descending order of $|E|$.

The statistics and distribution of normalised structure factors were compared with the theoretical values for centrosymmetrical structures in the following table.

	Theoretical	Observed
$<\|E\|>$	0.798	0.8162
$<\|E\|^2>$	1.000	0.9964
$<\|E^2-1\|>$	.968	0.9181
$\|E\|>3.0$	0.3%	0.3%
$\|E\|>2.0$	5.0%	3.82%
$\|E\|>1.0$	32%	32.31%

The table shows that the observed values are very close to the theoretical values and confirms the centrosymmetric space group.

The program SAP 3 searches for sets of reflections which satisfy the Σ_2 relationship

$$S\, E_{hk\ell} \sim S_{h'k'\ell'}{}_r\, E_{h'k'\ell'}\, E_{h-h',\, k-k',\, \ell-\ell'}$$

The program scans the input reflections for pairs shown on the R.H.S of Σ_2 expression which combine to give the indices on the L.H.S. SAP 3 also evaluates the arguments $(\sigma_3\, \sigma_2^{-3/2} |E_h E_k E_{h-k}|$ for each triplet.

The definition of σ_2 and σ_3 are as follows $\sigma_2 = \Sigma z_j^2$ and $\sigma_3 = \Sigma z_j^3$ where the sum is over the atom in the full unit cell, and z_j is the atomic number of atom j.

Next, the SAP 4 estimates the structure factor signs in two stages - SAP 4(a) and SAP 4(b).

SAP 4(a) performs three basic operations. It selects the origin defining reflections, estimates the tentative signs and final signs.

The program selects these reflections excluding those with eee parity and are sorted in descending order of associated number of E_2E_3 pairs.

Then a determinant of indices taken to modulo 2 is evaluated for the three reflections in the sorted list.

If the result is +1 or -1, the corresponding E values were given positive signs. If the result is other than +1 or -1, the determinant is re-evaluated with the fourth reflection replacing

the third. This operation is repeated until a non zero result is obtained.

The program is set to accept up to 400 reflections with $|E| > E_{opt}$. Sixteen separate counters are assigned to each reflection for accumulation of the sums

$$(\Sigma_k \sigma_3 \sigma_2^{-3/2} |E_n| E_k E_{h-k})$$

which relates to the probability of the sign of reflection being ±1, ±A, ±B, ±AB, ±C, ±AC, ±BC, ±ABC, ±D, ±AD, ±BD, ±ABC, ±CD, ±ACD, ±BCD, or ±ABCD .

At the beginning all counters are set to zero and the choice of the sign is decided by the sign of the sum accumulated in the counter. All accumulated sums are tested against the minimum acceptable limit.

After the procedure of tentative sign determination, the program tries to evaluate the signs of the symbols by comparing the accumulated sums in the 16 counters of each reflection. If there is consistent indication from several reflections that one of the symbols or symbol products is equivalent to one of the signs, then the real value of the symbol is determined. The Program always checks the determined sign as

Sign of A x Sign of B = Sign of AB

or Sign of ABC x Sign of BC = Sign of A

and when there is no such violation, the signs of the symbols are then accepted.

The final sign of each reflection is determined by substituting the symbols sign and adding the sums

$$(\sum_k \sigma_3 \sigma_2^{-3/2} |E_h| E_k E_{h-k})$$

which have been accumulated in the 16 counters of the reflection The final sign is accepted only when the total sum is above the minimum acceptable limit 1.75 which corresponds to a probability of 0.97.

The program determined the sign of the symbols in a very positive way. The estimated symbol, signs of symbols and cross-terms were

Symbol	Sign
1	+
A	-
B	+
AB	-

The program SAP 4(b) estimates the structure factor signs for with $|E| < E_{opt} > E_{min}$ by direct application of signs determined in SAP 4(a). A total of 908 signed normalised structure factors were for input to E-map calculation. The E-map was drawn using the Fourier program S42C. The Fourier summation was performed for the whole unit cell at the grid points of a 3-D mesh whose axes are parallel to the a_1, a_2, a_3 axes of the unit cell.

The direction of the sections was implied in order of the indices in the file, so the section was for constant values of y and printed with x horizontal and z down the page, where h_3 was the index varying most frequently, h_1 was that which varies least

frequently, and h_2 is the remaining index.

The main steps in computation were as follows:

1. Calculation of the coefficients $\pm F(hk\ell)$ [illegible] $\pm F(hk\bar{\ell})$ and apply the multiplicities to them, and store the results in the computer memory.

2. To perform the Fourier summation with a specific value of y, N_1(<25) values of x and N_2 (<121) values of z.

3. To repeat step 2 as many times as needed to cover all values of x, without varying y.

4. To repeat steps 2 and 3 for the remaining values of y.

5. To repeat steps 2, 3 and 4 for the next block of points, if there is more than one block.

The mathematical expression used for Fourier summation for the electron density map was in the form:

$$\rho(xyz) = \frac{1}{V}\{F(000) + \sum_{q} \Sigma_{ij} R_{ij}(hk\ell).trg\ 2\pi hx.trg\ 2\pi ky.trg\ 2\pi \ell z$$

where V = unit cell volume

trg = Cos or Sin

$R_{ij}(hk\ell)$ = m. $\pm A(hk\ell)$ $\pm A(hk\bar{\ell})$,

m = multiplicity (= 1,2,4 and 8)

$\Sigma = \sum_{q} \sum_{|h|} \sum_{|k|} \sum_{|\ell|}$ or all values of $|h|$, $|k|$, $|\ell|$

The number of the R terms and S terms may vary for the different groups of reflections, but multiplicity is assumed to be independent of group.

The molecule was traced from the 72 peaks located in the electron density map and atoms were easily identified from their peak height. As the electron density map was drawn for whole unit cell, there were 4 molecules, ie. (4 groups of 72 peaks) and they were all symmetry related.

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The table below lists the co-ordinates of the atoms as read from these peaks. A comparison of these co-ordinates with the final co-ordinates shows that the initial E-map was a very good representation of the structure.

TABLE 1. ATOMIC COORDINATES FROM E-MAP.

Atom Type	x/a	y/b	z/c
CD 1	.38	.38	.855
CD 2	.38	.09	.35
C 1	.215	.31	.83
C 2	.20	.30	.89
C 4	.11	.245	.95
C 5	.045	.19	.77
C 6	.11	.22	.81
C 7	.165	.275	.78
C 8	.41	.385	.715
C 9	.34	.41	.69
C 10	.32	.43	.64
C 11	.275	.465	.61
C 12	.48	.355	.555
C 13	.43	.375	.61
C 14	.445	.36	.66
C 15	.53	.50	.90
C 16	.57	.55	.94
C 17	.64	.58	.925
C 18	.69	.63	.96
C 19	.64	.55	.77
C 20	.61	.54	.835
C 21	.55	.50	.83
C 22	.22	.19	.40
C 23	.18	.225	.45
C 24	.12	.26	.44
C 25	.07	.29	.49

Atom Type	x/a	Y/b	z/c
C 26	.09	.31	.29
C 27	.125	.28	.34
C 28	.19	.22	.35
C 29	.31	.09	.20
C 30	.26	.07	.16
C 31	.27	.045	.11
C 32	.23	.005	.07
C 33	.46	.12	.08
C 34	.39	.11	.12
C 35	.38	.12	.19
C 36	.52	.000	.32
C 37	.54	.98	.38
C 38	.61	.94	.40
C 39	.64	.92	.465
C 40	.69	.90	.26
C 41	.63	.92	.30
C 42	.565	.96	.28
N 1	.34	.60	.88
N 2	.40	.165	.90
N 3	.36	.85	.38
N 4	.43	.29	.39
O 1	.28	.325	.82
O 2	.09	.22	.86
O 3	.425	.375	.77
O 4	.36	.405	.595
O 5	.48	.445	.91
O 6	.66	.58	.87
O 7	.36	.58	.83
O 8	.325	.51	.91
O 9	.31	.71	.885
O 10	.42	.18	.86
O 11	.37	.24	.93
O 12	.40	.12	.95
O 13	.28	.13	.41

Atom Type	x/a	y/b	z/c
O 13	.28	.13	.41
O 14	.09	.29	.39
O 15	.31	.12	.26
O 16	.34	.08	.09
O 17	.46	.035	.31
O 18	.65	.91	.355
O 19	.33	.89	.33
O 20	.345	.73	.395
O 21	.38	.92	.41
O 22	.39	.29	.35
O 23	.43	.19	.42
O 24	.455	.39	.41

Refinement of Structure.

The structure of the molecule was refined using the Least Squares Refinement program S42D using the block-diagonal approximation with one or two blocks per atom.

The quantity R which is minimized in this program is

$$R = \Sigma w(|F_o| - |F_c|)^2 = \Sigma w\Delta^2 \qquad \ldots (i)$$

where Σ = sum over all non-equivalent reflections

w = weight = $1/\sigma^2$ theoretically

$|F_c|$ = calculated structure amplitude on the scale of $|F_o|$

$$\Delta = |F_o| - |F_c| \qquad \ldots (ii)$$

If ρ_j ($j = 1, \ldots, N$) are the N parameters in $|F_c|$, then R is minimum when these parameters satisfy the N equations

$$\partial R/\partial \rho_i = 0 \qquad j = 1, \ldots N \qquad \ldots (iii)$$

$$\text{or } \Sigma w\Delta \partial|F_c|/\partial\rho_j = 0 \qquad \ldots (iv)$$

If the parameters ρ_j in the trial structure are close to the parameters $(\rho_j + \varepsilon_j)$ in the refined structure, then by Taylor's expansion

$$\Delta(\underline{\rho} + \underline{\varepsilon}) = \Delta(\underline{\rho}) \sum_{i=1}^{N} \varepsilon_i \partial|F_c|/\partial\rho_i$$

where $\underline{\rho}$ and $\underline{\varepsilon}$ stands for whole set of parameters and shifts. Substituting this value $\Delta(\underline{\rho} + \underline{\varepsilon})$ in equation (iv) we get the

Normal Equations

$$\sum_{i=1}^{N} \Sigma w \frac{\partial|F_c|}{\partial\rho_i} \frac{\partial|F_c|}{\partial\rho_j} \varepsilon_i = \Sigma w\Delta \frac{\partial|F_c|}{\partial\rho_j}$$

where the Δ on the r.h.s is calculated with the assumed parameter ρ_j and there are N equations for j = 1, ..., N.

The same equation can be written in the matrix form,

$$\Sigma_i a_{ij} \varepsilon_i = b_j \qquad j = 1, \ldots, N$$

where $$a_{ij} = \Sigma w \frac{\partial |F_c|}{\partial \rho_i} \frac{\partial |F_c|}{\partial \rho_j}$$

$$b_j = \Sigma w \Delta \frac{\partial |F_c|}{\partial \rho_j}$$

The estimated standard deviations are calculated according to the following formula, where the weights are assumed to be relative

$$\sigma(\rho_i) = (a^{-1})_{ii} (\Sigma w \Delta^2)/(m-n)^{\frac{1}{2}}$$

where $(a^{-1})_{ii}$ is a diagonal element in the matrix a_{ij}

m is the number of reflections

n is the number of variable parameters in the structure

The block diagonal approximation was made to reduce the amount of calculations and to allow for a large number of parameters to be refined simultaneously. The interactions between the parameters of different atoms were neglected.

The structure factors can be expressed in the following way

$$F(hk\ell) = \Sigma_r f_r(hk\ell) \exp 2\pi i(hx_r + ky_r + lz_r)$$

$$= A(hk\ell) + iB(hk\ell)$$

where $$A(hk\ell) = \Sigma_r f_r(hk\ell) \cos 2\pi(hx_r + ky_r + lz_r)$$

$$B(hk\ell) = \Sigma_r f_r(hk\ell) \sin 2\pi(hx_r + ky_r + lz_r)$$

where $|F|$, $|A|$, $|B|$ are on absolute scale; f_r is the atomic scattering factor corrected for atomic vibration.

The correction for atomic vibration was made in the following way:

If f_o is the atomic scattering factor at zero temperature, and q is the correction for thermal vibration, then

$$f_r(hk\ell) = f_r^{o}(hk\ell) \cdot q_r(hk\ell)$$

for isotropic vibration

$$q_r(hk\ell) = \exp\left[-B_r(\sin\theta/\lambda)^2\right]$$

for anisotropic vibration

$$q_r(hk\ell) = \exp\left[-(B_{11}h^2 + B_{22}k^2 + B_{33}b^2 + B_{23}k\ell + B_{13}h\ell + B_{12}hk)_r\right]$$

The isotropic temperature factor B expressed in terms of the mean square amplitude of vibration u is

$$B = 8\pi^2 U = 8\pi^2\overline{u^2} \quad (\mathring{A}^2)$$

The corresponding relationships in the case of anisotropic vibrations are

$$B_{11} = 2\pi^2 a^{*2}U_{11}\ , \quad B_{22} = 2\pi^2 b^{*2}U_{22}\ , \quad B_{33} = 2\pi^2 c^{*2}U_{33}\ ,$$

$$B_{23} = 4\pi^2 b^*c^*U_{23}, \quad B_{13} = 4\pi^2 a^*c^*U_{13}\ , \quad B_{12} = 4\pi^2 a^*b^*U_{12}$$

where $q_r(hk\ell) = \exp\ -2\pi^2(U_{11}h^2a^{*2} + \ldots\ldots + 2U_{23}k\ell b^*c^* + \ldots\ldots)$

The program refines B or B_{ij}

and also, since

$$(\sin\theta/\lambda)^2 = \tfrac{1}{4}\ h^2a^{*2} + \ldots\ldots + 2k\ .b^*c^*\cos\alpha^* + \ldots\ldots\ ,$$

Then the B_{ij}'s can be calculated from the isotropic B by the relations

$$B_{11} = B.a^{*2}/4\ , \quad B_{23} = B.b^*c^*\cos\alpha^*/2$$

During the least squares refinement, the observed structure amplitudes $|F_o|$ were kept on their original scale, and the calculated structure amplitudes $|F_c|$ are converted to the scale of the $|F_o|$ data by means of an overall scale factor G and before output on printer these amplitudes were converted to absolute scale by means of the scale K, where $K = \frac{1}{G}$.

After several cycles of refinements using isotropic thermal parameters for non-hydrogen atoms, the position of 48 hydrogen atoms were calculated and then the anisotropic temperature factors were used for the refinement of the non hydrogen atomic parameters. No attempt was made to refine the hydrogen parameters or temperature coefficients because of the possible swamping effect of the cadmium atom. Special programs were written for calculating the hydrogen atomic positions.

The procedure was repeated until no further improvement took place as shown by residual index

$$R = \Sigma(|F_o - F_c|)/\Sigma|F_o|$$

The S42D program utilises information either from the data reduction program S42A or from the previous run of structure factor least-squares program. The atomic parameters could either be read from previous program or supplied separately on the computer terminal.

The final value of the R index obtained was 0.0605 for 7325 observed reflections. The final positional co-ordinates and their thermal parameters and their standard deviations are given in Table 2.

Discussion:

The electron density map revealed the structure of the compound Cadmium Dinitrato Tris [2:6-Dimethyl-γ-Pyrone] as discrete molecules. There is no bonding between them, only Vander Waals forces hold them together.

The study of other structures of cadmium nitrate complexes are given below to compare with Cd-dimethyl-γ-pyrone structure.

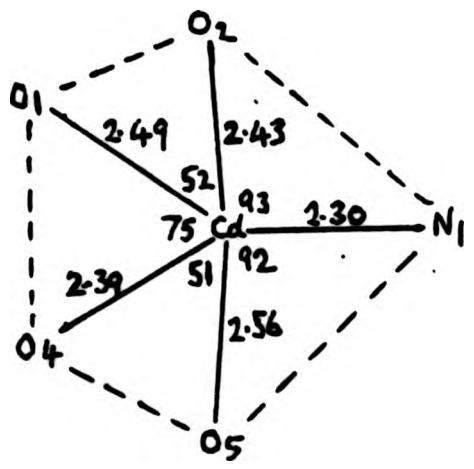
i) Aquodinitratobis (Quinoline) Cadmium (II)($C_{18}H_{16}Cd\ N_4O_7$)(Ref 5)

The compound was crystallised in monoclinic system with a = 11.018, b = 8.585 , c = 23.756 and β = 91.07 degree with space group $P2_1/c$.

The structure analysis revealed a seven co-ordinate monomeric structure in which the cadmium atom is surrounded by two non-equivalent, but bidentate nitrate groups by two quinoline molecules and by a water molecule. The cadmium-oxygen Cd - $O(NO_3)$ is 2.393 - 2.559 , Cd - $O(H_2O)$ is 2.346Å and cadmium-nitrogen Cd - N 2.296, 2.330Å .

The stereochemistry of the cadmium atom may be described as a pentagonal bipyramidal geometry. In this case the axial sites are occupied by the oxygen of the water O(7) and by the nitrogen of one of the quinoline molecules N(2) while the equatorial plane contains the nitrogen of the remaining quinoline molecule N(1) and the oxygen of the two nitratogroups O(1), O(2), O(4) and O(5) . The last four oxygens are virtually co-planar with the cadmium, although N(1) is displaced 0.88Å from this plane.

The figure below gives a diagrammatic representation of the equatorial plane of the pentagonal bipyramidal geometry of the molecule, showing the distortion from ideal geometry. The distances are in Å and the angles in deg.



(ii) Dinitratotrispyridinecadmium (II) $Cd(py)_3(NO_3)_2$ (Ref 4)

The compound was crystallised in a monoclinic system with space group C2/c with Z = 4 in a unit cell of dimensions a = 12.434 , b = 9.494 , c = 17.385 , $\beta = 115.42^{o}$.

Structure analysis revealed the crystalline $Cd(py)_3(NO_3)_2$ was monomeric. The cadmium atom was found to be surrounded by three pyridine and two bidentate nitrate groups. The molecule itself showed 2-fold symmetry.

The cadmium oxygen distances Cd-O(1) 2.444 , Cd-O(2) 2.491 Å indicate that the nitrate groups are almost symmetrically coordinated. Because of the large ionic radius cadmium can more

readily accommodate a regular seven co-ordinate environment.

(iii) Zinc bis(Nitrato 2,6-dimethyl-γ-pyrone) $Zn(NO_3)_2(C_7H_8O_2)_2$ (Ref 2)

The compound crystallises in the monoclinic system, space group A2/a with four molecules in a unit cell of dimensions

$a = 16.786\text{Å}$; $b = 12.922\text{Å}$; $c = 9.089\text{Å}$; $\beta = 106.80^{\circ}$

The structure was solved by Patterson and Fourier synthesis. The zinc atom displays distorted tetrahedral co-ordination.

The nitrate groups are found to be symmetrically positioned with respect to the zinc atom. The gamma pyrone ligands and nitrato groups are nearly planar. The nitrato groups were monodentate.

(iv) Copper Nitrato 2,6-dimethyl-γ-pyrone (Ref 3)

The unit cell is triclinic with $a = 15.872\text{Å}$, $b = 8.772\text{Å}$, $c = 8.384\text{Å}$, $\alpha = 124.35$, $\beta = 106.84$, $\gamma = 85.93^{\circ}$, space group $P\bar{1}$.

The structure was solved using Patterson and Fourier synthesis. The copper co-ordination is distorted square planar. The gamma pyrone ligands and the nitrato groups were found to be planar but not co-planar. The two gamma pyrones were cis to each other and the nitrato groups were monodentate.

The cadmium gamma pyrone complex showed 7-fold co-ordination and the stereo-chemistry of the cadmium atom may be described as a distorted pentagonal bipyramid.

The above type of co-ordination can be compared with calcium Naphthionate, $8H_2O$ structure determined by Brown, Ehrenberg and Yadav (1). In this case, the Ca^{2+} is surrounded by seven water molecules. Here each calcium ion is co-ordinated to seven oxygen

atoms, all ffom the water molecules. The oxygen atoms around the calcium are arranged in a distorted pentagonal bipyramid.

The reason of forming similar type of co-ordination polyhedra can be explained as Cd^{+2} and Ca^{+2} have atomic radii 0.97 and 0.99 and because of the large radii, they can easily accommodate seven atoms in the sphere of coordination.

The bidentate nature of the nitrate group in the cadmium gamma pyrone complex can be compared with the above mentioned nitrate groups of zinc-nitrato- 2,6-dimethyl-γ-pyrone and copper-nitrato 2,6-dimethyl-γ-pyrone, where nitrato groups are unidentate. In the former case, the sevenfold co-ordination enables the nitrate groups to be bidentate whereas in the later case Zn or Cu atoms, because of their smaller size can only accommodate four atoms within the coordination sphere. The planarity of the gamma pyrone rings shows the electron distribution of σ and π type. Table 5 shows the planarity of the ring.

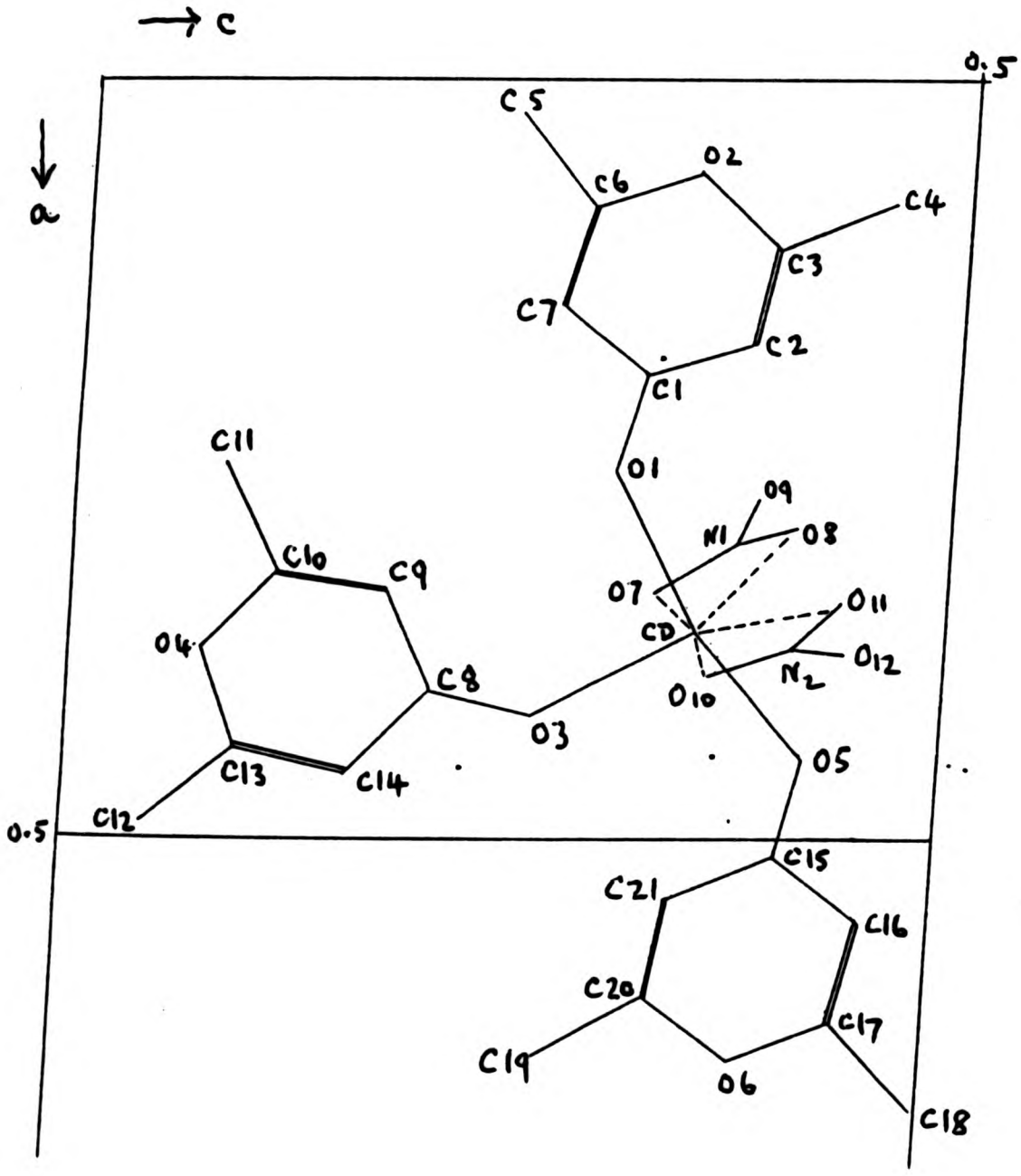


Figure 1. Orientation of one molecule in unit cell showing numbering of atoms. The numbering for molecule 2 is (C(n+21), N(n+2), O(n+12) and Cd(2).

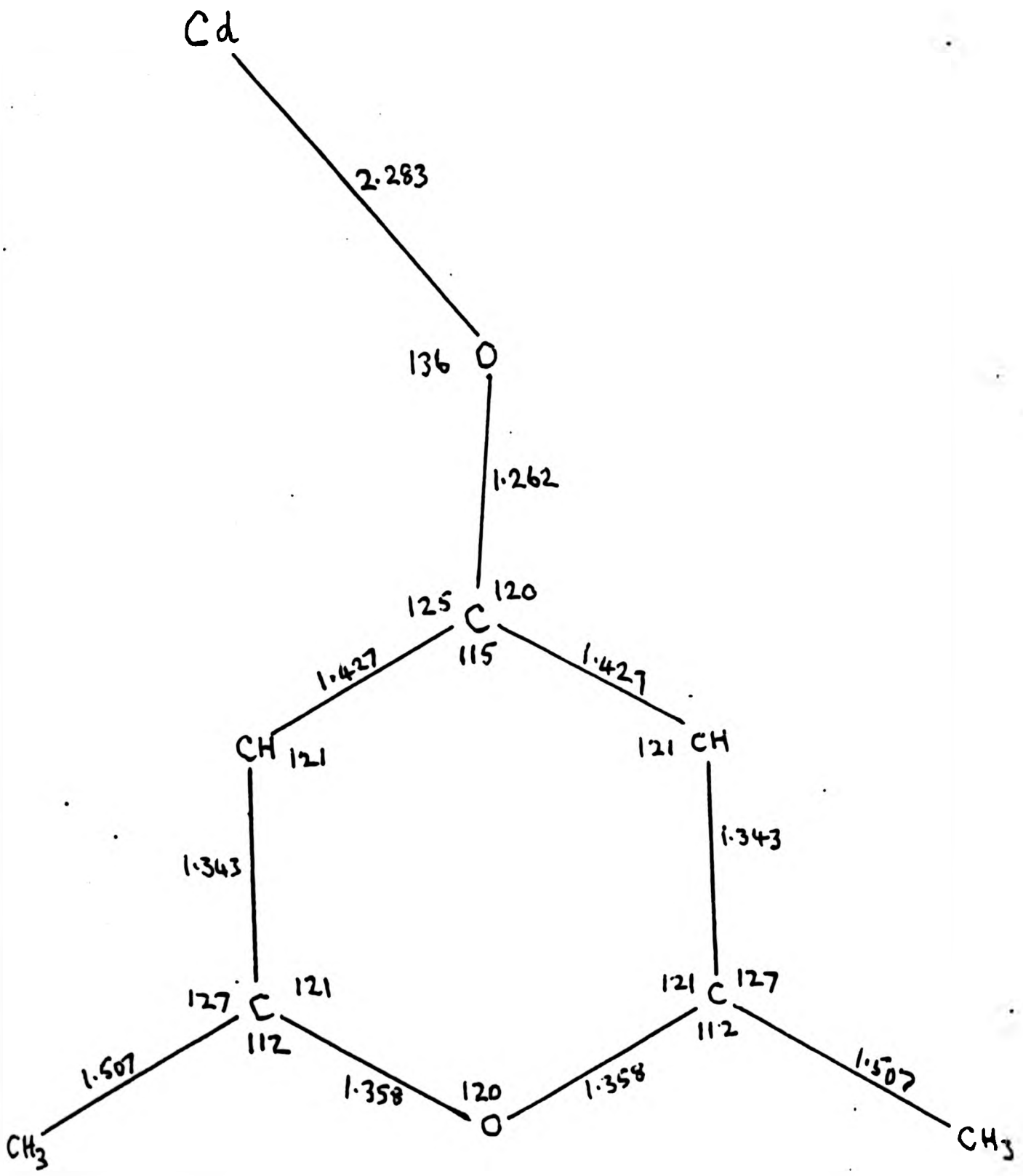


Figure 2. Averaged dimensions of the 2:6-dimethyl-γ-pyrone ring.

TABLE 2. ATOMIC PARAMETERS

CADMIUM DINITRATO TRIS(2,6,GAMMA-PYRONE)
ORIGINAL COORDINATES

ATOM	NO.	X/A	Y/B	Z/C	E.S.D.S SYG X/A	SYG Y/B	SYG Z/C
CD	1	0.38279	0.37833	0.35515	0.00002	0.00003	0.00001
CD	2	0.36140	0.08814	0.84911	0.00002	0.00003	0.00001
C	1	0.21750	0.29643	0.33131	0.00025	0.00041	0.00019
C	2	0.23126	0.28889	0.39048	0.00026	0.00046	0.00021
C	3	0.13854	0.25063	0.40350	0.00028	0.00046	0.00019
C	4	0.11328	0.23767	0.46401	0.00038	0.00079	0.00025
C	5	0.04388	0.18386	0.26635	0.00031	0.00069	0.00027
C	6	0.10513	0.22503	0.30515	0.00025	0.00043	0.00018
C	7	0.16644	0.26035	0.28728	0.00026	0.00043	0.00020
C	8	0.43891	0.37506	0.21329	0.00032	0.00050	0.00020
C	9	0.34100	0.41172	0.19230	0.00033	0.00048	0.00023
C	10	0.32352	0.42829	0.13514	0.00031	0.00044	0.00022
C	11	0.25315	0.46511	0.10951	0.00030	0.00055	0.00029
C	12	0.47786	0.35918	0.05980	0.00042	0.00075	0.00031
C	13	0.43556	0.37391	0.11180	0.00038	0.00050	0.00023
C	14	0.45565	0.35888	0.16819	0.00028	0.00047	0.00020
C	15	0.53121	0.40656	0.39597	0.00022	0.00046	0.00021
C	16	0.57833	0.54465	0.44019	0.00030	0.00048	0.00021
C	17	0.63986	0.58536	0.42549	0.00032	0.00051	0.00029
C	18	0.69674	0.63870	0.46493	0.00030	0.00056	0.00029
C	19	0.64397	0.55482	0.26677	0.00039	0.00070	0.00029
C	20	0.61443	0.54686	0.32603	0.00032	0.00047	0.00023
C	21	0.55308	0.50065	0.33780	0.00027	0.00041	0.00020
C	22	0.22240	0.18947	0.90558	0.00023	0.00043	0.00019
C	23	0.18111	0.20948	0.95371	0.00033	0.00047	0.00023
C	24	0.11831	0.25975	0.94759	0.00030	0.00056	0.00025
C	25	0.07894	0.28738	0.99459	0.00048	0.00088	0.00031
C	26	0.09072	0.31554	0.78989	0.00036	0.00068	0.00031
C	27	0.12818	0.27470	0.84556	0.00026	0.00043	0.00019
C	28	0.19083	0.22369	0.85074	0.00028	0.00043	0.00019
C	29	0.31259	0.09885	0.70780	0.00030	0.00055	0.00022
C	30	0.26078	0.06269	0.66496	0.00034	0.00058	0.00023
C	31	0.27319	0.04737	0.60908	0.00029	0.00050	0.00022
C	32	0.22415	0.00801	0.55961	0.00049	0.00083	0.00031
C	33	0.15410	0.11897	0.59677	0.00038	0.00084	0.00033
C	34	0.10996	0.10377	0.62990	0.00045	0.00053	0.00029
C	35	0.07833	0.12128	0.68623	0.00036	0.00056	0.00025
C	36	0.51811	-0.00114	0.82321	0.00025	0.00042	0.00019
C	37	0.54175	-0.01826	0.88301	0.00025	0.00052	0.00022
C	38	0.60553	-0.05728	0.89935	0.00029	0.00043	0.00019
C	39	0.63528	-0.07582	0.96099	0.00033	0.00074	0.00024
C	40	0.68752	-0.10857	0.76422	0.00037	0.00059	0.00029
C	41	0.63183	-0.07319	0.80183	0.00023	0.00044	0.00019
C	42	0.56883	-0.03348	0.78370	0.00028	0.00047	0.00023
N	1	0.32906	0.60129	0.37757	0.00028	0.00037	0.00021
N	2	0.43139	0.15620	0.40762	0.00030	0.00038	0.00023
N	3	0.35401	-0.15399	0.87259	0.00031	0.00058	0.00026
N	4	0.42497	0.29397	0.89367	0.00028	0.00041	0.00021
O	1	0.27596	0.33035	0.31445	0.00019	0.00033	0.00015
O	2	0.08950	0.21759	0.36156	0.00018	0.00031	0.00014
O	3	0.42834	0.35990	0.26643	0.00023	0.00038	0.00016
O	4	0.36954	0.40823	0.09445	0.00023	0.00035	0.00015
O	5	0.47393	0.45743	0.41058	0.00028	0.00041	0.00015
O	6	0.65925	0.58843	0.36978	0.00028	0.00034	0.00016
O	7	0.35719	0.57706	0.33294	0.00027	0.00037	0.00020
O	8	0.32248	0.52741	0.41232	0.00023	0.00039	0.00016
O	9	0.38955	0.70887	0.38686	0.00039	0.00053	0.00029
O	10	0.41832	0.17380	0.35824	0.00031	0.00044	0.00021
O	11	0.37427	0.23993	0.43214	0.00029	0.00045	0.00020
O	12	0.48773	0.06277	0.43320	0.00034	0.00049	0.00030
O	13	0.28978	0.14151	0.91114	0.00021	0.00039	0.00015
O	14	0.09265	0.29278	0.89406	0.00028	0.00032	0.00017
O	15	0.38293	0.11622	0.76089	0.00024	0.00043	0.00016
O	16	0.33757	0.06684	0.59216	0.00026	0.00042	0.00018
O	17	0.45956	0.03475	0.80571	0.00019	0.00040	0.00015
O	18	0.64984	-0.08695	0.85880	0.00018	0.00031	0.00016
O	19	0.32538.	-0.11248	0.82918	0.00026	0.00046	0.00020
O	20	0.35877	-0.25996	0.88424	0.00038	0.00058	0.00031
O	21	0.38140	-0.08403	0.90842	0.00025	0.00039	0.00018
O	22	0.38999	0.29004	0.84784	0.00026	0.00052	0.00021
O	23	0.43536	0.20251	0.92058	0.00023	0.00038	0.00018
O	24	0.44594	0.38886	0.91083	0.00042	0.00046	0.00031

TABLE 2 (contd.)

Calculated Hydrogen Parameters

	x/a	y/b	z/c	B_{iso}
H(1) (-C(2))	0.2392	0.3138	0.4248	5.00 Å^2
H(2) (-C(4))	0.1121	0.5232	0.4846	5.00
H(3) (-C(4))	0.0619	0.2004	0.4611	5.00
H(4) (-C(4))	0.1478	0.1802	0.4996	5.00
H(5) (-C(5))	0.0576	0.1046	0.2438	5.00
H(6) (-C(5))	0.0009	0.1658	0.2925	5.00
H(7) (-C(5))	0.0294	0.2518	0.2350	5.00
H(8) (-C(7))	0.1767	0.2614	0.2417	5.00
H(9) (-C(9))	0.3024	0.4266	0.2233	5.00
H(10) (-C(11))	0.2410	0.5519	0.1252	5.00
H(11) (-C(11))	0.2533	0.4668	0.0626	5.00
H(12) (-C(11))	0.2147	0.4030	0.1224	5.00
H(13) (-C(12))	0.4471	0.3834	0.0208	5.00
H(14) (-C(12))	0.5229	0.4152	0.0645	5.00
H(15) (-C(12))	0.4940	0.2684	0.0567	5.00
H(16) (-C(14))	0.5095	0.3339	0.1799	5.00
H(17) (-C(16))	0.5646	0.5482	0.4949	5.00
H(18) (-C(18))	0.7161	0.5736	0.4961	5.00
H(19) (-C(18))	0.7393	0.6676	0.4393	5.00
H(20) (-C(18))	0.6766	0.7132	0.4977	5.00
H(21) (-C(19))	0.6113	0.6106	0.2387	5.00
H(22) (-C(19))	0.6954	0.5917	0.2715	5.00
H(23) (-C(19))	0.6462	0.4678	0.2490	5.00
H(24) (-C(21))	0.5195	0.4658	0.3026	5.00
H(25) (-C(23))	0.2011	0.1832	0.9967	5.00
H(26) (-C(25))	0.0230	0.3227	0.9754	5.00
H(27) (-C(25))	0.0606	0.2078	1.0193	5.00
H(28) (-C(25))	0.0951	0.3515	1.0240	5.00
H(29) (-C(26))	0.0798	0.2407	0.7618	5.00
H(30) (-C(26))	0.0428	0.3575	0.7996	5.00
H(31) (-C(26))	0.1227	0.3777	0.7684	5.00
H(32) (-C(28))	0.2191	0.2082	0.8120	5.00
H(33) (-C(30))	0.2091	0.0469	0.6782	5.00
H(34) (-C(32))	0.2518	-0.0005	0.5206	5.00
H(35) (-C(32))	0.2020	-0.0761	0.5701	5.00
H(36) (-C(32))	0.1834	0.0724	0.5525	5.00

TABLE 2 (contd.)

	x/a	y/b	z/c	B_{iso}
H(37) (-C(33))	0.4417	0.1020	0.5511	5.00 Å^2
H(38) (-C(33))	0.4731	0.2080	0.6023	5.00
H(39) (-C(33))	0.4934	0.0577	0.6132	5.00
H(40) (-C(35))	0.4199	0.1531	0.7157	5.00
H(41) (-C(37))	0.5067	0.0011	0.9163	5.00
H(42) (-C(39))	0.6062	-0.1448	0.9813	5.00
H(43) (-C(39))	0.6887	-0.1036	0.9603	5.00
H(44) (-C(39))	0.6322	0.0041	0.9954	5.00
H(45) (-C(40))	0.7310	-0.1416	0.7912	5.00
H(46) (-C(40))	0.6689	-0.1767	0.7344	5.00
H(47) (-C(40))	0.7035	-0.0330	0.7399	5.00
H(48) (-C(42))	0.5549	-0.0259	0.7375	5.00

TABLE 2 (contd.)

Anisotropic Temperature Factors and their e.s.d's

	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
Cd(1)	0.00254 1	0.00709 3	0.00127 1	-0.00029 2	0.00097 1	-0.00059 3
Cd(2)	0.00252 1	0.00899 3	0.00129 1	0.00001 2	0.00096 1	0.00090 3
C(1)	0.00253 13	0.00654 36	0.00135 8	0.00031 30	-0.00002 16	0.00069 37
C(2)	0.00267 15	0.00723 40	0.00199 10	-0.00019 35	-0.00031 19	0.00028 40
C(3)	0.00366 16	0.00758 40	0.00103 8	0.00013 31	0.00015 18	0.00104 44
C(4)	0.00455 23	0.01746 88	0.00175 12	-0.00205 53	0.00245 27	-0.00090 11
C(5)	0.00273 17	0.01529 73	0.00223 13	-0.00463 52	-0.00095 23	-0.00079 56
C(6)	0.00263 13	0.00730 37	0.00116 8	-0.00039 30	-0.00009 16	-0.00055 39
C(7)	0.00294 15	0.00650 36	0.00162 9	-0.00036 32	0.00068 18	0.00150 41
C(8)	0.00375 19	0.01026 50	0.00094 8	-0.00130 32	0.00235 21	-0.00232 47
C(9)	0.00344 20	0.00728 40	0.00176 11	-0.00109 35	0.00079 23	-0.00045 45
C(10)	0.00359 17	0.00675 41	0.00156 10	0.00044 32	0.00039 20	0.00053 43
C(11)	0.00294 17	0.00975 47	0.00319 15	0.00175 47	0.00181 26	-0.00079 49
C(12)	0.00442 25	0.01625 92	0.00235 14	-0.00145 57	0.00350 32	-0.00376 76
C(13)	0.00494 24	0.00866 48	0.00135 10	-0.00056 34	0.00250 26	-0.00059 52
C(14)	0.00282 15	0.00903 44	0.00132 9	-0.00249 34	0.00200 19	-0.00192 43
C(15)	0.00168 11	0.00865 41	0.00077 9	0.00134 33	0.00079 16	0.00278 37
C(16)	0.00374 17	0.00846 42	0.00130 9	-0.00191 34	0.00234 20	0.00119 46

TABLE 2 (contd.)

	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
C(17)	0.00292 17	0.00746 43	0.00285 14	-0.00035 42	0.00190 25	-0.00051 44
C(18)	0.00223 15	0.01149 60	0.00270 14	0.00003 46	-0.00019 23	-0.00393 48
C(19)	0.00445 24	0.01376 73	0.00228 13	0.00284 53	0.00166 28	0.00532 73
C(20)	0.00423 19	0.00718 40	0.00179 11	-0.00016 36	0.00237 23	0.00404 50
C(21)	0.00332 16	0.00544 34	0.00146 9	0.00133 30	0.00037 19	-0.00019 39
C(22)	0.00199 12	0.00725 37	0.00137 8	0.00096 29	0.00150 16	0.00002 35
C(23)	0.00416 19	0.00686 41	0.00196 10	-0.00163 35	0.00241 23	0.00001 47
C(24)	0.00320 17	0.00967 51	0.00235 13	0.00013 43	0.00093 23	0.00148 51
C(25)	0.00585 31	0.01480 41	0.00253 15	-0.00113 59	0.00196 35	0.00477 86
C(26)	0.00370 21	0.01209 64	0.00299 16	0.00066 55	-0.00014 29	-0.00266 61
C(27)	0.00329 17	0.00612 34	0.00138 9	0.00077 30	0.00016 19	-0.00078 39
C(29)	0.00352 16	0.00624 36	0.00134 9	-0.00046 30	0.00128 19	0.00193 40
C(29)	0.00289 16	0.01139 50	0.00131 9	0.00145 36	0.00174 20	0.00257 47
C(30)	0.00404 20	0.01206 61	0.00126 9	-0.00123 40	0.00009 21	0.00218 58
C(31)	0.00310 16	0.00848 43	0.00167 10	0.00091 36	0.00047 19	-0.00004 46
C(32)	0.00658 36	0.01466 84	0.00252 16	-0.00166 63	-0.00010 38	0.00172 94
C(33)	0.00304 21	0.02170 113	0.00263 16	-0.00329 66	0.00225 31	-0.00231 72
C(34)	0.00609 31	0.00737 42	0.00259 15	0.00294 42	0.00466 37	0.00426 60
C(35)	0.00597 22	0.01121 59	0.00168 11	0.00040 40	0.00175 25	-0.00163 54
C(36)	0.00283 14	0.00628 35	0.00131 8	-0.00062 29	0.00085 17	0.00092 38
C(37)	0.00227 13	0.01021 49	0.00184 10	-0.00214 38	0.00099 19	-0.00120 44

TABLE 2 (contd.)

	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
C(38)	0.00391 18	0.00666 38	0.00105 8	-0.00063 29	0.00015 19	-0.00246 44
C(39)	0.00312 19	0.01875 98	0.00130 10	0.00074 49	-0.00044 21	-0.00069 65
C(40)	0.00370 21	0.01078 55	0.00259 14	-0.00166 47	0.00325 29	0.00101 55
C(41)	0.00172 11	0.00947 41	0.00130 9	-0.00055 30	-0.00006 15	0.00135 35
C(42)	0.00296 15	0.00705 39	0.00220 11	-0.00087 36	0.00226 22	-0.00105 43
N(1)	0.00304 16	0.00580 31	0.00230 11	-0.00154 31	-0.00085 21	0.00143 34
N(2)	0.00414 17	0.00528 31	0.00279 12	0.00092 33	0.00142 23	0.00034 40
N(3)	0.00394 19	0.00975 46	0.00305 14	0.00192 43	0.00212 26	0.00129 49
N(4)	0.00450 19	0.00702 36	0.00214 10	-0.00129 33	0.00216 22	0.00055 43
O(1)	0.00275 10	0.00935 33	0.00161 7	-0.00125 26	0.00186 13	-0.00110 30
O(2)	0.00261 10	0.00731 28	0.00180 7	-0.00134 24	0.00045 13	-0.00036 29
O(3)	0.00366 14	0.01123 39	0.00162 8	-0.00170 28	0.00141 17	-0.00094 37
O(4)	0.00399 14	0.00940 31	0.00160 7	-0.00072 26	0.00092 17	-0.00240 34
O(5)	- 0.00279 11	0.01294 42	0.00173 8	-0.00133 30	0.00095 14	-0.00226 37
O(6)	0.00284 12	0.00911 30	0.00203 8	-0.00147 26	0.00091 16	-0.00029 29
O(7)	0.00472 17	0.00907 39	0.00240 9	0.00313 32	0.00135 19	0.00088 41
O(8)	0.00403 15	0.01017 38	0.00165 7	-0.00111 29	0.00167 17	-0.00039 40
O(9)	0.00803 32	0.00986 47	0.00525 20	-0.00298 51	-0.00158 42	0.00063 64
O(10)	0.00569 21	0.01036 44	0.00290 11	0.00174 36	0.00154 24	0.00565 49
O(11)	0.00640 22	0.01022 42	0.00266 10	0.00173 35	0.00351 24	0.00212 50
O(12)	0.00523 21	0.01130 51	0.00522 19	0.00373 51	-0.00168 32	0.00114 55

TABLE 2 (contd.)

	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
O(13)	0.00301 12	0.01391 45	0.00121 6	-0.00053 27	0.00145 14	0.00367 37
O(14)	0.00318 12	0.00665 24	0.00232 9	-0.00068 26	0.00093 15	0.00027 30
O(15)	0.00379 14	0.01539 51	0.00109 6	0.00017 29	-0.00092 16	0.00337 41
O(16)	0.00491 17	0.01093 43	0.00215 9	0.00131 31	0.00073 20	0.00445 44
O(17)	0.00290 11	0.01195 40	0.00193 8	0.00094 30	0.00079 15	0.00292 37
O(18)	0.00237 11	0.00724 29	0.00144 7	-0.00170 24	-0.00022 13	-0.00103 27
O(19)	0.00368 15	0.01443 53	0.00225 10	-0.00066 34	0.00023 19	-0.00124 43
O(20)	0.00732 29	0.01019 51	0.00588 22	-0.00042 54	-0.00014 41	-0.00936 66
O(21)	0.00441 16	0.00934 36	0.00211 9	0.00132 29	0.00097 19	-0.00249 38
O(22)	0.00449 17	0.01582 55	0.00266 10	0.00348 39	-0.00143 21	-0.00294 47
O(23)	0.00389 14	0.00922 35	0.00239 9	-0.00043 30	0.00117 14	0.00001 37
O(24)	0.00736 31	0.00998 46	0.00457 19	-0.00264 47	0.00022 39	-0.00465 61

The temperature factor is given by the equation:-

$$T = \exp\left[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{23}hk + 2\beta_{31}lh + 2\beta_{12}hk)\right].$$

TABLE 3. BOND LENGTHS

CADMIUM DINITRATO TRIS(2,6,GAMMA-PYRONE)

INTRAMOLECULAR DISTANCES

ATOM	N1	ATOM	N2	DISTANCE	E.S.D.
C	1	C	2	1.421	0.0065
C	1	C	7	1.429	0.0067
C	1	O	1	1.288	0.0060
C	2	C	3	1.343	0.0074
C	3	C	4	1.514	0.0076
C	3	O	2	1.362	0.0059
C	5	C	6	1.511	0.0078
C	6	C	7	1.343	0.0069
C	6	O	2	1.355	0.0053
C	8	C	9	1.433	0.0086
C	8	C	14	1.434	0.0071
C	8	O	3	1.268	0.0060
C	9	C	10	1.350	0.0074
C	10	C	11	1.508	0.0083
C	10	O	4	1.354	0.0068
C	12	C	13	1.504	0.0097
C	13	C	14	1.341	0.0071
C	13	O	4	1.371	0.0083
C	15	C	16	1.438	0.0071
C	15	C	21	1.432	0.0067
C	15	O	5	1.261	0.0059
C	16	C	17	1.342	0.0084
C	17	C	18	1.508	0.0089
C	17	O	6	1.368	0.0076
C	19	C	20	1.516	0.0088
C	20	C	21	1.343	0.0079
C	20	O	6	1.370	0.0068
C	22	C	23	1.427	0.0072
C	22	C	28	1.420	0.0064
C	22	O	13	1.254	0.0061
C	23	C	24	1.343	0.0086
C	24	C	25	1.497	0.0101
C	24	O	14	1.351	0.0070
C	26	C	27	1.504	0.0085
C	27	C	28	1.343	0.0073
C	27	O	14	1.365	0.0060
C	29	C	30	1.421	0.0081
C	29	C	35	1.420	0.0089
C	29	O	15	1.264	0.0064
C	30	C	31	1.336	0.0074
C	31	C	32	1.502	0.0098
C	31	O	16	1.349	0.0075
C	33	C	34	1.510	0.0111
C	34	C	35	1.346	0.0089
C	34	O	16	1.358	0.0098
C	36	C	37	1.435	0.0067
C	36	C	42	1.419	0.0071
C	36	O	17	1.247	0.0061
C	37	C	38	1.343	0.0074
C	38	C	39	1.513	0.0073
C	38	O	18	1.352	0.0062
C	40	C	41	1.494	0.0083
C	41	C	42	1.342	0.0070
C	41	O	18	1.347	0.0057
N	1	O	7	1.226	0.0069
N	1	O	8	1.213	0.0061
N	1	O	9	1.215	0.0076
N	2	O	10	1.220	0.0073
N	2	O	11	1.239	0.0071
N	2	O	12	1.215	0.0075
N	3	O	19	1.206	0.0076
N	3	O	20	1.234	0.0081
N	3	O	21	1.239	0.0073
N	4	O	22	1.217	0.0070
N	4	O	23	1.218	0.0064
N	4	O	24	1.208	0.0075

DIRECTION COSINES

L	M	N
0.28448	0.26021	-0.95679
0.64560	0.28647	0.70791
-0.90450	-0.30050	0.30254
0.91969	0.32331	-0.22279
0.38377	0.29713	-0.91831
0.65095	0.27523	0.70747
-0.74615	-0.30901	-0.58972
-0.90424	-0.29828	0.30559
0.28639	0.86227	-0.95609
0.89585	-0.29023	0.33649
-0.67920	0.12804	0.72270
-0.23343	0.13565	-0.96287
0.18670	-0.13922	0.97250
0.87803	-0.27693	0.39035
-0.70378	0.16805	0.69026
0.59722	-0.11112	-0.79434
-0.22640	0.12711	-0.96569
0.91369	-0.28400	0.29072
-0.59148	-0.38150	-0.71036
-0.35742	-0.83241	0.93338
0.89737	0.35200	-0.26613
-0.90460	-0.34409	0.25159
-0.69122	-0.40136	-0.60094
-0.33810	-0.02560	0.94076
0.43651	0.85955	-0.89773
0.89834	0.39842	-0.20137
-0.58558	-0.34434	-0.73384
0.61167	-0.15911	-0.77495
0.37214	-0.27334	0.88702
-0.89518	0.43391	-0.10186
0.89922	-0.42478	0.10472
0.66048	-0.20936	-0.72114
0.38796	-0.27670	0.91027
-0.42649	0.38809	-0.85040
-0.89800	0.43098	-0.08862
0.55801	-0.14968	-0.81624
0.66083	0.28877	0.69276
-0.91984	-0.17928	0.34897
0.21161	-0.15589	-0.96484
-0.24333	0.13013	0.96117
0.58253	0.29724	0.75651
-0.94349	-0.16374	0.28814
0.85609	0.11428	-0.50405
0.23081	-0.14763	-0.96173
0.70528	0.38849	0.63838
-0.25688	0.13532	-0.95716
-0.72377	0.25860	0.63976
0.88847	-0.32657	0.32245
-0.98175	0.32969	-0.27956
-0.31934	0.14657	-0.93624
-0.68045	0.24903	0.68918
0.77041	-0.26861	-0.57820
0.88929	-0.33582	0.31047
-0.20648	0.11588	-0.97156
-0.49978	0.22438	0.83661
0.14974	0.73781	-0.65819
0.32272	-0.93004	-0.17570
-0.33004	-0.16364	0.92968
0.45385	-0.76645	-0.45451
-0.86922	0.87248	-0.48371
0.40527	-0.39037	0.82666
-0.86044	0.97432	-0.21690
-0.38456	-0.64074	-0.66450
0.49990	0.83664	0.86531
-0.13196	0.85223	-0.50625
-0.31482	-0.89127	-0.32638

TABLE 4. INTER-BOND ANGLES

CADMIUM DINITRATO TRIS(2,6-DIMETHYL-GAMMA-PYRONE)

	N1		N2		N3		ANGLE	E.S.D.
ANGLES INVOLVING ATOM CD 1								
	O	1	CD	1	O	3	97.24	0.139
	O	1	CD	1	O	5	165.73	0.137
	O	1	CD	1	O	7	88.10	0.147
	O	1	CD	1	O	8	85.83	0.135
	O	1	CD	1	O	10	91.83	0.156
	O	1	CD	1	O	11	92.47	0.154
	O	3	CD	1	O	5	101.81	0.145
	O	3	CD	1	O	7	88.95	0.155
	O	3	CD	1	O	8	140.21	0.144
	O	3	CD	1	O	10	79.22	0.164
	O	3	CD	1	O	11	130.84	0.162
	O	5	CD	1	O	7	83.67	0.153
	O	5	CD	1	O	8	79.44	0.141
	O	5	CD	1	O	10	98.73	0.161
	O	5	CD	1	O	11	86.12	0.159
	O	7	CD	1	O	8	51.39	0.151
	O	7	CD	1	O	10	168.17	0.170
	O	7	CD	1	O	11	140.18	0.169
	O	8	CD	1	O	10	140.41	0.160
	O	8	CD	1	O	11	88.92	0.158
	O	10	CD	1	O	11	51.65	0.176
ANGLES INVOLVING ATOM CD 2								
	O	13	CD	2	O	15	101.61	0.151
	O	13	CD	2	O	17	166.66	0.142
	O	13	CD	2	O	19	99.73	0.156
	O	13	CD	2	O	21	87.20	0.148
	O	13	CD	2	O	22	85.44	0.164
	O	13	CD	2	O	23	80.47	0.142
	O	15	CD	2	O	17	91.51	0.149
	O	15	CD	2	O	19	80.92	0.162
	O	15	CD	2	O	21	132.23	0.155
	O	15	CD	2	O	22	87.51	0.170
	O	15	CD	2	O	23	138.55	0.150
	O	17	CD	2	O	19	84.58	0.154
	O	17	CD	2	O	21	85.71	0.146
	O	17	CD	2	O	22	92.82	0.162
	O	17	CD	2	O	23	88.13	0.140
	O	19	CD	2	O	21	51.32	0.159
	O	19	CD	2	O	22	168.06	0.175
	O	19	CD	2	O	23	140.11	0.155
	O	21	CD	2	O	22	140.23	0.168
	O	21	CD	2	O	23	89.09	0.147
	O	22	CD	2	O	23	51.15	0.163

TABLE 4 (contd.)

ANGLES INVOLVING ATOM	C 1											
	C	2	C	1	C	7	118.45	0.426	C	2	C	7	2.448
	C	2	C	1	O	1	124.39	0.435	C	2	O	1	2.390
	C	7	C	1	O	1	117.12	0.420	C	7	O	1	2.313
ANGLES INVOLVING ATOM	C 2											
	C	1	C	2	C	3	119.62	0.460	C	1	C	3	2.389
ANGLES INVOLVING ATOM	C 3											
	C	2	C	3	C	4	126.08	0.506	C	2	C	4	2.547
	C	2	C	3	O	2	122.01	0.453	C	2	O	2	2.365
	C	4	C	3	O	2	111.91	0.460	C	4	O	2	2.384
ANGLES INVOLVING ATOM	C 4											
	NONE											
ANGLES INVOLVING ATOM	C 5											
	NONE											
ANGLES INVOLVING ATOM	C 6											
	C	5	C	6	C	7	125.92	0.467	C	5	C	7	2.544
	C	5	C	6	O	2	109.32	0.422	C	5	O	2	2.340
	C	7	C	6	O	2	124.73	0.430	C	7	O	2	2.391
ANGLES INVOLVING ATOM	C 7											
	C	1	C	7	C	6	116.93	0.436	C	1	C	6	2.363
ANGLES INVOLVING ATOM	C 8											
	C	9	C	8	C	14	113.73	0.401	C	9	C	14	2.401
	C	9	C	8	O	3	124.92	0.512	C	9	O	3	2.396
	C	14	C	8	O	3	121.33	0.496	C	14	O	3	2.356
ANGLES INVOLVING ATOM	C 9											
	C	8	C	9	C	10	122.34	0.522	C	8	C	10	2.439
ANGLES INVOLVING ATOM	C 10											
	C	9	C	10	C	11	125.60	0.513	C	9	C	11	2.544
	C	9	C	10	O	4	121.10	0.495	C	9	O	4	2.355
	C	11	C	10	O	4	113.27	0.463	C	11	O	4	2.392
ANGLES INVOLVING ATOM	C 11											
	NONE											
ANGLES INVOLVING ATOM	C 12											
	NONE											
ANGLES INVOLVING ATOM	C 13											
	C	12	C	13	C	14	128.15	0.504	C	12	C	14	2.560
	C	12	C	13	O	4	110.26	0.532	C	12	O	4	2.360
	C	14	C	13	O	4	121.59	0.532	C	14	O	4	2.368
ANGLES INVOLVING ATOM	C 14											
	C	8	C	14	C	13	121.86	0.506	C	8	C	13	2.426
ANGLES INVOLVING ATOM	C 15											
	C	16	C	15	C	21	116.96	0.436	C	16	C	21	2.420
	C	16	C	15	O	5	110.43	0.451	C	16	O	5	2.314
	C	21	C	15	O	5	125.49	0.451	C	21	O	5	2.395
ANGLES INVOLVING ATOM	C 16											
	C	15	C	16	C	17	119.10	0.505	C	15	C	17	2.391
ANGLES INVOLVING ATOM	C 17											
	C	16	C	17	C	18	127.75	0.560	C	16	C	18	2.560
	C	16	C	17	O	6	123.46	0.543	C	16	O	6	2.380
	C	18	C	17	O	6	108.75	0.490	C	18	O	6	2.333
ANGLES INVOLVING ATOM	C 18											
	NONE											
ANGLES INVOLVING ATOM	C 19											
	NONE											
ANGLES INVOLVING ATOM	C 20											
	C	19	C	20	C	21	125.60	0.533	C	19	C	21	2.556
	C	19	C	20	O	6	112.50	0.492	C	19	O	6	2.401
	C	21	C	20	O	6	120.85	0.489	C	21	O	6	2.359
ANGLES INVOLVING ATOM	C 21											
	C	15	C	21	C	20	121.45	0.465	C	15	C	20	2.421
ANGLES INVOLVING ATOM	C 22											
	C	23	C	22	C	28	116.68	0.433	C	23	C	28	2.396
	C	23	C	22	O	13	122.52	0.452	C	23	O	13	2.352
	C	28	C	22	O	13	122.83	0.438	C	28	O	13	2.350

TABLE 4 (contd.)

	N1	N2	3	ANGLE	E.S.D.	N1	N3	DISTANCE
ANGLES INVOLVING ATOM C 23								
	C 22	C 23	C 24	122.44	0.520	C 22	C 24	2.428
ANGLES INVOLVING ATOM C 24								
	C 23	C 24	C 25	127.39	0.596	C 23	C 25	2.547
	C 23	C 24	O 14	119.33	0.527	C 23	O 14	2.325
	C 25	C 24	O 14	113.27	0.544	C 25	O 14	2.388
ANGLES INVOLVING ATOM C 25	NONE							
ANGLES INVOLVING ATOM C 26	NONE							
ANGLES INVOLVING ATOM C 27								
	C 26	C 27	C 28	126.24	0.489	C 26	C 28	2.548
	C 26	C 27	O 14	114.21	0.453	C 26	O 14	2.410
	C 28	C 27	O 14	119.55	0.437	C 28	O 14	2.348
ANGLES INVOLVING ATOM C 28								
	C 22	C 28	C 27	122.05	0.458	C 22	C 27	2.417
ANGLES INVOLVING ATOM C 29								
	C 30	C 29	C 35	114.78	0.529	C 30	C 35	2.392
	C 30	C 29	O 15	125.00	0.548	C 30	O 15	2.382
	C 35	C 29	O 15	120.23	0.536	C 35	O 15	2.328
ANGLES INVOLVING ATOM C 30								
	C 29	C 30	C 31	122.86	0.559	C 29	C 31	2.421
ANGLES INVOLVING ATOM C 31								
	C 30	C 31	C 32	128.61	0.584	C 30	C 32	2.558
	C 30	C 31	O 16	119.03	0.520	C 30	O 16	2.313
	C 32	C 31	O 16	112.35	0.533	C 32	O 16	2.378
ANGLES INVOLVING ATOM C 32	NONE							
ANGLES INVOLVING ATOM C 33	NONE							
ANGLES INVOLVING ATOM C 34								
	C 33	C 34	C 35	131.72	0.673	C 33	C 35	2.607
	C 33	C 34	O 16	108.49	0.598	C 33	O 16	2.329
	C 35	C 34	O 16	119.78	0.627	C 35	O 16	2.339
ANGLES INVOLVING ATOM C 35								
	C 29	C 35	C 34	121.43	0.601	C 29	C 34	2.412
ANGLES INVOLVING ATOM C 36								
	C 37	C 36	C 42	113.08	0.435	C 37	C 42	2.381
	C 37	C 36	O 17	125.47	0.452	C 37	O 17	2.386
	C 42	C 36	O 17	121.41	0.450	C 42	O 17	2.326
ANGLES INVOLVING ATOM C 37								
	C 36	C 37	C 38	122.89	0.477	C 36	C 38	2.441
ANGLES INVOLVING ATOM C 38								
	C 37	C 38	C 39	126.73	0.498	C 37	C 39	2.553
	C 37	C 38	O 18	128.28	0.457	C 37	O 18	2.336
	C 39	C 38	O 18	113.84	0.450	C 39	O 18	2.391
ANGLES INVOLVING ATOM C 39	NONE							
ANGLES INVOLVING ATOM C 40	NONE							
ANGLES INVOLVING ATOM C 41								
	C 40	C 41	C 42	126.57	0.483	C 40	C 42	2.534
	C 40	C 41	O 18	111.81	0.433	C 40	O 18	2.354
	C 42	C 41	O 18	121.61	0.440	C 42	O 18	2.347
ANGLES INVOLVING ATOM C 42								
	C 36	C 42	C 41	122.13	0.475	C 36	C 41	2.416
ANGLES INVOLVING ATOM N 1								
	O 7	N 1	O 8	117.39	0.489	O 7	O 8	2.083
	O 7	N 1	O 9	121.12	0.554	O 7	O 9	2.125
	O 8	N 1	O 9	121.48	0.548	O 8	O 9	2.118
ANGLES INVOLVING ATOM N 2								
	O 10	N 2	O 11	116.55	0.535	O 10	O 11	2.092
	O 10	N 2	O 12	124.72	0.582	O 10	O 12	2.157
	O 11	N 2	O 12	118.78	0.565	O 11	O 12	2.111
ANGLES INVOLVING ATOM N 3								
	O 19	N 3	O 20	125.74	0.630	O 19	O 20	2.172
	O 19	N 3	O 21	117.07	0.566	O 19	O 21	2.086
	O 20	N 3	O 21	117.19	0.599	O 20	O 21	2.116

TABLE 4 (contd.)

			N1		N2		N3	ANGLE	E.S.D.	N1		N3		DISTANCE
ANGLES INVOLVING ATOM	N	4												
			O	22	N	4	O	23	118.22	0.515	O	22	O	23	2.NNN
			O	22	N	4	O	24	118.19	0.577	O	22	O	24	2.NNN
			O	23	N	4	O	24	123.56	0.567	O	23	O	24	2.137
ANGLES INVOLVING ATOM	O	1	NONE												
ANGLES INVOLVING ATOM	O	2	C	3	O	2	C	6	118.22	0.388	C	3	C	6	2.332
ANGLES INVOLVING ATOM	O	3	NONE												
ANGLES INVOLVING ATOM	O	4	C	13	O	4	C	13	119.35	0.449	C	10	C	13	2.352
ANGLES INVOLVING ATOM	O	5	NONE												
ANGLES INVOLVING ATOM	O	6	C	17	O	6	C	20	118.92	0.447	C	17	C	20	2.351
ANGLES INVOLVING ATOM	O	7	NONE												
ANGLES INVOLVING ATOM	O	8	NONE												
ANGLES INVOLVING ATOM	O	9	NONE												
ANGLES INVOLVING ATOM	O	10	NONE												
ANGLES INVOLVING ATOM	O	11	NONE												
ANGLES INVOLVING ATOM	O	12	NONE												
ANGLES INVOLVING ATOM	O	13	NONE												
ANGLES INVOLVING ATOM	O	14	C	24	O	14	C	27	121.99	0.428	C	24	C	27	2.375
ANGLES INVOLVING ATOM	O	15	NONE												
ANGLES INVOLVING ATOM	O	16	C	31	O	14	C	34	122.14	0.516	C	31	C	34	2.369
ANGLES INVOLVING ATOM	O	17	NONE												
ANGLES INVOLVING ATOM	O	18	C	38	O	18	C	41	120.02	0.398	C	38	C	41	2.337
ANGLES INVOLVING ATOM	O	19	NONE												
ANGLES INVOLVING ATOM	O	20	NONE												
ANGLES INVOLVING ATOM	O	21	NONE												
ANGLES INVOLVING ATOM	O	22	NONE												
ANGLES INVOLVING ATOM	O	23	NONE												
ANGLES INVOLVING ATOM	O	24	NONE												

TABLE 5. EQUATIONS OF MEAN PLANES

CADMIUM DINITRATO TRIS(2,6,GAMMA-PYRONE)

PLANE 1 IS (-0.3422)X + (0.9383)Y + (-0.0495)Z -(1.5814) = 0

CHI SQUARED = 11.0379

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	1	3.7119	3.3610	7.6112	0.307	0.0047
	C	2	3.3077	3.2775	8.9706	-0.301	0.0052
	C	3	2.0730	2.8434	9.2697	-0.301	0.0052
	O	2	1.1865	2.4686	8.3062	-0.302	0.0033
	C	6	1.5746	2.5530	7.0103	0.308	0.0049
	C	7	2.7894	2.9537	6.5997	-0.310	0.0049
					SUM OF P(I)	-0.2000	R.M.S. OF P(I) 0.006523
OTHER ATOMS							
	C	4	1.4920	2.6964	10.6598	-0.309	0.0060
	C	5	0.4468	2.8959	6.1189	0.000	0.0076
	O	1	4.8699	3.7478	7.2239	-0.308	0.0037

PLANE 2 IS (-0.2786)X + (-0.9576)Y + (-0.0726)Z -(-6.5542) = 0

CHI SQUARED = 17.6223

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	8	7.5985	4.2551	4.8999	0.000	0.0057
	C	9	6.3148	4.6710	4.4177	0.302	0.0055
	C	10	6.8627	4.8599	3.1046	-0.311	0.0051
	O	4	7.8150	4.6314	2.1698	0.308	0.0040
	C	13	8.2604	4.2420	2.5604	0.303	0.0058
	C	14	8.5722	4.0715	3.8639	-0.311	0.0053
					SUM OF P(I)	-0.3000	R.M.S. OF P(I) 0.000502
OTHER ATOMS							
	C	11	4.7302	5.2747	2.5158	0.000	0.0062
	C	12	9.1666	4.0749	1.3738	0.300	0.0085
	O	3	7.8945	4.0031	6.1207	0.302	0.0043

PLANE 3 IS (-0.4034)X + (0.9065)Y + (-0.1243)Z -(0.8752) = 0

CHI SQUARED = 29.6778

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	15	9.6912	5.6335	9.8967	-0.308	0.0051
	C	16	10.5371	6.1791	10.1126	0.310	0.0055
	C	17	11.7913	6.6489	9.7749	-0.311	0.0059
	O	6	12.2112	6.6757	8.4950	-0.305	0.0039
	C	20	11.4892	6.2041	7.4899	0.315	0.0055
	C	21	10.2029	5.6799	7.7603	-0.307	0.0040
					SUM OF P(I)	0.0000	R.M.S. OF P(I) 0.011095
OTHER ATOMS							
	C	18	12.7934	7.2461	10.6809	0.304	0.0063
	C	19	12.0712	6.2944	6.1286	-0.000	0.0079
	O	5	8.5594	5.1895	9.4323	0.303	0.0045

PLANE 4 IS (-0.4121)X + (-0.9027)Y + (-0.1238)Z -(-5.7129) = 0

CHI SQUARED = 18.3999

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	22	2.9375	2.1495	20.8841	-0.312	0.0040
	C	23	2.0647	2.3764	21.9998	0.305	0.0055
	C	24	0.8574	2.9469	21.7692	0.305	0.0063
	O	14	0.4414	3.3207	20.5394	-0.308	0.0037
	C	27	1.2031	3.1165	19.4292	-0.300	0.0049
	C	28	2.4089	2.5378	19.5442	0.310	0.0050
					SUM OF P(I)	-0.3000	R.M.S. OF P(I) 0.008171
OTHER ATOMS							
	C	25	-0.1313	3.2683	22.8489	-0.303	0.0091
	C	26	0.5617	3.5798	18.1463	0.304	0.0076
	O	13	4.0000	1.6054	20.9318	0.300	0.0044

PLANE 5 IS (-0.2345)X + (0.9527)Y + (-0.1902)Z -(-3.2002) = 0

CHI SQUARED = 15.9926

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	29	4.9848	1.1215	16.2804	-0.314	0.0062
	C	30	4.0452	0.7112	15.2762	0.304	0.0065
	C	31	4.3782	0.5374	13.9925	0.307	0.0056
	O	16	5.6430	0.7503	13.6038	-0.299	0.0040
	C	34	6.6000	1.1773	14.4700	-0.302	0.0062
	C	35	6.2982	1.3759	15.7649	0.313	0.0064
					SUM OF P(I)	-0.3000	R.M.S. OF P(I) 0.009033
OTHER ATOMS							
	C	32	3.4951	0.8989	12.8560	0.295	0.0093
	C	33	7.8934	1.3497	13.7097	0.301	0.0094
	O	15	4.7165	1.3185	17.4881	0.303	0.0040

TABLE 5 (contd.)

PLANE 6 IS (-3.3584)X + (-0.9342)Y + (-0.0277)Z -(-3.5864) = 0

CHI SQUARED = 15.0100

ATOMS IN PLANE	ATOM NO.	X	Y	Z	P	ESD(P)
	C 36	8.7907	-0.0129	18.9110	-0.305	0.0040
	C 37	9.1501	-2.2072	20.2055	0.309	0.0050
	C 38	10.3609	-0.6498	20.6609	-0.010	0.0050
	O 10	11.2007	-0.9864	19.7294	0.300	0.0035
	C 41	11.0105	-0.0303	18.4206	-0.304	0.0049
	C 42	9.0175	-0.3790	18.0041	0.303	0.0053
			SUM OF P(I)	0.3000	R.M.S. OF P(I)	0.007415

OTHER ATOMS

C 39	10.0519	-0.0715	22.0770	-0.311	0.0092
C 40	12.1610	-1.2317	17.5566	-0.300	0.0060
O 17	7.6029	0.3947	18.5097	0.012	0.0044

PLANE 7 IS (-0.8647)X + (-0.2203)Y + (-0.4514)Z -(0.0000000) = 0

CHI SQUARED = 0.0000

ATOMS IN PLANE	ATOM NO.	X	Y	Z	P	ESD(P)
	O 7	6.4155	6.5467	7.6487	0.300	0.0051
	O 8	5.6214	5.9267	9.4723	0.300	0.0043
	O 9	5.4110	7.9514	8.0074	0.300	0.0073
			SUM OF P(I)	0.3000	R.M.S. OF P(I)	0.000000

OTHER ATOMS

N 1	5.0030	6.0216	8.6740	0.306	0.0053

PLANE 8 IS (-2.0016)X + (-0.2001)Y + (+0.3739)Z -(0.0000000) = 0

CHI SQUARED = 0.0000

ATOMS IN PLANE	ATOM NO.	X	Y	Z	P	ESD(P)
	O 10	7.5614	1.9710	8.2299	0.300	0.0050
	O 11	6.5962	2.7220	9.9276	0.300	0.0055
	O 12	7.2420	0.7121	9.9570	0.300	0.0060
			SUM OF P(I)	0.3000	R.M.S. OF P(I)	0.000000

OTHER ATOMS

N 2	7.1507	1.7721	9.3643	-0.311	0.0056

PLANE 9 IS (1.0060)X + (-0.0501)Y + (-0.1609)Z -(-4.2437) = 0

CHI SQUARED = 0.0000

ATOMS IN PLANE	ATOM NO.	X	Y	Z	P	ESD(P)
	O 19	5.0401	-1.2761	19.0449	-0.300	0.0050
	O 20	5.0115	-2.9492	20.3138	-0.300	0.0073
	O 21	6.0133	-0.9533	20.0693	-0.300	0.0047
			SUM OF P(I)	-0.3000	R.M.S. OF P(I)	0.000000

OTHER ATOMS

N 3	5.5370	-1.7470	20.0462	-0.292	0.0060

PLANE 10 IS (0.8649)X + (-0.1405)Y + (-0.4820)Z -(-4.4257) = 0

CHI SQUARED = 0.0000

ATOMS IN PLANE	ATOM NO.	X	Y	Z	P	ESD(P)
	O 22	6.2714	3.2985	19.4776	0.300	0.0050
	O 23	7.0404	2.2975	21.1468	0.300	0.0044
	O 24	7.2599	4.4116	20.9247	-0.300	0.0079
			SUM OF P(I)	-0.3000	R.M.S. OF P(I)	0.000000

OTHER ATOMS

N 4	6.0797	3.1351	20.5304	0.312	0.0053

PLANE 1	PLANE 2	DIHEDRAL ANGLE (DEGREES)
1	2	143.897
1	3	5.836
2	3	138.309
4	5	137.637
4	6	6.822
5	6	143.498
7	8	5.986
9	10	5.456

TABLE 6. INTERMOLECULAR DISTANCES

CADMIUM DINITRATO TRIS(2,4,GAMMA-PYRONE)

INTERMOLECULAR DISTANCES, WITH THE COORDINATES USED IN THE COMPUTATION

ATOM	N1	X/A	Y/B	Z/C	ATOM	N2	X/A	Y/B	Z/C	DISTANCE	E.S.D.
C	40	0.68752	0.09143	0.76422		1	0.78250	0.78357	0.66069	3.647	0.0082
C	40	0.68752	0.09143	0.76422		1	0.71750	1.28357	0.83131	3.892	0.0081
C	41	0.63183	0.92681	0.08183		1	0.71750	1.28357	0.83131	3.682	0.0068
C	18	0.64984	0.91385	0.85888		1	0.71750	1.28357	0.83131	3.618	0.0058
C	18	0.64984	0.91385	0.85888		2	0.79874	0.71111	0.68952	3.847	0.0081
C	38	0.68553	0.94272	0.89935		2	0.70126	1.21111	0.89048	3.578	0.0073
C	39	0.63528	0.92318	0.96099		2	0.70126	1.21111	0.89048	3.988	0.0093
C	18	0.64984	0.91385	0.85888		2	0.70126	1.21111	0.89048	3.587	0.0063
C	11	0.29315	0.46511	0.10951		2	0.29874	0.78889	0.10952	3.778	0.0081
C	37	0.54175	0.98174	0.08301		3	0.63854	1.24937	0.90350	3.583	0.0077
C	38	0.68553	0.94272	0.89935		3	0.63854	1.24937	0.90350	3.537	0.0072
C	39	0.63528	0.92318	0.96099		3	0.63854	1.24937	0.90350	3.932	0.0096
C	18	0.64984	0.91385	0.85888		3	0.63854	1.24937	0.90350	3.962	0.0063
C	19	0.32352	0.42829	0.13514		3	0.36146	0.75063	0.09650	3.846	0.0072
C	11	0.29315	0.46511	0.10951		3	0.36146	0.75063	0.09650	3.882	0.0081
C	4	0.36954	0.40823	0.09445		3	0.36146	0.75063	0.09650	3.888	0.0064
C	4	0.42497	0.29397	0.89367		4	0.61328	0.26233	0.96401	3.909	0.0089
C	23	0.43536	0.20251	0.92050		4	0.61328	0.26233	0.96401	3.598	0.0086
C	24	0.44594	0.30886	0.91083		4	0.61328	0.26233	0.96401	3.601	0.0107
C	12	0.47786	0.35918	0.05988		4	0.61328	0.26233	-0.03599	3.703	0.0105
C	37	0.54175	0.98174	0.08301		4	0.61328	1.26233	0.96401	3.898	0.0099
C	38	0.68553	0.94272	0.89935		4	0.61328	1.26233	0.96401	3.919	0.0098
C	39	0.63528	0.92318	0.96099		4	0.61328	1.26233	0.96401	3.872	0.0123
C	4	0.36954	0.40823	0.09445		4	0.38672	0.73767	-0.03599	3.994	0.0095
C	3	0.35401	0.84601	0.87259		4	0.38672	0.73767	1.03599	3.969	0.0085
C	28	0.35877	0.74004	0.88424		4	0.38672	0.73767	1.03599	3.588	0.0092
C	21	0.38148	0.91597	0.98842		4	0.38672	0.73767	1.03599	3.562	0.0081
C	4	0.42697	0.29397	0.89367		5	0.54388	0.31614	0.76635	3.854	0.0088
C	17	0.45956	0.03475	0.08571		5	0.54388	0.31614	0.76635	3.725	0.0086
C	22	0.38999	0.29984	0.84784		5	0.54388	0.31614	0.76635	3.637	0.0079
C	36	0.51011	0.99886	0.82321		5	0.54388	1.31614	0.76635	3.874	0.0090
C	8	0.48891	0.37586	0.21329		5	0.45612	0.68386	0.23365	3.643	0.0096
C	9	0.34188	0.41172	0.19230		5	0.45612	0.68386	0.23365	3.898	0.0092
C	14	0.45565	0.35888	0.16819		5	0.45612	0.68386	0.23365	3.983	0.0092
C	19	0.64397	0.55482	0.26677		5	0.45612	0.68386	0.23365	3.958	0.0099
C	20	0.61443	0.54686	0.32603		5	0.45612	0.68386	0.23365	3.935	0.0086
C	21	0.55388	0.58865	0.33788		5	0.45612	0.68386	0.23365	3.606	0.0083
C	3	0.42034	0.35998	0.26643		5	0.45612	0.68386	0.23365	3.797	0.0088
C	7	0.35719	0.57706	0.33294		5	0.45612	0.68386	0.23365	3.388	0.0088
C	17	0.45956	0.03475	0.08571		6	0.60513	0.27497	0.08515	3.922	0.0064
C	36	0.51011	0.99886	0.82321		6	0.60513	1.27497	0.08515	3.594	0.0068
C	41	0.63183	0.92681	0.08183		6	0.60513	1.27497	0.08515	3.983	0.0070
C	42	0.56883	0.96652	0.78378		6	0.60513	1.27497	0.08515	3.690	0.0072
C	9	0.34188	0.41172	0.19230		6	0.39487	0.72503	0.19485	3.704	0.0074
C	19	0.32352	0.42829	0.13514		6	0.39487	0.72503	0.19485	3.858	0.0070
C	7	0.35719	0.57706	0.33294		6	0.39487	0.72503	0.19485	3.718	0.0063
C	40	0.68752	0.09143	0.76422		7	0.83356	0.73965	0.71272	3.588	0.0086
C	41	0.63183	0.92681	0.08183		7	0.66644	1.23965	0.78728	3.634	0.0070
C	42	0.56883	0.96652	0.78378		7	0.66644	1.23965	0.78728	3.636	0.0073
C	9	0.34188	0.41172	0.19230		7	0.33356	0.76035	0.21272	3.987	0.0073
C	7	0.35719	0.57706	0.33294		7	0.33356	0.76035	0.21272	3.468	0.0065
C	40	0.68752	0.09143	0.76422		8	0.59189	0.62494	0.78671	3.689	0.0090
C	41	0.63183	0.92681	0.08183		8	0.59189	0.62494	0.78671	3.523	0.0075
C	42	0.56883	0.96652	0.78378		8	0.59189	0.62494	0.78671	3.901	0.0078
C	18	0.64984	0.91385	0.85888		8	0.59189	0.62494	0.78671	3.806	0.0066
C	40	0.68752	0.09143	0.76422		8	0.65988	0.58028	0.88778	3.634	0.0086
C	41	0.63183	0.92681	0.08183		8	0.65988	0.58028	0.88778	3.888	0.0074
C	18	0.64984	0.91385	0.85888		8	0.65988	0.58028	0.88778	3.875	0.0065
C	2	0.88958	0.21759	0.36156		8	0.15988	-0.08028	0.38778	3.951	0.0066
C	18	0.64984	0.91385	0.85888		8	0.67648	0.57171	0.86486	3.988	0.0061
C	9	0.33955	0.70887	0.38686		8	0.17648	0.92829	0.36486	3.659	0.0088
C	2	0.88958	0.21759	0.36156		8	0.17648	-0.07171	0.36486	3.688	0.0063
C	9	0.33955	0.70887	0.38686		9	0.24685	0.96511	0.39049	3.238	0.0088
C	32	0.22415	0.08881	0.55961		9	0.24685	-0.03489	0.39049	3.977	0.0098
C	2	0.48139	0.15628	0.48762		9	0.24685	-0.03489	0.39049	3.696	0.0088
C	12	0.48773	0.86277	0.43328		9	0.24685	-0.03489	0.39049	3.392	0.0088
C	4	0.42497	0.29397	0.89367		10	0.47786	0.35918	1.05988	3.964	0.0087
C	23	0.43536	0.20251	0.92050		10	0.47786	0.35918	1.05988	3.712	0.0086
C	24	0.44594	0.30886	0.91083		10	0.47786	0.35918	1.05988	3.461	0.0101
C	38	0.68553	0.94272	0.89935		11	0.32214	0.64082	0.94020	3.927	0.0097
C	39	0.63528	0.92318	0.96099		11	0.32214	0.64082	0.94020	3.893	0.0114
C	28	0.35877	0.74004	0.88424		11	0.32214	0.64082	0.94020	3.522	0.0108
C	24	0.44594	0.30886	0.91083		11	0.32214	0.64082	0.94020	3.267	0.0103
C	38	0.68553	0.94272	0.89935		12	0.56444	0.62689	0.88820	3.685	0.0078
C	39	0.63528	0.92318	0.96099		12	0.56444	0.62689	0.88820	3.969	0.0097
C	18	0.64984	0.91385	0.85888		12	0.56444	0.62689	0.88820	3.734	0.0078
C	24	0.44594	0.30886	0.91083		12	0.56444	0.62689	0.88820	3.597	0.0092
C	2	0.88958	0.21759	0.36156		12	0.06444	-0.12689	0.38820	3.982	0.0067
C	38	0.68553	0.94272	0.89935		13	0.54435	0.64112	0.03181	3.918	0.0071
C	41	0.63183	0.92681	0.08183		13	0.54435	0.64112	0.03181	3.735	0.0072
C	42	0.56883	0.96652	0.78378		13	0.54435	0.64112	0.03181	3.890	0.0075
C	18	0.64984	0.91385	0.85888		13	0.54435	0.64112	0.03181	3.738	0.0064
C	24	0.44594	0.30886	0.91083		13	0.54435	0.64112	0.03181	3.948	0.0083
C	33	0.10111	0.20948	0.95371		14	0.03121	0.00344	0.09597	3.893	0.0076
C	34	0.11831	0.25975	0.94759		14	0.03121	0.00344	0.09597	3.529	0.0079
C	25	0.07894	0.28738	0.99459		14	0.03121	0.00344	0.09597	3.987	0.0099
C	27	0.12818	0.27478	0.04556		14	0.03121	0.00344	0.09597	3.825	0.0078
C	14	0.09265	0.29278	0.89486		14	0.03121	0.00344	0.09597	3.492	0.0063
C	33	0.45418	0.11897	0.59677		15	0.42167	0.45535	0.55981	3.951	0.0109
C	5	0.47393	0.45743	0.41858		15	0.42167	0.45535	0.55981	3.645	0.0061
C	8	0.32248	0.52241	0.41232		15	0.42167	0.45535	0.55981	3.866	0.0064
C	11	0.37427	0.23993	0.43214		15	0.42167	0.45535	0.55981	3.886	0.0078
C	22	0.22249	0.18947	0.90558		16	0.07833	-0.04465	0.94019	3.971	0.0073
C	23	0.19111	0.20948	0.95371		16	0.07833	-0.04465	0.94019	3.506	0.0088
C	24	0.11831	0.25975	0.94759		16	0.07833	-0.04465	0.94019	3.541	0.0084
C	25	0.07894	0.28738	0.99459		16	0.07833	-0.04465	0.94019	3.975	0.0104
C	14	0.09265	0.29278	0.89486		16	0.07833	-0.04465	0.94019	3.906	0.0065
C	33	0.45418	0.11897	0.59677		17	0.36914	0.41464	0.57451	3.835	0.0108
C	34	0.38996	0.18377	0.62998		17	0.36914	0.41464	0.57451	3.782	0.0085
C	8	0.32248	0.52241	0.41232		17	0.36914	0.41464	0.57451	3.951	0.0076
C	11	0.37427	0.23993	0.43214		17	0.36914	0.41464	0.57451	3.856	0.0088
C	16	0.33757	0.06684	0.59216		17	0.36914	0.41464	0.57451	3.994	0.0075
C	22	0.22248	0.18947	0.90558		17	0.13986	-0.08536	0.92549	3.548	0.0078
C	23	0.19111	0.20948	0.95371		17	0.13986	-0.08536	0.92549	3.498	0.0079

TABLE 6 (contd.)

ATOM	N1	X/A	Y/B	Z/C	ATOM	N2	X/A	Y/B	Z/C	DISTANCE	E.S.D.
C	24	0.11831	0.25975	0.94759	C	17	0.13984	-0.08536	0.92549	3.974	0.0086
C	13	0.20878	0.14151	0.91114	C	17	0.13984	-0.08536	0.92549	3.782	0.0074
C	14	0.38994	0.10377	0.62990	C	18	0.30326	0.36130	0.53507	3.955	0.0092
C	9	0.32240	0.52241	0.41232	C	18	0.30326	0.36130	0.53507	3.606	0.0076
C	11	0.37427	0.23993	0.43214	C	18	0.30326	0.36130	0.53507	3.136	0.0081
C	16	0.33757	0.06684	0.59216	C	18	0.30326	0.36130	0.53507	3.635	0.0079
N	3	0.35481	0.84681	0.87259	C	18	0.19674	0.86130	0.96493	3.832	0.0086
C	20	0.35877	0.74004	0.88424	C	18	0.19674	0.86130	0.96493	3.995	0.0094
C	21	0.38140	0.91597	0.99842	C	18	0.19674	0.86130	0.96493	3.935	0.0076
C	23	0.10111	0.20948	0.95371	C	18	0.19674	-0.13870	0.96493	3.969	0.0083
C	13	0.20878	0.14151	0.91114	C	18	0.19674	-0.13870	0.96493	3.815	0.0076
C	35	0.37033	0.12128	0.68623	C	19	0.35603	0.44518	0.73323	3.863	0.0101
C	15	0.30293	0.11622	0.76889	C	19	0.35603	0.44518	0.73323	3.934	0.0092
C	22	0.30999	0.29884	0.84784	C	19	0.35603	0.44518	0.73323	3.294	0.0088
C	19	0.32538	0.88752	0.82918	C	19	0.14397	0.90518	0.76677	3.765	0.0090
C	28	0.19883	0.22369	0.85074	C	19	0.14397	-0.05482	0.76677	3.783	0.0090
C	30	0.26078	0.86269	0.64496	C	19	0.14397	-0.05482	0.76677	3.618	0.0095
C	15	0.30293	0.11622	0.76889	C	19	0.14397	-0.05482	0.76677	3.658	0.0090
C	35	0.37033	0.12128	0.68623	C	20	0.30557	0.45314	0.67397	3.779	0.0083
C	22	0.22240	0.18947	0.99558	C	20	0.11443	-0.04686	0.82603	3.797	0.0073
C	27	0.12818	0.27478	0.84556	C	20	0.11443	-0.04686	0.82603	3.683	0.0072
C	28	0.19883	0.22369	0.85074	C	20	0.11443	-0.04686	0.82603	3.439	0.0074
C	26	0.09872	0.31554	0.78989	C	21	0.05308	-0.08065	0.83788	3.837	0.0090
C	27	0.12818	0.27478	0.84556	C	21	0.05308	-0.08065	0.83788	3.646	0.0068
C	28	0.19883	0.22369	0.85074	C	21	0.05308	-0.08065	0.83788	3.685	0.0072
C	14	0.89265	0.29278	0.89486	C	21	0.05308	-0.08065	0.83788	3.635	0.0059
C	6	0.65925	0.58843	0.36978	C	22	0.72240	0.31953	0.40558	3.662	0.0062
C	32	0.22415	0.89881	0.55961	C	22	0.27760	-0.31953	0.59442	3.038	0.0105
C	6	0.65925	0.58843	0.36978	C	23	0.60111	0.29852	0.45371	3.982	0.0066
O	8	0.32240	0.52241	0.41232	C	23	0.31889	0.70948	0.54629	3.749	0.0066
O	9	0.38955	0.70887	0.38686	C	23	0.31889	0.70948	0.54629	3.664	0.0085
O	32	0.22415	0.89881	0.55961	C	23	0.31889	-0.29052	0.54629	3.074	0.0110
O	5	0.47393	0.45743	0.41058	C	24	0.68831	0.24025	0.44759	3.702	0.0074
O	16	0.33757	0.06684	0.59216	C	24	0.38169	-0.24025	0.55241	3.716	0.0079
O	33	0.45418	0.11097	0.59677	C	25	0.57894	0.21262	0.49459	3.534	0.0114
O	2	0.49139	0.15628	0.40762	C	25	0.57894	0.21262	0.49459	3.787	0.0104
O	5	0.47393	0.45743	0.41058	C	25	0.57894	0.21262	0.49459	3.849	0.0096
O	11	0.37427	0.23993	0.43214	C	25	0.57894	0.21262	0.49459	3.995	0.0106
O	12	0.48773	0.86277	0.43320	C	25	0.57894	0.21262	0.49459	3.781	0.0111
O	9	0.38955	0.70887	0.38686	C	25	0.42906	0.78738	0.50541	3.597	0.0107
O	12	0.48773	0.86277	0.43320	C	25	0.42906	-0.21262	0.50541	3.550	0.0105
O	3	0.42834	0.35998	0.26643	C	26	0.59072	0.18446	0.28989	3.732	0.0084
O	10	0.41832	0.17388	0.35824	C	26	0.59072	0.18446	0.28989	3.786	0.0091
C	36	0.51811	0.99886	0.82321	C	26	0.40928	0.81554	0.71011	3.851	0.0086
C	12	0.96883	0.96652	0.78378	C	26	0.40928	0.81554	0.71011	3.810	0.0090
C	3	0.35481	0.84681	0.87259	C	26	0.40928	0.81554	0.71011	3.973	0.0093
C	19	0.32538	0.88752	0.82918	C	26	0.40928	0.81554	0.71011	3.378	0.0086
C	29	0.31259	0.99885	0.78788	C	26	0.40928	-0.18446	0.71011	3.719	0.0097
C	34	0.38996	0.10377	0.62990	C	26	0.40928	-0.18446	0.71011	3.762	0.0098
C	35	0.37033	0.12128	0.68623	C	26	0.40928	-0.18446	0.71011	3.557	0.0100
C	17	0.45956	0.83475	0.88571	C	26	0.40928	-0.18446	0.71011	3.428	0.0095
C	30	0.26078	0.86269	0.64496	C	27	0.37182	-0.22530	0.65444	3.928	0.0082
C	31	0.27319	0.84737	0.68908	C	27	0.37182	-0.22530	0.65444	3.748	0.0074
C	34	0.38996	0.10377	0.62990	C	27	0.37182	-0.22530	0.65444	3.796	0.0078
C	35	0.37033	0.12128	0.68623	C	27	0.37182	-0.22530	0.65444	4.000	0.0088
O	16	0.33757	0.06684	0.59216	C	27	0.37182	-0.22530	0.65444	3.655	0.0067
O	6	0.65925	0.58843	0.36978	C	28	0.69803	0.27831	0.35074	3.825	0.0062
C	30	0.26078	0.86269	0.64496	C	28	0.30917	-0.27831	0.64926	3.981	0.0082
C	31	0.27319	0.84737	0.68908	C	28	0.30917	-0.27831	0.64926	3.839	0.0074
N	3	0.35481	0.84681	0.87259	C	30	0.23922	0.56269	0.83504	3.971	0.0087
O	20	0.35877	0.74004	0.88424	C	30	0.23922	0.56269	0.83504	3.217	0.0093
O	14	0.89265	0.29278	0.89486	C	31	0.22681	0.54737	0.89092	3.898	0.0068
O	20	0.35877	0.74004	0.88424	C	31	0.22681	0.54737	0.89092	3.375	0.0088
N	4	0.42497	0.29397	0.89367	C	32	0.27505	0.50001	0.94039	3.976	0.0106
O	20	0.35877	0.74004	0.88424	C	32	0.27505	0.50001	0.94039	3.384	0.0111
O	24	0.44594	0.38886	0.91083	C	32	0.27505	0.50001	0.94039	3.666	0.0122
O	12	0.48773	0.86277	0.43320	C	33	0.54590	-0.11097	0.40323	3.483	0.0103
O	14	0.89265	0.29278	0.89486	C	33	0.04590	0.61097	0.90323	3.020	0.0101
O	6	0.65925	0.58843	0.36978	C	34	0.61004	0.89623	0.37010	3.620	0.0073
O	14	0.89265	0.29278	0.89486	C	34	0.11004	0.60377	0.87010	3.591	0.0071
O	6	0.65925	0.58843	0.36978	C	35	0.62167	0.87872	0.31377	3.994	0.0074
O	2	0.88958	0.21759	0.36156	C	36	0.01811	0.50114	0.32321	3.589	0.0059
O	2	0.88958	0.21759	0.36156	C	37	0.84175	0.51826	0.38301	3.577	0.0068
O	2	0.88958	0.21759	0.36156	C	38	0.10993	0.55728	0.39935	3.958	0.0088
O	4	0.36954	0.48823	0.89445	C	39	0.36472	0.87682	0.83981	3.979	0.0090
O	13	0.20878	0.14151	0.91114	C	39	0.36472	0.87682	1.03981	3.350	0.0069
O	23	0.43936	0.20251	0.92858	C	39	0.36472	0.87682	1.03981	3.441	0.0076
O	21	0.38140	0.91597	0.99842	C	39	0.36472	1.87682	1.03981	3.559	0.0077
O	9	0.32240	0.52241	0.41232	C	39	0.13528	0.57682	0.46099	3.913	0.0078
O	1	0.27596	0.33035	0.31445	C	40	0.31240	0.18057	0.23578	3.206	0.0076
O	3	0.42834	0.35998	0.26643	C	40	0.31240	0.18057	0.23578	3.668	0.0081
O	10	0.41832	0.17388	0.35824	C	40	0.31240	0.18057	0.23578	3.455	0.0086
N	1	0.32996	0.68129	0.37757	C	40	0.18752	0.68057	0.26422	3.660	0.0086
O	1	0.27596	0.33035	0.31445	C	40	0.18752	0.68057	0.26422	3.738	0.0077
O	7	0.39719	0.57706	0.33294	C	40	0.18752	0.68057	0.26422	3.571	0.0087
O	9	0.38955	0.70887	0.38686	C	40	0.18752	0.68057	0.26422	3.712	0.0097
O	3	0.42834	0.35998	0.26643	C	41	0.36092	0.87319	0.19017	3.760	0.0064
O	10	0.41832	0.17388	0.35824	C	41	0.36897	0.87319	0.19017	3.916	0.0066
O	1	0.27596	0.33035	0.31445	C	41	0.13103	0.57319	0.30103	3.931	0.0068
O	3	0.42834	0.35998	0.26643	C	42	0.43197	0.83340	0.21630	3.881	0.0068
O	10	0.41832	0.17388	0.35824	C	42	0.43197	0.83340	0.21630	3.668	0.0072
O	18	0.64984	0.91305	0.85888	C	1	0.02906	0.89071	0.87757	3.474	0.0065
O	14	0.89265	0.29278	0.89486	C	2	-0.09861	0.34388	0.90762	3.703	0.0078
O	6	0.65925	0.58843	0.36978	C	3	0.05481	0.65399	0.37259	3.842	0.0072
O	12	0.47786	0.35918	0.85988	C	4	0.42497	0.29397	-0.10633	3.964	0.0087
O	2	0.88958	0.21759	0.36156	C	6	-0.07503	0.28603	0.39367	3.321	0.0064
O	18	0.64984	0.91305	0.85888	C	1	0.77596	1.16965	0.81445	3.977	0.0051
O	17	0.45956	0.83475	0.88571	C	2	0.58958	0.20241	0.86156	3.932	0.0054
O	22	0.38909	0.29884	0.84784	C	2	0.58958	0.20241	0.86156	3.858	0.0061
O	23	0.43536	0.20251	0.92858	C	2	0.58958	0.20241	0.86156	3.488	0.0056
O	24	0.44594	0.38886	0.91083	C	2	0.58958	0.20241	0.86156	3.381	0.0084
O	4	0.36954	0.48823	0.89445	C	2	0.41958	0.71759	0.13844	3.724	0.0053
O	18	0.64984	0.91305	0.85888	C	2	0.63046	0.59177	0.99555	3.826	0.0053
O	14	0.89265	0.29278	0.89486	C	3	-0.02907	0.84257	0.91958	3.688	0.0057
O	19	0.32538	0.88752	0.82918	C	4	0.15925	0.91157	0.86978	3.421	0.0063
O	13	0.20878	0.14151	0.91114	C	6	0.15925	-0.08843	0.86978	3.644	0.0057
O	18	0.64984	0.91305	0.85888	C	8	0.02260	0.97759	0.91232	3.562	0.0056
O	18	0.64984	0.91305	0.85888	C	9	0.08955	0.79913	0.88606	3.376	0.0081
O	14	0.89265	0.29278	0.89486	C	10	-0.08168	0.32620	0.85824	3.444	0.0071
O	16	0.33757	0.06684	0.59216	C	14	0.48735	-0.29738	0.60594	3.398	0.0060
O	12	0.47786	0.35918	0.85988	C	23	0.43536	0.20251	-0.07950	3.712	0.0088
O	12	0.47786	0.35918	0.85988	C	24	0.44594	0.38886	-0.08917	3.461	0.0101

TABLE 7. STRUCTURE FACTORS

PAGE 0 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

UNIT CELL DIMENSIONS
A = 19.372
B = 11.345
C = 23.023
BETA = 93.77
SPACE GROUP P2(1)/N
NUMBER OF REFLEXIONS IN SPHERE = 7438
NUMBER OF REFLEXIONS USED IN REFINEMENT = 7325
R-FACTOR = 0.0606
SCALE FACTOR 10.459

PAGE 1 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 0, K= 0			13	439	-457	13	252	-297	16	585	585	H= 0, K= 9		
2	1735	-1746	14	126	105	14	1118	-1162	17	59	-66	1	342	368
4	7420	-7481	15	1400	1409	15	757	763	18	310	310	2	115	-77
6	311	245	16	266	-241	16	575	638	19	477	530	3	543	-598
8	1045	1083	17	164	-164	17	167	-150	20	637	-656	4	149	90
10	1338	-1385	18	139	-106	18	842	833	21	319	-325	5	266	-200
12	909	909	19	1150	-1203	19	676	-599	22	144	160	6	53	66
14	1160	1206	20	121	124	20	571	-599				7	707	769
16	1568	-1594	21	671	676	21	589	562	H= 0, K= 7			8	252	-289
18	690	-726	22	98	-67	22	246	-233	1	80	78	9	275	-312
20	1421	1478	23	563	578	23	148	101	2	729	-753	10	80	91
22	152	194	24	129	-68	24	488	504	3	83	-64	11	525	-541
24	751	-803	25	604	-593				4	281	286	12	80	59
						H= 0, K= 5			5	115	-140	13	518	534
H= 0, K= 1			H= 0, K= 3			1	1160	-1163	6	566	581	14	201	-202
1	2581	2584	1	193	130	2	857	-849	7	136	186	15	21	2
2	3062	3063	2	2498	-2537	3	1850	1850	8	1027	-971	16	265	217
3	3449	-3508	3	84	-77	4	164	173	9	389	405	17	320	-307
4	711	-722	4	1003	-1034	5	87	-78	10	36	-36	18	107	127
5	455	484	5	429	427	6	726	743	11	100	-113			
6	477	-543	6	1494	-1492	7	2299	-2347	12	1150	1109	H= 0, K= 10		
7	2111	2187	7	1076	-1106	8	397	452	13	203	-216	0	661	696
8	1191	1224	8	1850	1878	9	742	770	14	455	-462	1	281	292
9	1139	-1177	9	554	552	10	334	-336	15	127	156	2	203	-155
10	112	-146	10	41	42	11	1756	1822	16	435	-419	3	123	99
11	1066	-1149	11	930	954	12	132	127	17	38	44	4	566	-613
12	1703	-1758	12	1839	-1880	13	1463	-1528	18	574	570	5	177	-168
13	1662	1718	13	470	-550	14	276	-246	19	226	-254	6	408	419
14	1015	1059	14	657	712	15	338	-299	20	64	35	7	115	-149
15	216	-190	15	200	-242	16	124	-104	21	224	188	8	304	340
16	941	931	16	1055	1055	17	965	972				9	249	268
17	1076	-1122	17	304	356	18	91	-81	H= 0, K= 8			10	352	-389
18	982	-979	18	643	-637	19	152	-146	0	448	392	11	68	-33
19	43	51	19	225	229	20	439	401	1	341	-383	12	90	86
20	460	-458	20	338	-337	21	658	-684	2	185	-202	13	283	-262
21	794	820	21	421	-420	22	136	-170	3	84	-113	14	200	201
22	893	933	22	829	802	23	682	655	4	291	-234	15	61	108
23	459	-487	23	193	159				5	541	533			
24	140	-184	24	347	-329	H= 0, K= 6			6	341	337	H= 0, K= 11		
25	107	-69	25	93	118	0	2362	-2406	7	203	-179	1	245	241
						1	1745	-1683	8	349	328	2	412	454
H= 0, K= 2			H= 0, K= 4			2	223	268	9	459	-411	3	255	-275
0	762	807	0	2320	-2319	3	208	-228	10	572	-595	4	143	-163
1	2404	2460	1	1003	1000	4	1327	1329	11	468	485	5	87	-88
2	732	-719	2	1285	1294	5	991	1043	12	199	-158	6	346	-332
3	312	343	3	418	448	6	675	-592	13	180	179	7	211	253
4	826	-825	4	1400	1476	7	83	70	14	409	391	8	291	268
5	2529	-2599	5	1787	-1808	8	245	-292	15	611	-577	9	204	-209
6	84	83	6	1296	-1278	9	999	-1064	16	111	-99	10	35	54
7	491	463	7	314	-252	10	980	944	17	276	241	11	101	87
8	401	-374	8	1076	-1156	11	557	500	18	213	-208	12	201	-199
9	1024	1024	9	992	1051	12	135	-138	19	284	318	13	104	138
10	109	-135	10	1651	1639	13	655	597	20	178	163			
11	1359	-1343	11	107	-88	14	757	-782				H= 0, K= 12		
12	576	551	12	146	-141	15	710	-717				0	113	-120

PAGE 2 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H=	0,	K= 12	16	34	44	18	175	-131	20	143	-126	23	304	253
1	131	135	17	124	117	19	145	-117	21	272	-312	24	279	267
2	35	-13	18	172	-131	20	867	-881	22	90	120	-1	136	-95
3	70	82	19	449	-476	21	116*	-1	23	450	434	-2	939	970
4	61	61	20	59	-56	22	403	419	24	215	220	-3	805	811
5	133	-135	21	123*	-3	23	98	121	-1	544	-523	-4	408	-412
6	74	45	22	304	301	24	114	98	-2	526	510	-5	431	457
7	183	143	23	412	392	25	86	-114	-3	702	717	-6	1066	-1039
8	154	-172	24	278	-240	-1	517	-486	-4	1327	1353	-7	805	-806
			25	173	-202	-2	103	118	-5	491	-499	-8	1045	1060
H=	1,	K= 0	-1	305	-309	-3	489	433	-6	36	-57	-9	982	-984
1	222	271	-2	206	214	-4	175	125	-7	397	396	-10	521	498
3	1432	1472	-3	942	-894	-5	649	-601	-8	860	-857	-11	700	726
5	533	-534	-4	253	-280	-6	930	984	-9	172	-123	-12	867	-879
7	740	752	-5	416	408	-7	861	-839	-10	239	214	-13	623	612
9	55	-59	-6	354	-303	-8	191	244	-11	397	-398	-14	109	-111
11	1317	-1340	-7	147	-89	-9	62	72	-12	765	717	-15	471	-469
13	961	964	-8	31	-31	-10	559	-507	-13	94	-63	-16	425	434
15	489	569	-9	433	-497	-11	679	638	-14	31	-32	-17	226	215
17	376	-428	-10	190	-182	-12	307	-334	-15	160	124	-18	210	-202
19	140	-138	-11	82	62	-13	63*	0	-16	180	-143	-19	538	537
21	541	610	-12	487	439	-14	167	177	-17	138	143	-20	359	-363
23	132	-150	-13	133	186	-15	314	-301	-18	136	121	-21	393	-406
25	285	-293	-14	318	-297	-16	127	110	-19	115	86	-22	631	578
-1	63	69	-15	377	377	-17	129	114	-20	116	-119	-23	393	-308
-3	196	183	-16	141	176	-18	61	-69	-21	196	205	-24	34	-66
-5	767	-817	-17	36	58	-19	336	-271	-22	367	-326			
-7	20	-33	-18	187	124	-20	335	329	-23	82	59	H=	1,	K= 5
-9	105	-98	-19	423	-372	-21	219	-251	-24	275	269	0	153	-142
-11	502	534	-20	254	-252	-22	104	104	-25	264	-249	1	1515	-1520
-13	104	-110	-21	52	-42	-23	202	198				2	707	-704
-15	118	137	-22	115	117	-24	394	-376	H=	1,	K= 4	3	1108	1110
-17	654	625	-23	117	-87	-25	153	126	0	1296	-1293	4	86	71
-19	404	-386	-24	150	116				1	25	50	5	742	772
-21	139	-151	-25	240	-255	H=	1,	K= 3	2	521	580	6	364	356
-23	464	418				0	836	-860	3	826	842	7	1484	-1523
-25	237	-220	H=	1,	K= 2	1	276	279	4	1473	1469	8	61*	0
			0	440	-488	2	897	932	5	748	-752	9	191	-165
H=	1,	K= 1	1	867	872	3	722	783	6	1066	-1076	10	60	52
0	66	86	2	169	133	4	1066	-1069	7	450	-416	11	998	968
1	575	595	3	138	-146	5	88	76	8	952	-1034	12	83*	29
2	338	359	4	175	-171	6	941	-954	9	408	402	13	94	-67
3	79	87	5	1003	-1032	7	552	-589	10	500	514	14	573	-626
4	70	-75	6	114	-98	8	219	258	11	376	445	15	574	-556
5	175	190	7	45	45	9	204	-252	12	416	462	16	123*	-10
6	462	-514	8	90	-144	10	758	807	13	334	-406	17	389	456
7	47	44	9	101	87	11	479	494	14	114	-136	18	247	207
8	585	613	10	656	720	12	145	157	15	374	-420	19	203	200
9	209	253	11	97	-104	13	240	-236	16	340	351	20	88	-75
10	505	-536	12	489	-524	14	434	-515	17	358	414	21	474	-496
11	103	-158	13	31	-22	15	269	350	18	158	-152	22	154	-158
12	216	-271	14	76	62	16	142	113	19	243	263	23	55	53
13	732	-759	15	80	-93	17	325	389	20	285	-300	-1	697	774
14	512	501	16	690	706	18	210	200	21	236	-259	-2	523	483
15	448	522	17	131	147	19	481	-497	22	30	39	-3	1015	1009

PAGE 3 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H=	1, K=	5	-10	371	360	-19	37*	3	8	166	127	-9	242	227
-4	98*	-6	-11	712	-625	-20	35	68	9	369	321	-10	565	-597
-5	1421	-1436	-12	427	-388	-21	72	-61	10	338	-362	-11	92	85
-6	160	121	-13	335	378				11	839	-787	-12	523	563
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-10	687	-609	-17	767	-783	2	69	-39	15	417	368	-16	596	-646
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-13	587	-615	-20	454	-475	5	367	366	18	231	-233	0	416	-441
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11	490	512	19	132	127	-10	749	-738	3	141	144	-12	251	230
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17	457	-458	-4	717	-738	-16	119	-105	9	201	177	2	35	44
18	320	317	-5	212	196	-17	443	-467	10	391	-372	3	484	475
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20	37	24	-7	212	222	-19	250	-266	12	389	-397	5	388	-425
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-4	385	355	-13	22	-13	2	379	-365	-3	38	-27	-3	181	200
-5	882	-810	-14	634	595	3	387	-395	-4	97	-95	-4	35	-42
-6	498	-516	-15	251	-237	4	419	-449	-5	92	65	-5	387	413
-7	564	574	-16	136	143	5	654	-659	-6	800	863	-6	34	38
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-9	355	349	-18	278	-243	7	503	540	-8	258	-264	-8	107	-103

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2	1296	-1330	-4	1756	-1823	-6	564	587	-9	654	-661	-12	1599	1635
4	1202	1247	-5	3020	-3096	-7	2236	2237	-10	1390	-1440	-13	481	516
6	2654	2706	-6	792	816	-8	426	371	-11	721	733	-14	1004	-950
8	2518	-2598	-7	236	-288	-9	1505	-1494	-12	502	-517	-15	127	74
10	1076	1139	-8	397	413	-10	333	317	-13	204	189	-16	611	-575
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14	1014	-1035	-10	2080	-2143	-12	402	-418	-15	722	-692	-18	808	764
16	1129	-1144	-11	630	-698	-13	2153	2153	-16	1045	-1033	-19	556	557
18	663	749	-12	742	737	-14	42	48	-17	135	143	-20	284	309
20	305	299	-13	540	-574	-15	656	612	-18	742	-764	-21	384	386
22	783	-799	-14	1369	1413	-16	241	247	-19	387	388	-22	791	-776
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-16	1453	1426	-23	417	390	-25	185	-201	0	506	522	4	455	473
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-24	439	-450	H= 2, K= 2			1	4713	4800	4	1264	-1278	8	796	-778
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3	1087	-1133	5	1066	1093	7	156	-176	10	454	434	14	171	-160
4	1547	-1588	6	143	-133	8	1097	1101	11	751	-770	15	970	905
5	3135	3201	7	2456	2538	9	1045	1052	12	1421	-1433	16	111	106
6	230	202	8	1285	-1296	10	1599	-1640	13	776	860	17	351	-299
7	251	-254	9	742	-722	11	187	129	14	487	550	18	351	333
8	60	7	10	504	553	12	112	-125	15	102	-66	19	739	-660
9	941	-965	11	2195	-2229	13	810	-849	16	648	648	20	252	-224
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11	1212	1223	13	1463	1507	15	101	-65	18	856	-926	22	215	-245
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13	146	-82	15	252	325	17	324	329	20	250	-286	-1	2048	-2020
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15	1212	-1220	17	1087	-1129	19	84	-49	22	768	767	-3	788	-706
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17	147	191	19	268	319	21	148	164	24	33	-60	-5	2174	2186
18	609	-559	20	39	25	22	38	-38	-1	339	248	-6	250	-205
19	1042	1070	21	773	773	23	217	253	-2	2665	-2709	-7	397	-418
20	370	438	22	217	-218	24	630	-612	-3	1139	-1124	-8	59	-54
21	481	-530	23	817	-827	-1	405	367	-4	1379	1426	-9	1453	-1467
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23	348	-381	25	147	-154	-3	1515	-1541	-6	1693	1734	-11	1045	1058
24	552	-568	-1	1348	1355	-4	2184	-2220	-7	1181	1232	-12	351	-380
25	348	377	-2	106	-98	-5	570	639	-8	1818	-1883	-13	455	419
-1	2623	2708	-3	1494	-1538	-6	982	992	-9	587	-630	-14	129	-143

PAGE 5 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
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-14	211	309				8	191	-157	-4	306	279	5	103	76
-17	539	525	H= 2, K= 7			9	398	408	-5	442	-466	6	143	174
-18	191	200	0	1087	-1173	10	316	-328	-6	150	-152	7	142	-94
-19	747	706	1	146	170	11	382	399	-7	116	79	8	100	140
-20	127	-104	2	426	459	12	502	518	-8	162	-127	9	42	28
-21	719	-697	3	231	-170	13	561	-559	-9	293	327	10	222	-226
-22	245	-232	4	1005	1055	14	125	-150	-10	253	241	11	135	142
-23	412	-368	5	200	-217	15	39	-33	-11	222	-227	12	34	-48
			6	764	-815	16	381	-362	-12	121	-98	-1	39	42
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1	1066	-1086	9	219	-166	19	210	-207	-15	441	467	-4	175	-166
2	295	-292	10	936	951	-1	601	-596	-16	194	177	-5	80	49
3	418	338	11	368	-338	-2	429	436	-17	73	84	-6	186	198
4	863	-881	12	38	-61	-3	618	646	-18	179	165	-7	84	46
5	773	727	13	412	402	-4	494	-507				-8	190	192
6	124	-129	14	674	-638	-5	178	190	H= 2, K= 10			-9	138	-160
7	1254	-1247	15	90	93	-6	250	-196	0	123	-94	-10	190	-232
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13	733	-761	21	203	199	-12	405	-407	6	109	133	1	85	62
14	598	619	-1	205	232	-13	673	-691	7	307	356	2	131	123
15	31	2	-2	265	186	-14	115	-122	8	330	-274	3	50	-58
16	64	-60	-3	493	-497	-15	289	-261	9	39	27	4	41	24
17	904	869	-4	780	751	-16	314	307	10	138	168	5	57	-42
18	250	-201	-5	291	295	-17	553	576	11	258	-248	6	134	-122
19	201	-149	-6	584	-579	-18	36	68	12	303	300	7	127	110
20	334	294	-7	338	306	-19	35	24	13	86	94	8	31	-26
21	626	-570	-8	358	-366	-20	262	-271	14	284	-293	-1	124	49
22	167	139	-9	215	162				15	219	201	-2	36*	3
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-2	762	-774	-11	192	-204	0	259	-270	-2	294	321	-4	20	40
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-20	300	-293	5	36	31	-1	526	538	2	100	-101	23	621	-613

PAGE 6 CADMIUM DINITRATO TRIS[2:6-DIMETHYL GAMMA-PYRONE]

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
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-1	358	351	-15	366	321	-18	90	83	-21	119	-70			
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-9	1254	1288	-19	827	-798	-22	172	163	-25	33	-30	2	672	690
-11	250	-207	-20	39	-31	-23	463	-441				3	1348	1328
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-19	107	-85	-24	147	167	H= 3, K= 3			2	1275	1266	7	1055	-1093
-21	110	-62	-25	232	-247	0	385	-342	3	282	295	8	612	584
-23	384	-6				1	1024	1049	4	201	-233	9	185	150
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			0	1254	1257	3	827	892	6	941	-934	11	211	174
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2	123	88	4	117	-128	7	61	-64	10	143	-87	15	605	607
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5	930	-953	7	555	605	10	166	-92	13	346	-405	18	245	265
6	564	578	8	401	440	11	931	-992	14	202	163	19	352	-363
7	229	267	9	296	-334	12	714	-745	15	627	690	20	721	-701
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9	139	140	11	815	-819	14	585	592	17	279	-273	22	184	181
10	1338	-1343	12	86	-75	15	433	476	18	357	-342	23	306	283
11	680	-743	13	288	329	16	43	-47	19	300	-285	-1	1160	-1127
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15	388	456	17	135	-131	20	203	161	23	351	292	-5	899	909
16	878	-875	18	206	-227	21	403	456	-1	968	-974	-6	141	-187
17	585	-599	19	71	-59	22	459	419	-2	1076	-1099	-7	659	-649
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21	587	674	23	131	-168	-2	21	-18	-6	22	97	-11	746	698
22	341	-397	24	401	-390	-3	426	-421	-7	582	576	-12	127	-80
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-6	721	-738	-9	1244	-1277	-12	241	270	-16	86	79	-21	420	-439
-7	336	275	-10	185	-127	-13	401	338	-17	66	48	-22	81	45
-8	168	-154	-11	28	-70	-14	109	66	-18	98	68	-23	66	70
-9	205	202	-12	351	295	-15	156	-181	-19	276	243			
-10	622	554	-13	834	789	-16	533	-487	-20	359	-316	H= 3, K= 6		
-11	982	-1000	-14	318	-324	-17	524	495	-21	190	-148	0	1170	-1147
-12	624	594	-15	270	-255	-18	38	-35	-22	31	-36	1	717	-750

PAGE 7 CADMIUM DINITRATO TRIS[2:6-DIMETHYL GAMMA-PYRONE]

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H=	3,	K= 6	9	147	-137	19	233	221	-16	162	165	-5	44	-47
2	1212	1207	10	480	489	-1	357	350	-17	343	-344	-6	572	632
3	655	597	11	90	111	-2	543	555	-18	181	193	-7	415	497
4	115	75	12	477	476	-3	699	735				-8	44	-58
5	71	51	13	117	86	-4	439	-447	H=	3,	K= 10	-9	70	-45
6	572	-595	14	566	-593	-5	495	-422	0	632	676	-10	404	-483
7	107	-102	15	211	-209	-6	89	-85	1	310	342	-11	401	-475
8	74	-59	16	60	46	-7	629	-667	2	637	-612	-12	272	324
9	201	-174	17	189	147	-8	340	352	3	99	84			
10	515	546	18	265	255	-9	913	912	4	72	-42	H=	3,	K= 12
11	412	364	19	99	119	-10	448	-481	5	39	48	0	50	48
12	269	-206	20	92	-92	-11	191	161	6	620	677	1	552	597
13	266	-298	21	273	-263	-12	157	-213	7	39*	-1	2	100	130
14	516	-520	-1	208	-167	-13	601	-639	8	389	-373	3	332	-370
15	127	-107	-2	764	721	-14	215	224	9	345	-334	4	88*	-8
16	222	200	-3	103	123	-15	22	37	10	558	-550	5	347	-375
17	582	561	-4	209	211	-16	132	112	11	38	-33	6	147	-163
18	422	417	-5	108	-82	-17	467	484	12	545	540	7	331	391
19	147	-127	-6	674	-669	-18	166	-161	13	333	350	-1	36	63
20	153	-159	-7	40*	-5	-19	269	-252	14	266	264	-2	35	-33
21	344	-345	-8	312	344				15	19	26	-3	435	-469
22	19	-77	-9	36	-47	H=	3,	K= 9	-1	232	-263	-4	35	-41
-1	482	-442	-10	517	523	0	228	-239	-2	286	318	-5	238	287
-2	156	-167	-11	316	274	1	38	56	-3	410	-441	-6	34	-40
-3	1100	1153	-12	837	-839	2	640	-677	-4	832	-896	-7	291	324
-4	974	893	-13	39	-29	3	841	-862	-5	241	281	-8	57*	0
-5	408	-408	-14	288	-342	4	429	438	-6	68	56			
-6	384	410	-15	270	-250	5	709	693	-7	284	325	H=	4,	K= 0
-7	564	-580	-16	854	875	6	229	251	-8	761	849	0	6552	-6583
-8	622	-656	-17	71	51	7	293	280	-9	234	-235	2	1150	1164
-9	638	607	-18	183	214	8	527	-451	-10	443	-503	4	2654	2719
-10	359	319	-19	219	203	9	742	-753	-11	79*	4	6	2351	-2406
-11	359	-288	-20	670	-707	10	60	68	-12	364	-384	8	899	-956
-12	508	493	-21	73	-61	11	280	274	-13	47	80	10	2372	2380
-13	465	-460				12	401	379	-14	433	461	12	261	249
-14	709	-646	H=	3,	K= 8	13	611	626	-15	229	-251	14	1912	-1877
-15	527	508	0	58	67	14	220	-229				16	609	640
-16	39	31	1	1018	-1051	15	348	-338	H=	3,	K= 11	18	1055	1071
-17	433	410	2	665	-715	16	140	-100	0	272	282	20	763	-799
-18	444	431	3	287	227	17	301	-290	1	127	-117	22	124	-88
-19	252	-194	4	228	247	-1	853	885	2	149	197	24	543	610
-20	147	-143	5	507	553	-2	319	348	3	236	-285	-2	657	653
-21	308	-292	6	703	724	-3	365	-392	4	326	-353	-4	4253	4257
-22	220	-196	7	690	-699	-4	202	204	5	214	255	-6	1850	-1886
			8	426	-382	-5	902	-913	6	117	135	-8	1797	-1809
H=	3,	K= 7	9	77	-48	-6	296	-319	7	239	249	-10	2748	2748
0	578	-514	10	439	-418	-7	608	653	8	398	406	-12	136	-121
1	173	-213	11	647	658	-8	217	219	9	332	-325	-14	2132	-2212
2	407	-421	12	307	247	-9	380	360	10	326	-367	-16	1808	1783
3	715	742	13	39	-26	-10	107	121	11	59	-40	-18	930	904
4	752	744	14	50	55	-11	576	-611	12	278	-246	-20	1766	-1808
5	107	-75	15	678	-627	-12	205	-177	-1	142	174	-22	39	-53
6	157	-197	16	96	-57	-13	55	65	-2	471	-536	-24	910	861
7	357	-341	17	307	293	-14	135	-152	-3	212	-257			
8	610	-592	18	132	97	-15	368	364	-4	123	-69			

PAGE 8 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 4, K= 1			2	1202	1209	5	194	215	9	703	-754	14	119	-129
0	1275	1349	3	170	96	6	1505	1505	10	972	-967	15	39	-34
1	951	-963	4	21	42	7	899	904	11	1045	1074	16	352	326
2	1306	-1319	5	2309	2349	8	2393	-2439	12	108	-103	17	829	-890
3	3104	3095	6	596	-613	9	261	-254	13	186	-187	18	83	67
4	1150	-1158	7	617	-601	10	45	31	14	869	874	19	416	450
5	679	-682	8	264	341	11	763	-782	15	613	-589	20	395	-359
6	272	259	9	1630	-1679	12	1515	1570	16	318	-281	21	341	329
7	2383	-2453	10	65	110	13	451	490	17	178	184	22	70	67
8	1923	-1989	11	1202	1197	14	584	-649	18	482	-444	-1	2257	2253
9	721	797	12	606	-662	15	433	500	19	411	362	-2	256	-225
10	97	-85	13	637	719	16	259	-271	20	381	347	-3	2592	-2603
11	1317	1341	14	120	111	17	575	-620	21	436	-414	-4	733	691
12	1724	1748	15	1118	-1162	18	745	744	22	110	127	-5	113	-150
13	886	-978	16	85	76	19	32	45	23	35	-15	-6	41	47
14	403	-441	17	177	123	20	302	-311	-1	372	311	-7	2330	2373
15	105	-76	18	84	-66	21	265	247	-2	1045	-1039	-8	681	-615
16	857	-865	19	892	855	22	573	-518	-3	351	251	-9	1181	-1191
17	606	629	20	203	126	23	130	-88	-4	2362	-2368	-10	250	275
18	1066	1069	21	536	-554	24	333	292	-5	1264	-1293	-11	1202	-1121
19	324	-397	22	38	-58	-1	1348	-1273	-6	1526	1531	-12	216	191
20	91	-67	23	328	-332	-2	240	266	-7	104	-85	-13	1170	1222
21	182	-268	24	80	73	-3	843	-811	-8	1129	1145	-14	361	-318
22	617	-653	-1	303	297	-4	930	-943	-9	798	792	-15	273	283
23	405	461	-2	139	-130	-5	387	-397	-10	1599	-1598	-16	276	-263
24	190	226	-3	826	-833	-6	1588	-1646	-11	126	81	-17	1032	-1034
-1	2675	-2730	-4	293	-302	-7	1108	1133	-12	264	205	-18	141	116
-2	2090	2150	-5	1829	-1864	-8	1818	1893	-13	657	-663	-19	307	268
-3	1327	1349	-6	355	-352	-9	582	-610	-14	1160	1139	-20	305	289
-4	2121	-2124	-7	334	332	-10	773	815	-15	681	736	-21	725	693
-5	1014	1075	-8	361	-288	-11	867	-814	-16	721	-732	-22	192	-144
-6	1202	-1247	-9	2372	2420	-12	1735	-1768	-17	50	-73	-23	583	-587
-7	2330	-2375	-10	1066	999	-13	612	605	-18	723	-694			
-8	1735	1816	-11	937	-903	-14	428	394	-19	571	-518	H= 4, K= 6		
-9	1703	1790	-12	198	-186	-15	226	191	-20	939	881	0	2727	2672
-10	469	459	-13	1348	-1395	-16	1045	982	-21	375	315	1	2790	2846
-11	1933	2012	-14	742	-757	-17	149	-112	-22	109	67	2	133	104
-12	1735	-1729	-15	1693	1731	-18	584	-532	-23	323	298	3	415	452
-13	1850	-1925	-16	515	500	-19	245	-178	-24	672	-620	4	1244	-1284
-14	252	301	-17	71	81	-20	180	-134				5	1599	-1634
-15	376	-377	-18	532	530	-21	276	276	H= 4, K= 5			6	813	836
-16	575	585	-19	1087	-1124	-22	854	796	0	270	-292	7	204	-206
-17	1129	1166	-20	433	-452	-23	168	-163	1	1547	1567	8	529	-557
-18	628	-558	-21	601	561	-24	347	-321	2	480	461	9	909	923
-19	424	-453	-22	115	-56				3	2466	-2537	10	653	-630
-20	40	-39	-23	700	638	H= 4, K= 4			4	364	363	11	183	-147
-21	679	-662	-24	167	118	0	2163	2173	5	309	-331	12	438	412
-22	740	697	-25	818	-763	1	1008	-999	6	667	-734	13	455	-408
-23	729	692				2	659	-686	7	1672	1692	14	275	251
-24	249	-212	H= 4, K= 3			3	372	-325	8	498	-486	15	444	384
-25	113	94	0	529	-574	4	1641	-1642	9	705	-707	16	439	-482
			1	921	853	5	1400	1463	10	458	479	17	48	-33
H= 4, K= 2			2	521	460	6	1473	1452	11	614	-649	18	39	-42
0	614	624	3	84	83	7	515	-565	12	177	-206	19	445	-441
1	515	-535	4	1557	1584	8	899	910	13	985	989	20	486	453

PAGE 9 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H=	4,	K= 6	-9	60	38	H=	4,	K= 9	-3	199	-208	H=	5,	K= 0
21	217	254	-10	110*	-9	0	619	622	-4	451	530	1	920	-928
-1	749	-712	-11	136	130	1	328	-347	-5	184	-202	3	1560	1613
-2	620	-587	-12	1042	991	2	568	-547	-6	251	-250	5	310	355
-3	1032	-980	-13	131	-80	3	31	28	-7	178	209	7	564	-583
-4	1421	-1405	-14	299	-299	4	411	-439	-8	293	-291	9	268	-304
-5	941	872	-15	224	-221	5	165	168	-9	51	65	11	1703	1743
-6	412	341	-16	521	-559	6	130	132	-10	258	278	13	690	-675
-7	876	-785	-17	111	-71	7	468	-474	-11	203	-214	15	527	-559
-8	361	362	-18	447	434	8	319	333	-12	38	26	17	107	-112
-9	352	-323	-19	124	155	9	138	100	-13	87	-44	19	40	58
-10	655	-642	-20	250	295	10	206	-167	-14	358	-385	21	307	-361
-11	720	691	-21	34	20	11	403	408	-15	241	232	23	225	232
-12	142	88				12	126	-125				-1	805	788
-13	300	323	H=	4,	K= 8	13	270	-272	H=	4,	K= 11	-3	145	-160
-14	491	512	0	480	-449	14	436	381	0	310	309	-5	1651	-1594
-15	864	-840	1	478	494	15	300	-303	1	179	199	-7	300	-218
-16	500	-455	2	508	484	16	119	-115	2	314	-314	-9	2226	2298
-17	40	-30	3	209	170	17	399	356	3	97	102	-11	99	51
-18	282	-286	4	612	596	-1	110	85	4	105	-66	-13	1066	-1070
-19	690	694	5	616	-611	-2	87	-117	5	296	-313	-15	341	-399
-20	518	565	6	455	-439	-3	447	495	6	329	303	-17	752	-804
-21	294	-272	7	76	87	-4	108	109	7	31	40	-19	657	623
-22	34	65	8	486	-449	-5	127	60	8	148	-189	-21	139	-102
			9	247	285	-6	175	181	9	301	298	-23	55	-49
H=	4,	K= 7	10	225	217	-7	473	-522	10	185	-176	-25	75	77
0	445	418	11	396	-441	-8	145	-132	11	34	16			
1	98	-95	12	377	347	-9	319	391	-1	362	-368	H=	5,	K= 1
2	922	927	13	199	165	-10	97	-116	-2	330	341	0	139	97
3	78.	-48	14	387	-354	-11	370	423	-3	39	44	1	1672	-1685
4	655	-663	15	347	266	-12	76	85	-4	418	-430	2	784	826
5	443	424	16	22	-54	-13	606	-644	-5	210	252	3	1024	-1039
6	393	-367	17	210	-196	-14	135	145	-6	227	-247	4	187	239
7	205	-179	18	322	300	-15	211	-190	-7	178	-237	5	690	685
8	877	840	19	279	-221	-16	76	-89	-8	232	264	6	266	-302
9	356	-331	-1	590	-570	-17	484	505	-9	37	50	7	1787	1802
10	114	-130	-2	198	-169				-10	36	101	8	637	-669
11	339	284	-3	232	-247	H=	4,	K= 10	-11	124	135	9	1129	-1140
12	772	-753	-4	207	203	0	340	-367	-12	199	-219	10	648	659
13	213	219	-5	501	496	1	100*	-7				11	752	-747
14	429	433	-6	234	-201	2	232	212	H=	4,	K= 12	12	296	285
15	366	-369	-7	49	-57	3	363	-371	0	36	-40	13	454	537
16	416	390	-8	107	-140	4	292	307	1	232	-268	14	596	-583
17	45	67	-9	260	-231	5	228	210	2	35	16	15	482	-541
18	585	-594	-10	608	555	6	461	-483	3	35	-59	16	37	29
19	165	177	-11	164	142	7	243	233	4	20	-42	17	77	71
20	121	-82	-12	40	-44	8	184	-178	5	187	180	18	170	177
-1	84	-58	-13	277	265	9	298	-284	6	98	76	19	262	290
-2	816	-854	-14	390	-399	10	353	357	-1	246	240	20	166	-191
-3	242	-296	-15	428	-406	11	191	-156	-2	187	-213	21	56*	6
-4	606	655	-16	203	208	12	37	-29	-3	133	156	22	140	-154
-5	135	74	-17	75	71	13	338	346	-4	35	48	23	319	-336
-6	203	123	-18	213	214	14	202	-198	-5	167	-192	24	314	293
-7	89	50	-19	294	297	-1	461	453	-6	173	219	-1	371	360
-8	1170	-1114				-2	51	51	-7	34	15	-2	1766	1817

PAGE 10 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 5, K= 1			-5	259	-275	-9	88	-100	-14	466	478	-20	263	230
-3	192	153	-6	238	-169	-10	283	-320	-15	104	91	-21	184	164
-4	564	-572	-7	1045	-1124	-11	217	199	-16	574	-557	-22	103	-61
-5	836	843	-8	481	484	-12	621	-549	-17	429	-366	-23	387	-391
-6	1026	-982	-9	609	522	-13	281	350	-18	240	-268			
-7	397	-396	-10	295	321	-14	725	661	-19	535	-554	H= 5, K= 6		
-8	230	-220	-11	178	213	-15	262	231	-20	309	279	0	103	79
-9	554	-570	-12	395	-331	-16	140	-165	-21	450	449	1	189	-132
-10	575	604	-13	305	274	-17	351	-361	-22	145	-154	2	1233	1190
-11	433	-396	-14	318	-243	-18	457	-390	-23	36	62	3	865	853
-12	512	-525	-15	398	378	-19	264	-272	-24	34	-33	4	392	-371
-13	32	29	-16	90	133	-20	217	191				5	439	-375
-14	355	363	-17	343	-321	-21	115	85	H= 5, K= 5			6	172	-208
-15	103	144	-18	366	351	-22	134	90	0	844	794	7	99	-111
-16	126	-91	-19	239	214	-23	177	-159	1	1317	1315	8	723	641
-17	107	-120	-20	315	-327	-24	70	-90	2	554	569	9	346	359
-18	71	-97	-21	98*	9				3	540	-501	10	110	60
-19	89*	-5	-22	91	-74	H= 5, K= 4			4	201	-197	11	647	-642
-20	126	90	-23	38	-46	0	783	717	5	884	-879	12	633	-613
-21	306	281	-24	63	57	1	1076	1060	6	766	-673	13	183	-183
-22	102	63	-25	65	-70	2	316	248	7	920	975	14	268	278
-23	127	-128				3	1202	-1261	8	398	336	15	671	633
-24	43	-45	H= 5, K= 3			4	918	-1000	9	80	109	16	196	188
-25	154	171	0	321	348	5	126	-167	10	373	-428	17	89	80
			1	126	88	6	314	320	11	690	-694	18	303	-271
H= 5, K= 2			2	698	-729	7	1118	1110	12	268	-235	19	382	-351
0	724	677	3	1139	-1157	8	651	724	13	133	128	20	117	-121
1	74	66	4	408	-434	9	346	-362	14	881	892	21	262	239
2	899	-912	5	98	81	10	1055	-1045	15	489	540	-1	491	-507
3	159	-116	6	366	356	11	702	-714	16	76	-62	-2	444	-455
4	183	218	7	826	815	12	109	105	17	328	-299	-3	63	52
5	794	-783	8	283	278	13	794	787	18	433	-478	-4	627	-569
6	1369	1400	9	484	-538	14	660	723	19	176	-175	-5	648	640
7	310	337	10	721	-703	15	186	168	20	223	254	-6	568	519
8	809	-826	11	255	-281	16	667	-685	21	135	154	-7	268	-263
9	738	758	12	501	558	17	605	-652	22	280	296	-8	513	-467
10	1150	-1164	13	594	636	18	192	-257	-1	110	-63	-9	462	-439
11	243	296	14	449	477	19	40	-57	-2	926	-874	-10	191	-173
12	722	782	15	576	-594	20	568	599	-3	934	-926	-11	675	603
13	161	138	16	464	-481	21	743	727	-4	85	79	-12	767	737
14	79	80	17	445	-508	22	96	-76	-5	625	614	-13	38	-36
15	644	-672	18	177	-234	23	285	-284	-6	1101	28	-14	45*	27
16	655	-695	19	928	964	-1	289	280	-7	1055	1040	-15	589	-509
17	130	-112	20	274	324	-2	146	-146	-8	116	133	-16	462	-458
18	231	226	21	159	141	-3	941	-977	-9	1756	-1785	-17	543	545
19	299	358	22	38	43	-4	752	-754	-10	283	260	-18	127	-131
20	702	765	23	477	-481	-5	595	-571	-11	35	75	-19	154	117
21	118	-96	-1	1599	1626	-6	647	588	-12	575	563	-20	548	582
22	508	-584	-2	164	-163	-7	491	475	-13	752	759	-21	402	-439
23	37	-18	-3	63*	-9	-8	78	-63	-14	279	-236	-22	191	-183
24	120	-86	-4	585	-520	-9	318	286	-15	460	-470			
-1	79	81	-5	80	-104	-10	705	-731	-16	397	-402	H= 5, K= 7		
-2	679	688	-6	481	-498	-11	408	-414	-17	495	-448	0	724	-723
-3	387	388	-7	198	170	-12	642	631	-18	172	186	1	441	387
-4	169	-136	-8	1027	962	-13	449	469	-19	727	683	2	832	885

PAGE 11 CADMIUM DINITRATO TRİS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 5, K= 7			13	526	-510	-12	194	182	-4	507	-536	4	1202	1229
3	352	278	14	338	-351	-13	514	-548	-5	342	-355	5	2581	-2621
4	105	125	15	376	370	-14	247	-243	-6	31	-17	6	773	-786
5	307	-273	16	237	204	-15	331	371	-7	212	-216	7	1087	1076
6	677	-665	17	315	274	-16	46	53	-8	459	492	8	395	-457
7	223	-186	18	96	120	-17	365	380	-9	292	357	9	1285	1290
8	216	255	-1	872	-896				-10	193	-223	10	1108	1118
9	327	377	-2	71	80	H= 5, K= 10			-11	34*	-7	11	1150	-1169
10	380	384	-3	327	332	0	408	-398				12	176	-190
11	96	-67	-4	452	451	1	138	101	H= 5, K= 12			13	287	-334
12	396	-394	-5	560	526	2	305	-293	0	35*	12	14	762	-823
13	278	-289	-6	310	-347	3	124	111	1	61	-35	15	741	822
14	92	-107	-7	373	-386	4	599	601	2	64	-64	16	266	290
15	232	203	-8	241	-211	5	86	92	3	484	-511	17	251	-238
16	441	394	-9	154	-125	6	222	-236	4	129	-112	18	108	87
17	139	133	-10	309	347	7	268	-279	5	272	278	19	440	-409
18	193	-165	-11	638	640	8	425	-435	-1	366	367	20	217	-203
19	331	-297	-12	177	-183	9	223	-228	-2	67	29	21	342	303
20	219	-184	-13	261	-216	10	327	325	-3	135	-135	22	38	-31
-1	171	-173	-14	23	-26	11	81	125	-4	35*	4	23	188	198
-2	724	-710	-15	609	-629	12	296	294	-5	315	-339	24	326	285
-3	28	-36	-16	278	293	13	175	159	-6	19	-17	-1	167	170
-4	770	788	-17	329	360	14	371	-393				-2	2456	2508
-5	35	-72	-18	202	-203	-1	182	166	H= 6, K= 0			-3	2080	-2102
-6	342	327	-19	354	404	-2	760	795	0	89	-84	-4	1526	1499
-7	261	-247				-3	40	-85	2	1860	1906	-5	1745	1736
-8	943	-944	H= 5, K= 9			-4	40	48	4	1682	-1677	-6	2100	-2091
-9	205	-240	0	591	-660	-5	279	-299	6	1588	-1648	-7	350	352
-10	545	544	1	893	-916	-6	746	-776	8	2644	2684	-8	878	-895
-11	265	273	2	496	484	-7	177	180	10	69	-16	-9	1745	-1778
-12	464	406	3	739	771	-8	233	248	12	1777	-1811	-10	1630	1665
-13	219	208	4	114	155	-9	87	78	14	699	731	-11	951	897
-14	831	-829	5	149	169	-10	475	528	16	443	505	-12	141	-65
-15	40*	6	6	369	-388	-11	217	-220	18	978	-1018	-13	874	900
-16	65	24	7	612	-670	-12	361	-406	20	257	309	-14	1275	-1265
-17	88	-68	8	40	-24	-13	78	-62	22	537	518	-15	1390	-1443
-18	621	627	9	111	-87	-14	186	-221	24	91	-96	-16	637	651
-19	21	20	10	311	311	-15	163	171	-2	2957	-3017	-17	240	243
-20	300	-318	11	622	617				-4	1129	1176	-18	606	634
-21	80*	-5	12	146	-181	H= 5, K= 11			-6	2895	2953	-19	805	836
			13	73	-70	0	398	420	-8	2780	-2784	-20	930	-987
H= 5, K= 8			14	248	-210	1	391	-384	-10	1139	-1128	-21	653	-630
0	586	-554	15	373	-333	2	532	-542	-12	2351	2383	-22	40	45
1	162	-167	16	216	211	3	263	249	-14	763	-788	-23	492	-467
2	336	313	-1	170	-184	4	85	-98	-16	857	-888	-24	675	606
3	904	943	-2	78	58	5	237	288	-18	857	880			
4	382	380	-3	1066	1101	6	354	351	-20	347	-260	H= 6, K= 2		
5	110	-94	-4	505	532	7	154	-193	-22	842	-825	0	1338	1339
6	627	-645	-5	459	-437	8	107	-125	-24	435	400	1	1714	-1782
7	553	-503	-6	132	71	9	164	-160				2	98	120
8	328	-357	-7	612	-654	10	117	-98	H= 6, K= 1			3	2445	2499
9	489	445	-8	283	-308	11	162	150	0	1045	-1001	4	1150	-1144
10	413	456	-9	690	725	-1	105	54	1	2508	2585	5	721	715
11	106	-65	-10	61	-77	-2	270	302	2	1453	-1445	6	105	-114
12	273	208	-11	123	59	-3	303	310	3	144	-181	7	2205	-2251

PAGE 12 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 6, K= 2			12	236	203	17	399	387	-2	651	-595	-10	142	165
8	711	716	13	605	635	18	556	633	-3	720	709	-11	726	-696
9	642	660	14	783	-809	19	287	-279	-4	155	-159	-12	830	-783
10	123	132	15	118	-488	20	243	219	-5	1536	-1561	-13	1076	1095
11	1045	1065	16	467	506	21	97	-104	-6	352	304	-14	48*	8
12	129	88	17	106	-112	22	328	-350	-7	31	-30	-15	382	381
13	903	-969	18	280	311	-1	249	-245	-8	108	91	-16	716	694
14	311	296	19	323	382	-2	1275	1296	-9	1453	1472	-17	625	-580
15	131	-117	20	425	-478	-3	729	759	-10	64	46	-18	192	-231
16	116	-95	21	39	-26	-4	627	-627	-11	797	-743	-19	154	177
17	878	905	22	100	88	-5	558	-499	-12	37*	-2	-20	170	-149
18	632	-627	23	271	-223	-6	1275	-1311	-13	820	-811	-21	277	264
19	729	-704	-1	237	235	-7	961	-1011	-14	112	89	-22	344	385
20	210	220	-2	335	298	-8	1568	1589	-15	1191	1219			
21	139	-131	-3	737	704	-9	1014	932	-16	149	-134	H= 6, K= 7		
22	270	246	-4	1881	1930	-10	690	678	-17	70	57	0	943	961
23	510	500	-5	1024	-1046	-11	941	968	-18	74	-70	1	432	-462
-1	1359	-1348	-6	1453	-1466	-12	1170	-1194	-19	833	-780	2	289	-244
-2	633	-705	-7	189	-234	-13	732	-733	-20	130	125	3	621	661
-3	1975	2039	-8	742	-767	-14	346	380	-21	483	474	4	911	-912
-4	394	-310	-9	365	383	-15	542	-503	-22	36	-31	5	56	-47
-5	191	161	-10	1599	1629	-16	713	686	-23	448	446	6	924	891
-6	763	764	-11	247	257	-17	428	432				7	608	-624
-7	2842	-2857	-12	100	91	-18	1032	-1021	H= 6, K= 6			8	357	345
-8	381	-323	-13	247	-288	-19	109	81	0	1212	-1229	9	307	279
-9	1484	1554	-14	1327	-1383	-20	250	-273	1	1202	1154	10	590	-588
-10	82	-101	-15	488	537	-21	285	-289	2	105	99	11	586	602
-11	1588	1651	-16	836	841	-22	852	842	3	293	-286	12	23	-26
-12	649	691	-17	93	78	-23	264	249	4	1076	1119	13	551	-533
-13	1160	-1201	-18	920	945				5	365	-375	14	490	505
-14	73	-51	-19	939	-904	H= 6, K= 5			6	63*	10	15	114	-129
-15	700	-703	-20	1212	-1252	0	147	179	7	697	692	16	243	-228
-16	238	-244	-21	234	277	1	861	-846	8	905	-947	17	407	407
-17	1379	1426	-22	268	-241	2	445	399	9	201	-196	18	225	-204
-18	72	-62	-23	254	249	3	223	-263	10	398	374	19	143	-154
-19	437	-430	-24	855	841	4	679	-666	11	439	-427	-1	485	-447
-20	99*	5				5	1076	1066	12	638	667	-2	393	-440
-21	989	-955	H= 6, K= 4			6	61	52	13	413	399	-3	536	548
-22	121*	-2	0	257	-291	7	441	-463	14	581	-574	-4	1028	-1017
-23	673	635	1	329	-260	8	422	452	15	566	574	-5	105	-50
-24	99	85	2	1233	-1272	9	376	-401	16	98	-122	-6	1028	949
			3	756	785	10	29	-20	17	586	-589	-7	485	-533
H= 6, K= 3			4	723	797	11	913	966	18	429	371	-8	207	222
0	1693	-1710	5	58	-82	12	422	-451	19	117	-108	-9	38	45
1	714	-723	6	230	222	13	80	59	20	243	-246	-10	970	-929
2	416	459	7	596	-596	14	396	414	21	300	268	-11	305	292
3	94	-89	8	1379	-1400	15	908	-857	-1	527	-476	-12	48	-40
4	1651	1662	9	149	126	16	40	-20	-2	888	835	-13	40	41
5	888	943	10	120	66	17	81	-104	-3	1045	-967	-14	1055	1038
6	1871	-1887	11	185	230	18	433	-418	-4	31	40	-15	87	75
7	529	-614	12	1118	1165	19	596	582	-5	249	282	-16	148	-107
8	253	-266	13	554	-531	20	231	183	-6	733	-682	-17	124	-173
9	920	-932	14	240	-234	21	78	-70	-7	967	904	-18	550	-516
10	1306	1301	15	149	202	22	298	266	-8	576	525	-19	145	-183
11	347	374	16	859	-928	-1	1118	1085	-9	904	-979	-20	442	437

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 6, K= 8														
0	117	-107	14	261	-269	3	77	-70	-21	469	-519	1	150	-154
1	348	317	15	253	267	4	387	357	-23	58	66	2	402	343
2	714	709	16	237	-277	5	90	-139				3	87	93
3	849	-858	-1	642	-659	6	124	-135	H= 7, K= 1			4	1097	1121
4	118	-144	-2	89	-129	7	145	166	0	485	-508	5	84	79
5	227	227	-3	456	-450	8	201	-157	1	1037	-1040	6	100	71
6	608	-625	-4	265	-289	9	236	-198	2	242	305	7	770	-826
7	459	467	-5	627	699	10	237	238	3	1651	1661	8	1097	-1108
8	193	234	-6	119	-50	-1	103	-41	4	972	-973	9	672	723
9	394	-341	-7	228	-232	-2	194	203	5	585	591	10	773	765
10	468	459	-8	398	444	-3	271	-290	6	1097	-1083	11	436	473
11	400	-356	-9	224	-284	-4	242	237	7	1045	-1045	12	456	475
12	252	-239	-10	107	-148	-5	44	58	8	1400	1368	13	289	-324
13	292	246	-11	297	254	-6	394	-409	9	341	424	14	891	-951
14	280	-281	-12	242	-238	-7	265	274	10	1097	1107	15	71	-57
15	39	-33	-13	39*	17	-8	119	-103	11	564	569	16	249	278
16	387	381	-14	123	98	-9	36	-60	12	539	-609	17	52	-35
17	245	-285	-15	347	-312	-10	259	264	13	274	-271	18	445	523
18	34	43	-16	117	-109	-11	123	-84	14	373	-437	19	153	-129
-1	898	891	-17	157	177				15	700	-685	20	469	-533
-2	510	-533	H= 6, K= 10			H= 6, K= 12			16	465	503	21	144	179
-3	810	-772	0	207	211	0	443	493	17	398	408	22	296	-305
-4	432	421	1	389	-401	1	146	-140	18	41	-60	23	213	188
-5	182	-179	2	230	243	2	180	-189	19	266	278	-1	332	-382
-6	229	222	3	104	92	3	34	-94	20	458	-441	-2	878	895
-7	650	659	4	427	-423	4	274	-340	21	469	-477	-3	711	728
-8	577	-608	5	346	329	-1	35	22	22	477	454	-4	439	-440
-9	224	-205	6	52	44	-2	60	-47	23	95	91	-5	63	56
-10	137	165	7	205	-212	-3	157	192	-1	872	-796	-6	152	-134
-11	405	-396	8	309	305	-4	240	-261	-2	438	-376	-7	124	-182
-12	344	345	9	84	-57	-5	28	58	-3	283	-285	-8	443	-365
-13	287	322	10	194	-197	H= 7, K= 0			-4	1860	1832	-9	1035	1059
-14	205	-177	11	283	249	1	239	-271	-5	214	-147	-10	275	288
-15	233	259	12	202	-188	3	1202	1218	-6	888	-911	-11	148	-139
-16	180	-202	13	78	-49	5	1076	-1108	-7	376	-381	-12	314	-307
-17	530	-597	-1	52*	-11	7	402	-390	-8	1296	-1339	-13	397	-394
-18	363	322	-2	345	-399	9	888	871	-9	763	801	-14	203	-231
-19	34	51	-3	403	447	11	117	109	-10	388	373	-15	84	66
H= 6, K= 9			-4	276	281	13	562	-638	-11	470	484	-16	219	179
0	584	535	-5	70	-61	15	606	602	-12	581	511	-17	528	487
1	984	1049	-6	179	217	17	700	714	-13	217	-171	-18	201	175
2	116	97	-7	327	-328	19	1087	-1151	-14	181	111	-19	256	-295
3	211	234	-8	350	-381	21	40	-80	-15	225	-232	-20	252	-269
4	187	-123	-9	210	207	23	589	596	-16	193	128	-21	318	-308
5	624	-684	-10	140	-105	-1	1484	-1447	-17	91*	-22	-22	122	115
6	210	126	-11	38*	3	-3	226	238	-18	40	-32	-23	281	309
7	121	164	-12	210	207	-5	136	-152	-19	419	371	-24	87	60
8	327	-351	-13	122	-156	-7	1609	-1676	-20	245	-226			
9	407	400	-14	229	-248	-9	115	-103	-21	256	-272	H= 7, K= 3		
10	40	47	H= 6, K= 11			-11	324	318	-22	189	-133	0	92	-79
11	224	-197	0	338	-312	-13	391	455	-23	147	140	1	1202	-1193
12	261	295	1	245	282	-15	491	-505	-24	260	273	2	1275	-1318
13	251	-231	2	124	-51	-17	281	244				3	724	776
						-19	349	380	H= 7, K= 2			4	627	668
									0	951	-973	5	941	961

PAGE 14 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 7, K= 3			10	309	-339	17	230	-223	-6	93*	10	-18	210	-220
6	121	98	11	120	112	18	46	-48	-7	565	602	-19	209	-214
7	888	-888	12	401	403	19	233	253	-8	833	809	-20	672	684
8	585	-571	13	390	382	20	489	485	-9	459	-437			
9	439	-442	14	333	-351	21	210	187	-10	276	-276	H= 7, K= 8		
10	295	228	15	486	-507	-1	554	583	-11	186	-220	0	50	57
11	1275	1282	16	127	-165	-2	302	286	-12	404	-346	1	920	894
12	410	412	17	126	-96	-3	351	-405	-13	434	380	2	800	832
13	179	-197	18	365	371	-4	766	-733	-14	703	714	3	178	-165
14	435	-472	19	443	451	-5	773	-776	-15	137	-100	4	233	-276
15	514	-538	20	149	-171	-6	19	-15	-16	119	-93	5	642	-657
16	73	-115	21	37	-34	-7	588	526	-17	589	-563	6	567	-524
17	230	238	22	130	-127	-8	892	813	-18	578	-600	7	268	268
18	141	116	-1	1007	1024	-9	472	496	-19	447	432	8	517	507
19	102	-67	-2	1076	1017	-10	140	-136	-20	165	157	9	375	348
20	112	108	-3	270	198	-11	701	-666	-21	185	185	10	183	-189
21	415	-420	-4	899	-847	-12	232	-202				11	532	-501
22	135	-138	-5	487	-496	-13	286	237	H= 7, K= 7			12	262	-257
23	230	224	-6	527	-438	-14	140	86	0	336	335	13	56	-1
-1	437	403	-7	105	-107	-15	578	580	1	242	244	14	190	182
-2	633	614	-8	721	724	-16	47	-59	2	339	365	15	605	582
-3	1076	1095	-9	170	161	-17	491	-452	3	251	-277	16	87	76
-4	66	-57	-10	265	-289	-18	155	188	4	683	-677	17	271	-256
-5	899	-885	-11	586	-535	-19	150	-167	5	440	-473	-1	216	-176
-6	479	-472	-12	610	-566	-20	39	-57	6	156	192	-2	520	-571
-7	478	-431	-13	144	-158	-21	385	376	7	576	533	-3	620	-640
-8	973	917	-14	616	568	-22	150	-181	8	519	543	-4	148	158
-9	805	811	-15	623	600				9	167	167	-5	496	526
-10	253	304	-16	175	136	H= 7, K= 6			10	509	-528	-6	216	234
-11	419	-371	-17	125	-77	0	208	231	11	524	-529	-7	375	383
-12	857	-866	-18	232	-248	1	370	360	12	266	-254	-8	399	-405
-13	617	-585	-19	332	-296	2	523	-477	13	293	274	-9	703	-713
-14	246	-244	-20	282	265	3	491	-471	14	521	551	-10	41*	-11
-15	148	141	-21	39	53	4	56	-67	15	167	206	-11	70	33
-16	266	227	-22	97	94	5	214	-256	16	115	-118	-12	540	557
-17	252	-268	-23	35	57	6	521	503	17	349	-344	-13	433	442
-18	166	136				7	442	457	18	381	-402	-14	172	-223
-19	330	307	H= 7, K= 5			8	292	287	19	34	-26	-15	200	-212
-20	376	-393	0	351	359	9	62	-69	-1	232	258	-16	409	-401
-21	112	87	1	106	63	10	699	-705	-2	480	-480	-17	305	-333
-22	154	156	2	508	-454	11	318	-313	-3	357	-341	-18	370	418
-23	70	-46	3	593	-593	12	42	-38	-4	412	-447			
-24	91	95	4	246	252	13	357	334	-5	129	-74	H= 7, K= 9		
			5	33	34	14	594	550	-6	688	635	0	77	-38
H= 7, K= 4			6	905	875	15	41	40	-7	472	451	1	99	66
0	208	208	7	448	465	16	230	-173	-8	38	-41	2	407	436
1	705	-744	8	446	-413	17	479	-486	-9	75	-74	3	720	691
2	971	-1004	9	439	-449	18	236	-269	-10	698	-663	4	123	-58
3	451	-427	10	472	-486	19	321	322	-11	293	-258	5	450	-475
4	486	583	11	87	95	20	359	356	-12	334	369	6	354	-347
5	803	757	12	315	346	-1	305	268	-13	256	271	7	105	-43
6	784	876	13	396	408	-2	103	47	-14	497	490	8	539	475
7	142	146	14	421	396	-3	469	-467	-15	105	77	9	491	502
8	443	-492	15	361	-349	-4	699	-686	-16	665	-738	10	80	49
9	838	-891	16	674	-659	-5	274	241	-17	39	-71	11	328	-289

PAGE 15 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 7, K= 9			2	130	-145	3	1891	-1927	8	162	139	14	566	558
12	410	-419	3	314	339	4	909	-931	9	999	1059	15	447	-477
13	342	-346	4	368	382	5	55	-44	10	59	90	16	398	393
14	172	185	5	108	-143	6	439	-455	11	993	-1010	17	114	137
15	279	256	6	37	-50	7	826	812	12	263	327	18	679	-683
-1	610	-620	7	167	-187	8	1129	1147	13	200	-159	19	96	109
-2	343	-363	8	358	-342	9	343	-366	14	207	-217	20	179	184
-3	107	106	9	141	127	10	64	94	15	795	865	21	109	-120
-4	23	12	-1	385	-405	11	77	-126	16	265	-268	22	372	329
-5	590	597	-2	296	334	12	713	-713	17	235	-225	-1	166	-227
-6	320	279	-3	108	120	13	562	606	18	199	210	-2	1494	-1451
-7	448	-465	-4	193	212	14	180	189	19	428	-345	-3	617	639
-8	301	-323	-5	264	260	15	39	50	20	92	87	-4	575	572
-9	382	-383	-6	431	-475	16	424	478	21	283	202	-5	573	509
-10	111	99	-7	229	-272	17	763	-762	22	97	-104	-6	1369	1405
-11	576	578	-8	135	121	18	486	-520	-1	1348	-1386	-7	784	-715
-12	388	387	-9	104	-75	19	268	308	-2	282	-284	-8	1275	-1310
-13	39	-25	-10	230	263	20	61	39	-3	1139	-1156	-9	199	-194
-14	109	-125				21	228	276	-4	888	-894	-10	794	-797
-15	391	-415	H= 7, K= 12			22	224	229	-5	1881	1950	-11	409	396
-16	20	-60	0	205	-225	23	258	-248	-6	649	567	-12	1515	1546
			1	469	-514	-1	1254	1260	-7	29	36	-13	784	-682
H= 7, K= 10			-1	100	-87	-2	799	-814	-8	656	661	-14	54	-51
0	520	-533	-2	68	65	-3	1003	-1007	-9	1505	-1560	-15	32	-61
1	114	-95				-4	437	463	-10	448	-500	-16	1170	-1177
2	525	521	H= 8, K= 0			-5	217	-213	-11	646	636	-17	773	778
3	66	86	0	1703	1722	-6	909	891	-12	35	44	-18	602	595
4	205	193	2	116	-99	-7	2027	2101	-13	1055	1009	-19	558	-541
5	154	-155	4	1515	-1578	-8	1442	-1497	-14	390	346	-20	382	346
6	501	-454	6	1881	1907	-9	1108	-1142	-15	1097	-1133	-21	256	-275
7	39	-36	8	319	-372	-10	99	-67	-16	176	-185	-22	670	-687
8	59	61	10	1400	-1386	-11	1170	-1197	-17	237	-268	-23	353	337
9	176	199	12	111	75	-12	972	1020	-18	258	-236			
10	328	334	14	899	865	-13	1191	1205	-19	1055	1089	H= 8, K= 4		
11	37	-59	16	617	-638	-14	239	-211	-20	279	262	0	1076	-1087
12	138	-119	18	234	-283	-15	693	644	-21	561	-558	1	1139	1095
13	194	-185	20	373	444	-16	706	-749	-22	90	117	2	286	-231
-1	40	42	22	114	103	-17	1369	-1426	-23	737	-709	3	98	-124
-2	287	-289	-2	100	-72	-18	717	669	-24	199	-159	4	1181	1190
-3	247	293	-4	2936	-3030	-19	130	151				5	431	-474
-4	732	721	-6	1808	1830	-20	251	259	H= 8, K= 3			6	856	-882
-5	141	-159	-8	485	527	-21	882	867	0	359	315	7	419	423
-6	76	-77	-10	1588	-1656	-22	590	-592	1	652	-603	8	291	-294
-7	356	-397	-12	512	467	-23	420	-387	2	1097	1114	9	274	223
-8	679	-651	-14	1244	1248	-24	104	97	3	750	790	10	737	736
-9	148	156	-16	815	-838				4	1338	-1325	11	891	-941
-10	423	476	-18	1087	-1117	H= 8, K= 2			5	103	102	12	38	-54
-11	142	152	-20	1359	1424	0	694	652	6	909	-923	13	60	58
-12	205	234	-22	78	50	1	1202	1280	7	770	-783	14	764	-828
-13	144	-130	-24	1045	-1002	2	111	85	8	1233	1249	15	633	701
-14	454	-487				3	784	831	9	223	265	16	234	255
			H= 8, K= 1			4	98	-71	10	223	215	17	459	-469
H= 7, K= 11			0	42	33	5	2017	-2018	11	351	369	18	388	389
0	272	-301	1	492	552	6	169	153	12	951	-944	19	133	-73
1	85	38	2	1327	1360	7	538	561	13	894	-1	20	211	-186

PAGE 16 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 8, K= 4			-7	1369	-1383	-17	66	76	8	347	350	-11	238	-240
21	328	302	-8	599	593	-18	307	273	9	41	-53	-12	107	-136
-1	105	-101	-9	826	777	-19	678	-691	10	429	-378	-13	352	380
-2	669	713	-10	180	-179	-20	536	-508	11	401	396	-14	223	-235
-3	1055	-1071	-11	690	677	-21	205	215	12	216	-260	-15	36	36
-4	1693	1689	-12	39	38				13	279	-268	-16	196	255
-5	669	685	-13	1045	-1069	H= 8, K= 7			14	543	528			
-6	1233	-1282	-14	364	398	0	72	-62	15	214	-175	H= 8, K= 10		
-7	306	324	-15	280	-240	1	554	544	16	36	44	0	78	55
-8	717	-709	-16	47	-64	2	985	-983	17	119	122	1	153	-179
-9	833	-755	-17	1317	1321	3	85	89	-1	136	111	2	164	-198
-10	941	950	-18	675	-651	4	641	629	-2	382	-363	3	452	478
-11	248	230	-19	448	-472	5	651	-634	-3	317	333	4	47	-48
-12	65	-81	-20	134	129	6	391	310	-4	289	-232	5	103	-86
-13	388	378	-21	643	-655	7	297	236	-5	595	-609	6	338	328
-14	1191	-1204	-22	298	240	8	669	-685	-6	518	483	7	317	-318
-15	225	-236				9	125	118	-7	405	-392	8	21	-16
-16	857	798	H= 8, K= 6			10	41	25	-8	24	43	9	263	253
-17	100*	14	0	1181	-1178	11	464	-429	-9	781	818	10	270	-248
-18	627	649	1	466	-441	12	523	495	-10	338	-321	11	301	282
-19	298	243	2	628	608	13	41	49	-11	114	-80	12	111	148
-20	1108	-1129	3	711	-709	14	201	-169	-12	71	-74	-1	505	-498
-21	232	-199	4	312	311	15	401	367	-13	285	-289	-2	84	-63
-22	37	22	5	715	769	16	304	-272	-14	215	216	-3	137	111
-23	243	-236	6	652	-596	17	138	-120	-15	295	266	-4	233	-231
			7	287	-350	18	336	309	-16	58*	3	-5	320	302
H= 8, K= 5			8	480	487	-1	431	-416	-17	60	40	-6	266	266
0	1012	936	9	647	-616	-2	893	890	-18	114	-91	-7	184	-222
1	1150	-1134	10	351	326	-3	237	-218				-8	113	140
2	707	-701	11	485	457	-4	456	-395	H= 8, K= 9			-9	39	31
3	1379	1391	12	410	-419	-5	301	285	0	772	-792	-10	301	-321
4	164	-189	13	491	512	-6	631	-655	1	413	403	-11	218	222
5	40	35	14	123	-64	-7	99	93	2	308	292	-12	89	-35
6	668	697	15	579	-625	-8	1012	985	3	42	38	-13	122	-135
7	551	-616	16	359	403	-9	163	-111	4	796	823			
8	37	-39	17	159	-135	-10	408	340	5	161	-184	H= 8, K= 11		
9	603	598	18	82	-62	-11	156	150	6	95	-123	0	38	-50
10	375	-345	19	433	433	-12	832	-800	7	274	291	1	38	-39
11	641	591	20	199	-216	-13	318	-236	8	334	-359	2	291	289
12	170	-92	-1	1160	1080	-14	109	174	9	98	-93	3	109	-117
13	645	-629	-2	561	-516	-15	148	-137	10	69	117	4	188	-173
14	317	321	-3	35	90	-16	352	343	11	260	-246	5	273	272
15	227	-238	-4	1202	1206	-17	220	183	12	38	55	6	279	-286
16	257	-247	-5	1139	-1142	-18	393	-408	13	206	166	7	35	-34
17	643	698	-6	112	-133	-19	204	185	14	321	-262	8	106	114
18	301	-269	-7	784	792				-1	263	-231	-1	276	276
19	363	-353	-8	593	-604	H= 8, K= 8			-2	85	-55	-2	209	-211
20	256	235	-9	599	577	0	367	355	-3	472	-497	-3	69	-99
21	122	-128	-10	782	752	1	461	-442	-4	214	212	-4	240	270
-1	1630	-1640	-11	759	-675	2	516	506	-5	47	33	-5	37	-84
-2	101	119	-12	140	-127	3	239	204	-6	265	-190	-6	37	68
-3	1473	1420	-13	257	-250	4	612	-605	-7	556	549	-7	36	-40
-4	846	-833	-14	668	-586	5	570	521	-8	78	-77	-8	229	-253
-5	854	767	-15	468	479	6	52	-38	-9	139	-140	-9	70	67
-6	117	-97	-16	704	671	7	268	-275	-10	170	171			

PAGE 17 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 9, K= 0			-7	143	-130	-13	264	-249	-21	238	-268	4	586	633
1	1327	1303	-8	160	134	-14	248	195	-22	243	-247	5	396	394
3	1191	-1192	-9	888	919	-15	380	-358	-23	168	187	6	238	-231
5	912	-972	-10	763	-773	-16	306	-259				7	606	-581
7	700	703	-11	543	501	-17	260	247	H= 9, K= 4			8	821	-818
9	222	195	-12	243	206	-18	110	-69	0	829	-792	9	224	-253
11	1035	-1049	-13	163	158	-19	182	157	1	820	-847	10	773	780
13	102	87	-14	236	264	-20	100	59	2	598	589	11	372	405
15	867	857	-15	620	-588	-21	247	-266	3	781	785	12	439	365
17	433	-451	-16	408	-338	-22	132	-90	4	835	852	13	226	177
19	118	-128	-17	169	159	-23	73	-84	5	73	65	14	533	-479
21	491	532	-18	71	66				6	1359	-1430	15	401	-398
-1	200	-168	-19	268	253	H= 9, K= 3			7	1014	-993	16	122	49
-3	1599	-1542	-20	242	195	0	558	-596	8	69	-57	17	84	49
-5	1359	1346	-21	217	-200	1	469	-417	9	658	653	18	296	275
-7	1202	1261	-22	260	-256	2	211	265	10	1160	1129	19	61	76
-9	1170	-1223	-23	126	-143	3	1400	1408	11	551	539	20	240	-224
-11	138	-156	-24	20	-12	4	443	533	12	136	-60	-1	179	126
-13	622	595				5	700	-713	13	421	-435	-2	556	515
-15	137	-135	H= 9, K= 2			6	616	-666	14	416	-458	-3	578	508
-17	115	-136	0	401	-386	7	474	-541	15	308	-339	-4	35	-28
-19	190	143	1	115	115	8	35	53	16	162	144	-5	1076	-1021
-21	40	57	2	1432	1500	9	827	875	17	494	478	-6	726	-720
-23	230	-255	3	122	-159	10	502	495	18	120	144	-7	362	-302
			4	577	-589	11	38	-32	19	39*	-6	-8	488	446
H= 9, K= 1			5	523	605	12	148	-177	20	314	-278	-9	888	893
0	210	144	6	1850	-1818	13	478	-503	21	450	-391	-10	461	458
1	679	707	7	486	548	14	367	-354	-1	514	-495	-11	223	213
2	124	137	8	877	937	15	530	518	-2	80	-36	-12	842	-780
3	1097	1098	9	130	-91	16	384	360	-3	1212	1190	-13	396	-348
4	617	-603	10	643	682	17	402	467	-4	784	774	-14	58	-60
5	530	-544	11	465	-540	18	157	158	-5	200	-238	-15	98	-48
6	286	280	12	511	-539	19	820	-828	-6	1003	-1028	-16	751	697
7	895	-949	13	64	-79	20	401	-404	-7	1338	-1370	-17	329	354
8	85	44	14	257	-303	21	37	-37	-8	35	54	-18	220	-236
9	805	795	15	435	447	-1	1473	-1457	-9	750	716	-19	419	-425
10	491	-489	16	914	965	-2	113*	-14	-10	899	920	-20	332	-370
11	76	69	17	117	-136	-3	103	99	-11	261	223	-21	111	-113
12	362	-358	18	332	-313	-4	793	800	-12	102*	-18			
13	478	-524	19	96	-138	-5	530	478	-13	365	-324	H= 9, K= 6		
14	799	869	20	500	-479	-6	512	-521	-14	432	-449	0	116	-118
15	414	409	21	153	159	-7	521	-463	-15	41	-37	1	403	-398
16	41	-38	22	525	499	-8	303	-302	-16	363	382	2	444	-400
17	318	315	-1	678	-665	-9	276	210	-17	322	262	3	61	-60
18	627	-620	-2	835	-837	-10	449	442	-18	146	206	4	484	471
19	420	-488	-3	209	-185	-11	280	261	-19	182	164	5	444	433
20	386	404	-4	1076	1068	-12	144	86	-20	461	-454	6	466	445
21	99	52	-5	507	467	-13	38	-41	-21	339	-299	7	382	-384
22	334	328	-6	242	-189	-14	683	-617	-22	72	81	8	771	-778
-1	584	-609	-7	707	676	-15	428	-454				9	187	-152
-2	873	-871	-8	1045	-1032	-16	145	-145	H= 9, K= 5			10	268	-307
-3	328	-358	-9	162	-153	-17	418	379	0	183	-195	11	621	609
-4	230	221	-10	107	100	-18	705	708	1	454	-392	12	558	580
-5	433	-393	-11	35	46	-19	41	49	2	214	-256	13	41	-33
-6	1233	1203	-12	478	486	-20	77	-58	3	616	563	14	116	95

PAGE 18 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 9, K= 6			-10	266	-309	7	601	623	4	78*	5	17	242	258
15	387	-323	-11	263	-251	8	40	28	5	176	-197	18	203	-196
16	324	-328	-12	441	-437	9	40	30	6	350	-343	19	306	282
17	114	100	-13	213	-236	10	292	-307	-1	37	-15	20	243	208
18	61	58	-14	489	519	11	384	-368	-2	248	-250	21	183	-170
19	31	-21	-15	205	213	12	37	20	-3	219	-236	22	89	83
-1	470	444	-16	218	215	13	66	67	-4	231	253	-1	669	643
-2	609	596	-17	252	-261	14	368	317	-5	270	304	-2	1076	-1134
-3	185	147	-18	377	-389	-1	133	116	-6	21	-37	-3	1390	1404
-4	365	-363	-19	35	20	-2	41	-52	-7	129	122	-4	867	-885
-5	725	-731				-3	539	-578	-8	279	-318	-5	1432	-1504
-6	675	-636	H= 9, K= 8			-4	347	-352				-6	1560	1599
-7	161	115	0	435	513	-5	327	344	H= 10, K= 0			-7	271	-225
-8	710	699	1	70	51	-6	132	177	0	470	-500	-8	83	-57
-9	874	867	2	230	-211	-7	254	273	2	942	-946	-9	1191	1229
-10	232	263	3	532	-526	-8	342	378	4	1536	1522	-10	978	-959
-11	379	-310	4	402	-416	-9	536	-607	6	439	454	-11	349	-339
-12	531	-551	5	272	271	-10	140	-167	8	1170	-1173	-12	137	0
-13	396	-380	6	402	362	-11	79*	6	10	152	-132	-13	831	-802
-14	110	-90	7	357	353	-12	201	-206	12	982	984	-14	899	908
-15	283	252	8	206	170	-13	711	710	14	275	-252	-15	644	593
-16	41	29	9	461	-452	-14	284	294	16	741	-807	-16	236	-174
-17	125	-135	10	347	-338	-15	95	-143	18	489	450	-17	165	208
-18	157	-177	11	112	-91				20	87	154	-18	457	-403
-19	156	-162	12	253	277	H= 9, K= 10			22	300	-299	-19	960	-917
-20	157	-137	13	315	276	0	356	351	-2	1453	1471	-20	682	634
			14	155	178	1	40	4	-4	472	-408	-21	534	510
H= 9, K= 7			15	37*	3	2	40	45	-6	1202	-1194	-22	38	40
0	345	379	16	386	-387	3	228	227	-8	1223	1253	-23	611	590
1	144	-195	-1	485	554	4	436	-465	-10	520	524			
2	506	-529	-2	81	69	5	236	-242	-12	2059	-2071	H= 10, K= 2		
3	397	-360	-3	260	-287	6	147	149	-14	103	94	0	1205	-1281
4	112	-85	-4	518	-586	7	38	27	-16	1568	1644	1	1039	1026
5	560	584	-5	470	-500	8	360	389	-18	1035	-1067	2	116*	-19
6	548	471	-6	255	231	9	173	172	-20	350	-329	3	1891	-1902
7	131	198	-7	654	661	10	283	-289	-22	983	998	4	752	733
8	85	83	-8	162	189	11	185	-164				5	71	63
9	607	-630	-9	168	199	-1	148	-153	H= 10, K= 1			6	433	-490
10	404	-373	-10	109	-111	-2	418	-428	0	1118	1132	7	832	902
11	92	102	-11	738	-788	-3	33	-29	1	1223	-1218	8	317	-354
12	103	-98	-12	141	125	-4	137	-138	2	637	631	9	274	-260
13	565	535	-13	146	139	-5	130	104	3	121	109	10	310	365
14	95	-42	-14	112	118	-6	653	675	4	972	-960	11	504	-496
15	395	-376	-15	417	465	-7	93	-60	5	846	837	12	405	397
16	38	-32	-16	162	-185	-8	207	-183	6	472	482	13	236	217
17	201	-152	-17	230	-269	-9	95	-57	7	506	-569	14	227	-271
-1	386	404				-10	525	-594	8	254	291	15	145	155
-2	388	419	H= 9, K= 9			-11	148	205	9	431	-427	16	42	31
-3	39	-34	0	245	243	-12	340	389	10	388	-443	17	464	-488
-4	561	-637	1	355	349				11	1024	1048	18	163	173
-5	378	-351	2	41	-62	H= 9, K= 11			12	92	26	19	246	189
-6	134	-70	3	509	-522	0	217	-232	13	73	106	20	163	-151
-7	180	179	4	75*	-7	1	216	230	14	307	337	21	206	214
-8	436	445	5	208	-220	2	501	493	15	920	-922	-1	1202	1222
-9	388	376	6	136	148	3	82*	-7	16	212	-195	-2	553	534

PAGE 19 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 10, K= 2			-10	805	-810	-20	281	336	7	642	-590	-4	743	691
-3	1348	-1356	-11	523	511	-21	482	457	8	353	414	-5	100	-61
-4	763	778	-12	235	230	-22	588	-582	9	121	116	-6	543	-513
-5	163	-110	-13	89	57				10	246	-226	-7	562	557
-6	658	-665	-14	587	523	H= 10, K= 5			11	437	398	-8	230	-193
-7	1536	1577	-15	196	-137	0	182	-170	12	258	-272	-9	318	-330
-8	34	31	-16	644	-627	1	991	947	13	200	-161	-10	737	697
-9	1003	-1000	-17	80	-75	2	381	-333	14	563	496	-11	335	-323
-10	219	223	-18	890	-835	3	487	551	15	389	-333	-12	76	-94
-11	951	-962	-19	469	467	4	512	567	16	39	-52	-13	196	203
-12	304	-336	-20	872	815	5	887	-840	17	247	165	-14	802	-801
-13	1254	1284	-21	199	-174	6	106	113	18	272	-270	-15	254	210
-14	173	148	-22	236	242	7	328	271	-1	38	23	-16	380	366
-15	491	508				8	224	-199	-2	771	-705	-17	73	-66
-16	439	484	H= 10, K= 4			9	249	233	-3	1007	925	-18	370	344
-17	1630	-1608	0	55	59	10	301	305	-4	285	-281			
-18	259	-270	1	301	280	11	810	-799	-5	248	-203	H= 10, K= 8		
-19	141	166	2	1233	1230	12	238	232	-6	826	804	0	326	293
-20	403	-367	3	631	-625	13	122	83	-7	857	-853	1	86	-78
-21	990	933	4	750	-779	14	343	-327	-8	319	-235	2	562	-525
-22	317	337	5	278	263	15	650	627	-9	304	312	3	300	363
-23	511	-462	6	562	-605	16	240	-217	-10	352	-321	4	42	58
			7	74	54	17	97	-85	-11	423	404	5	306	-293
H= 10, K= 3			8	498	507	18	347	322	-12	783	805	6	630	604
0	1500	1560	9	312	-349	19	272	-216	-13	795	-799	7	262	-245
1	431	414	10	64	52	-1	867	-886	-14	80	-45	8	166	-134
2	402	-383	11	297	310	-2	226	195	-15	104	-95	9	370	306
3	553	527	12	376	-368	-3	1139	-1072	-16	606	-665	10	564	-535
4	969	-907	13	258	219	-4	130*	-12	-17	734	684	11	266	206
5	689	-730	14	203	214	-5	1191	1214	-18	141	139	12	334	309
6	795	856	15	136	-125	-6	690	-665	-19	412	-382	13	259	-197
7	50	42	16	473	410	-7	524	533	-20	289	285	14	222	195
8	138	-176	17	163	-147	-8	378	379				15	165	151
9	458	454	18	385	-349	-9	1025	-964	H= 10, K= 7			-1	387	-414
10	612	-600	19	165	159	-10	299	231	0	752	-724	-2	157	168
11	115	-126	20	222	-175	-11	502	510	1	321	358	-3	362	355
12	376	340	-1	751	732	-12	155	-181	2	99	122	-4	260	-242
13	270	-288	-2	1129	-1131	-13	322	339	3	557	-563	-5	105	123
14	206	173	-3	345	-336	-14	31	-37	4	681	625	-6	42	-33
15	229	312	-4	231	169	-15	821	-813	5	288	275	-7	373	-363
16	473	-554	-5	350	-305	-16	236	198	6	460	-400	-8	276	321
17	101	101	-6	1285	1287	-17	41	-59	7	415	414	-9	59	49
18	40	48	-7	491	489	-18	40*	3	8	351	-318	-10	284	-275
19	292	-314	-8	951	-974	-19	740	698	9	367	-369	-11	579	571
20	283	299	-9	157	-145	-20	192	-209	10	643	552	-12	394	-365
21	76	-48	-10	299	-334	-21	336	-290	11	463	-441	-13	154	-122
-1	758	-726	-11	481	-418				12	95	-46	-14	413	407
-2	32	-5	-12	797	764	H= 10, K= 6			13	468	438	-15	265	-288
-3	345	-305	-13	703	669	0	684	651	14	364	-349	-16	176	193
-4	1839	-1887	-14	552	-523	1	946	-889	15	142	129			
-5	575	596	-15	355	309	2	524	520	16	217	215	H= 10, K= 9		
-6	1012	957	-16	603	-652	3	390	366	17	528	-457	0	64	47
-7	489	-468	-17	664	-694	4	840	-851	-1	391	363	1	134	-169
-8	811	753	-18	837	819	5	444	478	-2	126	100	2	307	309
-9	426	-360	-19	176	-149	6	46	-51	-3	479	-468	3	41	-47

PAGE 20 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 10, K= 9			-1	273	-291	-1	312	280	-10	730	-660	-20	193	171
4	279	-272	-2	100	-106	-2	844	855	-11	550	-505	-21	38	25
5	304	332	-3	205	252	-3	632	-590	-12	745	724	-22	140	-117
6	391	-345	-4	146	-170	-4	1223	-1177	-13	96	102			
7	87	-50	-5	35	27	-5	113	-60	-14	318	280	H= 11, K= 4		
8	285	274	-6	312	312	-6	164	-126	-15	644	580	0	146	137
9	417	-385				-7	575	601	-16	707	-671	1	265	233
10	163	115	H= 11, K= 0			-8	1108	1063	-17	492	-492	2	510	525
11	154	123	1	462	380	-9	281	-301	-18	42	-38	3	616	650
12	253	-250	3	1484	-1466	-10	357	-298	-19	24	-35	4	629	-628
13	273	255	5	362	400	-11	338	344	-20	333	300	5	750	-784
-1	287	274	7	226	207	-12	830	-776	-21	222	219	6	205	-152
-2	276	-291	9	461	-495	-13	42	-44	-22	93	-60	7	246	-253
-3	80	50	11	186	233	-14	303	296				8	243	208
-4	63	34	13	432	520	-15	152	-155	H= 11, K= 3			9	696	722
-5	386	-391	15	426	-499	-16	136	137	0	252	267	10	41	-34
-6	257	242	17	523	-511	-17	126	-119	1	1359	1337	11	224	-226
-7	138	138	19	513	504	-18	381	-360	2	356	415	12	54	-50
-8	294	-313	21	36	34	-19	79	65	3	769	-693	13	664	-665
-9	377	398	-1	1735	1713	-20	274	226	4	424	-420	14	432	459
-10	121	44	-3	480	413	-21	90	78	5	791	-878	15	604	591
-11	375	-377	-5	1317	-1323	-22	171	202	6	531	-543	16	92	-113
-12	381	397	-7	536	556	-23	146	-163	7	1129	1125	17	243	194
-13	80	-114	-9	379	405				8	475	531	18	266	-281
-14	144	-127	-11	951	-953	H= 11, K= 2			9	68*	-16	19	498	-479
			-13	42	45	0	826	837	10	265	261	-1	825	-775
H= 10, K= 10			-15	657	592	1	213	282	11	1066	-1057	-2	534	-554
0	69	-83	-17	449	-482	2	396	406	12	344	-373	-3	80	-63
1	409	397	-19	42	-43	3	289	-249	13	296	322	-4	439	387
2	148	-155	-21	402	382	4	1494	-1504	14	42	-38	-5	1045	1032
3	64	-37	-23	50	50	5	252	249	15	588	630	-6	299	237
4	285	309				6	534	561	16	347	347	-7	551	-487
5	456	-400	H= 11, K= 1			7	60	-69	17	582	-625	-8	140	-92
6	62*	-9	0	554	603	8	867	891	18	217	-221	-9	479	-434
7	205	165	1	835	888	9	445	-509	19	88*	2	-10	110	92
8	240	-260	2	1014	-1011	10	1202	-1199	20	93	-50	-11	596	605
9	220	188	3	412	-437	11	111	145	-1	352	-293	-12	509	492
-1	80	-65	4	124	-154	12	243	-262	-2	857	-861	-13	327	313
-2	31	28	5	210	-253	13	145	179	-3	1254	-1234	-14	390	-357
-3	299	-312	6	1233	1242	14	1108	1090	-4	559	495	-15	705	-634
-4	110	-65	7	505	576	15	24	89	-5	745	694	-16	543	-534
-5	147	125	8	398	-450	16	318	-338	-6	656	589	-17	285	295
-6	159	-196	9	95	-110	17	167	-181	-7	705	679	-18	347	291
-7	206	216	10	481	-480	18	551	-598	-8	541	-556	-19	284	275
-8	348	383	11	448	-485	19	87	109	-9	831	-748	-20	99	108
-9	263	-255	12	271	264	20	379	424	-10	70	93	-21	230	-214
-10	284	264	13	223	259	-1	296	308	-11	149	195			
-11	91	77	14	306	374	-2	292	-283	-12	440	459	H= 11, K= 5		
			15	512	491	-3	282	-364	-13	534	481	0	426	-452
H= 10, K= 11			16	585	-583	-4	523	-557	-14	67	-49	1	84	79
0	264	240	17	554	-554	-5	109	-93	-15	281	-314	2	625	566
1	206	-211	18	41	33	-6	1139	1071	-16	400	-364	3	308	273
2	141	154	19	79	-66	-7	153	183	-17	470	-504	4	22	-45
3	357	343	20	305	345	-8	577	-504	-18	129	137	5	427	-355
4	367	-345	21	249	286	-9	106	-104	-19	353	321	6	585	-555

PAGE 21 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 11, K= 5			-3	356	381	H= 11, K= 8			-10	63	-80	4	606	612
7	188	-196	-4	596	591	0	123	-134	-11	547	-571	5	316	-362
8	169	140	-5	69*	-1	1	361	-358	-12	229	-226	6	408	412
9	408	409	-6	428	-437	2	658	-633	-13	71	-66	7	344	-396
10	428	422	-7	382	-385	3	116	140				8	460	-470
11	42	61	-8	586	-581	4	457	487	H= 11, K= 10			9	232	278
12	403	-370	-9	194	169	5	336	351	0	226	254	10	133*	-25
13	24	-63	-10	435	391	6	148	154	1	39	-25	11	97	118
14	242	-224	-11	235	174	7	277	-285	2	116	-147	12	543	549
15	144	-85	-12	400	439	8	434	-465	3	123	-106	13	108*	-14
16	657	621	-13	297	-293	9	230	-184	4	189	-150	14	114	-161
17	198	169	-14	401	-360	10	247	250	5	132	122	15	42	-46
18	226	-178	-15	118	-123	11	436	379	6	340	289	16	145	-166
-1	500	-426	-16	48	-31	12	196	229	7	21	73	17	181	210
-2	270	-219	-17	521	519	13	126	75	8	45	27	18	92	147
-3	388	381	-18	321	337	14	280	-267	-1	217	215	19	66	-102
-4	828	805	-19	179	-173	-1	127	95	-2	64	43	20	42	52
-5	623	541				-2	561	549	-3	228	-210	-1	1024	-1010
-6	39	-62	H= 11, K= 7			-3	317	317	-4	270	-319	-2	762	832
-7	698	-675	0	672	-663	-4	106	41	-5	38	-45	-3	982	987
-8	666	-592	1	490	-456	-5	771	-756	-6	38	47	-4	166	-207
-9	228	-233	2	41	45	-6	238	-240	-7	230	255	-5	440	462
-10	33	-32	3	269	252	-7	113	85	-8	266	285	-6	463	-468
-11	593	614	4	775	753	-8	321	293	-9	36	-37	-7	961	-985
-12	327	290	5	322	294	-9	594	582	-10	250	-250	-8	1030	949
-13	148	-129	6	468	-430	-10	78	56				-9	125	117
-14	191	-156	7	611	-582	-11	41	-48	H= 12, K= 0			-10	80	-43
-15	292	-292	8	338	-344	-12	501	-477	0	1557	-1554	-11	721	669
-16	133	-85	9	134	169	-13	496	-482	2	505	518	-12	827	-816
-17	373	324	10	513	531	-14	204	200	4	524	463	-13	1038	-987
-18	297	286	11	527	483	-15	170	203	6	481	-535	-14	348	341
-19	38	29	12	81	37	-16	297	313	8	191	157	-15	140	-104
-20	172	-145	13	263	-239				10	711	684	-16	524	567
			14	240	-225	H= 11, K= 9			12	315	-374	-17	1076	1128
H= 11, K= 6			15	339	-335	0	63	93	14	211	-254	-18	353	-366
0	390	-409	16	82*	5	1	323	-308	16	418	440	-19	220	-222
1	256	-248	-1	204	-203	2	374	-335	18	89	140	-20	258	-254
2	48	-23	-2	410	358	3	219	-215	20	345	-358	-21	748	-684
3	628	617	-3	622	546	4	164	229	-2	34	27	-22	502	489
4	294	309	-4	292	280	5	297	300	-4	1390	1403			
5	41	33	-5	83	-76	6	259	234	-6	1076	-1067	H= 12, K= 2		
6	397	-371	-6	933	-870	7	40	-22	-8	653	-610	0	187	-226
7	562	-527	-7	568	-513	8	397	-352	-10	1003	1006	1	1421	-1391
8	154	-120	-8	283	235	9	353	-348	-12	311	-293	2	84	109
9	362	285	-9	205	222	10	94*	-19	-14	424	-362	3	387	-388
10	428	404	-10	720	713	11	219	217	-16	794	714	4	261	-270
11	42	66	-11	130	91	-1	455	441	-18	613	642	5	1181	1164
12	155	129	-12	210	-226	-2	368	354	-20	805	-860	6	397	-406
13	266	-199	-13	41	-22	-3	31	24	-22	121	-166	7	38	-57
14	459	-422	-14	327	-330	-4	326	-349				8	135	197
15	190	171	-15	109	-76	-5	441	-434	H= 12, K= 1			9	460	-468
16	156	158	-16	419	429	-6	183	-175	0	416	-445	10	127	-165
17	291	266	-17	180	206	-7	372	412	1	690	-683	11	617	628
-1	323	-323	-18	35	4	-8	291	326	2	686	-767	12	42	-19
-2	68	-73				-9	370	414	3	1024	1023	13	31	42

PAGE 22 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 12,	K= 2		-4	293	-291	-17	385	401	9	627	566	-12	734	814
14	64	94	-5	303	311	-18	518	-470	10	334	-259	-13	204	-217
15	512	-506	-6	1108	-1094	-19	302	-308	11	109	-42	-14	108	-156
16	110	72	-7	439	455	-20	665	642	12	466	416	-15	281	254
17	58	79	-8	564	570				13	386	-347	-16	491	-497
18	99	-46	-9	319	-288	H= 12,	K= 5		14	61	-58	-17	35	-42
19	147	199	-10	564	581	0	570	-631	15	169	162			
20	35	-49	-11	435	-378	1	105	116	16	341	-336	H= 12,	K= 8	
-1	920	928	-12	1275	-1270	2	191	164	-1	601	-628	0	483	-490
-2	408	403	-13	497	506	3	357	-395	-2	285	274	1	536	510
-3	891	972	-14	42	-41	4	146	173	-3	144	-149	2	334	-311
-4	465	516	-15	325	244	5	352	338	-4	490	-521	3	124	-115
-5	1724	-1710	-16	1139	1079	6	465	-428	-5	728	663	4	645	635
-6	36	47	-17	500	-472	7	344	338	-6	115	-113	5	520	-461
-7	138	127	-18	566	-555	8	160	-178	-7	458	-424	6	159	135
-8	635	-654	-19	117	145	9	498	-473	-8	457	444	7	329	264
-9	1264	1231	-20	396	-341	10	579	584	-9	504	-441	8	494	-498
-10	101	67	-21	176	185	11	347	-304	-10	334	-304	9	144	148
-11	506	-521				12	42	-44	-11	902	898	10	280	249
-12	209	272	H= 12,	K= 4		13	413	361	-12	238	-245	11	292	-278
-13	700	-732	0	765	808	14	327	-294	-13	59	-46	12	257	248
-14	398	-376	1	397	-370	15	70*	-21	-14	541	531	13	119	119
-15	614	564	2	88	77	16	331	299	-15	513	-565	-1	142*	-9
-16	146	132	3	514	516	17	231	-210	-16	130	-116	-2	149	156
-17	262	256	4	533	-500	18	77	71	-17	134	99	-3	477	-523
-18	255	203	5	72	57	-1	330	370	-18	495	-527	-4	225	179
-19	773	-778	6	225	202	-2	39	24				-5	362	366
-20	329	-282	7	513	-548	-3	379	-380	H= 12,	K= 7		-6	531	-500
-21	382	346	8	421	351	-4	567	561	0	69	51	-7	593	653
-22	71	-69	9	164	-156	-5	40	69	1	165	-201	-8	168	194
			10	446	-421	-6	40	-49	2	702	665	-9	568	-623
H= 12,	K= 3		11	774	745	-7	460	449	3	122	-117	-10	436	450
0	387	-423	12	122	86	-8	299	-330	4	478	-433	-11	171	-159
1	387	384	13	69	-52	-9	577	-526	5	517	475	-12	283	-271
2	899	-888	14	278	241	-10	344	329	6	404	-370	-13	268	302
3	150	-219	15	326	-342	-11	881	-826	7	374	-348	-14	191	-194
4	1150	1143	16	228	-235	-12	347	346	8	432	370	-15	58	-81
5	523	-516	17	264	259	-13	993	959	9	417	-401			
6	428	448	18	249	-206	-14	429	-454	10	67	70	H= 12,	K= 9	
7	355	370	-1	167	-170	-15	372	380	11	707	660	0	577	554
8	658	-676	-2	329	-347	-16	67*	-24	12	293	-254	1	323	-277
9	125	92	-3	470	430	-17	943	-1016	13	95	71	2	91	-99
10	58	-82	-4	825	-763	-18	270	278	14	37	52	3	133	121
11	272	-276	-5	144	-160	-19	37	40	15	443	-381	4	602	-592
12	355	367	-6	757	741				-1	245	233	5	200	192
13	122	124	-7	380	-327	H= 12,	K= 6		-2	496	-460	6	236	228
14	161	-191	-8	216	204	0	347	339	-3	34	49	7	362	-316
15	76	139	-9	607	525	1	348	346	-4	119	131	8	247	247
16	123	-153	-10	720	-693	2	250	-240	-5	372	-363	9	37*	14
17	240	-262	-11	167	125	3	273	256	-6	596	568	10	304	-294
18	54	119	-12	164	87	4	110	145	-7	98	149	-1	47	64
19	21	-21	-13	721	-671	5	636	-578	-8	716	-709	-2	41	44
-1	139	161	-14	805	811	6	398	374	-9	418	409	-3	352	332
-2	993	998	-15	103	92	7	189	-152	-10	251	-252	-4	309	-308
-3	366	-391	-16	547	-484	8	400	-372	-11	206	-256	-5	112	-163

PAGE 23 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 12, K= 9			5	553	632	17	186	213	-11	524	-558	3	298	-271
-6	310	305	6	180	-239	18	262	268	-12	145	-182	4	794	-761
-7	390	-388	7	186	145	19	51*	-5	-13	378	356	5	87	-40
-8	39	23	8	551	-597	-1	426	415	-14	610	598	6	152	84
-9	129	147	9	568	-656	-2	888	814	-15	310	312	7	270	226
-10	322	-352	10	794	784	-3	403	378	-16	131	-90	8	581	577
-11	321	341	11	396	408	-4	754	-719	-17	425	-402	9	82	-50
-12	62	72	12	104	134	-5	458	-485	-18	240	-247	10	415	-405
			13	523	566	-6	119	-151	-19	39	-55	11	291	-253
H= 12, K= 10			14	920	-900	-7	240	-163	-20	137	118	12	300	-271
0	211	-196	15	390	-376	-8	699	619				13	24*	-5
1	38	24	16	280	305	-9	145	146	H= 13, K= 4			14	503	504
2	210	214	17	257	-227	-10	333	-287	0	619	583	15	249	227
3	429	-411	18	418	403	-11	345	-397	1	508	480	16	108	-111
4	74	87	19	297	280	-12	680	-672	2	688	-710	-1	168	126
5	220	217	-1	614	595	-13	81	39	3	386	-394	-2	372	-395
6	108	-91	-2	568	514	-14	555	569	4	427	-444	-3	249	-235
-1	431	392	-3	164	204	-15	209	228	5	130	-105	-4	457	-461
-2	88	-101	-4	794	786	-16	355	337	6	953	871	-5	680	727
-3	240	-236	-5	591	-640	-17	42	41	7	652	638	-6	810	866
-4	183	208	-6	1223	-1170	-18	478	-555	8	84	94	-7	34	-16
-5	255	-247	-7	405	421	-19	110	-65	9	42	-32	-8	212	-218
-6	37	-31	-8	152	-86	-20	108	104	10	479	-471	-9	429	-439
-7	345	359	-9	235	234	-21	36	57	11	502	-458	-10	629	-692
-8	91	-90	-10	652	621				12	42	21	-11	208	-202
			-11	693	-705	H= 13, K= 3			13	131	140	-12	379	385
H= 13, K= 0			-12	535	-572	0	530	562	14	142	167	-13	325	339
1	1348	-1307	-13	42	56	1	38	-34	15	461	412	-14	218	256
3	308	261	-14	101*	-21	2	97	-97	16	125	-166	-15	41	-66
5	647	717	-15	558	563	3	1118	-1170	17	519	-494	-16	320	-377
7	930	-923	-16	826	830	4	227	-253	-1	272	268	-17	248	-314
9	164	175	-17	428	-513	5	593	614	-2	116*	19	-18	31	66
11	1076	1058	-18	96	-121	6	282	326	-3	898	-856	-19	283	297
13	342	-313	-19	161	-186	7	481	474	-4	385	-403			
15	711	-758	-20	369	-396	8	34	31	-5	40	33	H= 13, K= 6		
17	568	608	-21	234	308	9	825	-819	-6	594	580	0	42	41
19	107	65				10	261	-262	-7	920	919	1	466	426
-1	101*	23	H= 13, K= 2			11	249	253	-8	146	150	2	628	610
-3	1484	1535	0	501	552	12	115	-85	-9	348	-310	3	356	-305
-5	171	-127	1	237	-216	13	413	444	-10	640	-601	4	334	-300
-7	1170	-1172	2	1244	-1312	14	266	274	-11	321	-296	5	203	-210
-9	827	833	3	93	-118	15	561	-580	-12	43	-78	6	671	-613
-11	103	112	4	86	96	16	80	-53	-13	234	268	7	341	319
-13	805	-794	5	93	53	17	218	-196	-14	276	276	8	397	354
-15	270	350	6	968	1018	18	209	-197	-15	178	143	9	217	210
-17	899	947	7	137	-144	-1	1129	1164	-16	167	-195	10	277	226
-19	721	-736	8	532	-526	-2	191	118	-17	449	-503	11	448	-399
-21	117	-164	9	79	-57	-3	142	-179	-18	149	-151	12	371	-335
			10	495	-462	-4	603	-603	-19	191	158	13	31	-31
H= 13, K= 1			11	217	251	-5	919	-850	-20	301	273	14	44*	-6
0	585	-618	12	712	723	-6	583	578				15	373	299
1	85	-85	13	200	-244	-7	408	379	H= 13, K= 5			-1	291	-256
2	510	-522	14	119	127	-8	378	390	0	258	312	-2	591	-583
3	810	-872	15	42	-43	-9	138	150	1	109	135	-3	316	-345
4	972	955	16	742	-745	-10	805	-841	2	422	451	-4	353	315

PAGE 24 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 13, K= 6			7	414	-348	6	438	-514	-18	266	270	9	384	-413
-5	597	573	8	40	48	8	416	445	-19	515	581	10	488	439
-6	344	355	9	139	141	10	43	-36	-20	341	-352	11	43	-74
-7	162	152	10	222	190	12	355	-375				12	225	-188
-8	161	-162	11	47	62	14	193	206	H= 14, K= 2			13	152	145
-9	644	-645	-1	404	-412	16	222	184	0	39	46	14	41	42
-10	43	23	-2	183	-151	18	139	-79	1	214	-263	15	93	-51
-11	84	81	-3	264	248	-2	815	-842	2	130	-77	16	132	158
-12	183	171	-4	521	520	-4	494	437	3	543	570	17	100	-87
-13	363	358	-5	228	278	-6	929	881	4	99	58	-1	195	225
-14	84	-49	-6	303	-319	-8	531	-531	5	139	-188	-2	118	-61
-15	163	-212	-7	86	-107	-10	516	-554	6	182	178	-3	240	186
-16	343	-366	-8	217	-230	-12	1244	1318	7	588	-583	-4	675	621
-17	77	-83	-9	111	-91	-14	113	99	8	190	196	-5	350	-324
			-10	317	328	-16	909	-940	9	148	183	-6	479	-481
H= 13, K= 7			-11	346	348	-18	491	488	10	500	-508	-7	291	285
0	143	-201	-12	97	-89	-20	198	222	11	298	335	-8	617	-634
1	370	330	-13	135	-173				12	70	-71	-9	289	235
2	164	166				H= 14, K= 1			13	43	-60	-10	747	683
3	462	456	H= 13, K= 9			0	529	-605	14	266	299	-11	774	-753
4	298	226	0	304	-288	1	582	643	15	41*	6	-12	231	205
5	581	-532	1	242	-254	2	363	373	16	130	-163	-13	92	114
6	193	-171	2	60	-37	3	112	-92	17	90	67	-14	493	-467
7	147	-133	3	124	160	4	217	261	18	36	-71	-15	611	598
8	138	-88	4	274	258	5	335	-305	-1	543	-581	-16	344	339
9	468	397	5	150	199	6	384	-401	-2	310	-298	-17	142	-166
10	66*	5	6	220	-201	7	171	183	-3	667	641	-18	432	491
11	157	-131	7	203	-190	8	249	-278	-4	293	336	-19	185	-180
12	158	-121	8	109	-77	9	214	180	-5	84	-88			
13	336	-328	-1	122	-168	10	143	90	-6	465	538	H= 14, K= 4		
-1	600	-632	-2	92	87	11	553	-639	-7	564	-557	0	92	-130
-2	249	-265	-3	362	335	12	137	143	-8	393	-363	1	165	-138
-3	141	136	-4	206	172	13	101	87	-9	571	516	2	459	-461
-4	239	256	-5	51	-63	14	42	-34	-10	652	-660	3	480	465
-5	353	409	-6	190	-188	15	195	223	-11	932	984	4	266	257
-6	193	171	-7	315	-272	16	66*	16	-12	74	-74	5	552	-533
-7	410	-392	-8	38	-39	17	38	-51	-13	1097	-1100	6	452	458
-8	123	-103	-9	315	265	18	76	136	-14	261	306	7	138	-147
-9	166	-191	-10	182	197	-1	258	-235	-15	243	-251	8	407	-409
-10	76	80				-2	118	103	-16	116	-103	9	421	390
-11	457	455	H= 13, K= 10			-3	554	-556	-17	804	819	10	158	-178
-12	377	329	0	289	-251	-4	456	440	-18	83	51	11	120	-163
-13	99	80	1	191	-171	-5	961	926	-19	111	-106	12	519	524
-14	407	-400	2	175	-149	-6	237	-242	-20	209	238	13	41*	6
-15	341	-368	3	139	146	-7	109	113				14	57	-38
-16	124	-138	-1	36	26	-8	150	-150	H= 14, K= 3			15	135	157
			-2	317	276	-9	976	-929	0	561	-580	16	242	-187
H= 13, K= 8			-3	131	114	-10	633	667	1	428	-423	-1	216	-177
0	348	-283	-4	120	117	-11	228	170	2	468	522	-2	479	457
1	423	-422	-5	186	-175	-12	148	-164	3	135	-89	-3	226	210
2	450	413				-13	796	743	4	31	-26	-4	342	-323
3	577	531	H= 14, K= 0			-14	758	-745	5	447	427	-5	247	269
4	76	-47	0	150	112	-15	540	-481	6	544	-581	-6	419	-416
5	41	36	2	763	752	-16	352	351	7	436	423	-7	286	-302
6	208	-156	4	506	-526	-17	232	-232	8	43	47	-8	822	770

PAGE 25 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 14, K= 4			5	334	-329	H= 14, K= 8			-7	42	34	7	160	-197
-9	392	-382	6	35	-33	0	480	-465	-9	885	-961	8	679	-749
-10	101	151	7	504	422	1	259	-279	-11	349	328	9	363	386
-11	368	397	8	332	-341	2	300	261	-13	147	118	10	753	813
-12	815	-888	9	42	69	3	120	-113	-15	431	-406	11	43	22
-13	43	57	10	277	262	4	120	83	-17	386	383	12	81	95
-14	386	369	11	528	-459	5	446	415	-19	251	262	13	368	-347
-15	291	-272	12	91	57	6	382	-331				14	857	-827
-16	506	560	13	135	142	7	39*	10	H= 15, K= 1			15	105	88
-17	227	235	14	238	-239	8	116	75	0	417	-472	16	280	305
-18	431	-455	-1	55	77	9	382	-308	1	353	-436	17	108	119
-19	88	93	-2	369	320	10	341	302	2	909	891	-1	325	-344
			-3	566	-540	-1	263	266	3	135	151	-2	590	590
H= 14, K= 5			-4	163	189	-2	21	-23	4	83	92	-3	79	93
0	355	356	-5	351	341	-3	84	-66	5	139	153	-4	883	871
1	724	-713	-6	504	-523	-4	523	567	6	684	-747	-5	42	33
2	454	407	-7	459	448	-5	138	-128	7	308	-328	-6	1139	-1178
3	55	-84	-8	117	126	-6	208	-210	8	200	216	-7	254	-226
4	410	-380	-9	333	-363	-7	200	195	9	43	47	-8	74	53
5	647	607	-10	367	412	-8	272	-291	10	512	531	-9	137	-174
6	459	-421	-11	373	-402	-9	39	-55	11	314	344	-10	788	769
7	155	-130	-12	487	-525	-10	201	210	12	493	-451	-11	495	451
8	461	419	-13	412	456	-11	234	-260	13	316	-338	-12	553	-578
9	352	-367	-14	118	-142	-12	276	286	14	171	-192	-13	136	-99
10	156	-138	-15	38	53				15	177	-189	-14	270	-287
11	338	365	-16	448	519	H= 14, K= 9			16	491	463	-15	54	-98
12	127	-136				0	122	-75	17	390	383	-16	656	688
13	133	98	H= 14, K= 7			1	122	114	-1	135	-92	-17	293	306
14	210	194	0	555	498	2	344	-344	-2	417	-421	-18	80	81
15	281	-254	1	137	-139	3	149	148	-3	653	655	-19	37	-47
-1	280	301	2	95	-71	4	284	255	-4	442	446			
-2	238	-214	3	605	569	5	185	-165	-5	160	-178	H= 15, K= 3		
-3	473	446	4	274	-207	6	154	153	-6	72	62	0	108	72
-4	299	-278	5	206	-197	-1	396	-385	-7	303	-303	1	892	-846
-5	774	-763	6	64	85	-2	442	443	-8	571	-539	2	583	-617
-6	718	742	7	458	-445	-3	89	77	-9	60	64	3	831	841
-7	247	-265	8	217	180	-4	142	-178	-10	201	164	4	166	134
-8	150	-139	9	336	274	-5	348	375	-11	129*	28	5	279	332
-9	813	807	10	330	-246	-6	302	-318	-12	728	734	6	481	504
-10	412	-393	11	304	280	-7	214	-219	-13	155	-206	7	1139	-1104
-11	127	-116	12	124	106	-8	238	232	-14	508	-500	8	305	-305
-12	323	349	-1	496	-515				-15	42	30	9	138	79
-13	504	-537	-2	154	-150	H= 15, K= 0			-16	293	-309	10	144	-159
-14	121	-110	-3	485	501	1	21	22	-17	333	391	11	763	830
-15	382	419	-4	456	-467	3	907	938	-18	529	551	12	293	292
-16	320	-343	-5	216	211	5	106	-74	-19	242	-253	13	289	-289
-17	163	184	-6	295	320	7	664	-637				14	23*	1
-18	115	122	-7	442	-451	9	699	690	H= 15, K= 2			15	416	-389
			-8	153	144	11	134	-112	0	680	-732	16	185	-185
H= 14, K= 6			-9	250	236	13	512	-521	1	79	-73	-1	59	-33
0	434	-428	-10	310	-342	15	482	524	2	403	-384	-2	456	440
1	496	514	-11	251	286	17	251	252	3	384	435	-3	935	922
2	171	-214	-12	99	-48	-1	1097	-1089	4	975	1059	-4	282	-225
3	230	-158	-13	322	-371	-3	41*	9	5	237	-295	-5	609	-576
4	591	548	-14	292	321	-5	1264	1258	6	345	-343	-6	239	-256

PAGE 26 CADMIUM DINITRATO TRIS[2:6-DIMETHYL GAMMA-PYRONE]

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 15, K= 3			5	116	-116	-15	76	56	-3	37	-55	-14	136	-49
-7	436	-452	6	533	470				-4	219	211	-15	268	292
-8	285	225	7	253	231	H= 15, K= 7			-5	195	220	-16	362	-395
-9	1055	1016	8	238	-193	0	326	301				-17	530	-540
-10	43	-56	9	68	-71	1	441	413	H= 16, K= 0			-18	162	221
-11	106	-122	10	380	-331	2	42	54	0	357	376			
-12	350	-355	11	41	-30	3	570	-558	2	208	-254	H= 16, K= 2		
-13	745	-791	12	304	209	4	76	-67	4	74	97	0	70	46
-14	121	127	13	103	74	5	189	-180	6	424	452	1	607	657
-15	281	312	14	76	100	6	90	90	8	70	52	2	458	-467
-16	495	543	-1	273	229	7	518	511	10	502	-477	3	105	70
-17	489	538	-2	481	434	8	75	60	12	160	134	4	183	156
-18	231	-264	-3	43	-29	9	79	-44	14	100	54	5	445	-452
			-4	688	-628	10	134	-139	16	59	-52	6	448	484
H= 15, K= 4			-5	355	-361	-1	173	212	-2	109	82	7	35	-57
0	362	-350	-6	213	236	-2	260	-247	-4	407	-386	8	193	-226
1	366	-375	-7	160	-115	-3	371	-382	-6	172	193	9	353	369
2	154	-125	-8	624	620	-4	238	-232	-8	387	410	10	38	47
3	614	-563	-9	241	275	-5	146	159	-10	818	-749	11	213	-246
4	259	303	-10	326	-321	-6	377	411	-12	43	-30	12	112	92
5	852	787	-11	334	-383	-7	198	198	-14	773	756	13	118	-120
6	50	37	-12	470	-494	-8	122	121	-16	512	-477	14	131	-148
7	168	124	-13	152	123	-9	342	-332	-18	364	-422	15	91	67
8	259	-287	-14	348	354	-10	344	-392				-1	237	-175
9	715	-675	-15	255	253	-11	63	-56	H= 16, K= 1			-2	43	39
10	206	292	-16	94	120	-12	53	46	0	77	62	-3	423	-399
11	216	189				-13	240	258	1	144	195	-4	171	-160
12	113	132	H= 15, K= 6						2	155	162	-5	700	710
13	352	376	0	574	553	H= 15, K= 8			3	159	-153	-6	348	-336
14	331	-356	1	43	22	0	178	134	4	217	208	-7	96	94
15	282	-286	2	129	-124	1	291	298	5	251	284	-8	351	342
-1	766	702	3	262	-282	2	234	245	6	294	-330	-9	895	-884
-2	433	377	4	547	-495	3	96	52	7	191	168	-10	43	44
-3	85	-93	5	206	161	4	325	-310	8	257	250	-11	315	317
-4	156	172	6	289	325	5	239	-225	9	276	-296	-12	113	-146
-5	820	-791	7	232	185	6	38	43	10	43*	-10	-13	641	692
-6	714	-686	8	266	223	7	52	35	11	60	-57	-14	260	257
-7	553	612	9	372	-300	8	250	256	12	345	-375	-15	570	-629
-8	234	208	10	277	-245	-1	103*	-23	13	148	168	-16	40	-52
-9	517	530	11	63	-45	-2	341	-381	14	40	36	-17	138	-166
-10	199	197	12	37	16	-3	251	-266	15	38	-83	-18	193	-190
-11	799	-849	-1	338	392	-4	103*	-4	16	104	103			
-12	381	-366	-2	126*	14	-5	347	406	-1	154	115	H= 16, K= 3		
-13	77	-106	-3	321	-308	-6	264	299	-2	260	-223	0	318	302
-14	345	321	-4	321	-292	-7	74	-96	-3	309	-280	1	374	-372
-15	630	691	-5	289	-296	-8	189	-197	-4	326	356	2	288	264
-16	127	148	-6	211	229	-9	348	-372	-5	43	-49	3	134	101
-17	121	-131	-7	531	552	-10	134	-149	-6	137	136	4	429	-423
			-8	69	51				-7	236	256	5	400	353
H= 15, K= 5			-9	42	68	H= 15, K= 9			-8	711	-705	6	293	-274
0	273	231	-10	348	-425	0	149	-115	-9	43	-36	7	268	-234
1	311	302	-11	458	-502	1	239	224	-10	312	-272	8	370	348
2	939	-884	-12	33	50	2	281	243	-11	651	-702	9	230	-214
3	285	-286	-13	216	212	-1	234	-226	-12	610	657	10	94	-80
4	291	218	-14	319	345	-2	262	-255	-13	525	519	11	289	269

PAGE 27 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 16, K= 3			H= 16, K= 5			-13	111	134	-17	421	-489	-4	714	670
12	129	-128	0	367	351							-5	232	252
13	39	-63	1	43	-71	H= 16, K= 7			H= 17, K= 1			-6	330	-378
14	140	138	2	220	-241	0	95	-106	0	627	594	-7	389	322
15	36	-92	3	204	153	1	312	290	1	209	213	-8	622	-592
-1	105	-60	4	114*	0	2	389	-365	2	83	99	-9	286	-316
-2	374	-349	5	141	-129	3	93*	10	3	452	473	-10	455	441
-3	404	377	6	245	227	4	152	143	4	465	-507	-11	118	155
-4	248	223	7	238	-196	5	403	-340	5	529	-552	-12	508	631
-5	286	-263	8	41	-51	6	116	92	6	131	112	-13	150	226
-6	590	590	9	153	151	7	359	319	7	127	-75	-14	538	-556
-7	231	-210	10	344	-317	8	160	-144	8	539	545	-15	162	-167
-8	309	-371	11	39	-45	-1	250	-235	9	507	527	-16	255	-277
-9	268	227	12	158	150	-2	512	476	10	451	-484			
-10	551	-579	-1	259	-265	-3	202	-189	11	158	-142	H= 17, K= 3		
-11	266	277	-2	75	86	-4	196	-181	12	223	-223	0	390	-411
-12	700	704	-3	424	441	-5	486	510	13	270	-260	1	333	297
-13	251	-234	-4	472	-432	-6	271	-288	14	394	478	2	31	27
-14	154	166	-5	86	62	-7	89	-57	-1	583	-627	3	367	377
-15	31	21	-6	147	165	-8	318	304	-2	472	-526	4	506	480
-16	523	-543	-7	354	-358	-9	365	-409	-3	141	110	5	341	-347
-17	176	189	-8	141	102	-10	38	19	-4	698	-700	6	161	-158
			-9	194	151	-11	116	137	-5	338	398	7	247	-173
H= 16, K= 4			-10	229	-199				-6	1020	1078	8	424	-418
0	431	-430	-11	328	306	H= 16, K= 8			-7	349	-379	9	514	478
1	390	366	-12	233	-222	0	349	314	-8	44	40	10	248	210
2	79	106	-13	371	-354	1	356	-353	-9	345	-382	11	95	-136
3	340	-332	-14	368	372	2	308	286	-10	736	-767	12	102	78
4	185	167	-15	112	-54	3	223	195	-11	559	616	13	357	-316
5	326	-245				4	340	-274	-12	368	366	-1	752	-750
6	172	-156	H= 16, K= 6			5	111	118	-13	120	50	-2	153	131
7	310	304	0	304	-267	-1	38	-32	-14	311	358	-3	194	211
8	231	-228	1	228	-210	-2	468	-448	-15	356	-351	-4	153	184
9	54	38	2	284	301	-3	400	385	-16	386	-345	-5	709	700
10	141	84	3	186	-192	-4	135	-115	-17	139	126	-6	94	-50
11	311	-321	4	131	-128	-5	210	-178				-7	512	-544
12	82*	12	5	295	232	-6	467	471	H= 17, K= 2			-8	75	-69
13	224	191	6	134	-100	-7	246	-256	0	314	-321	-9	404	-446
14	47*	5	7	131	127				1	158	-137	-10	126	124
-1	359	356	8	348	284	H= 17, K= 0			2	1191	1170	-11	621	618
-2	347	338	9	273	-250	1	803	792	3	44	-63	-12	48*	5
-3	526	-505	10	38	-63	3	972	-949	4	233	-288	-13	57	-66
-4	456	457	11	60	-39	5	385	-374	5	71	39	-14	243	-259
-5	154	153	-1	460	450	7	1170	1131	6	726	-747	-15	434	-424
-6	349	-387	-2	273	-291	9	60	47	7	50	58	-16	192	236
-7	222	178	-3	43	-52	11	846	-814	8	460	486			
-8	296	-277	-4	404	426	13	213	237	9	81	30	H= 17, K= 4		
-9	364	-342	-5	459	-482	-1	149	154	10	403	380	0	249	-214
-10	442	428	-6	77	45	-3	888	-880	11	97	-142	1	456	-464
-11	130	-132	-7	271	270	-5	530	524	12	473	-443	2	397	369
-12	72*	2	-8	322	-319	-7	548	555	13	96	49	3	488	499
-13	336	328	-9	160	171	-9	943	-955	14	37	14	4	144	-143
-14	380	-354	-10	46	81	-11	291	-278	-1	90	108	5	266	258
-15	39	-21	-11	403	-426	-13	775	833	-2	520	-540	6	270	-238
-16	289	278	-12	164	157	-15	40	-40	-3	327	-324	7	552	-550

PAGE 28 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 17,	K= 4		5	298	273	4	109	-139	H= 18,	K= 3		H= 18,	K= 5	
8	109	118	6	249	209	5	220	207	0	141	111	0	109	-162
9	40	-30	7	414	-380	6	203	200	1	126	-77	1	418	381
10	147	131	8	249	-247	7	122	96	2	199	-271	2	374	-365
11	512	462	9	94	-51	8	83	111	3	181	218	3	41	-59
12	232	-175	-1	72	64	9	127	-114	4	127	115	4	230	175
-1	75	-78	-2	351	350	10	46	-60	5	24	-45	5	153	-118
-2	75	-76	-3	300	294	11	144	140	6	242	236	6	200	207
-3	594	591	-4	311	-261	12	38	-28	7	121	56	7	91	83
-4	257	224	-5	258	-265	-1	118	105	8	94	-88	8	269	-257
-5	312	-302	-6	209	-228	-2	159	-191	9	94	81	-1	176	-161
-6	138	-169	-7	114	-151	-3	464	444	10	86	-70	-2	465	456
-7	334	-306	-8	397	394	-4	243	-243	11	37	-64	-3	311	-274
-8	356	-339	-9	302	291	-5	343	-348	-1	382	-363	-4	24	27
-9	232	206	-10	44	51	-6	144	-143	-2	137	169	-5	407	356
-10	257	288	-11	37	-37	-7	167	-239	-3	246	260	-6	439	-419
-11	300	320				-8	224	203	-4	264	-274	-7	47	48
-12	273	296	H= 17,	K= 7		-9	416	434	-5	249	252	-8	100	95
-13	362	-395	0	101	56	-10	117	-109	-6	138	110	-9	397	-422
-14	336	-350	1	165	-205	-11	110	-70	-7	234	-259	-10	204	208
-15	84	-59	2	127	-172	-12	33	-49	-8	279	250	-11	136	111
			3	421	-351	-13	252	-238	-9	144	-93			
H= 17,	K= 5		4	38	28	-14	254	283	-10	420	-377	H= 18,	K= 6	
0	436	-390	5	338	288	-15	342	338	-11	178	195	0	335	305
1	133	-166	6	140	103				-12	72	-62	1	312	-296
2	184	-161	-1	441	420	H= 18,	K= 2		-13	21	19	2	64	-61
3	60	44	-2	157	147	0	272	-281	-14	330	361	3	181	158
4	563	515	-3	93	75	1	150	132				4	210	-180
5	72	65	-4	143	-166	2	133	136	H= 18,	K= 4		5	298	252
6	41	-70	-5	398	-396	3	83	-78	0	147	-114	6	123	133
7	33	-47	-6	139	-133	4	122	-120	1	396	-383	-1	91	-45
8	490	-460	-7	234	247	5	118	79	2	194	228	-2	160	-175
9	113	-75	-8	192	203	6	98*	-5	3	116	-97	-3	420	425
10	474	431				7	161	133	4	108	89	-4	220	-240
11	70	87	H= 18,	K= 0		8	41	37	5	435	399	-5	155	-143
-1	89	-46	0	167	-156	9	99	-125	6	88	-69	-6	293	315
-2	441	438	2	366	-407	10	83	95	7	122	-105	-7	204	-224
-3	212	201	4	43	62	11	59*	17	8	134	119	-8	37	-29
-4	371	376	6	354	356	12	109*	-21	9	127	-115	-9	216	193
-5	250	-246	8	157	-154	-1	115	102	10	80*	-15			
-6	625	-659	10	33*	3	-2	42	40	-1	280	284	H= 18,	K= 7	
-7	153	-124	12	132	148	-3	331	-344	-2	342	-319	0	157	-109
-8	145	-152	-2	478	484	-4	173	202	-3	217	239	1	99	76
-9	138	167	-4	142	-122	-5	61	-36	-4	575	564	-1	329	294
-10	542	561	-6	484	-483	-6	232	-245	-5	320	-304	-2	37	20
-11	40	53	-8	138	92	-7	253	274	-6	116	131	-3	323	-343
-12	147	-205	-10	404	399	-8	255	217	-7	72	-56	-4	188	211
-13	48	-73	-12	414	-397	-9	104	-119	-8	562	-585			
			-14	87	108	-10	163	147	-9	91	97	H= 19,	K= 0	
H= 17,	K= 6					-11	307	-334	-10	146	-153	1	462	-443
0	150	118	H= 18,	K= 1		-12	132	-146	-11	220	-232	3	307	-238
1	413	-364	0	287	306	-13	377	371	-12	375	397	5	489	497
2	432	-396	1	326	-345	-14	38	-24	-13	67	55	7	41	86
3	245	224	2	127	-120	-15	123	151				9	548	-554
4	145	116	3	219	-227							11	63	82

PAGE 29 CADMIUM DINITRATO TRIS(2:6-DIMETHYL GAMMA-PYRONE)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC
H= 19, K= 0			-9	91	-101	2	443	444	H= 20, K= 2			-4	167	-178
-1	721	709	-10	682	-738	3	31	98	0	52*	8			
-3	255	-217	-11	253	-291	4	76	57	1	211	-213			
-5	682	-709	-12	405	440	5	48	-50	2	159	153			
-7	633	633	-13	60	70	-1	39	34	3	131	-157			
-9	471	471				-2	298	-320	4	140	-126			
-11	577	-593	H= 19, K= 3			-3	130	96	5	108	52			
-13	124	-176	0	100	-103	-4	307	322	6	79*	-7			
			1	661	631	-5	84	-92	7	89	67			
H= 19, K= 1			2	224	221	-6	159	118	-1	202	203			
0	210	249	3	497	-547	-7	38	-55	-2	207	-192			
1	123	169	4	145	-182	-8	357	-411	-3	185	183			
2	826	-803	5	299	-357				-4	187	175			
3	418	-415	6	130	-126	H= 19, K= 6			-5	220	-194			
4	198	237	7	650	650	0	230	-281	-6	204	179			
5	21	-24	8	246	264	1	37	46	-7	40	-21			
6	528	565	9	37	-52	-1	257	-304	-8	159	-204			
7	311	326	-1	91	146	-2	160	161	-9	175	234			
8	282	-329	-2	217	-199	-3	118	60	-10	48	-42			
9	87	-87	-3	695	-654	-4	204	250						
10	291	-291	-4	326	343				H= 20, K= 3					
-1	129	124	-5	305	295	H= 20, K= 0			0	216	-233			
-2	346	374	-6	98	155	0	246	-252	1	185	166			
-3	564	-563	-7	322	370	2	41	-43	2	39	-43			
-4	501	-456	-8	434	-481	4	70*	3	3	121	-131			
-5	413	439	-9	471	-514	6	39	16	4	138	134			
-6	252	235	-10	71	-95	8	179	180	5	44	-100			
-7	423	429	-11	110	-72	-2	41	28	6	107	61			
-8	459	429	-12	312	349	-4	311	336	-1	40	-23			
-9	370	-369				-6	108	-101	-2	176	168			
-10	220	-227	H= 19, K= 4			-8	145	-124	-3	248	-263			
-11	143	-173	0	265	280	-10	255	220	-4	97	71			
-12	488	-482	1	124	145				-5	140	140			
-13	295	327	2	127	130	H= 20, K= 1			-6	212	-228			
			3	424	411	0	34	43	-7	38	30			
H= 19, K= 2			4	224	-214	1	41	31	-8	82	77			
0	643	683	5	352	-389	2	131	-166	-9	118	-171			
1	43	-69	6	31	-28	3	41	37						
2	24	-32	7	165	-142	4	124	-108	H= 20, K= 4					
3	202	-223	8	160	137	5	137	-157	0	138	113			
4	504	-530	-1	549	-583	6	81	32	1	22	-43			
5	246	262	-2	205	-206	7	143	158	2	90	-43			
6	266	238	-3	99	-97	8	21	11	3	78	71			
7	120	100	-4	301	-270	-1	234	-265	4	222	-208			
8	440	423	-5	501	547	-2	198	225	-1	207	-189			
9	189	-193	-6	365	408	-3	182	207	-2	155	-163			
10	503	-536	-7	97	-153	-4	168	-167	-3	199	219			
-1	284	274	-8	135	-98	-5	220	250	-4	131	-87			
-2	539	-581	-9	450	-487	-6	94	-124	-5	93	-59			
-3	133	-135	-10	166	-172	-7	144	-124	-6	57	59			
-4	445	-497	-11	416	456	-8	301	287	-7	209	-231			
-5	124	64				-9	32	-23						
-6	826	889	H= 19, K= 5			-10	38	-29	H= 21, K= 3					
-7	146	184	0	292	-306	-11	37	52	-2	129	-168			
-8	34	37	1	32	-41				-3	60	-60			

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CHAPTER 4.

PICRIC ACID - NAPHTHALENE 1:1 COMPLEX

Introduction

The compound was prepared by Dr J V Westwood (Department of Chemistry, City of London Polytechnic) by crystallisation from an alcoholic solution of equimolecular proportions of naphthalene and picric acid. This was one of a series of molecular complexes which could be used for separation of homologues of an isomorphous series, because of their clathrate-type structures.

Some of the structures of the molecular complexes which have already been published are as follows:

1. Equimolar Aromatic Hydrocarbon: 2,4,6-Trinitrobenzene Molecular Compounds.

Crystal data are reported (1) for the 1:1 molecular compounds with naphthalene, anthracene, phenanthrene, acenaphthene, fluoranthene, pyrene or triphenylene as donor and 1,3,5-trinitrobenzene, picric acid, picryl chloride or picryl bromide as acceptor (33 different crystals have been studied). In three out of seven pairs, the trinitrobenzene and picric acid molecular compounds of a particular hydrocarbon are isomorphous, and in four out of seven pairs the picryl chloride and picryl bromide molecular compounds are isomorphous.

a) The Structure of fluoranthene: picryl bromide, polymorph I (monoclinic; $a = 7.664(8)$, $b = 8.035(2)$, $c = 31.631(8)$Å, $\beta = 91.8(1)^{\circ}$, space group $P2_1/c$, $Z = 4$) has been solved by Patterson and Fourier methods and refined by block-diagonal least squares calculations to a final R of 0.062. The structure consists of quasi-hexagonally close-packed mixed stacks of alternating donor and acceptor molecules. The stack axes are parallel to $[\bar{1}00]$. The interplanar spacing is 3.49Å which suggests a rather weak charge-transfer interaction.

b) The Crystal Structure of Anthracene: Picric Acid (Ref 2)

The structure of the molecular complex anthracene; picric acid (monoclinic; a = 7.180, b = 12.901, c = 19.205Å, β = 90.52° space group $P2_1/c$, Z = 4) has been solved by direct methods, and refined to R = 5.0% with the intensities of 2092 non-zero reflections measured on a diffractometer using graphite-monochromatized Mo Kα . The components are arranged alternately in quasi-hexagonally close-packed mixed stacks, extending in the [100] direction. The picric acid molecule has an intra-molecular hydrogen bond between the phenol OH group and an adjacent nitro group. The individual six-membered rings of the anthracene molecule are planar but the molecule as a whole deviates significantly from planarity and is also almost but not quite centrosymmetric. The bond lengths and angles conform to mm symmetry.

2. Experimental measurements of Picric Acid-Naphthalene

(i) Preparation of Crystals. The compound was dissolved in absolute alcohol and warmed at 60°C to make a saturated solution. The solution was then filtered and transferred into a crystallising dish. The temperature gradient was maintained in such a way that yellow crystals of a suitable size for diffractometry were formed.

(ii) Selection of Crystal. The crystals were carefully examined under the optical microscope. Under cross polars, it was made sure that the selected crystal was free from twinning. One extinction direction was found parallel to the needle axis.

3. Space-Group and Unit Cell Dimensions

The chosen crystal was mounted on a glass-fibre parallel to the direction of the needle axis. First the crystal was aligned by optical methods but the final adjustment was made using double oscillations. The oscillation photographs showed mirror symmetry perpendicular to the axis which was 6.871Å. A full rotation photograph was taken along that axis. The crystal was then transferred to a Stoe Weissenberg camera, and zero and first layer photographs were taken. Similarly the crystal was mounted along the other two axes and rotation and zero layer Weissenberg photographs were taken.

The space group was determined from the following systematic absences

Reflection	Condition limiting possible reflections
hkℓ	No absences
hoℓ	h = 2n
oko	k = 2n

so the space group was $P2_1/a$.

The cell parameters were measured as accurately as possible from the rotation photographs, along the three chosen crystallographic axes. Later on final corrections were made on the Stadi-2 system diffractometer before making intensity measurements.

The final values were

$a = 16.248\text{Å}$, $b = 6.871\text{Å}$, $c = 14.306\text{Å}$

$\beta = 96.62^{\circ}$.

4. Measurement of density

The density of the crystal was measured by the method of flotation. The density of the liquid (Na I solution in water) was adjusted in such a way that the crystal neither sank nor floated in the liquid. Then the density of the liquid was measured on a Westphal balance and this value was taken to be the same as the density of the crystal.

The measured density of the crystal was 1.470 gm/cc. It was very close to the calculated density (1.492 gm/cc).

5. Intensity Measurement

Intensities were measured using the 2-circle-Stoe-Automated-Xray-Weissenberg-Diffractometer-STADI-2. This diffractometer is based on the Weissenberg equi-inclination geometry. It works on the same principle as Weissenberg equi-inclination camera.

A high stabilized X-ray generator and a counting chain with scaler, timer, discriminator, ratemeter, high and low voltage power supplies and scintillation counter, belong to the complete system. It is advisable to use a strip chart recorder system for inspection of reflection profiles versus background to help to detect faulty results. In this system Mo-radiation and a graphite mono-chromator are used.

The instrument is mounted on a stable aluminium casting base. The diffractometer height can be adjusted to align with the X-ray beam by four adjustable legs.

The two circles of the diffractometer are driven by two motors. The ω-system is connected to the rotating shaft with goniometer head and crystal. The 2θ-system is connected with the rotating arm for the scintillation counter.

The telescope is mounted on a rack and pinion support which was mounted on the frame. This can be rotated about a vertical axis in order to change the equi-inclination angle μ . The automatic operations of the diffractometer require a very precise setting of the inclination angle μ .

The two motors can be disconnected from their gears. In this state the circles can be quickly rotated by hand, which is convenient specially for the ω-system for adjusting purposes.

It was found necessary to position the ω-circle to an angular accuracy of 0.01^{o} , at the peak intensity of a particular reflection. This can be achieved in the following way. The maximum peak intensity was found by moving the circle slowly through the reflection in the manual mode of operation.

In that position the allan screw on drum of the ω-circle is loosened and the drum is placed to the required position to an accuracy of 0.01^{o} . The scale of the 2θ-circle cannot be disconnected. This scale should read 0.00^{o} for the position of the maximum intensity of the primary beam, otherwise necessary corrections should be made, using same method as described above for the ω-circle.

6. Adjustment of the crystal on the diffractometer

Prior to intensity measurements on the STADI-2 system the space-group of the crystal was determined from Weissenberg and precession photographs. The photographs also helped to observe the quality of the crystal from the shape of the reflections. If the quality of the crystal is poor or if the exposure time is too long, another crystal should be used. It is worth spending time to have a perfect crystal for intensity measurement.

The accuracy of the lattice parameters which were obtained by the photographic method was of order of 0.5^{o} . So it was therefore necessary to improve the accuracy of adjustment on the diffractometer prior to remeasuring the lattice constants. For this purpose the STADI 2 system was used in its manual mode of operation.

For the adjustment of the orientation, the Weissenberg photographs of the zero layer line with hol reflections were used to select one strong reference reflection at low 2θ value. The counter was set to the 2θ - position of the reference reflection and the ω-circle slowly rotated until a strong reflection was found. To ensure that the maximum of the desired reflection had been achieved, the next reflection on the same reciprocal axis was selected on the Weissenberg photograph. For that reflection the 2θ and the ω-values were calculated. The reflection maximum was found at these angular positions, which proved that the orientation of the crystal was correct.

The next step was to improve the crystal setting. This was achieved by studying the reflections from lattice planes (001). The (001) plane was brought into the symmetrical reflecting position between the primary beam and the reflecting beam by setting the μ-angle

with an accuracy of 0.02^{o} on the diffractometer and on the counter.

If the crystal was perfectly adjusted, the reflecting maximum should occur at these positions. If the adjustment was not perfect, considerable fluctuations of intensities would be observed during the rotation of the ω circle. These fluctuations could be reduced very often by slight adjustments of the axes of the goniometer head. If the absorption was large, some fluctuations of intensity would remain during the rotation of the ω circle, but these fluctuations were symmetrical with respect to the azimuthal angle. This was also minimised by readjusting the axes. After the necessary adjustment, the crystal centring was checked using a microscope.

7. Preparation of the input tape

Once the crystal was set, the next step was to prepare the input tape for the intensity data collection.

The following information was required:

a) Lattice constant or reciprocal lattice constant of sufficient accuracy.

b) Equi-inclination angle μ (which remains the same for all reflections on each layer)

c) Wave length of the radiation used.

d) Information about whether the 2θ scan or ω scan is to be used.

e) Specification about the $\Delta\omega$ minimum and maximum limits.

The STOET program was used. The input paper-tape was prepared for each layer at the computer terminal.

The listing of the input tape would be required to measure the intensities. Since the crystal was set at an arbitrary azimuthal position, the actual values would not coincide with those listed on the input tape. To make it co incide one particular (known reflection) was selected and the counter was positioned to its 2θ value. When the reflection maximum was found, the ω-scale and the drum were placed to the ω-position according to the input list. Before starting the automatic measurements, the angular positions of few other reflections were also checked.

8. Structure Determination

The STOWK program was used to convert the intensity measurements to structure amplitudes (F's). About 1200 structure amplitudes (F) were produced, and then both the NRC and Multan direct-methods programs were used but the structure could not be solved. The reason was not known.

Later a new set of intensity data was collected using the photographic method. A new crystal was used on this occasion. The structure was solved using trial-and-error methods as the direct methods had failed. A Patterson map indicated layer structure with b/4 separation.

From considerations of the $P2_1/a$ space group symmetry, it was evident that each layer of the unit cell at $y = \frac{1}{8}$ (and also at $y = \frac{3}{8}$, $\frac{5}{8}$ and $\frac{7}{8}$) would contain one picric acid and one naphthalene molecule. Paper models of these two molecules were made and used for fitting in an ac projection of the unit cell, and structure factors were calculated using a special computer program for many combinations of orientation and translation of the two molecules.

Two points were taken into consideration:

1) There must be about 3.5Å separation between the atoms of the two molecules;

2) There was a possibility of hydrogen bonding between the OH groups of centrosymmetrically-related picric acid molecules (this was later found to be true).

Repetitive structure factor calculations were then carried out noticing particularly the requirement for high F values for planes 002, 202, $40\bar{1}$ and $20\bar{3}$, until there was some semblance of overall agreement between F_c and F_o. Commencing coordinates for three-dimensional refinement were then fixed with $y = 0.125$ and the best x and z from the trial-and-error. Several cycles of structure factors and least-squares were carried out using initially B_{iso} temperature parameters and $\sqrt{w} = 1/F_o$. Hydrogen atoms were also included at calculated positions with $B_{iso} = 8.0 Å^2$ but not refined. The lowest R factor was 23% .

The difference Fourier map showed alternative sites for the O atom of the picric acid -OH . Site occupation refinement was introduced into the least squares calculations with the O atom in each of the alternative positions which then converged to 0.80

for O(1) position and 0.20 for O(8). These values were used in further refinement until the final shifts are less than 0.30σ . The final R factor was 0.066 and R_w (weighted) = 0.136 . The maximum electron density in the final difference Fourier was $\pm 0.2eA^{-3}$. The atomic scattering factors were taken from International Tables for X-ray Crystallography, Vol III, pp 202-207.

9. Discussion

The final atomic co-ordinates and equivalent isotropic temperature parameters are listed in Table 1. Bond lengths and inter bond angles are given in Table 2. The arrangements of the molecules in unit cell together with the atomic numbering and hydrogen bonds are shown in Figure 1.

Molecules of both picric acid and naphthalene lie approximately parallel to (010) at y = $\frac{1}{8}$, $\frac{3}{8}$, $\frac{5}{8}$ and $\frac{7}{8}$. The overlap diagram is shown (Figure 2)as a normal projection along b. C(14) of the naphthalene ring lies almost exactly above the mid-point of the picric acid ring. There are hydrogen bond links between picric acid molecules which are related by centre of symmetry either through O(1) - O(1') 2.844 Å across (0,0,0) with 80% site occupation, or through O(8) - O(8") 3.082 Å across (0,0,$\frac{1}{2}$) with 20% site occupation. These alternative sites can be occupied with little change to the co-ordinates of the other picric acid atoms.

The H-bonds through OH - HO leave a spare proton from half the picric acid molecules and this appears to be transferred to either

O(4) or O(6) which enables H-bonding to take place between O(4) ..... H ..... O(6) 2.901 Å .

The hydrogen bonding between the nitro groups were confirmed by the bond lengths N(2) - O(4) 1.291 and N(3) - O(6) 1.304 Å which were longer than the four other N-O bonds (Mean 1.194 Å). These hydrogen bonds have a stabilising influence which is reflected by the Biso factor ≈7.5Å^2 for O(4) and O(5) and ≈8.5 for O(6) and O(7). For O(2) and O(3) which were not H-bonded the value of Biso was ≈12.2Å . The dihedral angles between the planes of the nitrogroups and the phenyl rings were found to be noticeably different. N(1) O(2) O(3) makes an angle of 13.61° with the C(1) - (6) ring whereas N(2) O(4) O(5) makes 7.56° and N(3) O(6) O(7) makes 5.67° with the phenyl ring (C(1) - (6)).

The bond lengths (Table 2) result from the superposition of the two disordered picric acid molecules as a result of which precise positioning of the naphthalene molecules is affected. Attempts were made to separate the two arrangements, but no progress was made along these lines. The results presented are the best interpretation which can be made at present.

To improve the resolution of the atomic parameters, two additional complete sets of intensity data were collected on the diffractometer using Mo Kα radiation. At the same time a considerable amount of photographic data on the Weissenberg camera was collected.

A large variation in values of F was noticed which led to a conclusion that no two crystals used had the same proportion of

disorder. The results presented here are for one particular crystal under one particular set of experimental condition and these are not necessarily reproducible.

disorder. The results presented here are for one particular crystal under one particular set of experimental condition and these are not necessarily reproducible.

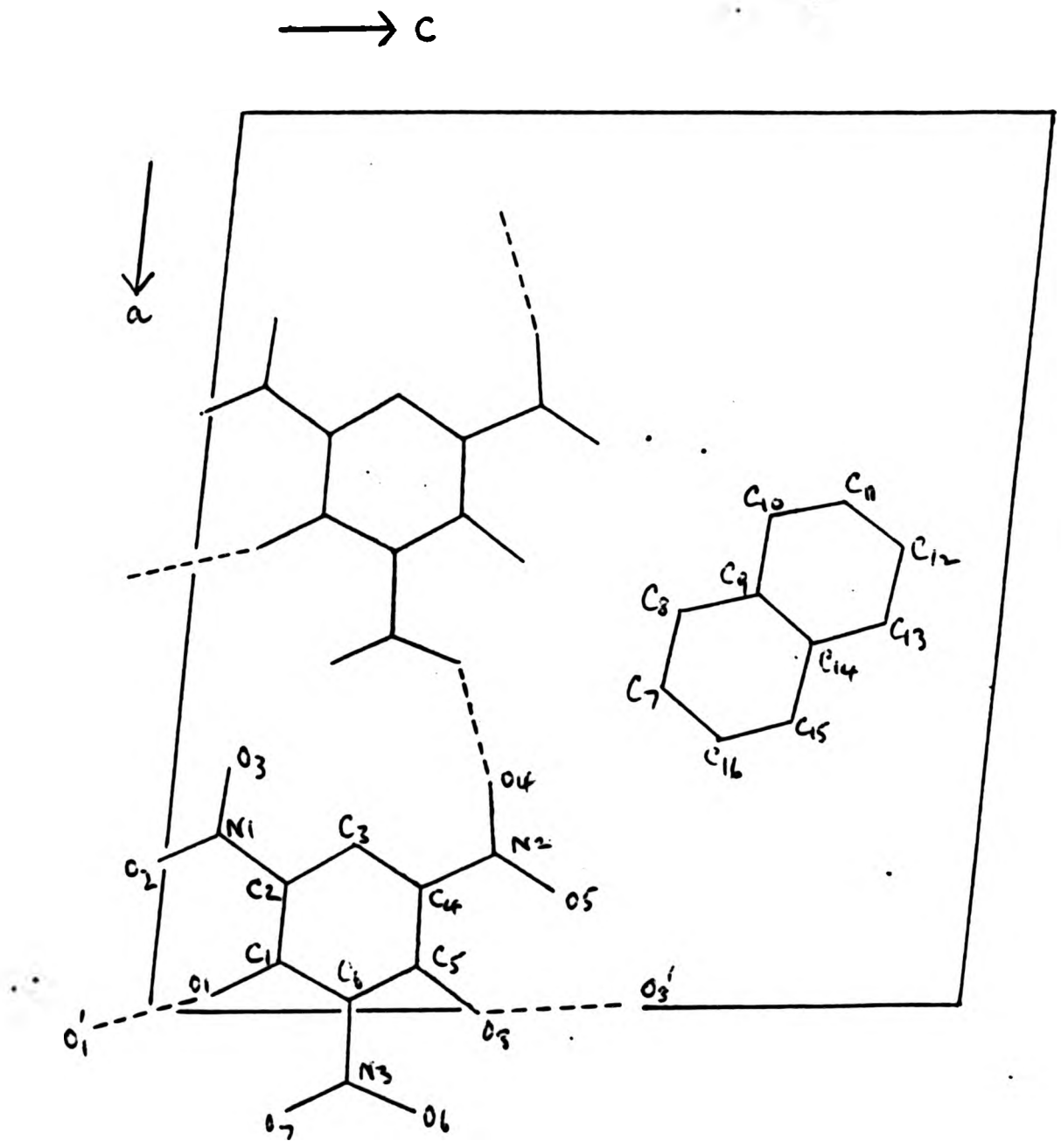


Figure 1. Molecules in Unit Cell showing numbering of atoms and potential hydrogen bonds.

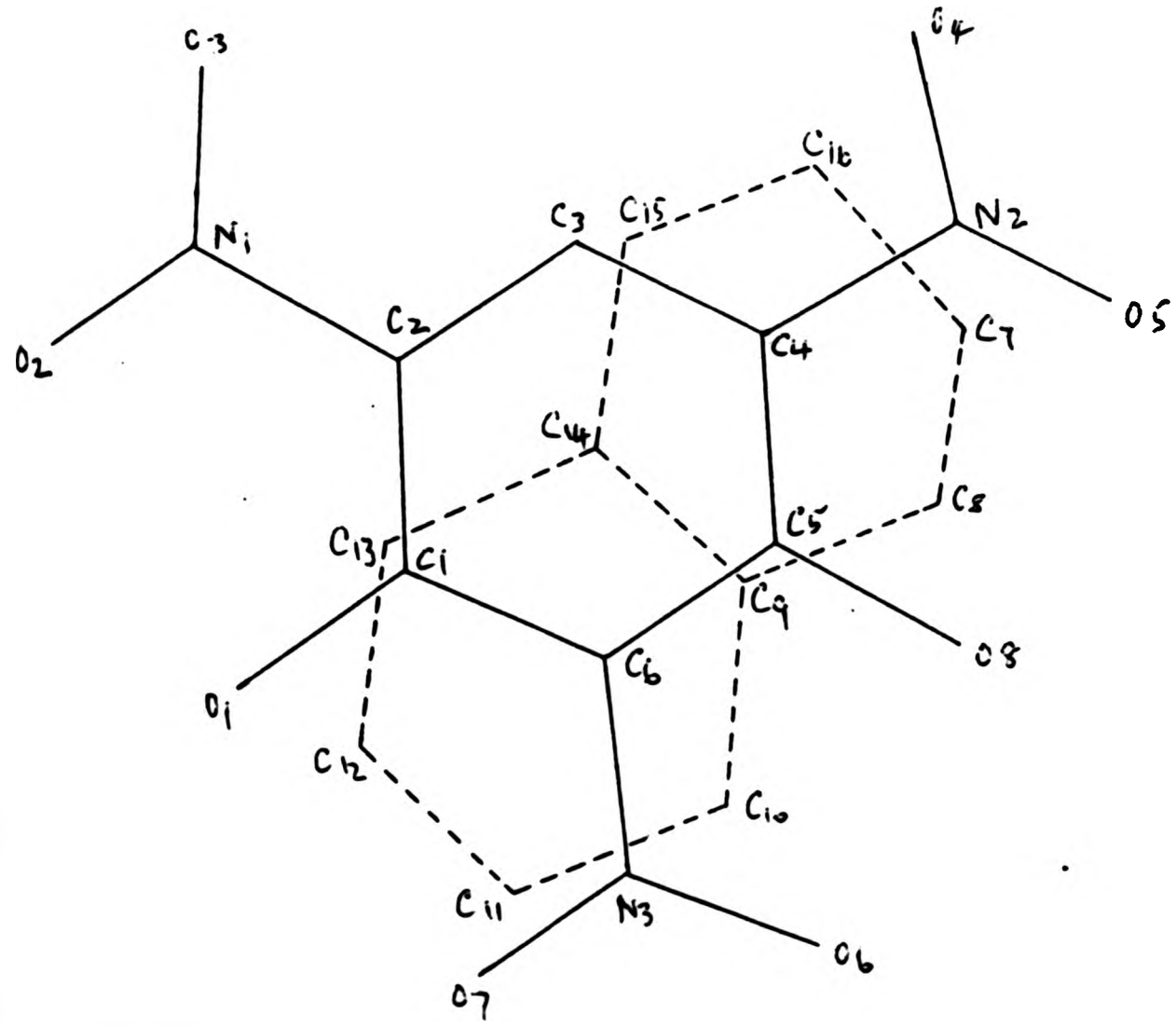


Figure 2. Overlap of molecules in projection along b, separated by b/2.

TABLE 1. ATOMIC PARAMETERS

PICRIC ACID - NAPHTHALENE COMPLEX
ORIGINAL COORDINATES

ATOM	NO.	X/A	Y/B	Z/C	E.S.D.S SYG X/A	SYG Y/B	SYG Z/C
C	1	0.94127	0.11824	0.15417	0.00045	0.00080	0.00045
C	2	0.85414	0.10274	0.15260	0.00047	0.00099	0.00044
C	3	0.81450	0.11048	0.23466	0.00033	0.00073	0.00031
C	4	0.86231	0.11887	0.31902	0.00041	0.00089	0.00044
C	5	0.95034	0.10963	0.32720	0.00044	0.00107	0.00053
C	6	0.98724	0.11234	0.24556	0.00033	0.00092	0.00051
C	7	0.63952	0.12107	0.59267	0.00057	0.00089	0.00055
C	8	0.55522	0.13252	0.60611	0.00054	0.00139	0.00047
C	9	0.53466	0.12307	0.70024	0.00035	0.00084	0.00039
C	10	0.45083	0.14129	0.70926	0.00046	0.00108	0.00050
C	11	0.43283	0.11524	0.79575	0.00047	0.00099	0.00056
C	12	0.48754	0.12293	0.87427	0.00069	0.00110	0.00057
C	13	0.56712	0.12941	0.86473	0.00059	0.00115	0.00055
C	14	0.59474	0.12591	0.77418	0.00042	0.00091	0.00045
C	15	0.67953	0.11159	0.75925	0.00046	0.00097	0.00063
C	16	0.69984	0.11805	0.66720	0.00047	0.00106	0.00051
N	1	0.79906	0.09962	0.06192	0.00049	0.00104	0.00053
H	2	0.82638	0.10934	0.41110	0.00039	0.00076	0.00042
H	3	1.07720	0.12588	0.25459	0.00043	0.00092	0.00061
O	1	0.97993	0.11307	0.07749	0.00053	0.00102	0.00052
O	2	0.93007	0.14717	-0.00632	0.00056	0.00135	0.00054
O	3	0.72620	0.07445	0.06780	0.00052	0.00127	0.00051
O	4	0.74730	0.08545	0.39350	0.00041	0.00085	0.00046
O	5	0.86846	0.12928	0.48389	0.00047	0.00087	0.00039
O	6	1.11498	0.13894	0.33984	0.00040	0.00098	0.00055
O	7	1.13980	0.14569	0.18509	0.00043	0.00090	0.00060
O	8	1.08126	0.13021	0.41248	0.00158	0.00413	0.00169
H	3	0.74773	0.10979	0.23086	0.00000	0.00000	0.00000
H	7	0.65672	0.11456	0.52203	0.00000	0.00000	0.00000
H	8	0.50776	0.14793	0.54720	0.00000	0.00000	0.00000
H	10	0.42472	0.17379	0.65078	0.00000	0.00000	0.00000
H	11	0.36896	0.08520	0.80470	0.00000	0.00000	0.00000
H	12	0.46652	0.12374	0.94319	0.00000	0.00000	0.00000
H	13	0.61191	0.13748	0.92654	0.00000	0.00000	0.00000
H	15	0.72720	0.09604	0.81794	0.00000	0.00000	0.00000
H	16	0.76416	0.12069	0.65484	0.00000	0.00000	0.00000
H	1	0.99517	0.02719	0.01864	0.00000	0.00000	0.00000
H	20	1.03038	0.03895	0.47382	0.00000	0.00000	0.00000

TABLE 1 (contd.)

Anisotropic Temperature Factors and their e.s.d's

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
C(1)	0.00567 37	0.01715 171	0.00538 39	0.00075 138	0.00320 59	0.00185 131
C(2)	0.00542 37	0.03111 221	0.00432 36	0.00473 147	-0.00019 57	-0.00602 146
C(3)	0.00349 27	0.01557 151	0.00095 22	0.00321 109	-0.00029 40	0.00479 105
C(4)	0.00420 33	0.02329 198	0.00546 39	-0.00432 144	0.00314 57	0.00132 126
C(5)	0.00395 33	0.03723 255	0.00729 46	-0.00068 169	0.00033 62	-0.00241 145
C(6)	0.00095 23	0.02562 198	0.00922 44	0.00232 156	-0.00103 51	-0.00148 116
C(7)	0.00962 56	0.01790 196	0.00796 52	-0.00597 159	0.00711 89	-0.00749 156
C(8)	0.00752 47	0.04200 246	0.00344 35	0.00977 166	0.00048 62	0.00572 175
C(9)	0.00267 27	0.02467 187	0.00391 32	-0.00129 138	0.00050 45	-0.00129 117
C(10)	0.00517 41	0.03562 245	0.00702 49	0.00133 176	0.00076 69	0.01152 153
C(11)	0.00478 37	0.03373 269	0.00901 55	-0.01136 191	0.00444 72	-0.01279 161
C(12)	0.01225 73	0.05203 243	0.00612 47	-0.01040 196	0.00539 91	-0.00262 197
C(13)	0.00761 50	0.05058 303	0.00659 49	0.00976 210	0.00476 80	0.00662 205
C(14)	0.00435 34	0.02590 201	0.00596 42	-0.00225 154	-0.00075 60	0.00296 133
C(15)	0.00413 39	0.02384 209	0.01194 64	0.00390 190	-0.00190 78	0.00146 138
C(16)	0.00464 39	0.04106 257	0.00642 45	-0.00449 175	-0.00221 65	-0.00165 163
N(1)	0.00793 46	0.04819 256	0.00934 49	0.00719 182	-0.00033 75	0.00252 169
N(2)	0.00563 33	0.02791 170	0.00730 39	0.00456 132	0.00455 58	0.00132 116
N(3)	0.00444 35	0.03926 233	0.01506 67	-0.00105 186	0.00559 76	-0.00099 138

TABLE 1 (Contd.)

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
O(1) *	0.00965 50	0.04483 247	0.00931 50	-0.00755 169	0.01036 84	0.00879 164
O(2)	0.01042 53	0.10121 392	0.01114 51	0.01520 231	-0.00159 84	-0.00459 200
O(3)	0.01070 53	0.09351 363	0.01020 46	0.00766 240	-0.00301 79	-0.01455 231
O(4)	0.00454 38	0.04140 144	0.01231 49	-0.00376 151	0.00903 73	0.00121 129
O(5)	0.01037 45	0.06390 250	0.00618 33	-0.01019 154	-0.00079 57	-0.00052 170
O(6)	0.00594 36	0.05960 255	0.01040 66	-0.00464 183	-0.00024 77	-0.00198 137
O(7)	0.00722 39	0.05077 233	0.01914 75	0.01349 190	0.01108 92	0.00326 143
O(8) **	0.00334 115	0.06123 1032	0.00591 139	0.00384 635	-0.01143 232	-0.00094 544

* 80% site occupation ** 20% site occupation

$$T = \exp\left[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{23}hk + 2\beta_{31}lh + 2\beta_{12}hk)\right].$$

TABLE 2. BOND LENGTHS AND ANGLES

PICRIC ACID - NAPHTHALENE COMPLEX

INTRAMOLECULAR DISTANCES

ATOM	N1	ATOM	N2	DISTANCE	E.S.D.
C	1	C	2	1.417	0.0106
C	1	C	6	1.430	0.0096
C	1	O	1	1.326	0.0103
C	2	C	3	1.404	0.0082
C	2	N	1	1.489	0.0101
C	3	C	4	1.359	0.0079
C	3	H	3	1.000	0.0054
C	4	C	5	1.423	0.0098
C	4	N	2	1.503	0.0088
C	5	C	6	1.373	0.0101
C	5	O	8	1.400	0.0256
C	6	H	3	1.455	0.0089
C	7	C	8	1.407	0.0128
C	7	C	16	1.363	0.0112
C	7	H	7	1.000	0.0000
C	8	C	9	1.426	0.0089
C	8	H	8	1.000	0.0076
C	9	C	10	1.389	0.0094
C	9	C	14	1.354	0.0087
C	10	C	11	1.316	0.0107
C	10	H	10	1.000	0.0073
C	11	C	12	1.351	0.0121
C	11	H	11	1.000	0.0077
C	12	C	13	1.317	0.0148
C	12	H	12	1.000	0.0087
C	13	C	14	1.419	0.0124
C	13	H	13	1.000	0.0085
C	14	C	15	1.422	0.0102
C	15	C	16	1.395	0.0114
C	15	H	15	1.000	0.0084
C	16	H	16	1.000	0.0077
H	1	O	2	1.195	0.0112
H	1	O	3	1.209	0.0117
H	2	O	4	1.291	0.0092
H	2	O	5	1.186	0.0086
H	3	O	6	1.304	0.0114
H	3	O	7	1.187	0.0115
O	1	H	1	1.000	0.0074
O	8	H	28	1.000	0.0256

DIRECTION COSINES

L	M	N
0.99705	0.07514	0.01574
-0.41704	0.02836	-0.90845
-0.56912	0.02679	0.82182
0.55528	-0.03789	-0.83080
0.50065	0.01440	0.86553
-0.46920	-0.04242	-0.88207
0.99874	0.00439	0.05000
-0.99566	0.04462	-0.08169
0.48960	0.04358	-0.87086
-0.53471	-0.01356	0.84493
-0.49047	-0.13100	-0.86558
-0.99405	-0.06392	-0.08817
0.98917	-0.05591	-0.13573
-0.62903	0.01523	-0.77723
-0.36662	0.04142	0.92945
0.34318	0.04554	-0.93817
0.62408	-0.09004	0.77518
0.99164	-0.09016	-0.09231
-0.63076	-0.01441	-0.77585
0.33062	0.13601	-0.93391
0.60435	-0.20675	0.76942
-0.56222	-0.03912	-0.82606
0.97448	0.19110	-0.11775
-0.99411	-0.03382	0.10298
0.42147	-0.00515	-0.90683
-0.42144	0.01695	0.90670
-0.57950	-0.05135	-0.81336
-0.98638	0.06921	0.14924
-0.34546	-0.03103	0.93789
-0.62756	0.09893	-0.77226
-0.98654	-0.01680	0.16263
-0.51598	-0.27349	0.81176
0.98730	0.14306	-0.06912
0.97278	0.12715	0.19373
-0.47529	-0.11553	-0.87221
-0.36301	-0.06883	-0.92924
-0.54278	-0.11467	0.83201
-0.31915	0.54637	0.77435
0.10691	0.58061	-0.80713

TABLE 2 (contd.)

PICRIC ACID - NAPHTHALENE COMPLEX

	#1		#2		#3		ANGLE	E.S.D.	#1		#3		DISTANCE
ANGLES INVOLVING ATOM	C	1											
	C	2	C	1	C	6	115.34	0.500	C	2	C	6	2.496
	C	2	C	1	O	1	123.54	0.645	C	2	O	1	2.417
	C	6	C	1	O	1	120.56	0.620	C	6	O	1	2.394
ANGLES INVOLVING ATOM	C	2											
	C	1	C	2	C	3	122.53	0.594	C	1	C	3	2.474
	C	1	C	2	N	1	120.92	0.616	C	1	N	1	2.529
	C	3	C	2	N	1	116.21	0.586	C	3	N	1	2.456
ANGLES INVOLVING ATOM	C	3											
	C	2	C	3	C	4	118.39	0.526	C	2	C	4	2.372
	C	2	C	3	N	3	120.86	0.494	C	2	N	3	2.166
	C	4	C	3	N	3	120.86	0.491	C	4	N	3	2.126
ANGLES INVOLVING ATOM	C	4											
	C	3	C	4	C	5	122.50	0.586	C	3	C	5	2.439
	C	3	C	4	N	3	122.45	0.543	C	3	N	3	2.389
	C	5	C	4	N	3	114.40	0.568	C	5	N	3	2.461
ANGLES INVOLVING ATOM	C	5											
	C	4	C	5	C	6	117.57	0.625	C	4	C	6	2.391
	C	4	C	5	O	6	123.66	1.319	C	4	O	6	2.499
	C	6	C	5	O	6	117.89	1.319	C	6	O	6	2.376
ANGLES INVOLVING ATOM	C	6											
	C	1	C	6	N	5	123.02	0.610	C	1	N	5	2.463
	C	1	C	6	N	5	119.83	0.586	C	1	N	5	2.493
	C	5	C	6	N	5	117.29	0.612	C	5	N	5	2.415
ANGLES INVOLVING ATOM	C	7											
	C	8	C	7	C	10	121.17	0.710	C	8	C	10	2.413
	C	8	C	7	N	7	119.41	0.711	C	8	N	7	2.154
	C	10	C	7	N	7	119.42	0.710	C	10	N	7	2.114
ANGLES INVOLVING ATOM	C	8											
	C	7	C	8	O	8	117.66	0.683	C	7	O	8	2.424
	C	9	C	8	O	8	121.17	0.700	C	9	O	8	2.173
	C	9	C	8	N	8	121.17	0.660	C	9	N	8	2.109
ANGLES INVOLVING ATOM	C	9											
	C	8	C	9	C	10	115.01	0.583	C	8	C	10	2.374
	C	8	C	9	C	14	120.71	0.581	C	8	C	14	2.417
	C	10	C	9	C	14	123.54	0.595	C	10	C	14	2.417
ANGLES INVOLVING ATOM	C	10											
	C	9	C	10	N	11	113.69	0.659	C	9	N	11	2.265
	C	9	C	10	N	10	123.16	0.654	C	9	N	10	2.176
	C	11	C	10	N	10	123.15	0.705	C	11	N	10	2.110
ANGLES INVOLVING ATOM	C	11											
	C	10	C	11	N	12	125.47	0.762	C	10	N	12	2.371
	C	10	C	11	N	11	117.27	0.697	C	10	N	11	2.050
	C	12	C	11	N	11	117.26	0.725	C	12	N	11	2.090
ANGLES INVOLVING ATOM	C	12											
	C	11	C	12	C	13	119.37	0.631	C	11	N	12	2.291
	C	11	C	12	N	12	120.02	0.622	C	11	N	12	2.110
	C	13	C	12	N	12	120.61	0.651	C	13	N	12	2.087
ANGLES INVOLVING ATOM	C	13											
	C	12	C	13	N	14	120.70	0.701	C	12	N	14	2.370
	C	12	C	13	N	13	119.61	0.613	C	12	N	13	2.075
	C	14	C	13	N	13	119.61	0.736	C	14	N	13	2.107
ANGLES INVOLVING ATOM	C	14											
	C	9	C	14	N	13	113.97	0.619	C	9	N	13	2.252
	C	9	C	14	N	15	122.36	0.611	C	9	N	15	2.409
	C	13	C	14	N	15	123.92	0.656	C	13	N	15	2.503
ANGLES INVOLVING ATOM	C	15											
	C	14	C	15	N	16	118.39	0.677	C	14	C	16	2.422
	C	14	C	15	N	15	122.70	0.605	C	14	N	15	2.101
	C	16	C	15	N	15	120.71	0.707	C	16	N	15	2.156
ANGLES INVOLVING ATOM	C	16											
	C	1	C	16	N	15	120.01	0.706	C	1	N	15	2.398
	C	1	C	16	N	16	119.68	0.694	C	1	N	16	2.118
	C	15	C	16	N	16	119.68	0.684	C	15	N	16	2.145
ANGLES INVOLVING ATOM	N	1											
	C	2	N	1	O	1	118.13	0.746	C	2	O	1	2.301
	C	2	N	1	O	1	115.08	0.726	C	2	O	1	2.291
	O	1	N	1	O	1	127.30	0.862	O	1	O	1	2.193
ANGLES INVOLVING ATOM	N	3											
	C	4	N	3	O	4	120.25	0.342	C	4	O	4	2.207
	C	4	N	3	O	5	121.46	0.594	C	4	O	5	2.250
	O	4	N	3	O	5	118.14	0.869	O	4	O	5	2.240

TABLE 2 (contd.)

	#1	#2	#3	ANGLE	E.S.D.	#1	#3	DISTANCE
ANGLES INVOLVING ATOM N 3								
	C 6	N 3	O 6	119.56	0.660	C 6	O 6	2.340
	C 6	N 3	O 7	118.26	0.707	C 6	O 7	2.272
	O 6	N 3	O 7	124.63	0.767	O 6	O 7	2.296
ANGLES INVOLVING ATOM O 1								
	C 1	O 1	N 1	146.37	0.764	C 1	N 1	2.304
ANGLES INVOLVING ATOM O 2	NONE							
ANGLES INVOLVING ATOM O 3	NONE							
ANGLES INVOLVING ATOM O 4	NONE							
ANGLES INVOLVING ATOM O 5	NONE							
ANGLES INVOLVING ATOM O 6	NONE							
ANGLES INVOLVING ATOM O 7	NONE							
ANGLES INVOLVING ATOM O 8								
	C 5	O 8	N 20	129.99	2.213	C 5	N 20	2.214
ANGLES INVOLVING ATOM N 3	NONE							
ANGLES INVOLVING ATOM N 7	NONE							
ANGLES INVOLVING ATOM N 8	NONE							
ANGLES INVOLVING ATOM N 10	NONE							
ANGLES INVOLVING ATOM N 11	NONE							
ANGLES INVOLVING ATOM N 12	NONE							
ANGLES INVOLVING ATOM N 13	NONE							
ANGLES INVOLVING ATOM N 19	NONE							
ANGLES INVOLVING ATOM N 16	NONE							
ANGLES INVOLVING ATOM N 1								
	O 1	N 1	N 1	179.99	0.489	O 1	N 1	1.764
ANGLES INVOLVING ATOM N 20								
	O 8	N 20	N 20	179.99	1.306	O 8	N 20	2.892

TABLE 3. EQUATIONS OF MEAN PLANES

PLANE 1 IS (-0.0230)X + (-0.9996)Y + (-0.0165)Z -(-1.2070) = 0

CHI SQUARED = 444.9778

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	7	9.4151	0.0302	0.4220	0.021	0.0061
	C	8	8.0172	0.9116	0.6139	-0.010	0.0075
	C	9	7.9330	0.0460	9.9507	0.023	0.0050
	C	10	6.1547	0.9607	10.0787	-0.009	0.0074
	C	11	5.7103	0.7942	11.3040	0.005	0.0060
	C	12	6.4011	0.0446	12.4261	0.008	0.0076
	C	13	7.7090	0.0074	12.2078	-0.002	0.0079
	C	14	8.3000	0.0044	11.0030	-0.031	0.0063
	C	15	9.7910	0.7660	10.7009	0.037	0.0067
	C	16	10.2720	0.0070	9.4004	0.007	0.0073

SUM OF P(I) 0.0000 R.M.S. OF P(I) 0.047903

PLANE 2 IS (-0.0069)X + (0.9999)Y + (-0.0112)Z -(0.6345) = 0

CHI SQUARED = 196.9836

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	C	1	15.0410	0.0116	2.1919	0.049	0.0059
	C	2	13.6251	0.7062	2.1656	-0.045	0.0060
	C	3	12.8461	0.7592	3.3340	-0.000	0.0050
	C	4	13.4050	0.0190	4.5344	0.042	0.0062
	C	5	14.9017	0.7534	4.6499	-0.035	0.0074
	C	6	15.6369	0.7709	3.4907	-0.009	0.0063

SUM OF P(I) 0.0000 R.M.S. OF P(I) 0.035763

OTHER ATOMS

N	1	12.0036	0.6874	0.0017	-0.045	0.0071
N	2	12.7471	0.7494	5.0390	-0.037	0.0053
N	3	17.0021	0.0650	3.6153	0.073	0.0064
O	1	10.7926	0.7703	1.0993	0.023	0.0071
O	8	15.5993	0.9006	5.0572	0.093	0.0204

PLANE 3 IS (0.1060)X + (-0.9723)Y + (-0.1406)Z -(1.5664) = 0

CHI SQUARED = 55.2737

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	N	1	12.0036	0.6874	0.0017	0.047	0.0072
	C	2	13.6251	0.7062	2.1656	-0.012	0.0060
	O	2	13.4963	1.0129	-0.0093	-0.017	0.0093
	O	3	11.6050	0.5123	0.9630	-0.017	0.0087

SUM OF P(I) -0.0000 R.M.S. OF P(I) 0.027619

PLANE 4 IS (-0.1357)X + (0.9908)Y + (-0.0374)Z -(-1.1921) = 0

CHI SQUARED = 9.6982

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	N	2	12.7471	0.7494	5.0390	-0.014	0.0053
	C	4	13.4050	0.0190	4.5344	0.003	0.0062
	O	4	11.4963	0.5004	5.5941	0.004	0.0059
	O	5	13.3125	0.0060	6.8779	0.005	0.0060

SUM OF P(I) 0.0000 R.M.S. OF P(I) 0.008469

PLANE 5 IS (0.1030)X + (-0.9946)Y + (-0.0116)Z -(0.0144) = 0

CHI SQUARED = 99.9959

ATOMS IN PLANE	ATOM NO.		X	Y	Z	P	ESD(P)
	N	3	17.0021	0.0650	3.6153	0.241	0.0064
	C	6	15.6369	0.7709	3.4907	-0.211	0.0063
	O	8	17.5572	0.9562	4.8317	-0.013	0.0060
	O	9	17.7259	1.0012	2.6296	-0.015	0.0062

SUM OF P(I) -0.0000 R.M.S. OF P(I) 0.023863

PLANE 1	PLANE 2	DIHEDRAL ANGLE (DEGREES)
1	2	177.667
2	3	166.387
2	4	7.563
2	5	174.330

TABLE 4. INTERMOLECULAR DISTANCES

PICRIC ACID - NAPHTHALENE 1:1 COMPLEX A.BANERJEE

INTERMOLECULAR DISTANCES, WITH THE COORDINATES USED IN THE COMPUTATION

ATOM	N1	X/A	Y/B	Z/C	ATOM	N2	X/A	Y/B	Z/C	DISTANCE	E.S.D.
C	9	0.53466	0.12307	0.70024	C	1	0.55073	0.61024	0.84583	3.989	0.0081
C	12	0.40754	0.12293	0.87427	C	1	0.55073	0.61024	0.84583	3.632	0.0099
C	13	0.36712	0.12941	0.86473	C	1	0.55073	0.61024	0.84583	3.371	0.0096
C	14	0.39474	0.12591	0.77418	C	1	0.55073	0.61024	0.84583	3.602	0.0085
C	12	0.40754	0.12293	0.87427	C	1	0.55073	-0.38176	0.84583	3.693	0.0099
C	13	0.36712	0.12941	0.86473	C	1	0.55073	-0.38176	0.84583	3.524	0.0096
C	14	0.39474	0.12591	0.77418	C	1	0.55073	-0.38176	0.84583	3.701	0.0085
C	13	0.36712	0.12941	0.86473	C	2	0.64586	0.60274	0.84748	3.514	0.0107
C	14	0.39474	0.12591	0.77418	C	2	0.64586	0.60274	0.84748	3.510	0.0093
C	15	0.47953	0.11159	0.75925	C	2	0.64586	0.60274	0.84748	3.666	0.0097
C	13	0.36712	0.12941	0.86473	C	2	0.64586	-0.39726	0.84748	3.856	0.0107
C	14	0.39474	0.12591	0.77418	C	2	0.64586	-0.39726	0.84748	3.809	0.0093
C	15	0.47953	0.11159	0.75925	C	2	0.64586	-0.39726	0.84748	3.778	0.0097
O	7	1.10980	0.14569	0.10509	C	3	1.31430	0.38952	0.23466	3.719	0.0087
C	14	0.39474	0.12591	0.77418	C	3	0.68550	0.61048	0.76534	3.650	0.0081
C	15	0.47953	0.11159	0.75925	C	3	0.68550	0.61048	0.76534	3.430	0.0083
C	16	0.49984	0.11805	0.66720	C	3	0.68550	0.61048	0.76534	3.681	0.0088
C	14	0.39474	0.12591	0.77418	C	3	0.68550	-0.38952	0.76534	3.844	0.0081
C	15	0.47953	0.11159	0.75925	C	3	0.68550	-0.38952	0.76534	3.449	0.0083
C	16	0.49984	0.11805	0.66720	C	3	0.68550	-0.38952	0.76534	3.777	0.0088
C	7	0.63952	0.12107	0.59267	C	4	0.63769	0.61807	0.68098	3.648	0.0088
C	8	0.55522	0.13252	0.60611	C	4	0.63769	0.61807	0.68098	3.712	0.0098
C	9	0.53466	0.12307	0.70024	C	4	0.63769	0.61807	0.68098	3.820	0.0085
C	14	0.39474	0.12591	0.77418	C	4	0.63769	0.61807	0.68098	3.735	0.0088
C	15	0.47953	0.11159	0.75925	C	4	0.63769	0.61807	0.68098	3.709	0.0093
C	16	0.49984	0.11805	0.66720	C	4	0.63769	0.61807	0.68098	3.590	0.0096
C	7	0.63952	0.12107	0.59267	C	4	0.63769	-0.38113	0.68098	3.676	0.0088
C	8	0.55522	0.13252	0.60611	C	4	0.63769	-0.38113	0.68098	3.892	0.0098
C	9	0.53466	0.12307	0.70024	C	4	0.63769	-0.38113	0.68098	3.871	0.0085
C	14	0.39474	0.12591	0.77418	C	4	0.63769	-0.38113	0.68098	3.823	0.0088
C	15	0.47953	0.11159	0.75925	C	4	0.63769	-0.38113	0.68098	3.606	0.0093
C	16	0.49984	0.11805	0.66720	C	4	0.63769	-0.38113	0.68098	3.587	0.0096
C	7	0.63952	0.12107	0.59267	C	5	0.54966	0.60963	0.67200	3.806	0.0101
C	8	0.55522	0.13252	0.60611	C	5	0.54966	0.60963	0.67200	3.418	0.0105
C	9	0.53466	0.12307	0.70024	C	5	0.54966	0.60963	0.67200	3.378	0.0093
C	10	0.45003	0.14129	0.70926	C	5	0.54966	0.60963	0.67200	3.660	0.0104
C	14	0.39474	0.12591	0.77418	C	5	0.54966	0.60963	0.67200	3.666	0.0097
C	8	0.55522	0.13252	0.60611	C	5	0.54966	-0.39037	0.67200	3.721	0.0105
C	9	0.53466	0.12307	0.70024	C	5	0.54966	-0.39037	0.67200	3.561	0.0093
C	14	0.39474	0.12591	0.77418	C	5	0.54966	-0.39037	0.67200	3.870	0.0097
C	9	0.53466	0.12307	0.70024	C	6	0.51276	0.61234	0.75444	3.477	0.0086
C	10	0.45003	0.14129	0.70926	C	6	0.51276	0.61234	0.75444	3.429	0.0097
C	11	0.43283	0.11926	0.79575	C	6	0.51276	0.61234	0.75444	3.729	0.0093
C	12	0.40754	0.12293	0.87427	C	6	0.51276	0.61234	0.75444	3.818	0.0101
C	13	0.36712	0.12941	0.86473	C	6	0.51276	0.61234	0.75444	3.736	0.0103
C	14	0.39474	0.12591	0.77418	C	6	0.51276	0.61234	0.75444	3.597	0.0089
C	9	0.53466	0.12307	0.70024	C	6	0.51276	-0.38766	0.75444	3.620	0.0086
C	10	0.45003	0.14129	0.70926	C	6	0.51276	-0.38766	0.75444	3.886	0.0097
C	11	0.43283	0.11926	0.79575	C	6	0.51276	-0.38766	0.75444	3.762	0.0093
C	12	0.40754	0.12293	0.87427	C	6	0.51276	-0.38766	0.75444	3.947	0.0101
C	13	0.36712	0.12941	0.86473	C	6	0.51276	-0.38766	0.75444	3.944	0.0102
C	14	0.39474	0.12591	0.77418	C	6	0.51276	-0.38766	0.75444	3.771	0.0089

TABLE 4 (contd.)

PICRIC ACID - NAPHTHALENE III COMPLEX A.BANERJEE

INTERMOLECULAR DISTANCES. WITH THE COORDINATES USED IN THE COMPUTATION

ATOM	M1	X/A	Y/B	Z/C	ATOM	M2	X/A	Y/B	Z/C	DISTANCE	E.S.D.
O	6	1.11498	0.13894	0.33904	C	7	1.13952	0.37093	0.59267	3.933	0.0107
O	8	1.00126	0.13021	0.41240	C	7	1.13952	0.37093	0.59267	3.644	0.0288
N	2	0.82638	0.10934	0.41110	C	7	0.86048	0.62107	0.40733	3.561	0.0001
O	5	0.86046	0.12928	0.40389	C	7	0.86048	0.62107	0.40733	3.550	0.0007
N	2	0.82638	0.10934	0.41110	C	7	0.86048	-0.37093	0.40733	3.402	0.0001
O	4	0.74730	0.09549	0.39350	C	7	0.86048	-0.37093	0.40733	3.677	0.0093
O	5	0.86046	0.12928	0.40389	C	7	0.86048	-0.37093	0.40733	3.637	0.0086
C	8	0.55522	0.13252	0.60611	C	8	0.44478	-0.13252	0.39389	3.888	0.0102
O	5	0.86046	0.12928	0.40389	C	8	1.05522	0.36748	0.60611	3.704	0.0107
O	8	1.00126	0.13021	0.41240	C	8	1.05522	0.36748	0.60611	3.247	0.0262
O	5	0.86046	0.12928	0.40389	C	8	0.94478	0.63252	0.39389	3.940	0.0090
O	8	1.00126	0.13021	0.41240	C	8	0.94478	0.63252	0.39389	3.573	0.0292
N	2	0.82638	0.10934	0.41110	C	8	0.94478	-0.36748	0.39389	3.822	0.0096
O	5	0.86046	0.12928	0.40389	C	8	0.94478	-0.36748	0.39389	3.900	0.0090
O	8	1.00126	0.13021	0.41240	C	8	0.94478	-0.36748	0.39389	3.543	0.0292
N	3	1.07720	0.12588	0.29459	C	9	0.96334	0.62307	0.29976	3.958	0.0087
O	8	1.00126	0.13021	0.41240	C	9	0.96334	0.62307	0.29976	3.767	0.0282
N	3	1.07720	0.12588	0.29459	C	9	0.96334	-0.37693	0.29976	3.991	0.0087
O	8	1.00126	0.13021	0.41240	C	9	0.96334	-0.37693	0.29976	3.816	0.0282
O	4	0.74730	0.09549	0.39350	C	10	0.54917	-0.14129	0.29074	3.723	0.0099
O	5	0.86046	0.12928	0.40389	C	10	0.73083	0.39871	0.70926	3.698	0.0092
N	3	1.07720	0.12588	0.29459	C	10	1.04917	0.64129	0.29074	3.615	0.0098
O	6	1.11498	0.13894	0.33904	C	10	1.04917	0.64129	0.29074	3.658	0.0100
O	7	1.10988	0.14569	0.18509	C	10	1.04917	0.64129	0.29074	3.898	0.0099
N	3	1.07720	0.12588	0.29459	C	10	1.04917	-0.35871	0.29074	3.488	0.0098
O	6	1.11498	0.13894	0.33904	C	10	1.04917	-0.35871	0.29074	3.627	0.0100
O	7	1.10988	0.14569	0.18509	C	10	1.04917	-0.35871	0.29074	3.951	0.0099
O	8	1.00126	0.13021	0.41240	C	10	1.04917	-0.35871	0.29074	3.903	0.0284
O	3	0.72620	0.07445	0.06700	C	11	0.56717	-0.11324	0.20425	3.694	0.0112
O	4	0.74730	0.09549	0.39350	C	11	0.56717	-0.11324	0.20425	3.996	0.0101
O	2	0.83007	0.14717	0.99360	C	11	0.93283	0.38676	0.79575	3.818	0.0114
N	3	1.07720	0.12588	0.29459	C	11	1.06717	0.61324	0.20425	3.439	0.0094
O	6	1.11498	0.13894	0.33904	C	11	1.06717	0.61324	0.20425	3.838	0.0100
O	7	1.10988	0.14569	0.18509	C	11	1.06717	0.61324	0.20425	3.318	0.0093
N	3	1.07720	0.12588	0.29459	C	11	1.06717	-0.38676	0.20425	3.582	0.0094
O	7	1.10988	0.14569	0.18509	C	11	1.06717	-0.38676	0.20425	3.726	0.0093
C	12	0.48754	0.12293	0.87427	C	12	0.91246	-0.12293	1.12573	3.933	0.0113
O	3	0.72620	0.07445	0.06700	C	12	0.91246	-0.12293	0.12573	3.905	0.0137
O	2	0.83007	0.14717	0.99360	C	12	0.98754	0.37707	0.87427	3.601	0.0134
O	1	0.97993	0.11307	0.07749	C	12	0.98754	0.37707	-0.12573	3.441	0.0109
N	3	1.07720	0.12588	0.29459	C	12	1.01246	0.62293	0.12573	3.965	0.0105
O	1	0.97993	0.11307	0.07749	C	12	1.01246	0.62293	0.12573	3.598	0.0104
O	7	1.10988	0.14569	0.18509	C	12	1.01246	0.62293	0.12573	3.697	0.0103
O	1	0.97993	0.11307	0.07749	C	12	1.01246	-0.37707	0.12573	3.466	0.0104
O	7	1.10988	0.14569	0.18509	C	12	1.01246	-0.37707	0.12573	3.977	0.0104
O	3	0.72620	0.07445	0.06700	C	13	0.56712	0.12941	-0.13527	3.679	0.0115
O	1	0.97993	0.11307	0.07749	C	13	1.06712	0.37059	-0.13527	3.924	0.0112
O	1	0.97993	0.11307	0.07749	C	13	0.93288	0.62941	0.13527	3.742	0.0107
N	1	0.79906	0.09982	0.06192	C	13	0.93288	-0.37059	0.13527	3.967	0.0112
O	1	0.97993	0.11307	0.07749	C	13	0.93288	-0.37059	0.13527	3.929	0.0107
O	2	0.83007	0.14717	0.99360	C	15	0.67993	0.11159	0.79925	3.924	0.0118
O	7	1.10988	0.14569	0.18509	C	15	1.32007	-0.11159	0.24075	3.853	0.0102
O	6	1.11498	0.13894	0.33904	C	16	1.30016	-0.11008	0.33200	3.390	0.0101
O	7	1.10988	0.14569	0.18509	C	16	1.30016	-0.11008	0.33200	3.976	0.0105
N	2	0.82638	0.10934	0.41110	C	16	0.80016	0.61008	0.33200	3.698	0.0090
O	4	0.74730	0.09549	0.39350	C	16	0.80016	0.61008	0.33200	3.888	0.0094
N	2	0.82638	0.10934	0.41110	C	16	0.80016	-0.38992	0.33200	3.367	0.0090
O	4	0.74730	0.09549	0.39350	C	16	0.80016	-0.38992	0.33200	3.481	0.0074
O	2	0.83007	0.14717	0.99360	N	1	0.70094	0.39968	0.93808	3.783	0.0110
O	3	0.72620	0.07445	0.06700	N	1	0.70094	-0.40038	-0.06192	3.792	0.0111
O	6	1.11498	0.13894	0.33904	N	2	1.17362	-0.10938	0.58090	3.966	0.0096
O	8	1.00126	0.13021	0.41240	N	2	1.17362	-0.10938	0.58090	3.911	0.0263
O	6	1.11498	0.13894	0.33904	N	2	1.32638	0.39066	0.41110	3.876	0.0091
O	4	0.74730	0.09549	0.39350	N	3	0.92720	0.37412	0.29459	3.778	0.0090
O	2	0.83007	0.14717	0.99360	O	1	1.02007	-0.11307	0.92251	3.806	0.0123
O	1	0.97993	0.11307	0.07749	O	1	1.02007	-0.11307	-0.07749	2.044	0.0105
O	7	1.10988	0.14569	0.18509	O	2	1.16993	-0.14717	0.00632	3.401	0.0114
O	3	0.72620	0.07445	0.06700	O	2	0.66993	-0.35283	0.00632	3.169	0.0126
C	13	0.56712	0.12941	0.86473	O	3	0.72620	0.07445	1.06700	3.679	0.0115
O	7	1.10988	0.14569	0.18509	O	3	1.22620	0.42555	0.06700	3.290	0.0110
O	6	1.11498	0.13894	0.33904	O	4	1.24730	0.41455	0.39350	2.901	0.0092
O	7	1.10988	0.14569	0.18509	O	4	1.24730	0.41455	0.39350	3.970	0.0101
O	6	1.11498	0.13894	0.33904	O	5	1.13154	-0.12928	0.31611	3.110	0.0094
O	8	1.00126	0.13021	0.41240	O	5	1.13154	-0.12928	0.31611	3.022	0.0272
O	8	1.00126	0.13021	0.41240	O	8	0.99874	-0.13021	0.58752	3.002	0.0362

TABLE 5. STRUCTURE FACTORS

PAGE 0 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

MONOCLINIC
UNIT CELL DIMENSIONS
A = 16.248
B = 6.871
C = 14.306
BETA = 96.62
SPACE GROUP P2(1)/A
NUMBER OF REFLEXIONS IN SPHERE = 1559
NUMBER USED IN THE REFINEMENT = 918
R-FACTOR 0.0663
SCALE FACTOR USED = 10.286

PAGE 1 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC
H= 0, L= 0			2	632	660	H= 16, L= 0			1	172	176	H= 8, L= 1		
2	370	384	3	215	-201	0	97	107	2	34	30	0	268	283
4	1627	-1599	4	0*	29	1	0*	38	3	35	33	1	191	-169
			5	70	-72				4	83	66	2	0*	55
H= 1, L= 0			6	127	-133	H= 0, L= 1			5	77	-85	3	74	-94
1	466	468				0	59	63				4	131	-132
2	1716	1713	H= 8, L= 0			1	412	-393	H= 4, L= -1					
3	121	-126	0	127	-149	2	77	76	0	1036	1039	H= 8, L= -1		
4	332	304	1	531	524	3	325	-338	1	319	-295	0	745	-760
5	83	-87	2	0*	-1	4	73	82	2	80	58	1	432	409
6	202	-199	3	282	281				3	48	50	2	0*	-34
			4	129	135	H= 1, L= 1			4	322	-294	3	228	212
H= 2, L= 0			5	91	-91	1	248	262				4	262	253
0	535	547				2	2364	-2345	H= 5, L= 1			5	74	-55
1	44	42	H= 9, L= 0			3	54	85	1	407	390			
2	251	-250	1	72	71	4	100	-77	2	132	105	H= 9, L= 1		
3	140	162	2	170	189	6	353	354	3	126	-135	1	120	143
4	272	-250	3	94	-83				4	132	-115	2	0*	2
5	52	59	4	56	46	H= 1, L= -1						3	56	-47
6	74	58				1	117	-134	H= 5, L= -1			4	0*	46
			H= 10, L= 0			2	2153	2127	1	0*	-10	5	55	-48
H= 3, L= 0			0	80	-70	3	167	-161	2	64	62			
1	504	-527	1	57	-35	4	0*	34	3	62	-63	H= 9, L= -1		
2	280	-283	2	58	-57	6	263	-261	4	128	111	1	329	-334
3	331	328	3	0*	-4							2	327	-315
4	62	-50	4	0*	37	H= 2, L= 1			H= 6, L= 1			3	174	150
5	46	63				0	887	861	0	574	586	4	79	-79
6	78	-55	H= 11, L= 0			1	417	391	1	160	148	5	55	55
			1	86	92	2	236	235	2	0*	-13			
H= 4, L= 0			2	0*	6	3	202	203	3	44	51	H= 10, L= 1		
0	197	-185	3	0*	-31	4	407	-393	4	159	-147	0	99	103
1	334	-327	4	0*	29	6	91	-119				1	0*	-50
2	253	280							H= 6, L= -1			2	0*	30
3	80	-72	H= 12, L= 0			H= 2, L= -1			0	495	-502	3	0*	29
4	109	115	0	0*	-24	0	689	-689	1	270	274	4	84	-69
5	0*	47	1	91	-76	1	56	-64	2	0*	-32			
			2	0*	10	2	175	-154	3	173	168	H= 10, L= -1		
H= 5, L= 0			3	64	-43	3	55	-37	4	181	183	0	219	204
1	0*	-31	4	0*	5	4	184	181	5	79	-67	1	62	62
2	83	-88				6	77	57				2	0*	0
3	67	54	H= 13, L= 0						H= 7, L= 1			3	0*	47
4	0*	50	1	0*	8	H= 3, L= 1			1	226	244	4	59	-56
5	42	-49	2	67	54	1	344	-325	2	160	172			
			3	0*	-29	2	72	77	3	153	-170	H= 11, L= 1		
H= 6, L= 0			4	0*	21	3	154	142	4	0*	-14	1	87	-93
0	123	129				4	77	58	5	50	-51	2	0*	-17
1	258	272	H= 14, L= 0									3	63	88
2	0*	5	0	0*	-31	H= 3, L= -1			H= 7, L= -1			4	0*	16
3	121	122	1	0*	-9	1	302	327	1	342	-331			
4	100	-94	2	0*	13	2	342	-314	2	45	-44	H= 11, L= -1		
5	46	-62	3	0*	-41	3	148	-165	3	277	272	1	76	105
6	44	-53				4	0*	-14	4	0*	9	2	106	92
			H= 15, L= 0						5	85	81	3	62	-53
H= 7, L= 0			1	67	66	H= 4, L= 1						4	0*	39
1	486	465	2	0*	-2	0	163	-150						

K	FO	FC
H= 12, L= 1		
0	159	185
1	0*	-1
2	0*	10
3	0*	-23
4	108	-114
H= 12, L= -1		
0	64	-54
1	64	67
2	64	-74
3	0*	45
4	0*	42
H= 13, L= 1		
1	68	-92
2	95	79
3	0*	52
4	0*	-22
H= 13, L= -1		
1	0*	24
2	54	-66
3	0*	-15
4	0*	-16
H= 14, L= 1		
0	0*	48
1	0*	-49
2	0*	8
3	0*	-53
H= 14, L= -1		
0	69	54
1	0*	28
2	0*	26
3	0*	21
H= 15, L= 1		
1	0*	17
2	0*	-27
H= 15, L= -1		
1	0*	2
2	96	91
H= 16, L= 1		
0	0*	27
1	0*	-34
H= 16, L= -1		
0	0*	-77
1	96	80

K	FO	FC
H= 0, L= 2		
0	1047	-1036
1	91	-105
2	63	61
3	44	-26
4	425	438
5	39	48
H= 1, L= 2		
1	369	347
2	314	312
3	85	-89
4	108	-91
6	42	52
H= 1, L= -2		
1	76	-93
2	329	328
3	0*	13
4	0*	11
H= 2, L= 2		
0	1175	-1162
1	563	539
2	58	-55
3	152	129
4	288	280
5	47	-38
6	0*	-2
H= 2, L= -2		
0	661	-655
1	516	493
2	0*	-32
3	119	142
4	116	88
H= 3, L= 2		
1	72	-72
2	82	-82
3	0*	6
4	127	-112
H= 3, L= -2		
1	266	-262
2	622	643
3	64	50
4	0*	-51
H= 4, L= 2		
0	380	407
1	297	-308
2	54	62
3	76	-69
4	89	-79

K	FO	FC
H= 4, L= -2		
0	0*	-3
1	84	-86
2	34	45
3	93	-73
4	0*	42
H= 5, L= 2		
1	241	245
2	0*	10
3	178	-174
4	44	48
H= 5, L= -2		
1	136	140
2	66	-61
3	82	-71
4	0*	35
H= 6, L= 2		
0	214	210
1	215	-236
2	124	-123
3	102	-106
4	67	-61
H= 6, L= -2		
0	494	499
1	91	-95
2	71	-69
3	44	43
4	86	-80
5	46	42
H= 7, L= 2		
1	132	117
2	166	-160
3	0*	-33
4	0*	-36
H= 7, L= -2		
1	244	-265
2	359	-342
3	130	138
4	0*	-39
5	107	128
H= 8, L= 2		
0	143	131
1	0*	15
2	52	56
3	0*	-8
4	55	-59

K	FO	FC
H= 8, L= -2		
0	443	443
1	235	-256
2	0*	73
3	158	-156
4	183	-202
5	74	74
H= 9, L= 2		
1	123	-120
2	0*	-45
3	0*	47
4	0*	-1
H= 9, L= -2		
1	167	-170
2	0*	34
3	113	93
4	0*	9
H= 10, L= 2		
0	117	-116
1	0*	-40
2	0*	4
3	61	-61
4	60	59
H= 10, L= -2		
0	0*	-39
1	0*	-24
2	0*	-9
3	0*	-2
4	0*	-12
H= 11, L= 2		
1	136	146
2	200	-214
3	0*	-45
4	0*	-9
H= 11, L= -2		
1	113	-113
2	0*	8
3	62	37
4	0*	-53
H= 12, L= 2		
0	0*	22
1	187	203
2	0*	-11
3	160	150
4	0*	21
H= 12, L= -2		
0	0*	-2

K	FO	FC
1	0*	17
2	0*	-14
3	0*	0
4	0*	14
H= 13, L= 2		
1	97	-122
2	215	-202
3	0*	43
4	0*	-10
H= 13, L= -2		
1	0*	8
2	0*	-3
3	0*	14
4	0*	-18
H= 14, L= 2		
0	70	71
1	0*	15
2	0*	-14
3	0*	3
H= 14, L= -2		
0	0*	-1
1	0*	36
2	0*	-17
3	62	65
H= 15, L= 2		
1	0*	-19
2	0*	-25
H= 15, L= -2		
1	98	-103
2	0*	11
H= 16, L= 2		
0	0*	-28
1	0*	-5
H= 16, L= -2		
0	62	-62
1	62	-67
H= 0, L= 3		
0	321	320
1	291	-274
2	0*	6
3	313	-302
4	0*	1
5	49	-50
H= 1, L= 3		
1	652	650

PAGE 3 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC
H=	1, L=	3
2	210	203
3	0*	-17
4	0*	16
5	62	-74
H=	1, L=	-3
1	532	-523
2	181	182
3	70	69
4	53	52
5	60	75
H=	2, L=	3
0	165	144
1	259	254
2	100	-115
3	118	142
4	54	-54
6	37	37
H=	2, L=	-3
0	1041	-1044
1	382	-368
2	31	-31
3	381	-371
4	226	218
H=	3, L=	3
1	246	-260
2	278	-288
3	38	31
4	0*	-22
H=	3, L=	-3
1	412	420
2	214	-214
3	35	30
4	37	-33
H=	4, L=	3
0	819	815
1	210	-198
2	56	68
3	82	-86
4	202	-195
H=	4, L=	-3
0	72	-65
1	196	203
2	64	78
3	136	146
4	0*	41

K	FO	FC
H=	5, L=	3
1	93	95
2	74	74
3	82	89
4	82	83
H=	5, L=	-3
1	195	-205
2	297	297
3	0*	-33
4	62	47
H=	6, L=	3
0	90	90
1	0*	0
2	46	-63
3	48	43
4	0*	-33
H=	6, L=	-3
0	184	178
1	0*	15
2	0*	-33
3	111	-115
4	82	-59
H=	7, L=	3
1	85	-80
2	0*	-42
3	0*	-1
4	0*	-15
H=	7, L=	-3
1	93	99
2	441	438
3	0*	-39
4	51	43
H=	8, L=	3
0	0*	-42
1	0*	38
2	76	59
3	0*	19
4	0*	11
H=	8, L=	-3
0	130	-149
1	55	-51
2	0*	-11
3	91	-67
4	0*	-8
5	53	52
H=	9, L=	3
1	0*	-64

K	FO	FC
2	0*	15
3	0*	23
4	0*	29
H=	9, L=	-3
1	0*	-15
2	0*	32
3	112	-89
4	0*	5
H=	10, L=	3
0	159	-173
1	105	-78
2	0*	-7
3	0*	-7
4	61	73
H=	10, L=	-3
0	81	77
1	81	-95
2	0*	33
3	82	-84
4	0*	-16
H=	11, L=	3
1	64	55
2	129	142
3	91	-77
4	0*	27
H=	11, L=	-3
1	61	57
2	0*	-26
3	0*	-17
4	0*	-17
H=	12, L=	3
0	335	-361
1	0*	7
2	0*	-3
3	0*	1
4	240	234
H=	12, L=	-3
0	0*	57
1	0*	-2
2	0*	9
3	0*	7
4	0*	-45
H=	13, L=	3
1	93	95
2	0*	-31
3	93	-82
4	0*	2

K	FO	FC
H=	13, L=	-3
1	68	-64
2	0*	4
3	0*	-3
4	0*	-22
H=	14, L=	3
0	172	-181
1	72	75
2	0*	-9
3	0*	59
4	81	80
H=	14, L=	-3
0	0*	2
1	69	-73
2	0*	3
3	72	-62
4	0*	-6
H=	15, L=	3
1	0*	-3
2	0*	-31
H=	15, L=	-3
1	0*	23
2	0*	6
H=	16, L=	3
0	0*	-24
1	0*	4
H=	16, L=	-3
0	0*	-30
1	68	-68
H=	0, L=	4
0	163	-152
1	84	-58
2	136	-120
3	82	-99
4	57	47
H=	1, L=	4
1	222	213
2	0*	-17
3	75	-84
4	56	-55
H=	1, L=	-4
1	96	79
2	130	-117
3	116	-104
4	67	-74

K	FO	FC
H=	2, L=	4
0	336	-334
1	202	197
2	84	71
3	79	-62
4	108	107
H=	2, L=	-4
0	131	139
1	290	293
2	88	78
3	65	77
4	0*	-43
H=	3, L=	4
1	39	33
2	411	-415
3	61	-57
4	0*	13
5	89	-61
6	74	99
H=	3, L=	-4
1	0*	-54
2	226	215
3	150	140
4	41	41
H=	4, L=	4
0	117	-114
1	62	-60
2	61	51
3	0*	-29
4	0*	-1
H=	4, L=	-4
0	530	-524
1	112	-137
2	98	-82
3	102	-94
4	123	111
H=	5, L=	4
1	308	310
2	580	-580
3	76	-67
4	0*	22
H=	5, L=	-4
1	0*	20
2	243	-249
3	44	-50
4	0*	-16

PAGE 4 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC
H= 6, L= 4		
0	442	439
1	104	128
2	0*	32
3	102	99
4	105	-106
H= 6, L= -4		
0	0*	45
1	111	-104
2	0*	-29
3	67	-84
4	57	45
H= 7, L= 4		
1	52	-56
2	0*	36
3	0*	8
4	55	33
H= 7, L= -4		
1	59	62
2	0*	41
3	102	-78
4	0*	42
H= 8, L= 4		
0	95	-89
1	0*	2
2	0*	0
3	0*	-39
4	58	51
H= 8, L= -4		
0	198	-209
1	73	57
2	74	-81
3	0*	-11
4	62	64
H= 9, L= 4		
1	103	79
2	0*	-22
3	0*	-15
4	60	-74
H= 9, L= -4		
1	0*	17
2	0*	29
3	0*	44
4	0*	-26
H= 10, L= 4		
0	246	247
1	0*	40
2	62	61
3	72	77
4	151	-162
H= 10, L= -4		
0	58	55
1	59	80
2	0*	47
3	60	58
4	0*	-11
H= 11, L= 4		
1	66	53
2	329	328
3	0*	-22
4	0*	19
H= 11, L= -4		
1	0*	-28
2	141	141
3	0*	11
4	0*	38
H= 12, L= 4		
0	136	144
1	0*	23
2	0*	11
3	66	53
4	61	-65
H= 12, L= -4		
0	0*	18
1	0*	9
2	0*	-2
3	0*	-20
4	0*	-34
H= 13, L= 4		
1	0*	-7
2	322	318
3	0*	4
H= 13, L= -4		
1	96	105
2	68	63
3	62	-67
H= 14, L= 4		
0	99	-116
1	0*	-73
2	0*	-16
3	0*	-53
H= 14, L= -4		
0	0*	26
1	70	53
2	0*	34
3	0*	33
H= 15, L= 4		
1	62	58
2	0*	12
H= 15, L= -4		
1	0*	48
2	0*	3
H= 16, L= 4		
0	0*	46
1	0*	18
H= 16, L= -4		
0	62	-56
1	0*	34
H= 0, L= 5		
0	124	131
1	151	-150
2	0*	19
3	83	-72
4	43	-31
H= 1, L= 5		
1	137	165
2	148	147
3	0*	-56
4	63	54
H= 1, L= -5		
1	297	-294
2	138	120
3	59	41
4	43	42
H= 2, L= 5		
0	313	-310
1	82	-79
2	73	-68
3	0*	-12
4	128	101
5	66	72
H= 2, L= -5		
0	588	-581
1	229	-208
2	0*	7
3	146	-138
4	170	173
H= 3, L= 5		
1	264	-265
2	140	-127
3	46	-30
4	48	-51
5	69	82
H= 3, L= -5		
1	299	305
2	231	-230
3	94	-81
4	64	-60
5	92	-115
H= 4, L= 5		
0	528	-514
1	584	-578
2	0*	25
3	375	-364
4	251	239
5	71	76
H= 4, L= -5		
0	135	124
1	147	137
2	87	70
3	157	169
4	47	-45
H= 5, L= 5		
1	377	400
2	258	-266
3	111	-128
4	72	-85
5	74	-94
H= 5, L= -5		
1	266	252
2	206	191
3	179	-185
4	49	30
H= 6, L= 5		
0	227	-229
1	52	36
2	53	51
3	54	43
4	55	62
H= 6, L= -5		
0	94	89
1	336	330
2	84	-90
3	190	215
4	0*	5
H= 7, L= 5		
1	0*	3
2	0*	23
3	0*	-34
4	58	-16
H= 7, L= -5		
1	72	-66
2	89	82
3	0*	51
4	82	-88
H= 8, L= 5		
0	116	-124
1	0*	54
2	0*	4
3	0*	25
4	0*	44
H= 8, L= -5		
0	75	-89
1	107	-99
2	0*	1
3	79	-71
4	0*	48
H= 9, L= 5		
1	62	-58
2	108	-101
3	0*	47
4	0*	-29
H= 9, L= -5		
1	0*	18
2	58	-43
3	0*	31
4	0*	-4
H= 10, L= 5		
0	91	100
1	238	-221
2	65	-48
3	189	-191
4	87	-60
H= 10, L= -5		
0	104	-113
1	0*	-40
2	0*	-3
3	62	50
4	0*	25
H= 11, L= 5		
1	227	234
2	335	-308

PAGE 5 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC
H= 11, L= 5		
3	93	-89
4	0*	-64
H= 11, L= -5		
1	90	-81
2	91	93
3	91	65
4	0*	34
H= 12, L= 5		
0	485	501
1	0*	11
2	0*	35
3	0*	23
4	233	-254
H= 12, L= -5		
0	115	-132
1	115	102
2	0*	-9
3	65	82
4	88	79
H= 13, L= 5		
1	0*	0
2	96	77
3	0*	20
H= 13, L= -5		
1	119	-122
2	136	126
3	93	95
H= 14, L= 5		
0	69	70
1	0*	-12
2	0*	-10
H= 14, L= -5		
0	140	-138
1	99	86
2	0*	-13
3	0*	16
H= 15, L= 5		
1	0*	-21
H= 15, L= -5		
1	0*	-53
2	47	-69
H= 16, L= -5		
0	96	90

K	FO	FC
H= 0, L= 6		
0	134	-127
1	151	-133
2	142	-136
3	114	-95
4	68	74
H= 1, L= 6		
1	242	246
2	0*	-46
3	0*	-8
4	69	-73
H= 1, L= -6		
1	106	90
2	323	-324
3	207	-209
4	48	-58
H= 2, L= 6		
0	72	-66
1	178	210
2	100	123
3	188	203
4	0*	-4
H= 2, L= -6		
0	335	349
1	264	281
2	96	91
3	65	82
4	69	-66
5	109	-111
H= 3, L= 6		
1	549	-548
2	344	333
3	255	239
4	73	91
5	73	88
H= 3, L= -6		
1	0*	30
2	0*	-11
3	64	58
4	70	85
H= 4, L= 6		
0	609	-634
1	173	-181
2	102	-98
3	105	-83
4	167	163

K	FO	FC
H= 4, L= -6		
0	456	-449
1	457	462
2	67	-43
3	420	398
4	51	44
5	102	-99
H= 5, L= 6		
1	165	-149
2	0*	33
3	190	175
4	72	-64
5	77	70
H= 5, L= -6		
1	666	-666
2	0*	-39
3	293	295
4	0*	10
5	189	194
H= 6, L= 6		
0	95	101
1	174	-157
2	56	-46
3	162	-148
4	58	52
H= 6, L= -6		
0	276	-295
1	72	-65
2	42	33
3	79	-105
4	173	152
H= 7, L= 6		
1	144	135
2	118	102
3	85	-63
4	0*	-40
H= 7, L= -6		
1	58	56
2	77	-71
3	0*	4
4	0*	-56
H= 8, L= 6		
0	0*	36
1	61	47
2	0*	-28
3	0*	40
4	0*	-2

K	FO	FC
H= 8, L= -6		
0	56	71
1	98	84
2	0*	13
3	0*	33
4	59	-58
H= 9, L= 6		
1	91	-71
2	0*	24
3	64	64
4	62	36
H= 9, L= -6		
1	62	55
2	104	84
3	61	-47
4	0*	-19
H= 10, L= 6		
0	258	-260
1	94	-72
2	67	-43
3	0*	-55
4	144	144
H= 10, L= -6		
0	62	-68
1	0*	-16
2	0*	-1
3	0*	26
4	87	70
H= 11, L= 6		
1	137	-133
2	245	-251
3	66	82
4	0*	-10
H= 11, L= -6		
1	185	-192
2	0*	18
3	89	89
4	0*	16
H= 12, L= 6		
0	70	-73
1	98	-111
2	0*	4
3	92	-117
4	0*	48
H= 12, L= -6		
0	377	-380
1	262	-247

K	FO	FC
2	67	-61
3	186	-196
4	195	181
H= 13, L= 6		
1	0*	35
2	134	-160
H= 13, L= -6		
1	69	-56
2	226	-228
3	62	61
4	85	-58
H= 14, L= 6		
0	0*	43
H= 14, L= -6		
0	0*	15
1	98	-118
2	0*	26
H= 15, L= -6		
1	0*	-4
H= 0, L= 7		
0	93	-91
1	167	-151
2	70	-72
3	0*	-3
4	0*	-26
H= 1, L= 7		
1	98	-117
2	0*	-14
3	0*	49
4	0*	-24
H= 1, L= -7		
1	136	-126
2	70	-72
3	155	129
4	52	-57
H= 2, L= 7		
0	203	205
1	0*	-8
2	51	49
3	75	-68
4	54	-60
H= 2, L= -7		
0	429	424
1	253	230
2	49	41

K	FO	FC
H= 2, L= -7		
3	100	102
4	149	-149
H= 3, L= 7		
1	104	116
2	265	264
3	77	-50
4	0*	14
H= 3, L= -7		
1	570	-560
2	112	126
3	256	274
4	75	55
5	129	156
H= 4, L= 7		
0	53	52
1	209	183
2	0*	-32
3	118	115
4	99	-73
H= 4, L= -7		
0	69	-79
1	441	-455
2	126	-119
3	264	-270
4	77	52
5	54	64
H= 5, L= 7		
1	0*	-18
2	110	100
3	0*	1
4	0*	46
H= 5, L= -7		
1	62	-50
2	272	-289
3	72	65
4	0*	-31
H= 6, L= 7		
0	131	128
1	59	52
2	0*	-16
3	0*	-38
4	0*	-20
H= 6, L= -7		
0	121	110
1	94	-119
2	0*	-4
3	98	-81
4	0*	-22
H= 7, L= 7		
1	0*	-71
2	0*	29
3	72	67
4	0*	61
H= 7, L= -7		
1	80	-95
2	0*	2
3	62	55
4	0*	-32
H= 8, L= 7		
0	64	-60
1	64	-70
2	0*	-11
3	0*	-62
4	0*	-11
H= 8, L= -7		
0	144	-139
1	84	68
2	60	60
3	0*	0
4	0*	37
H= 9, L= 7		
1	0*	-7
2	0*	69
3	0*	9
4	0*	50
H= 9, L= -7		
1	0*	-33
2	108	108
3	62	61
4	0*	13
H= 10, L= 7		
0	97	-122
1	69	56
2	0*	5
3	0*	62
4	61	51
H= 10, L= -7		
0	351	-351
1	112	-86
2	0*	-18
3	91	-103
4	196	185
H= 11, L= 7		
1	70	-81
2	118	99
3	0*	16
4	0*	16
H= 11, L= -7		
1	150	172
2	361	-368
3	155	-154
4	88	-74
H= 12, L= 7		
0	99	-115
1	0*	36
2	0*	-5
3	0*	9
4	79	84
H= 12, L= -7		
0	179	178
1	92	-111
2	0*	27
3	132	-122
4	61	-63
H= 13, L= 7		
1	0*	-16
H= 13, L= -7		
1	241	262
2	153	-142
3	130	-140
H= 14, L= -7		
0	62	58
1	0*	20
2	0*	15
H= 16, L= -7		
0	65	-72
H= 0, L= 8		
0	53	45
1	106	99
2	0*	-36
3	0*	5
4	0*	-33
H= 1, L= 8		
1	54	49
2	0*	27
3	0*	40
4	0*	-9
H= 1, L= -8		
1	75	-70
2	132	130
3	55	51
4	0*	-13
H= 2, L= 8		
0	77	85
1	110	106
2	0*	40
3	0*	42
4	0*	-21
H= 2, L= -8		
0	0*	-45
1	167	-159
2	0*	9
3	157	-150
4	0*	45
H= 3, L= 8		
1	113	-126
2	0*	-42
3	0*	32
4	0*	38
H= 3, L= -8		
1	76	71
2	155	170
3	0*	-1
4	0*	-72
H= 4, L= 8		
0	0*	16
1	165	-156
2	0*	-27
3	0*	-46
4	72	-73
H= 4, L= -8		
0	121	-137
1	155	-161
2	0*	-16
3	81	-94
4	0*	32
H= 5, L= 8		
1	0*	9
2	72	-84
3	0*	-43
4	0*	17
H= 5, L= -8		
1	62	70
2	68	-64
3	58	-44
4	0*	-65
H= 6, L= 8		
0	53	69
1	0*	-7
2	0*	31
3	0*	30
4	0*	-18
H= 6, L= -8		
0	58	74
1	164	-141
2	0*	28
3	85	-85
4	0*	-12
H= 7, L= 8		
1	65	-80
2	0*	-20
3	0*	1
4	0*	-6
H= 7, L= -8		
1	60	45
2	64	-75
3	0*	29
4	0*	9
H= 8, L= 8		
0	67	56
1	67	68
2	0*	54
3	0*	6
4	0*	-45
H= 8, L= -8		
0	119	-124
1	0*	-41
2	0*	26
3	0*	-5
4	87	84
H= 9, L= 8		
1	0*	-22
2	0*	48
3	0*	42
4	0*	4
H= 9, L= -8		
1	0*	22
2	251	-242
3	0*	10
4	0*	4

PAGE 7 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC
H= 10, L= 8		
0	70	78
1	0*	18
2	0*	25
3	0*	27
4	0*	-60
H= 10, L= -8		
0	94	85
1	133	133
2	0*	24
3	66	70
4	88	-61
H= 11, L= 8		
1	0*	0
2	72	55
3	0*	-23
4	0*	3
H= 11, L= -8		
1	68	70
2	173	-175
3	0*	-8
4	0*	-12
H= 12, L= 8		
0	46	42
1	0*	8
2	0*	-5
4	0*	-5
H= 12, L= -8		
0	349	327
1	207	216
2	0*	29
3	134	157
4	188	-171
H= 13, L= 8		
1	0*	11
H= 13, L= -8		
1	0*	-20
2	96	74
H= 14, L= -8		
0	0*	41
1	0*	38
H= 0, L= 9		
0	63	-61
1	0*	-40
2	59	-63
3	60	-57
4	0*	-10
H= 1, L= 9		
1	69	-65
2	0*	-4
3	0*	-1
4	0*	-25
H= 1, L= -9		
1	50	44
2	55	-52
3	0*	10
4	0*	-46
H= 2, L= 9		
0	72	74
1	102	-95
2	60	58
3	122	-98
4	0*	20
H= 2, L= -9		
0	57	-59
1	58	46
2	83	87
3	62	61
4	0*	43
H= 3, L= 9		
1	86	72
2	61	40
3	0*	-17
4	0*	33
H= 3, L= -9		
1	0*	-5
2	59	-41
3	0*	4
4	0*	5
H= 4, L= 9		
0	102	-94
1	62	61
2	0*	0
3	0*	54
4	0*	17
H= 4, L= -9		
0	104	-130
1	51	-52
2	0*	19
3	0*	-21
4	0*	27
H= 5, L= 9		
1	0*	-18
2	64	-42
3	0*	0
4	0*	-11
H= 5, L= -9		
1	104	107
2	122	-107
3	0*	-34
4.	0*	-17
H= 6, L= 9		
0	82	84
1	66	68
2	0*	23
3	0*	58
4	0*	-40
H= 6, L= -9		
0	123	135
1	0*	-13
2	0*	38
3	0*	-13
4	0*	-14
H= 7, L= 9		
1	0*	20
2	0*	66
3	0*	-35
4	0*	-8
H= 7, L= -9		
1	0*	23
2	0*	-31
3	0*	-29
4	0*	25
H= 8, L= 9		
0	98	-99
1	69	61
2	0*	-24
3	0*	65
4	0*	44
H= 8, L= -9		
0	65	68
1	0*	3
2	0*	-12
3	0*	10
4	57	-51
H= 9, L= 9		
1	0*	21
2	134	-132
3	0*	-9
4	0*	-41
H= 9, L= -9		
1	150	-124
2	95	-96
3	109	114
4	0*	-4
H= 10, L= 9		
0	103	98
1	0*	16
2	0*	7
3	0*	35
H= 10, L= -9		
0	265	246
1	68	-69
2	0*	21
3	0*	-2
4	150	-148
H= 11, L= 9		
1	0*	8
H= 11, L= -9		
1	161	-162
2	153	142
3	92	105
H= 12, L= -9		
0	62	55
1	0*	-54
2	0*	-1
H= 13, L= -9		
1	0*	-29
2	66	78
H= 0, L= 10		
0	62	62
1	121	121
2	0*	-18
3	89	62
4	0*	-12
H= 1, L= 10		
1	229	-235
2	110	-125
3	127	130
4	0*	-40
H= 1, L=-10		
1	82	-93
2	125	98
3	89	72
4	0*	12
H= 2, L= 10		
0	241	235
1	0*	0
2	64	48
3	0*	-15
4	138	-136
H= 2, L=-10		
0	107	-87
1	0*	-12
2	0*	-17
3	0*	-46
4	72	64
H= 3, L= 10		
1	92	-72
2	103	101
3	112	86
4	0*	7
H= 3, L=-10		
1	88	-97
2	93	-90
3	0*	47
4	0*	-36
H= 4, L= 10		
0	62	-62
1	66	-62
2	0*	-6
3	0*	4
4	62	39
H= 4, L=-10		
0	63	69
1	63	-60
2	0*	40
3	64	-52
4	0*	-39
H= 5, L= 10		
1	0*	-16
2	93	-96
3	0*	-16
4	0*	-29
H= 5, L=-10		
1	64	76
2	0*	15
3	0*	-7
4	0*	38

PAGE 8 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC
H=	6, L= 10	
0	82	87
1	68	-94
2	0*	8
3	93	-90
4	0*	2
H=	6, L=-10	
0	0*	-38
1	0*	5
2	0*	-15
3	0*	16
4	0*	5
H=	7, L= 10	
1	98	100
2	0*	-19
3	66	-63
4	0*	-4
H=	7, L=-10	
1	0*	0
2	92	-81
3	0*	-15
4	0*	-19
H=	8, L= 10	
0	0*	-28
1	0*	-23
2	0*	0
3	0*	-33
H=	8, L=-10	
0	135	132
1	135	-118
2	0*	22
3	93	-101
4	62	-57
H=	9, L= 10	
1	69	61
2	0*	56
3	63	-55
H=	9, L=-10	
1	69	70
2	95	84
3	66	-60
H=	10, L= 10	
0	154	-153
1	0*	15
2	0*	-2
3	0*	-12
H=	10, L=-10	
0	0*	-43
1	120	-132
2	0*	-8
3	92	-89
H=	11, L=-10	
1	70	77
2	0*	44
H=	12, L=-10	
0	0*	-28
1	0*	-43
2	0*	25
H=	0, L= 11	
0	384	-413
1	209	235
2	66	-67
3	201	202
4	240	222
H=	1, L= 11	
1	66	-45
2	257	-239
3	0*	-28
4	0*	-36
H=	1, L=-11	
1	93	65
2	243	-241
3	65	-55
4	93	-96
H=	2, L= 11	
0	95	-77
1	139	125
2	0*	5
3	0*	45
4	87	65
H=	2, L=-11	
0	93	88
1	181	-183
2	66	75
3	161	-169
4	0*	-54
H=	3, L= 11	
1	68	66
2	200	-229
3	0*	-2
4	0*	-12
H=	3, L=-11	
1	114	140
2	0*	-19
3	0*	-62
4	0*	17
H=	4, L= 11	
0	128	127
1	137	124
2	0*	-2
3	114	112
4	86	-83
H=	4, L=-11	
0	0*	10
1	0*	9
2	0*	20
3	65	72
4	0*	-18
H=	5, L= 11	
1	98	-94
2	103	98
3	66	64
4	0*	9
H=	5, L=-11	
1	0*	-24
2	0*	-35
3	0*	-6
4	0*	20
H=	6, L= 11	
0	154	-154
1	0*	15
2	0*	-21
3	0*	23
4	103	94
H=	6, L=-11	
0	66	60
1	0*	-6
2	0*	-6
3	0*	-27
4	0*	-9
H=	7, L= 11	
1	120	-121
2	0*	-54
3	90	86
H=	7, L=-11	
1	76	89
2	0*	-62
3	66	-62
H=	8, L= 11	
0	0*	30
1	69	-94
2	0*	7
3	87	-63
H=	8, L=-11	
0	123	124
1	69	64
2	0*	19
3	0*	23
H=	9, L=-11	
1	103	96
2	0*	13
3	65	-56
H=	10, L= 11	
0	0*	52
H=	10, L=-11	
0	0*	23
1	70	50
2	0*	5
H=	11, L=-11	
1	0*	44
H=	12, L=-11	
0	0*	4
H=	0, L= 12	
0	146	159
1	82	83
2	0*	36
3	85	110
4	86	-75
H=	1, L= 12	
1	0*	57
2	342	369
3	93	-92
4	0*	34
H=	1, L=-12	
1	335	341
2	84	-93
3	186	-177
4	0*	8
H=	2, L= 12	
0	403	-434
1	0*	-36
2	0*	-13
3	0*	-32
4	207	228
H=	2, L=-12	
0	235	217
1	134	151
2	0*	48
3	117	126
4	113	-106
H=	3, L= 12	
1	100	118
2	93	-82
3	0*	-57
H=	3, L=-12	
1	0*	-2
2	115	122
3	0*	-38
H=	4, L= 12	
0	70	-67
1	0*	-11
2	0*	17
3	0*	5
H=	4, L=-12	
0	69	-54
1	0*	12
2	0*	-15
3	0*	2
H=	5, L= 12	
1	98	89
2	0*	-7
3	90	-77
H=	5, L=-12	
1	0*	-41
2	96	-78
3	0*	25
H=	6, L= 12	
0	57	69
1	98	83
2	0*	-16
3	88	61
H=	6, L=-12	
0	171	172
1	0*	-40
2	0*	16
3	0*	-36
H=	7, L= 12	
1	0*	11

PAGE 9 NAPHTHALENE - PICRIC ACID 1:1 COMPLEX A.BANERJEE

K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC
H= 7, L=-12			2	0*	-23	H= 4, L=-14								
1	0*	22	3	0*	45	0	0*	-33						
2	96	100				1	0*	29						
3	0*	-1	H= 4, L= 13											
			0	0*	-36	H= 5, L=-14								
H= 8, L=-12			1	72	-73	1	0*	45						
0	99	102												
1	0*	55	H= 4, L=-13			H= 0, L= 15								
2	0*	13	0	0*	53	0	0*	20						
			1	70	-68									
H= 9, L=-12			2	0*	4	H= 2, L=-15								
1	0*	-31	3	0*	-48	0	0*	-19						
2	118	120												
			H= 5, L= 13			H= 0, L= 16								
H= 10, L=-12			1	0*	28	0	0*	-25						
0	0*	-2												
1	0*	19	H= 5, L=-13			H= 4, L= 16								
			1	70	74	0	88	94						
H= 0, L= 13			2	72	70									
0	191	219	3	63	-59	H= 0, L= 17								
1	121	-117				0	48	52						
2	0*	11	H= 6, L=-13											
3	91	-68	0	0*	5									
4	115	-119	1	0*	-15									
			2	0*	-9									
H= 1, L= 13														
1	0*	24	H= 7, L=-13											
2	96	98	1	0*	30									
4	0*	5	2	133	134									
4	0*	5												
			H= 8, L=-13											
H= 1, L=-13			0	119	-128									
1	0*	30	1	0*	12									
2	164	167												
3	0*	14	H= 0, L= 14											
4	0*	50	0	0*	11									
			1	0*	-27									
H= 2, L= 13														
0	101	112	H= 1, L= 14											
1	0*	-36	1	0*	14									
2	0*	-18												
4	56	-75	H= 1, L=-14											
			1	0*	-38									
H= 2, L=-13														
0	121	-113	H= 2, L= 14											
1	121	124	0	0*	49									
2	0*	-22												
3	91	90	H= 2, L=-14											
4	0*	59	0	0*	-12									
			1	0*	3									
H= 3, L= 13														
1	0*	-26	H= 3, L=-14											
			1	0*	-4									
H= 3, L=-13														
1	68	-63												

References

1. F H Herbstein and M Kaftory; Acta Cryst., (1975), B31, 60.

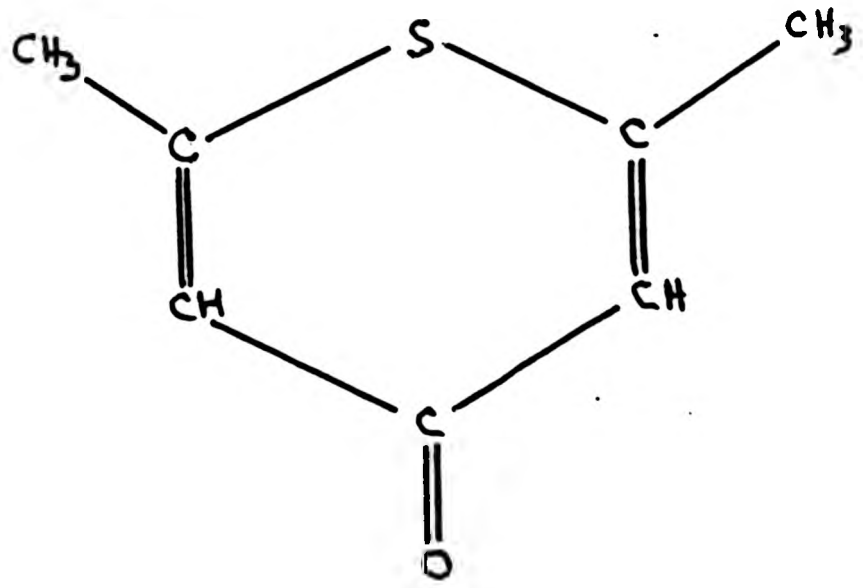
2. F H Herbstein and M Kaftory; Acta Cryst., (1976), B32, 387.

CHAPTER 5.

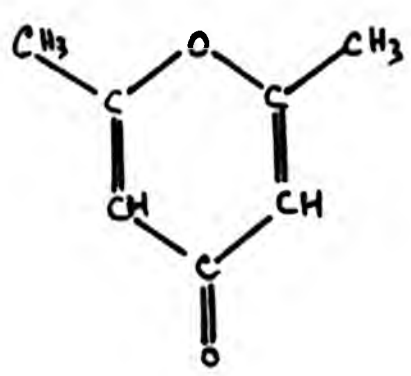
1-THIA-2,6-DIMETHYL-4H-PYRAN-4-ONE

Introduction

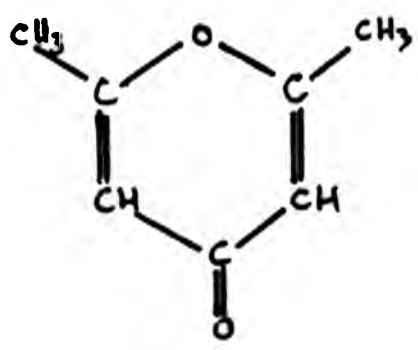
The above material was prepared by Dr Arthur Hill as a part of a program to investigate the anti-corrosion properties of gamma-pyrones. The chemical formula and configuration are as follows:-



The other homologues are given below:-

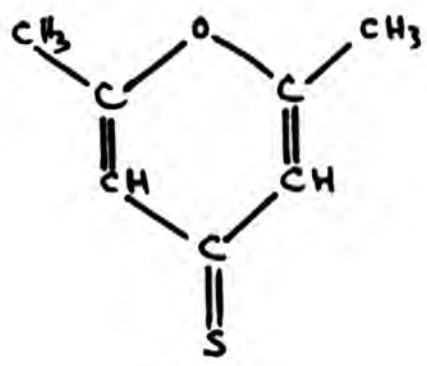


2,6 dimethyl γ-pyrone
(A)



HBr , H_2O

2,6-dimethyl-γ-pyrone hydrobromide monohydrate
(B)



2,6 dimethyl 4-thio-γ-pyrone
(C)

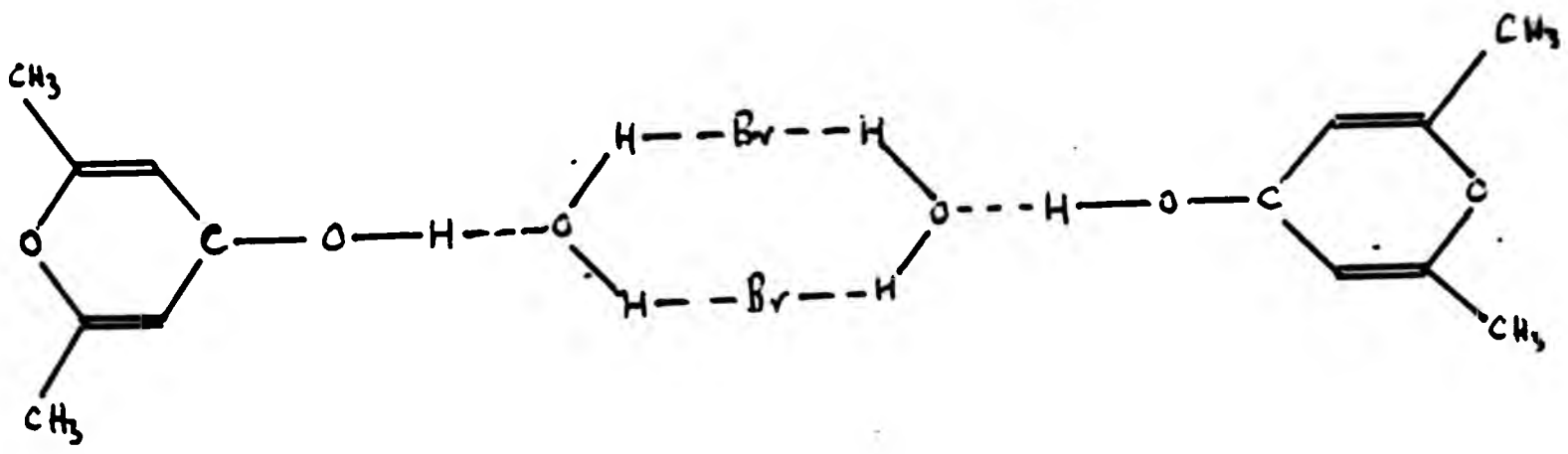
(A) 2:6 dimethyl γ-pyrone (Ref 3)

This crystallizes in the monoclinic system with a = 7.672Å, b = 7.212Å, c = 13.92Å, $\beta = 121^{o}$, $z = 4$, R = 0.104, $P2_1/c$. Only the provisional

results have been given for this structure, and the final results do not seem to have been published yet. The unit cell parameters correspond to those reported by Toussaint but with a different orientation and space group $P2_1/n$.

(B) 2,6-dimethyl-γ-pyrone hydrobromide monohydrate (Ref 4)

This crystallises in the triclinic system with a = 7.00, b = 8.33, c = 9.47Å , α = 109.9, β = 92.9 and γ = 106.0° , space group $P\bar{1}$. The structure was solved from two projections giving R factors of 11% and 7.6% for 307 terms. The results suggest that the keto oxygen has acquired a proton and pairs of molecules are linked by a hydrogen bonding system:-



(C) 2,6-dimethyl-4-thio pyrone (Ref 5)

This structure was solved by J Toussaint. The compound was recrystallised from absolute alcohol. It crystallised in the triclinic system with unit cell dimensions a = 7.66Å , b = 9.69Å , c = 5.39Å , α = 88.5° , β = 105.25° , γ = 108.25° , Z = 2.

The structure was solved by electron density projections on the (001) and (100) planes. Bond lengths of the pyrone ring were interpreted in terms of a resonance structure and values were deduced for the weights of the forms contributing to the electronic structure of the molecules.

The atomic co-ordinates, bond lengths and bond angles are given below in Tables1, 2 and 3.

Fig 1 gives the projection along c showing the position of the molecules in the unit cell.

Table 1.

Atoms	$X_{/A}$	$Y_{/B}$	$Z_{/C}$
C2	0.2682	0.1986	0.570
C3	0.1432	0.1624	0.335
C4	0.0672	0.2637	0.192
C5	0.1342	0.4056	0.327
C6	0.2590	0.4371	0.551
C7	0.3538	0.0999	0.732
C8	0.3408	0.5786	0.704
O	0.3306	0.3359	0.687

Table 2. Bond Lengths

Bond	Length Å
C_1 - C_2	1.472
C_2 - C_3	1.347
C_3 - C_4	1.410
C_4 - C_5	1.421
C_5 - C_6	1.342
C_6 - 0	1.373
C_6 - C_8	1.478
C_4 - S	1.663

Table 3.

Bond	Angles°
C_2 - C_3- C_4	122.4°
C_3 - C_4- C_5	114.2°
C_4 - C_5- C_6	121.4°
C_5 - C_6- 0	123.1°
C_6 - 0 - C_2	116.5
0 - C_2- C_3	122.5
C_1 - C_2- 0	111.5
0 - C_6- C_8	109.3
C_8 - C_6- C_5	127.5
C_5 - C_4- S	121.0
S - C_4- C_3	125.0
C_3 - C_2- C_1	125.5

A. Preparation of the Sample.

The sample was recrystallised by vacuum sublimation of thio-pyranone trihydrate, m.p (66-68°C) at 0.4 mm of Hg pressure at 100°C. This way, pale yellow crystals were formed. The melting point of these crystals was found to be 104°C. These crystals are not very stable. They decompose in atmosphere by taking up water from it. So the crystal was coated with silicone grease and then mounted in a sealed tube for Xray photography and diffractometry.

B. Unit Cell and space group determination.

The unit cell and space group were determined on a Stoe-reciprocal lattice explorer camera from one setting of the crystal (about C axis). Later the cell parameters were refined on the diffractometer giving $a = 9.036\text{Å}$, $b = 11.348\text{Å}$, $c = 7.360\text{Å}$, $\beta = 104.40^{\circ}$, so the cell volume $= abc \sin\beta = 730.988\text{A}^3$

The space group was determined from the following systematic absences.

Reflections	Conditions limiting following absences.
hk1	h + k + 1 = 2n
ho1	h = 2n (1 = 2n)
hoo	(h = 2n)
oko	(k = 2n)
oo1	(1 = 2n)

the space group was Ia with equivalent positions, $0,0,0;\ \frac{1}{2},\frac{1}{2},\frac{1}{2} + [x,y,z;\ \frac{1}{2}+x,\ \bar{y},\ z]$.

C. Density Measurement.

The density was measured by the flotation method in sodium iodide solution. It was 1.275 g/cc and was very close to the calculated density which is 1.274 g/cc confirming that there are four molecules per unit cell.

D. Intensity Data Collection.

Intensity data were obtained using the Stoe-Stadi-2 diffractometer which uses Weissenberg geometry. The crystal was mounted about the C axis. MoK_{α} radiation was used. Intensity data were collected for 0-5 layers. It was possible to collect data from planes with diffraction angles up to 30^{o} for 0 to 2nd layer and up to 20^{o} for the rest of the layers. 571 reflections were recorded. Out of that 114 reflections were small or zero, which were neglected.

E. Structure Determination.

The structure was solved by Multan which used 136 E(hkl) > 1.2 and gave nine clearly defined peaks at reasonable inter-atomic distances.

The Table below lists the co-ordinates of the atoms as read from these peaks. A comparison of these co-ordinates with the final co-ordinates shows that the initial E-map was a very good representation of the structure.

Table 4. E-Map Coordinates

Atom No.	X/A	Y/B	Z/C
S	0.5832	0.6421	1.0004
O	0.5811	1.0368	0.9997
Cl	0.8823	0.6450	1.0758
C2	0.7344	0.7301	1.0391
C3	0.7327	0.8449	0.0394
C4	0.5859	0.9367	0.9995
C5	0.4335	0.8561	0.9581
C6	0.4337	0.7341	0.9598
C7	0.2789	0.6627	0.9503

F. REFINEMENT OF THE STRUCTURE.

The structure was refined using least squares on F. The R-factor went down to 6.11% . The final positional co-ordinates, thermal parameters and their standard deviations are given in Table 5. Bond lengths and angles are given in Tables 6 and 7, and the equation of the mean plane in Table 8.

The fractional coordinates were adjusted so that X' and Z' for sulphur were at 0.5 to fix the unit cell origin of the non-centrosymmetrical lattice, by taking

$$X' = X - 0.08 \text{ approx.}$$

$$Y' = 1.5 - Y$$

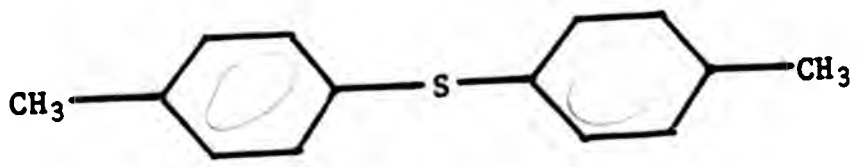
$$Z' = Z - 0.5$$

where X' Y' Z' are the new coordinates listed in Table 5. In the refinement process X' and Z' for sulphur were kept constant.

DISCUSSION

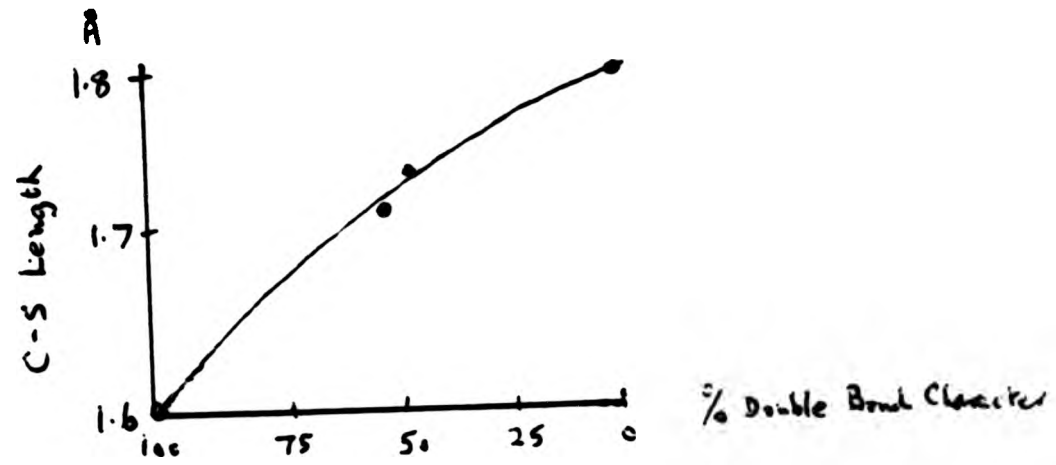
c axis projection in Fig 1 shows the molecules in the unit cell between $z = \frac{1}{4}$ and $z = \frac{3}{4}$. Fig 2 shows how two molecules stack above each other separated by C/2 = 3.68Å . Fig 3 shows the electron density map with contours through each atom.

The sulphur bonding was compared with the C-S list given by S C Abrahams (1) and there were general agreement with values found in comparable molecules. Comparison was also made with the structure of di-p-tolyl sulphide



published by W R Blackmore and S C Abrahams (2). The C-S-C angle and C-S bond lengths were respectively 109.0° , 1.76Å and 1.74Å .

S C Abrahams plotted the double bond character of the C-S bond on the basis of about 40 crystal structure determinations:



According to that, in this structure the C-S bond is approximately 50% double bond. All C-C bonds are only slightly shorter than the normal single bond length 1.54Å and both C=C bonds agree with normal double bond 1.33Å . The molecules are separate, with no intermolecular distances less than 3.6Å .

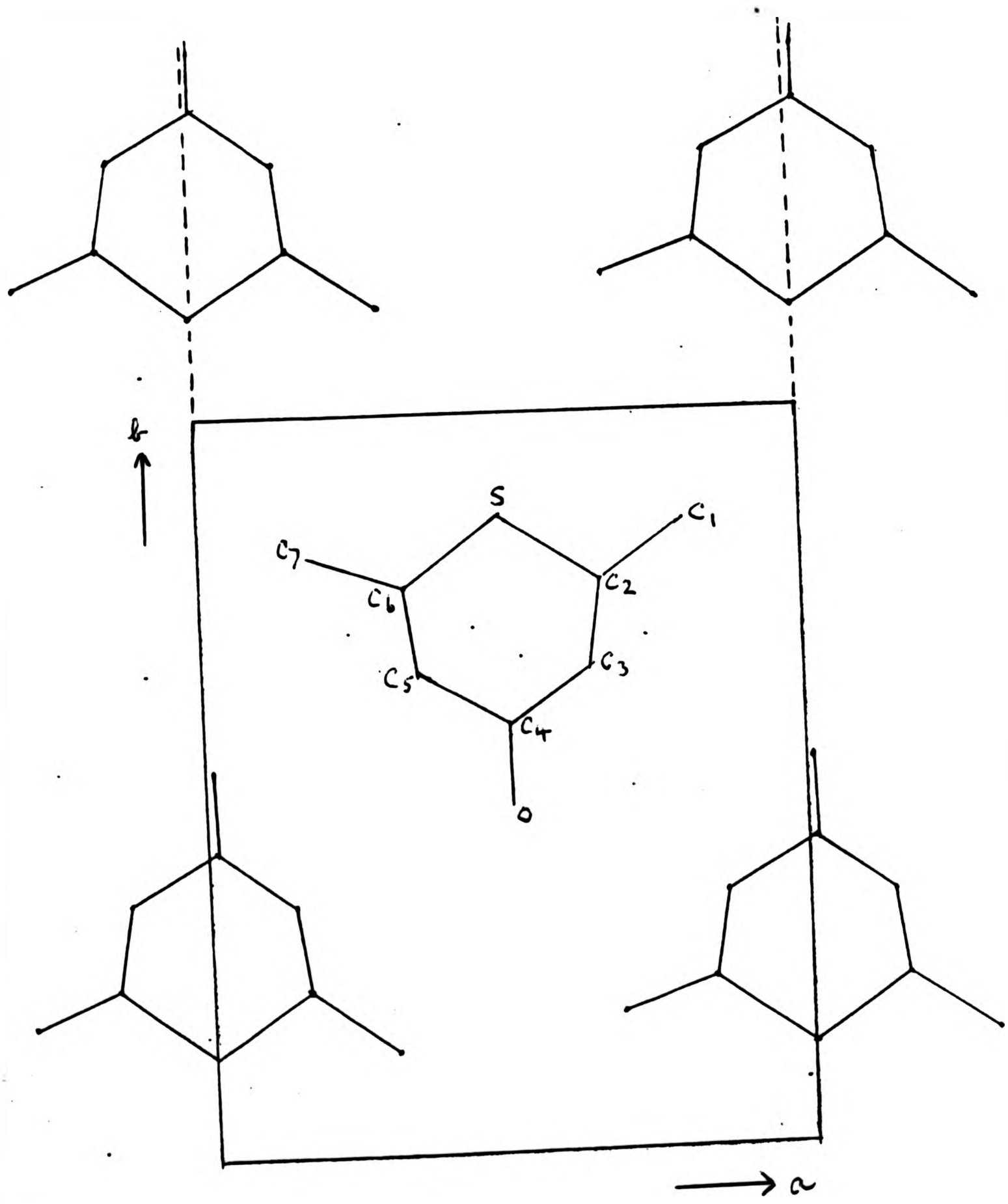


Figure 1. Arrangement of molecules in half of the unit cell (Z = ½ to ⅔) showing numbering of atoms and close packing.

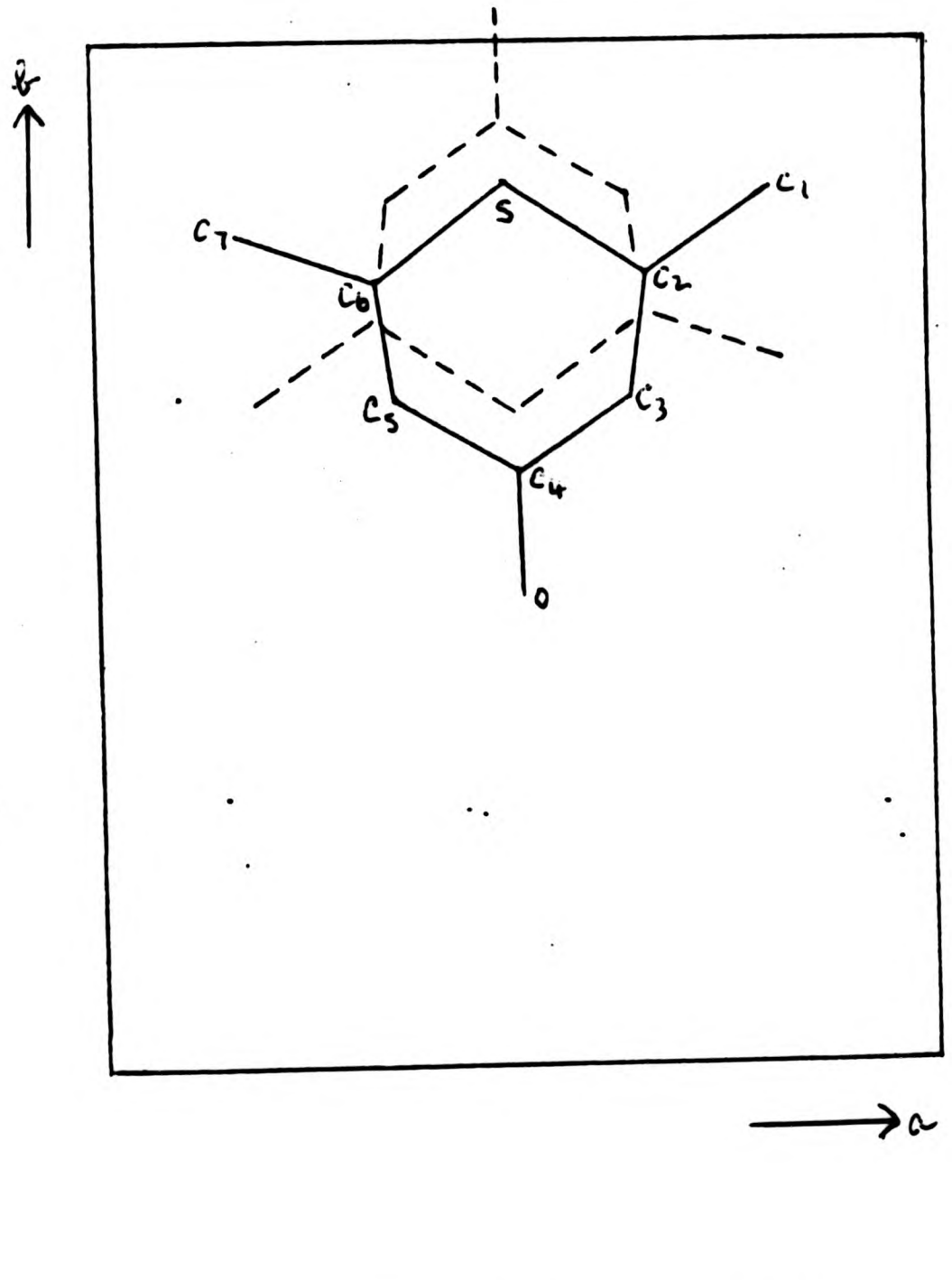


Figure 2. Overlap of molecules in stack separated by C/2 = 3.68Å .

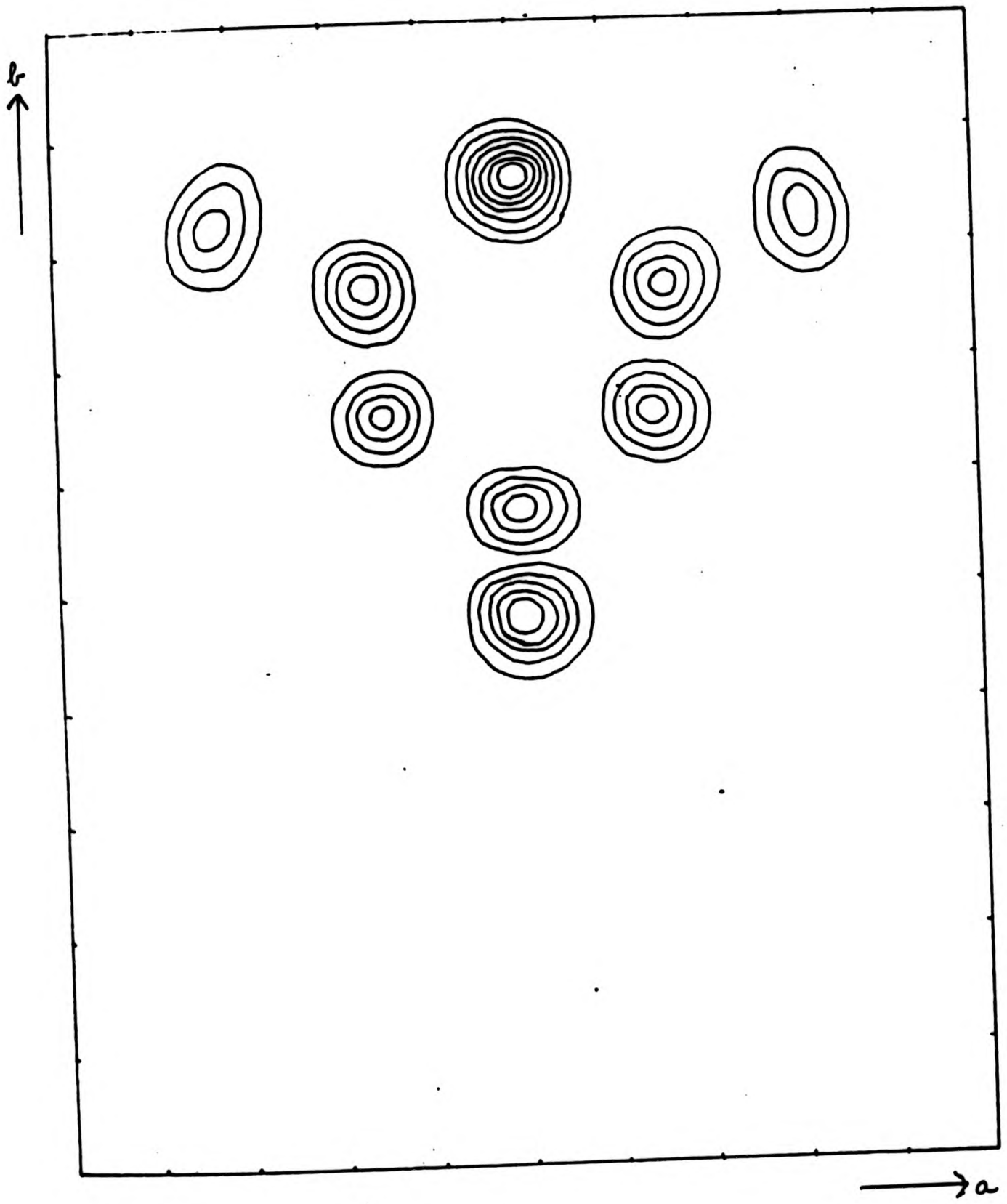


Figure 3. Electron density sections of atoms. Contours are at intervals of approx $1e.\text{Å}^3$ starting at $2e.\text{Å}^3$ for C and O and intervals of $2e.\text{Å}^3$ for S.

TABLE 5. ATOMIC PARAMETERS

Table 1. Final Atomic Parameters and e.s.d's.

	x/a	y/b	z/c	B_{eq} ($Å^2$)
S	0.5000	0.8592(1)	0.5000	1.55(2)
O	0.5081(12)	0.4735(4)	0.5195(14)	2.55(10)
C(1)	0.8064(13)	0.8508(9)	0.5581(23)	3.23(19)
C(2)	0.6650(9)	0.7724(7)	0.5351(17)	2.59(14)
C(3)	0.6439(11)	0.6569(6)	0.5405(17)	2.06(13)
C(4)	0.5054(16)	0.5796(6)	0.5154(23)	2.22(15)
C(5)	0.3587(12)	0.6516(9)	0.4891(19)	3.12(21)
C(6)	0.3434(8)	0.7666(8)	0.4768(15)	2.16(14)
C(7)	0.1771(10)	0.8072(8)	0.4388(19)	2.54(14)

$$B_{eq} = \frac{4}{3}\left(\beta_{11}/a^2 + \beta_{22}/b^2 + \beta_{33}/c^2\right).$$

Calculated Hydrogen Parameters

	x/a	y/b	z/c	$B_{iso}(Å^2)$
H(1) (-C(1))	0.7714	0.9414	0.5597	5.00
H(2) (-C(1))	0.8565	0.8355	0.4424	5.00
H(3) (-C(1))	0.8871	0.8296	0.6885	5.00
H(4) (-C(3))	0.7484	0.6059	0.5590	5.00
H(5) (-C(5))	0.2540	0.6014	0.4692	5.00
H(6) (-C(7))	0.1313	0.7909	0.5606	5.00
H(7) (-C(7))	0.1744	0.9047	0.4384	5.00
H(8) (-C(7))	0.1095	0.7735	0.3160	5.00

TABLE 5 (contd.)

Anisotropic Temperature Factors and their e.s.d's

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
S	0.00505 14	0.00304 8	0.00910 63	0.00069 85	0.00332 50	-0.00077 59
O	0.00990 73	0.00474 35	0.01182 193	-0.00145 249	0.00658 207	-0.00198 163
C(1)	0.01000 123	0.00692 74	0.01617 389	-0.00086 254	0.01098 389	0.00072 169
C(2)	0.00556 89	0.00481 51	0.00930 269	0.00392 243	0.00088 303	0.00118 121
C(3)	0.00692 83	0.00389 51	0.00763 332	0.00068 211	0.00197 292	-0.00124 121
C(4)	0.00625 71	0.00539 51	0.01133 267	0.00174 363	0.00297 248	-0.00013 228
C(5)	0.01105 142	0.00567 80	0.01379 457	0.00015 225	0.00432 451	0.00085 157
C(6)	0.00403 79	0.00369 54	0.00892 363	-0.00159 167	0.00397 274	-0.00134 86
C(7)	0.00615 91	0.00472 57	0.00850 276	0.00075 219	0.00091 276	0.00084 119

$$T = \exp\left[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{23}kl + 2\beta_{31}lh + 2\beta_{12}hk)\right].$$

TABLE 6. BOND LENGTHS

2:6-DIMETHYL GAMMA 1-THIAPYRONE A BANERJEE JUNE 1980

INTRAMOLECULAR DISTANCES

ATOM	N1	ATOM	N2	DISTANCE	E.S.D.	DIRECTION COSINES L	M	N
S	1	C	2	1.752	0.0120	-0.81424	0.56263	-0.14302
S	1	C	6	1.736	0.0115	0.79048	0.60500	0.09547
O	1	C	4	1.204	0.0101	0.01392	-0.99961	0.02439
C	1	C	2	1.532	0.0185	0.80686	0.58101	0.10681
C	1	H	1	1.080	0.0126	0.27565	-0.96096	0.02396
C	1	H	2	1.080	0.0175	-0.64219	0.18125	0.74481
C	1	H	3	1.080	0.0184	-0.44033	0.19869	-0.87557
C	2	C	3	1.326	0.0135	0.15050	0.98820	-0.02860
C	3	C	4	1.502	0.0206	0.80287	0.58413	0.11915
C	3	H	4	1.080	0.0116	-0.83481	0.51707	-0.18904
C	4	C	5	1.527	0.0222	0.83516	-0.53507	0.12731
C	5	C	6	1.314	0.0174	0.08945	-0.99409	0.06148
C	5	H	5	1.080	0.0132	0.84919	0.52273	0.07498
C	6	C	7	1.529	0.0175	0.93721	-0.30059	0.17685
C	7	H	6	1.080	0.0154	0.59319	0.30135	-0.74653
C	7	H	7	1.080	0.0121	0.02381	-0.99775	0.06270
C	7	H	8	1.080	0.0160	0.32019	0.39583	0.86070
H	1	H	2	1.764	0.0000	-0.56207	0.69943	0.44144
H	1	H	3	1.764	0.0000	-0.43846	0.71013	-0.55088
H	2	H	3	1.764	0.0000	0.12361	0.01068	-0.99227
H	6	H	7	1.764	0.0000	-0.34867	-0.79553	0.49555
H	6	H	8	1.764	0.0000	-0.16719	0.05785	0.98423
H	7	H	8	1.764	0.0000	0.18149	0.85340	0.48865

TABLE 7. INTER-BOND ANGLES

	N1	N2	N3	ANGLE	E.S.D.	N1	N3	DISTANCE
ANGLES INVOLVING ATOM S 1								
	C 2	S 1	C 6	108.48	0.590	C 2	C 6	2.830
ANGLES INVOLVING ATOM O 1	NONE							
ANGLES INVOLVING ATOM C 1								
	C 2	C 1	H 1	109.47	1.278	C 2	H 1	2.149
	C 2	C 1	H 2	109.47	1.278	C 2	H 2	2.149
	C 2	C 1	H 3	109.47	1.278	C 2	H 3	2.149
	H 1	C 1	H 2	109.47	1.375	H 1	H 2	1.764
	H 1	C 1	H 3	109.47	1.375	H 1	H 3	1.764
	H 2	C 1	H 3	109.47	1.375	H 2	H 3	1.764
ANGLES INVOLVING ATOM C 2								
	S 1	C 2	C 1	110.20	0.959	S 1	C 1	2.698
	S 1	C 2	C 3	119.99	1.003	S 1	C 3	2.619
	C 1	C 2	C 3	133.83	1.259	C 1	C 3	2.631
ANGLES INVOLVING ATOM C 3								
	C 2	C 3	C 4	134.00	1.274	C 2	C 4	2.604
	C 2	C 3	H 4	113.00	1.193	C 2	H 4	2.011
	C 4	C 3	H 4	113.00	1.150	C 4	H 4	2.166
ANGLES INVOLVING ATOM C 4								
	O 1	C 4	C 3	124.74	1.487	O 1	C 3	2.402
	O 1	C 4	C 5	123.34	1.492	O 1	C 5	2.409
	C 3	C 4	C 5	111.91	1.293	C 3	C 5	2.510
ANGLES INVOLVING ATOM C 5								
	C 4	C 5	C 6	127.91	1.343	C 4	C 6	2.354
	C 4	C 5	H 5	116.04	1.279	C 4	H 5	2.224
	C 6	C 5	H 5	116.05	1.296	C 6	H 5	2.034
ANGLES INVOLVING ATOM C 6								
	S 1	C 6	C 5	121.66	1.030	S 1	C 5	2.671
	S 1	C 6	C 7	125.16	0.992	S 1	C 7	2.908
	C 5	C 6	C 7	113.17	1.184	C 5	C 7	2.376
ANGLES INVOLVING ATOM C 7								
	C 6	C 7	H 6	109.47	1.163	C 6	H 6	2.146
	C 6	C 7	H 7	109.47	1.163	C 6	H 7	2.146
	C 6	C 7	H 8	109.47	1.163	C 6	H 8	2.146
	H 6	C 7	H 7	109.47	1.237	H 6	H 7	1.764
	H 6	C 7	H 8	109.47	1.237	H 6	H 8	1.764
	H 7	C 7	H 8	109.47	1.237	H 7	H 8	1.764
ANGLES INVOLVING ATOM H 1								
	C 1	H 1	H 2	35.27	0.842	C 1	H 2	1.000
	C 1	H 1	H 3	35.27	0.842	C 1	H 3	1.000
	H 2	H 1	H 3	60.00	0.000	H 2	H 3	1.764
ANGLES INVOLVING ATOM H 2								
	C 1	H 2	H 1	35.26	0.842	C 1	H 1	1.000
	C 1	H 2	H 3	35.26	0.842	C 1	H 3	1.000
	H 1	H 2	H 3	60.00	0.000	H 1	H 3	1.764
ANGLES INVOLVING ATOM H 3								
	C 1	H 3	H 1	35.26	0.842	C 1	H 1	1.000
	C 1	H 3	H 2	35.26	0.842	C 1	H 2	1.000
	H 1	H 3	H 2	60.00	0.000	H 1	H 2	1.764
ANGLES INVOLVING ATOM H 4	NONE							
ANGLES INVOLVING ATOM H 5	NONE							
ANGLES INVOLVING ATOM H 6								
	C 7	H 6	H 7	35.26	0.758	C 7	H 7	1.000
	C 7	H 6	H 8	35.26	0.758	C 7	H 8	1.000
	H 7	H 6	H 8	60.00	0.000	H 7	H 8	1.764
ANGLES INVOLVING ATOM H 7								
	C 7	H 7	H 6	35.26	0.758	C 7	H 6	1.000
	C 7	H 7	H 8	35.26	0.758	C 7	H 8	1.000
	H 6	H 7	H 8	60.00	0.000	H 6	H 8	1.764
ANGLES INVOLVING ATOM H 8								
	C 7	H 8	H 6	35.27	0.758	C 7	H 6	1.000
	C 7	H 8	H 7	35.27	0.758	C 7	H 7	1.000
	H 6	H 8	H 7	60.00	0.000	H 6	H 7	1.764

TABLE 8. EQUATION OF MEAN PLANE

2,6-DIMETHYL GAMMA 1-THIAPYRONE A.BANERJEE

PLANE 1 IS (0.1541)X + (-0.0342)Y + (-0.9875)Z -(-3.2997) = 0

CHI SQUARED = 191.9177

ATOMS IN PLANE	ATOM NO.	X	Y	Z	D	ESD(D)
	C 1	6.2655	9.6592	3.9786	0.005	0.0144
	C 2	5.0290	8.7649	3.8149	0.006	0.0197
	C 3	4.0294	7.4544	3.8528	-0.016	0.0195
	C 4	3.6232	6.5768	3.6738	0.004	0.0208
	C 5	2.3479	7.3939	3.4794	-0.020	0.0164
	C 6	2.2304	8.6999	3.3986	-0.011	0.0144
	C 7	0.7971	9.1597	3.1282	0.019	0.0178
	O 1	3.6400	5.3732	3.7032	0.019	0.0126
	S 1	3.6028	9.7503	3.5644	0.000	0.0001

SUM OF D(I) 0.0000 R.M.S. OF D(I) 0.014993

TABLE 9. INTERMOLECULAR DISTANCES

2:6-DIMETHYL GAMMA 1-THIÂPYRONE À BANERJEE JUNE 1980

INTERMOLECULAR DISTANCES, WITH THE COORDINATES USED IN THE COMPUTATION

ATOM	NI	X/A	Y/B	Z/C	ATOM	NI	X/A	Y/B	Z/C	DISTANCE	E.S.D.
C	1	0.00644	0.03003	0.55010	S	1	1.00000	1.14079	0.50000	3.000	0.0132
C	3	0.64394	0.65609	0.54046	S	1	0.50000	0.64079	0.00000	3.070	0.0134
C	4	0.50937	0.57956	0.51535	S	1	0.50000	0.64079	0.00000	3.043	0.0130
C	3	0.64394	0.65609	0.54046	S	1	0.50000	0.64079	1.00000	3.910	0.0147
C	4	0.50937	0.57956	0.51535	S	1	0.50000	0.64079	1.00000	3.646	0.0130
C	5	0.35071	0.65156	0.48000	S	1	0.00000	0.64079	1.00000	3.667	0.0164
C	5	0.35071	0.65156	0.48000	O	1	0.00006	0.92651	0.31947	3.531	0.0102
C	7	0.17710	0.00716	0.43001	O	1	0.00006	0.92651	0.31947	3.644	0.0130
C	3	0.64394	0.65609	0.54046	O	1	1.00006	0.92651	0.31947	3.644	0.0171
C	7	0.17710	0.00716	0.43001	O	1	0.00006	0.97349	0.81947	3.416	0.0193
C	1	0.00644	0.03003	0.55010	O	1	1.00006	0.97349	1.81947	3.703	0.0219
C	7	0.17710	0.00716	0.43001	C	1	-0.19356	0.03003	0.55010	3.701	0.0203
C	2	0.66499	0.77237	0.53914	C	1	0.00644	0.64917	1.05010	3.995	0.0241
C	3	0.64394	0.65609	0.54046	C	1	0.00644	0.64917	1.05010	3.728	0.0240
C	2	0.66499	0.77237	0.53914	C	2	0.66499	0.72763	0.03914	3.719	0.0219
C	3	0.64394	0.65609	0.54046	C	2	0.66499	0.72763	0.03914	3.699	0.0215
C	2	0.66499	0.77237	0.53914	C	2	0.66499	0.72763	1.03914	3.719	0.0219
C	3	0.64394	0.65609	0.54046	C	2	0.66499	0.72763	1.03914	3.697	0.0217
C	6	0.34341	0.76665	0.47679	C	3	0.35071	0.84064	0.98000	3.849	0.0215
C	7	0.17710	0.00716	0.43001	C	3	0.35071	0.84064	0.98000	3.994	0.0235
C	6	0.34341	0.76665	0.47679	C	3	0.35071	0.84064	-0.01192	3.730	0.0214
C	6	0.34341	0.76665	0.47679	C	6	0.34341	0.73335	0.97679	3.699	0.0202
C	7	0.17710	0.00716	0.43001	C	6	0.34341	0.73335	0.97679	3.959	0.0221
C	6	0.34341	0.76665	0.47679	C	6	0.34341	0.73335	-0.02325	3.699	0.0202
C	1	0.00644	0.03003	0.55010	C	7	1.17710	0.00716	0.43001	3.701	0.0203
C	7	0.17710	0.00716	0.43001	C	7	0.17710	0.69284	0.93001	3.902	0.0231
C	7	0.17710	0.00716	0.43001	C	7	0.17710	0.69284	-0.06119	3.902	0.0231

TABLE 10. STRUCTURE FACTORS

PAGE 0 2,6-DIMETHYL GAMMA 1-THIAPYRONE A.BANERJEE

MONOCLINIC
UNIT CELL DIMENSIONS
A = 9.036
B = 11.348
C = 7.360
BETA = 104.40
SPACE GROUP I(A)
NUMBER OF REFLEXIONS IN SPHERE = 571
NUMBER USED IN THE REFINEMENT = 457
R-FACTOR 0.0611
SCALE FACTOR USED = 10.363

PAGE 1 2,6-DIMETHYL GAMMA 1-THIAPYRONE A.BANERJEE

H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC
K= 0, L= 0			K= 1, L= 2			3	701	705	K= 3, L= 0			-7	0*	33
2	398	401	1	249	271	5	242	213	1	250	241			
4	322	294	3	145	147	7	106	122	3	391	378	K= 3, L= 5		
6	765	766	5	324	296	9	173	187	5	105	111	0	235	222
8	262	277	7	241	249	11	94	114	7	104	91	2	377	384
10	124	134	9	104	88	-1	234	244	9	179	158	-2	206	224
12	175	200	11	0*	93	-3	714	738	11	0*	74	-4	423	441
			-1	1032	1057	-5	198	215				-6	210	210
K= 0, L= 2			-3	119	87	-7	209	197	K= 3, L= 1					
0	1922	1905	-5	88	65	-9	143	152	0	751	765	K= 4, L= 0		
2	347	371	-7	500	476	-11	103	131	2	519	532	0	394	390
4	274	248	-9	93	92				4	436	436	2	273	257
6	636	672	-11	0*	42	K= 2, L= 2			6	355	349	4	262	253
8	197	204				0	302	286	8	102	109	6	178	154
10	77	87	K= 1, L= 3			2	376	355	10	142	182	8	196	190
-2	461	478	0	133	137	4	268	259	-2	336	354	10	158	161
-4	415	414	2	109	127	6	130	163	-4	605	603			
-6	668	644	4	0*	48	8	0*	70	-6	360	378	K= 4, L= 1		
-8	361	354	6	93	98	10	100	78	-8	119	112	1	0*	73
-10	179	172	8	0*	56	-2	322	341	-10	252	222	3	304	301
-12	181	169	-2	207	197	-4	319	345	-12	119	143	5	119	112
			-4	148	147	-6	93	82				7	0*	51
K= 0, L= 4			-6	0*	9	-8	0*	37	K= 3, L= 2			9	117	94
0	840	845	-8	0*	26	-10	97	81	1	189	184	11	80	70
2	269	278	-10	102	78	-12	0*	29	3	313	321	-1	98	100
4	250	223							5	0*	83	-3	269	290
6	469	465	K= 1, L= 4			K= 2, L= 3			7	119	115	-5	0*	72
-2	549	570	1	96	103	1	164	133	9	155	141	-7	106	110
-4	353	359	3	114	113	3	472	458	11	0*	46	-9	0*	59
-6	406	385	5	321	334	5	223	206	-1	113	141	-11	0*	60
-8	336	408	-1	735	737	7	0*	70	-3	358	333			
			-3	0*	58	-1	310	321	-5	181	169	K= 4, L= 2		
K= 1, L= 0			-5	0*	54	-3	416	404	-7	0*	53	0	303	318
1	740	722	-7	440	435	-5	249	246	-9	188	158	2	287	284
3	188	183	-9	98	90	-7	229	247	-11	104	99	4	179	169
5	207	192				-9	76	98				6	149	170
7	415	390	K= 1, L= 5						K= 3, L= 3			8	209	190
9	112	93	0	104	98	K= 2, L= 4			0	445	421	10	131	120
11	0*	57	2	143	149	0	124	111	2	475	489	-2	196	194
			4	0*	58	2	222	209	4	277	282	-4	294	302
K= 1, L= 1			-2	114	107	4	121	112	6	238	251	-6	143	136
0	90	84	-4	136	126	6	97	117	8	146	125	-8	201	189
2	104	109	-6	0*	40	-2	0*	79	-2	252	266	-10	182	184
4	0*	43				-4	241	216	-4	616	617	-12	0*	52
6	93	93	K= 2, L= 0			-6	0*	59	-6	306	300			
8	0*	35	0	542	543	-8	0*	30	-8	154	140	K= 4, L= 3		
10	0*	80	2	479	479				-10	203	217	1	0*	23
-2	282	256	4	402	409	K= 2, L= 5						3	192	213
-4	80	96	6	147	153	1	157	165	K= 3, L= 4			5	148	134
-6	0*	69	8	0*	70	3	259	263	1	192	171	7	0*	12
-8	0*	34	10	93	90	-1	254	255	3	229	221	-1	209	178
-10	87	90	12	0*	52	-3	157	193	5	0*	54	-3	135	120
-12	0*	47				-5	257	271	-1	0*	65	-5	0*	71
			K= 2, L= 1			-7	216	220	-3	212	234	-7	149	157
			1	0*	79				-5	153	184	-9	0*	25

H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC
K= 4, L= 4			5	0*	19	-8	119	101	-1	129	144	-6	191	161
1	213	205	3	0*	70	-10	100	115	-3	98	89	-8	119	135
2	232	262	-2	167	174				-5	176	182	-10	0*	29
4	122	132	-4	119	108	K= 6, L= 3			-7	0*	74			
6	179	151	-6	0*	72	1	194	177	-9	0*	46	K= 8, L= 3		
-2	214	195	-8	93	79	3	248	259				1	250	229
-4	265	274				5	98	116	K= 7, L= 3			3	397	409
-6	111	103	K= 5, L= 4			7	111	115	0	105	124	5	104	96
-8	173	172	1	227	226	-1	124	118	2	374	384	-1	125	129
			3	257	242	-3	282	266	4	257	273	-3	367	391
K= 4, L= 5			5	175.	167	-5	208	204	6	100	88	-5	327	296
1	0*	32	-1	392	371	-7	0*	34	-2	238	226	-7	85	96
3	124	114	-3	300	239	-9	146	132	-4	426	435			
-1	170	168	-5	220	195				-6	136	150	K= 8, L= 4		
-3	0*	43	-7	239	231	K= 6, L= 4			-8	119	140	0	195	200
-5	95	106				0	114	142				2	0*	81
			K= 5, L= 5			2	151	161	K= 7, L= 4			-2	196	171
K= 5, L= 0			0	99	87	4	0*	37	1	125	135	-4	0*	48
1	428	442	2	0*	65	-2	96	113	3	93	93	-6	179	156
3	477	452	-2	93	86	-4	185	185	-1	77	80			
5	172	161	-4	78	82	-6	0*	30	-3	119	121	K= 9, L= 0		
7	262	260	-6	89	77				-5	135	124	1	210	202
9	224	211				K= 6, L= 5						3	0*	52
11	89	62	K= 6, L= 0			1	173	197	K= 7, L= 5			5	134	119
			0	319	316	-1	0*	68	0	158	141	7	181	177
K= 5, L= 1			2	130	133	-3	215	197	-2	146	159	9	0*	62
0	114	95	4	115	96	-5	221	207						
2	203	228	6	131	135				K= 8, L= 0			K= 9, L= 1		
4	142	162	8	114	100	K= 7, L= 0			0	229	216	0	103	122
6	0*	36	10	93	83	1	211	211	2	195	183	2	77	94
8	104	101				3	0*	39	4	119	133	4	96	109
10	0*	60	K= 6, L= 1			5	156	177	6	157	148	6	67	75
-2	254	272	1	119	133	7	90	83	8	105	122	8	0*	9
-4	171	166	3	311	293	9	0*	25	10	0*	43	-2	0*	32
-6	0*	58	5	156	138	11	0*	61				-4	154	162
-8	97	104	7	114	92				K= 8, L= 1			-6	0*	70
-10	0*	60	9	146	142	K= 7, L= 1			1	207	217	-8	0*	23
			11	91	84	0	96	90	3	496	501	-10	0*	78
K= 5, L= 2			-1	155	141	2	411	402	5	191	167			
1	291	317	-3	293	295	4	418	396	7	114	104	K= 9, L= 2		
3	359	364	-5	147	163	6	103	100	9	196	189	1	174	172
5	173	172	-7	0*	62	8	179	167	-1	192	195	3	0*	19
7	211	212	-9	127	147	10	196	200	-3	499	495	5	155	140
9	179	181	-11	128	114	-2	340	334	-5	260	247	7	180	163
-1	456	460				-4	462	467	-7	102	106	9	0*	40
-3	426	407	K= 6, L= 2			-6	117	123	-9	188	180	-1	139	211
-5	153	167	0	234	250	-8	167	155				-3	92	68
-7	287	273	2	162	162	-10	217	220	K= 8, L= 2			-5	105	104
-9	198	208	4	0*	47				0	228	217	-7	145	164
-11	119	95	6	148	139	K= 7, L= 2			2	131	143	-9	74	74
			8	105	98	1	214	202	4	149	143			
K= 5, L= 3			10	0*	44	3	0*	70	6	143	135	K= 9, L= 3		
0	0*	78	-2	119	113	5	134	120	8	114	95	0	119	97
2	144	138	-4	154	167	7	0*	76	-2	163	192	2	143	132
4	131	114	-6	93	97	9	0*	19	-4	93	90	4	0*	47

H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC	H	FO	FC
K= 9, L= 3			5	104	88	-6	0*	38	K= 15, L= 1					
-2	0*	39	7	171	153	-8	0*	3	0	98	92			
-4	136	169							2	101	115			
-6	0*	48	K= 11, L= 1			K= 12, L= 3			-2	104	101			
			0	250	245	1	90	108	-4	141	109			
K= 9, L= 4			2	170	157	-1	119	130						
1	179	155	4	191	169	-3	98	86	K= 15, L= 2					
-1	198	186	6	179	179				-1	95	116			
-3	68	69	8	119	99	K= 13, L= 0			-3	0*	39			
			-2	173	184	1	146	153						
K= 10, L= 0			-4	179	150	3	158	157						
0	325	293	-6	192	190	5	115	132						
2	179	157	-8	143	125	7	119	97						
4	106	112												
6	175	174	K= 11, L= 2			K= 13, L= 1								
8	136	134	1	176	164	0	71	67						
			3	0*	42	2	0*	72						
K= 10, L= 1			5	88	88	4	104	95						
1	265	279	7	143	136	6	0*	53						
3	86	81	-1	228	200	-2	0*	68						
5	218	202	-3	0*	63	-4	0*	85						
7	176	175	-5	104	82	-6	0*	63						
9	0*	36	-7	146	141									
-1	322	304	-9	0*	53	K= 13, L= 2								
-3	110	99				1	143	137						
-5	186	190	K= 11, L= 3			3	133	146						
-7	179	197	0	238	221	5	119	120						
-9	0*	61	2	117	120	-1	138	152						
			-2	176	193	-3	153	150						
K= 10, L= 2			-4	143	121	-5	131	130						
0	279	277				-7	130	108						
2	143	126	K= 12, L= 0											
4	119	102	0	0*	12	K= 14, L= 0								
6	148	174	2	0*	60	0	191	165						
8	86	93	4	104	84	2	107	111						
-2	209	177	6	0*	26	4	89	101						
-4	85	99	8	0*	4									
-6	188	159				K= 14, L= 1								
-8	163	163	K= 12, L= 1			1	0*	5						
			1	100	124	3	0*	42						
K= 10, L= 3			3	74	80	-1	0*	24						
1	214	205	5	115	126	-3	0*	53						
3	70	77	7	119	94	-5	0*	26						
5	166	180	-1	118	138									
-1	250	275	-3	98	85	K= 14, L= 2								
-3	102	123	-5	149	128	0	121	154						
-5	135	146	-7	107	104	2	108	101						
						4	110	100						
K= 10, L= 4			K= 12, L= 2			-2	113	119						
0	238	231	0	0*	20	-4	91	96						
-2	215	184	2	96	73	-5	131	112						
			4	0*	65									
K= 11, L= 0			6	0*	10	K= 15, L= 0								
1	175	203	-2	0*	41	1	143	111						
3	0*	41	-4	107	90	3	0*	32						

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SUMMARY and ACKNOWLEDGEMENTS

This research project has resulted in the determination of four new crystal structures. The results have been communicated to those who suggested the research in the first place, and three of the structures have already been published; it is expected that the fourth will appear in print within a few months.

Two of the structures have produced some interesting results. First, the cadmium complex contains bidentate nitrate groups with variations in the N - O bond lengths. Secondly, the picric acid - naphthalene molecular complex presents an unusual disordered system with the picric acid molecules occuping alternative sites which are mirror-related to each other. Several kinds of disorder are already known to exist in picric acid complexes, and this work adds to existing knowledge.

I should like to acknowledge the assistance given to me in this project by technical and computer staff of the City of London Polytechnic, including particularly those who have helped with the operation of highly complicated scientific equipment. Also to my supervisor, Dr C J Brown, who has not only initiated and directed the work, but has also seen the results taken to the stage of being accepted for publication by the editors of Acta Crystallographica.

2744

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N-Nitrosodiphenylamine

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(*Received* 13 *January* 1982; *accepted* 27 *April* 1982)

Abstract. $C_{12}H_{10}N_2O$, $M_r = 198{\cdot}08$, monoclinic, $C2/c$, $a = 16{\cdot}283$ (20), $b = 8{\cdot}827$ (10), $c = 16{\cdot}508$ (20) Å, $\beta = 117{\cdot}53$ (15)°, $V = 2104{\cdot}03$ Å^3, $D_m = 1{\cdot}25$, $D_c = 1{\cdot}251$ Mg m^{-3}, $Z = 8$, λ(Cu $K\alpha$) = 1·5418 Å, $F(000) = 832$, $R = 0{\cdot}067$ for 807 observed reflexions. The structure comprises discrete molecules with no intermolecular interactions other than van der Waals forces.

Introduction. *N*-Nitrosodiphenylamine is a brown crystalline solid readily obtainable by reacting diphenylamine with nitrous acid, and is used industrially as an anti-oxidant to slow the curing of rubber. Our sample was supplied by Imperial Chemical Industries Limited (trade name 'Vulcatard A') and the commerical material was purified by recrystallization from absolute ethanol. Unit-cell dimensions were obtained first from layer-line measurements on rotation photographs about several axes, but as the crystals were equi-dimensional with very similar a, c and [101]-axis lengths, their identification was difficult. No reflexions were observable in the high-angle region of Weissenberg photographs so the best values of the lattice parameters were calculated from the 2θ measurements of specific indexed reflexions.

Intensities were obtained from visual estimations of multiple-film Weissenberg photographs of layers $h0l$–$h5l$ and $hk0$ which was also used for inter-layer scaling. The data were corrected for Lp effects but not for absorption, which was small. The structure was determined with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1978) and refined using the NRC suite of programs (Ahmed, Hall, Pippy & Huber, 1973). There were 1067 reflexions within the region examined, of which 260 were < 1·0 on the scale used.

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Table 1. *Final atomic coordinates and isotropic thermal parameters*

	x	y	z	B_{iso}(Å^2)
C(1)	0·2986 (3)	0·1977 (4)	0·3424 (2)	4·55 (18)
C(2)	0·2366 (3)	0·1377 (4)	0·3718 (3)	5·52 (22)
C(3)	0·1493 (3)	0·1037 (4)	0·3071 (4)	6·22 (25)
C(4)	0·1233 (3)	0·1121 (5)	0·2133 (3)	4·89 (20)
C(5)	0·1814 (3)	0·1754 (5)	0·1788 (4)	6·65 (25)
C(6)	0·2724 (3)	0·2114 (5)	0·2529 (3)	4·73 (20)
C(7)	0·4720 (3)	0·1769 (5)	0·4070 (3)	5·20 (22)
C(8)	0·5433 (3)	0·2725 (5)	0·4159 (3)	5·38 (22)
C(9)	0·6206 (3)	0·2028 (6)	0·4153 (3)	4·78 (20)
C(10)	0·6199 (3)	0·0573 (6)	0·3902 (4)	5·34 (22)
C(11)	0·5449 (4)	−0·0405 (6)	0·3742 (4)	6·77 (28)
C(12)	0·4665 (3)	0·0329 (6)	0·3774 (3)	7·05 (29)
N(1)	0·3920 (2)	0·2401 (3)	0·4108 (2)	4·51 (14)
N(2)	0·4110 (3)	0·3442 (4)	0·4765 (3)	5·96 (19)
O(1)	0·3441 (2)	0·3973 (3)	0·4793 (2)	8·18 (22)

The positions of the H atoms were calculated (C–H=1·08Å) and used in the structure-factor calculations with B_{iso} = 8·0 Å^2 but were not refined. After a number of cycles of least-squares calculations, using initially isotropic and finally anisotropic temperature factors, the shifts indicated were all less than 0·5σ. The final positional and isotropic thermal parameters are listed in Table 1.*

Discussion. The numbering of the atoms is shown in Fig. 1, and a list of bond lengths and bond angles is in Table 2. The molecules in the crystal are discrete with no strong intermolecular bonds; the nearest approach of the O atom is to C(2) at 3·293 (7), and to C(3) at 3·477 (6) Å. The mean C–C distance in the benzene rings is 1·397 Å, and the N=O bond of the nitroso group is 1·206 Å. Pauling (1944) predicted the N=O bond length at 1·18 Å, but the majority of values which have been determined are greater than this, probably because of ionization or hybridization; *e.g.* 1·26 Å in *N*-nitrosodimethylamine (Krebs & Mandt, 1975) and 1·234 Å in 5-nitrososalicyclic acid (Talberg, 1977). The best agreement with our value is 1·199 Å in *S*-nitroso-*N*-acetyl-DL-penicillamine (Carnahan, Lenhert & Ravichandran, 1978).

The molecule lies in three planes defined by:
(*a*) C(1), C(2), C(3), C(4), C(5), C(6) $0·3743X - 0·9271Y - 0·0199Z = -0·8719$ from which the perpendicular distances of the atoms are, respectively, −0·003 (4), +0·017 (4), −0·033 (4), +0·035 (5), −0·020 (5), and +0·005 (5) Å.
(*b*) C(1), C(7), N(1), N(2), O(1) $0·3044X + 0·7335Y - 0·6077Z = -1·0887$ and the out-of-plane distances are, respectively, +0·007 (4), +0·007 (4), −0·023 (3), +0·008 (4) and −0·010 (3) Å.
(*c*) C(7), C(8), C(9), C(10), C(11), C(12) $0·0400X + 0·2463Y - 0·9684Z = -5·1355$ and the atom deviations are, respectively, −0·066 (4), +0·058 (5), −0·033 (5), +0·013 (6), −0·016 (5) and +0·045 (5) Å. The angle between planes (*a*) and (*b*) is 123·64 (5), between (*b*) and (*c*) 38·62 (5), and between (*a*) and (*c*) 101·19 (5)°.

* Lists of structures factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36858 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

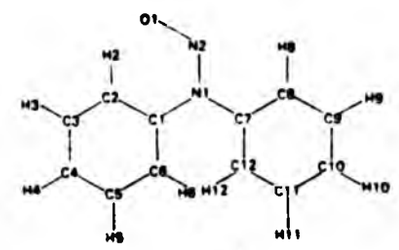

Fig. 1. Chemical formula showing numbering of atoms.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·411 (7)	C(7)–C(8)	1·387 (7)
C(2)–C(3)	1·360 (7)	C(8)–C(9)	1·405 (8)
C(3)–C(4)	1·406 (7)	C(9)–C(10)	1·349 (7)
C(4)–C(5)	1·425 (8)	C(10)–C(11)	1·418 (8)
C(5)–C(6)	1·456 (7)	C(11)–C(12)	1·454 (8)
C(6)–C(1)	1·341 (5)	C(12)–C(7)	1·350 (7)
C(1)–N(1)	1·462 (6)	N(1)–N(2)	1·344 (5)
C(7)–N(1)	1·445 (6)	N(2)–O(1)	1·206 (7)

C(1)–C(2)–C(3)	117·8 (4)	C(7)–C(8)–C(9)	116·2 (5)
C(2)–C(3)–C(4)	121·8 (4)	C(8)–C(9)–C(10)	122·6 (5)
C(3)–C(4)–C(5)	122·9 (5)	C(9)–C(10)–C(11)	121·4 (5)
C(4)–C(5)–C(6)	110·9 (4)	C(10)–C(11)–C(12)	114·7 (5)
C(5)–C(6)–C(1)	126·0 (4)	C(11)–C(12)–C(7)	121·3 (5)
C(6)–C(1)–C(2)	120·0 (4)	C(12)–C(7)–C(8)	121·6 (5)
N(1)–C(1)–C(2)	118·9 (4)	C(1)–N(1)–C(7)	120·2 (3)
N(1)–C(1)–C(6)	121·1 (4)	C(1)–N(1)–N(2)	124·6 (3)
N(1)–C(7)–C(8)	119·2 (4)	C(7)–N(1)–N(2)	115·0 (3)
N(1)–C(7)–C(12)	117·9 (4)	N(1)–N(2)–O(1)	114·9 (4)

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Tris(2,6-dimethyl-4*H*-pyran-4-one)dinitratocadmium(II), $[Cd(NO_3)_2(C_7H_8O_2)_3]$

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Abstract. $M_r = 608{\cdot}8$, monoclinic, $P2_1/n$, $a = 19{\cdot}372\ (5)$, $b = 11{\cdot}345\ (4)$, $c = 23{\cdot}023\ (5)$ Å, $\beta = 93{\cdot}77\ (5)°$, $V = 5048{\cdot}9$ Å^3, $Z = 8$, $D_m = 1{\cdot}60\ (1)$, $D_x = 1{\cdot}602$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}93$ mm^{-1}, $F(000) = 2464$, $T = 293$ K, $R = 0{\cdot}061$ for 7325 observed reflexions ($I > \sigma I$). The structure comprises two discrete non-equivalent molecules in each of which cadmium is seven-coordinated, to three oxygen atoms of the dimethyl-4-pyrone ligands, and bidentately to four oxygen atoms of two nitrato groups. The pyrone rings are planar and there are no strong intermolecular interactions.

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Introduction. The material was originally prepared by Drs E. Briggs and A. E. Hill of the Chemistry Department in the City of London Polytechnic (Hill, 1971) as part of a research project to investigate the protective properties of compounds of this class against the corrosion of steel. The molecular formula and the atomic numbering used are shown in Fig. 1.

Experimental. Equidimensional crystals (*ca* 0·2 mm) by reacting hot solutions of cadmium nitrate and 2,6-dimethyl-4-pyrone in 2,2-dimethoxypropane and cooling during addition of ethyl acetate, density by flotation in carbon tetrachloride/methylene bromide, lattice parameters initially from zero-level Weissenberg photographs and refined on the diffractometer, 7438 measured reflexions of which 113 considered unobserved; absences 0*k*0 for *k* odd and *h*0*l* for *h* + *l* odd, intensities on an Enraf–Nonius CAD-4 diffractometer at Queen Mary College with crystal enclosed in sealed glass tube, corrections made for Lp effects and for fading (maximum 8%; 3 standard reflections monitored) owing to deterioration of crystals, but not for absorption or extinction, index range *h* 0 to 21, *k* 0 to 12, *l* 0 to ±25, $2\theta_{max} = 120°$; structure solved from Patterson and successive Fourier syntheses; refinement on *F* by least squares using initially B_{iso} and $w^{1/2} = 1/F_o$ and finally B_{ij} until all shifts were $<0{\cdot}1\sigma$, approximate H positions determined from difference syntheses but included in structure factor calculations in idealized positions with $B_{iso} = 5{\cdot}0$ Å^2 without refining, $\Delta\rho$ in final difference synthesis generally within ±0·4 e Å^{-3} but down to −0·9 e Å^{-3} in regions around Cd atoms, $R_w = 0{\cdot}16$, scattering factors from *International Tables for X-ray Crystallography* (1962), computer programs of the *NRC* series (Ahmed, Hall, Pippy & Huber, 1970) used on our DEC-10 computer.

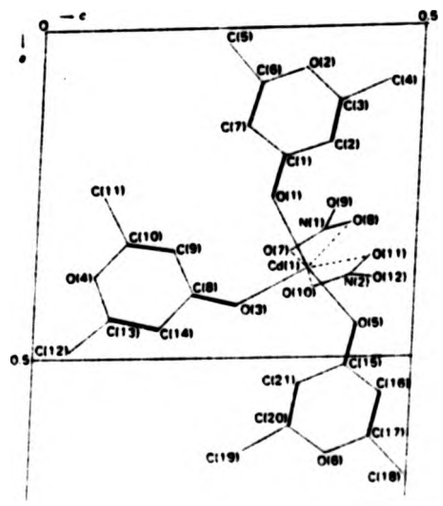

Fig. 1. Projection of part of the unit cell on (010) showing one of the molecules and the atomic numbering used. Atoms of the second molecule are numbered C(*n* + 21), N(*n* + 2), O(*n* + 12).

Table 1. *Final atomic parameters and e.s.d.'s*

$$B_{eq} = \tfrac{4}{3}\left(\frac{\beta_{11}}{a^{*2}} + \frac{\beta_{22}}{b^{*2}} + \frac{\beta_{33}}{c^{*2}}\right).$$

	x	*y*	*z*	B_{eq}(Å^2)
Cd(1)	0·38279 (2)	0·37833 (3)	0·35515 (1)	3·08 (1)
Cd(2)	0·36140 (2)	0·08814 (3)	0·84911 (1)	3·40 (1)
C(1)	0·2175 (2)	0·2964 (4)	0·3313 (2)	2·59 (8)*
C(2)	0·2013 (3)	0·2889 (5)	0·3905 (2)	3·43 (9)
C(3)	0·1385 (3)	0·2506 (5)	0·4035 (2)	3·62 (8)
C(4)	0·1133 (4)	0·2377 (8)	0·4640 (2)	5·81 (13)
C(5)	0·0439 (3)	0·1839 (7)	0·2664 (3)	4·66 (11)
C(6)	0·1051 (2)	0·2250 (4)	0·3052 (2)	2·87 (8)
C(7)	0·1664 (3)	0·2604 (4)	0·2873 (2)	3·48 (8)
C(8)	0·4089 (3)	0·3751 (5)	0·2133 (2)	4·14 (9)
C(9)	0·3410 (3)	0·4117 (5)	0·1923 (2)	4·07 (10)
C(10)	0·3235 (3)	0·4283 (4)	0·1351 (2)	3·75 (9)
C(11)	0·2532 (3)	0·4651 (6)	0·1095 (3)	5·18 (11)
C(12)	0·4779 (4)	0·3592 (8)	0·0598 (3)	7·94 (13)
C(13)	0·4356 (4)	0·3739 (5)	0·1118 (2)	4·23 (10)
C(14)	0·4556 (3)	0·3589 (5)	0·1682 (2)	3·53 (8)
C(15)	0·5312 (2)	0·4966 (5)	0·3960 (2)	3·22 (8)
C(16)	0·5783 (3)	0·5446 (5)	0·4402 (2)	4·10 (8)
C(17)	0·6399 (3)	0·5854 (5)	0·4255 (3)	4·35 (10)
C(18)	0·6967 (3)	0·6387 (6)	0·4649 (3)	4·59 (11)
C(19)	0·6440 (4)	0·5548 (7)	0·2668 (3)	6·16 (13)
C(20)	0·6144 (3)	0·5469 (5)	0·3260 (2)	4·15 (9)
C(21)	0·5531 (3)	0·5006 (4)	0·3378 (2)	3·40 (8)
C(22)	0·2224 (2)	0·1895 (4)	0·9056 (2)	2·99 (7)
C(23)	0·1811 (3)	0·2095 (5)	0·9537 (2)	4·09 (9)
C(24)	0·1183 (3)	0·2598 (6)	0·9476 (2)	5·07 (11)
C(25)	0·0709 (5)	0·2874 (8)	0·9946 (3)	7·08 (15)
C(26)	0·0907 (4)	0·3155 (7)	0·7899 (3)	5·35 (14)
C(27)	0·1282 (3)	0·2747 (4)	0·8456 (2)	3·46 (8)
C(28)	0·1908 (3)	0·2237 (4)	0·8507 (2)	4·12 (8)
C(29)	0·3126 (3)	0·0988 (6)	0·7078 (2)	4·18 (9)
C(30)	0·2608 (3)	0·0827 (6)	0·6650 (2)	4·26 (10)
C(31)	0·2732 (3)	0·0474 (5)	0·6091 (2)	4·16 (9)
C(32)	0·2242 (5)	0·0080 (8)	0·5596 (3)	7·37 (17)
C(33)	0·4541 (4)	0·1190 (8)	0·5948 (3)	7·86 (15)
C(34)	0·3900 (4)	0·1038 (5)	0·6299 (3)	4·75 (11)
C(35)	0·3783 (4)	0·1213 (6)	0·6862 (2)	5·27 (11)
C(36)	0·5181 (2)	−0·0011 (4)	0·8232 (2)	2·99 (8)
C(37)	0·5418 (2)	−0·0183 (5)	0·8830 (2)	3·97 (9)
C(38)	0·6055 (3)	−0·0573 (4)	0·8994 (2)	3·52 (8)
C(39)	0·6353 (3)	−0·0768 (7)	0·9610 (2)	4·96 (11)
C(40)	0·6875 (4)	−0·1086 (6)	0·7642 (3)	4·70 (11)
C(41)	0·6310 (2)	−0·0732 (4)	0·8018 (2)	2·91 (8)
C(42)	0·5680 (3)	−0·0335 (5)	0·7837 (2)	3·94 (9)
N(1)	0·3291 (3)	0·6013 (4)	0·3776 (2)	3·30 (8)
N(2)	0·4814 (3)	0·1562 (4)	0·4076 (2)	4·38 (9)
N(3)	0·3540 (3)	−0·1540 (5)	0·8726 (3)	5·13 (11)
N(4)	0·4250 (3)	0·2940 (4)	0·8937 (2)	4·06 (9)
O(1)	0·2760 (2)	0·3304 (3)	0·3144 (2)	3·72 (6)
O(2)	0·0895 (2)	0·2176 (3)	0·3616 (1)	3·46 (6)
O(3)	0·4283 (2)	0·3599 (4)	0·2664 (2)	5·03 (8)
O(4)	0·3695 (2)	0·4082 (4)	0·0944 (2)	4·37 (7)
O(5)	0·4739 (2)	0·4574 (4)	0·4106 (2)	4·80 (7)
O(6)	0·6592 (2)	0·5884 (3)	0·3698 (2)	3·68 (7)
O(7)	0·3572 (3)	0·5771 (4)	0·3329 (2)	5·87 (8)
O(8)	0·3224 (2)	0·5224 (4)	0·4123 (2)	4·99 (8)
O(9)	0·3096 (4)	0·7009 (5)	0·3869 (3)	7·06 (13)
O(10)	0·4183 (3)	0·1738 (4)	0·3582 (2)	6·04 (10)
O(11)	0·3743 (3)	0·2399 (4)	0·4321 (2)	6·24 (9)
O(12)	0·4077 (3)	0·0628 (5)	0·4332 (3)	6·95 (12)
O(13)	0·2808 (2)	0·1415 (4)	0·9111 (2)	4·18 (7)
O(14)	0·0926 (2)	0·2927 (3)	0·8941 (2)	3·90 (7)
O(15)	0·3029 (2)	0·1162 (4)	0·7609 (2)	4·91 (8)
O(16)	0·3376 (3)	0·0668 (4)	0·5922 (2)	5·46 (9)
O(17)	0·4598 (2)	0·0348 (4)	0·8057 (2)	4·86 (7)
O(18)	0·6498 (2)	−0·0870 (3)	0·8588 (2)	3·94 (6)
O(19)	0·3254 (3)	−0·1125 (5)	0·8292 (2)	5·39 (10)
O(20)	0·3588 (4)	−0·2600 (5)	0·8842 (3)	7·60 (13)
O(21)	0·3814 (2)	−0·0840 (4)	0·9084 (2)	4·43 (8)
O(22)	0·3900 (3)	0·2900 (5)	0·8478 (2)	6·15 (10)
O(23)	0·4354 (2)	0·2025 (4)	0·9205 (2)	4·70 (8)
O(24)	0·4459 (4)	0·3889 (5)	0·9108 (3)	7·71 (13)

Table 2. *Bond lengths* (Å) *and selected inter-bond angles* (°)

Angles subtended at Cd(1) and Cd(2) have been deposited (see deposition footnote).

Cd(1)–O(1)	2·281 (4)	Cd(2)–O(13)	2·267 (4)
Cd(1)–O(3)	2·287 (4)	Cd(2)–O(15)	2·282 (4)
Cd(1)–O(5)	2·292 (4)	Cd(2)–O(17)	2·287 (4)
Cd(1)–O(7)	2·357 (4)	Cd(2)–O(19)	2·416 (5)
Cd(1)–O(8)	2·444 (4)	Cd(2)–O(21)	2·400 (4)
Cd(1)–O(10)	2·420 (5)	Cd(2)–O(22)	2·357 (6)
Cd(1)–O(11)	2·382 (5)	Cd(2)–O(23)	2·476 (4)
O(1)–C(1)	1·280 (6)	O(13)–C(22)	1·254 (6)
C(1)–C(2)	1·421 (6)	C(22)–C(23)	1·427 (7)
C(1)–C(7)	1·429 (7)	C(22)–C(28)	1·420 (6)
C(2)–C(3)	1·343 (7)	C(23)–C(24)	1·343 (9)
C(3)–C(4)	1·514 (8)	C(24)–C(25)	1·497 (10)
C(3)–O(2)	1·362 (6)	C(24)–O(14)	1·351 (7)
O(2)–C(6)	1·355 (5)	O(14)–C(27)	1·365 (6)
C(5)–C(6)	1·511 (8)	C(26)–C(27)	1·504 (8)
C(6)–C(7)	1·343 (7)	C(27)–C(28)	1·343 (7)
O(3)–C(8)	1·268 (6)	O(15)–C(29)	1·264 (6)
C(8)–C(9)	1·433 (9)	C(29)–C(30)	1·421 (8)
C(8)–C(14)	1·434 (7)	C(29)–C(35)	1·420 (9)
C(9)–C(10)	1·350 (7)	C(30)–C(31)	1·336 (7)
C(10)–C(11)	1·508 (8)	C(31)–C(32)	1·502 (10)
C(10)–O(4)	1·354 (7)	C(31)–O(16)	1·369 (8)
O(4)–C(13)	1·371 (8)	O(16)–C(34)	1·358 (9)
C(12)–C(13)	1·504 (10)	C(33)–C(34)	1·510 (11)
C(13)–C(14)	1·341 (7)	C(34)–C(35)	1·346 (9)
O(5)–C(15)	1·261 (6)	O(17)–C(36)	1·247 (6)
C(15)–C(16)	1·430 (7)	C(36)–C(37)	1·435 (7)
C(15)–C(21)	1·432 (7)	C(36)–C(42)	1·419 (7)
C(16)–C(17)	1·342 (8)	C(37)–C(38)	1·343 (7)
C(17)–C(18)	1·508 (9)	C(38)–C(39)	1·513 (7)
C(17)–O(6)	1·360 (8)	C(38)–O(18)	1·352 (6)
O(6)–C(20)	1·370 (7)	O(18)–C(41)	1·347 (6)
C(19)–C(20)	1·516 (9)	C(40)–C(41)	1·494 (8)
C(20)–C(21)	1·343 (8)	C(41)–C(42)	1·342 (7)
N(1)–O(7)	1·226 (7)	N(3)–O(19)	1·206 (8)
N(1)–O(8)	1·213 (6)	N(3)–O(20)	1·234 (8)
N(1)–O(9)	1·215 (8)	N(3)–O(21)	1·239 (7)
N(2)–O(10)	1·220 (7)	N(4)–O(22)	1·217 (7)
N(2)–O(11)	1·239 (7)	N(4)–O(23)	1·218 (6)
N(2)–O(12)	1·215 (8)	N(4)–O(24)	1·208 (8)

Cd(1)–O(1)–C(1)	138·1 (3)	Cd(2)–O(13)–C(22)	134·9 (3)
Cd(1)–O(3)–C(8)	137·9 (4)	Cd(2)–O(15)–C(29)	137·9 (4)
Cd(1)–O(5)–C(15)	130·4 (3)	Cd(2)–O(17)–C(36)	135·2 (3)
C(2)–C(1)–C(7)	118·4 (4)	C(23)–C(22)–C(28)	114·6 (4)
C(2)–C(1)–O(1)	124·4 (4)	C(23)–C(22)–O(13)	122·5 (5)
C(7)–C(1)–O(1)	117·1 (4)	C(28)–C(22)–O(13)	122·8 (4)
C(1)–C(2)–C(3)	119·6 (5)	C(22)–C(23)–C(24)	122·4 (5)
C(2)–C(3)–C(4)	126·1 (5)	C(23)–C(24)–C(25)	127·4 (6)
C(2)–C(3)–O(2)	122·0 (5)	C(23)–C(24)–O(14)	119·3 (5)
C(4)–C(3)–O(2)	111·9 (5)	C(25)–C(24)–O(14)	113·3 (5)
C(3)–O(2)–C(6)	118·2 (4)	C(24)–O(14)–C(27)	122·0 (4)
C(5)–C(6)–C(7)	125·9 (5)	C(26)–C(27)–C(28)	126·2 (5)
C(5)–C(6)–O(2)	109·3 (4)	C(26)–C(27)–O(14)	114·2 (5)
C(7)–C(6)–O(2)	124·7 (4)	C(28)–C(27)–O(14)	119·6 (4)
C(1)–C(7)–C(6)	116·9 (4)	C(22)–C(28)–C(27)	122·0 (4)
C(9)–C(8)–C(14)	113·7 (5)	C(30)–C(29)–C(35)	114·7 (5)
C(9)–C(8)–O(3)	124·9 (5)	C(30)–C(29)–O(15)	125·0 (5)
C(14)–C(8)–O(3)	121·3 (5)	C(35)–C(29)–O(15)	120·2 (5)
C(8)–C(9)–C(10)	122·3 (5)	C(29)–C(30)–C(31)	122·9 (6)
C(9)–C(10)–C(11)	125·6 (5)	C(30)–C(31)–C(32)	128·6 (6)
C(9)–C(10)–O(4)	121·1 (5)	C(30)–C(31)–O(16)	119·0 (5)
C(11)–C(10)–O(4)	113·3 (5)	C(32)–C(31)–O(16)	112·4 (5)
C(10)–O(4)–C(13)	119·4 (4)	C(31)–O(16)–C(34)	122·1 (5)
C(12)–C(13)–C(14)	128·2 (6)	C(33)–C(34)–C(35)	131·7 (7)
C(12)–C(13)–O(4)	110·3 (5)	C(33)–C(34)–O(16)	108·5 (6)
C(14)–C(13)–O(4)	121·6 (5)	C(35)–C(34)–O(16)	119·8 (6)
C(8)–C(14)–C(13)	121·9 (5)	C(29)–C(35)–C(34)	121·4 (6)
C(16)–C(15)–C(21)	116·1 (4)	C(37)–C(36)–C(42)	113·1 (4)
C(16)–C(15)–O(5)	118·4 (5)	C(37)–C(36)–O(17)	125·5 (5)
C(21)–C(15)–O(5)	125·5 (5)	C(42)–C(36)–O(17)	121·4 (4)
C(15)–C(16)–C(17)	119·2 (5)	C(36)–C(37)–C(38)	122·9 (5)
C(16)–C(17)–C(18)	127·8 (6)	C(37)–C(38)–C(39)	126·7 (5)
C(16)–C(17)–O(6)	123·5 (5)	C(37)–C(38)–O(18)	120·2 (5)
C(18)–C(17)–O(6)	108·8 (5)	C(39)–C(38)–O(18)	113·0 (4)
C(17)–O(6)–C(20)	118·9 (4)	C(38)–O(18)–C(41)	120·0 (4)
C(19)–C(20)–C(21)	126·6 (5)	C(40)–C(41)–C(42)	126·6 (5)
C(19)–C(20)–O(6)	112·5 (5)	C(40)–C(41)–O(18)	111·8 (4)
C(21)–C(20)–O(6)	120·8 (5)	C(42)–C(41)–O(18)	121·6 (4)
C(15)–C(21)–C(20)	121·4 (5)	C(36)–C(42)–C(41)	122·1 (5)
O(7)–N(1)–O(8)	117·4 (5)	O(19)–N(3)–O(20)	125·7 (6)
O(7)–N(1)–O(9)	121·1 (6)	O(19)–N(3)–O(21)	117·1 (6)

Table 2 (*cont.*)

O(8)–N(1)–O(9)	121·5 (5)	O(20)–N(3)–O(21)	117·2 (6)
O(10)–N(2)–O(11)	116·6 (5)	O(22)–N(4)–O(23)	118·2 (5)
O(10)–N(2)–O(12)	124·7 (6)	O(22)–N(4)–O(24)	118·2 (6)
O(11)–N(2)–O(12)	118·7 (6)	O(23)–N(4)–O(24)	123·6 (6)

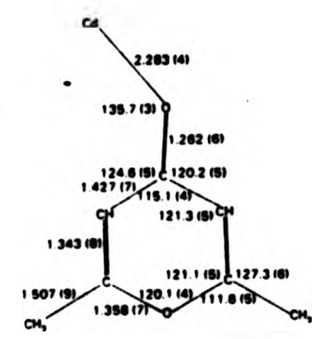

Fig. 2. 2,6-Dimethyl-4-pyrone ring showing averaged bond lengths (Å) and angles (°).

Discussion. The final atomic coordinates and equivalent isotropic temperature parameters are given in Table 1.* Bond lengths and inter-bond angles are given in Table 2.

Although prepared in the same way as the copper and zinc compounds, this cadmium compound is different in composition, probably because of the greater size of the Cd atom. Three 2,6-dimethyl-4-pyrone ligands coordinate to the metal instead of two, and the two nitrato groups are bidentate in coordination whereas in the Cu and Zn compounds they are unidentate (Brown & Lewis, 1984*a*,*b*). These differences confer seven-coordination on the Cd whereas the Cu and Zn are both four-coordinated. Seven O atoms lie at the corners of a slightly distorted pentagonal bipyramid around each of the two Cd atoms (Fig. 1), in each of which four nitrato O atoms and one 4-pyrone O atom form the equatorial plane and the two other 4-pyrone O atoms lie at the apices. The mean Cd–O distance for the 2,6-dimethyl-4-pyrone ligands is 2·283 (4) Å, while the distance for the nitrato oxygens is 2·406 (5) Å.

The six 4-pyrone rings are each closely planar, the r.m.s. deviation being 0·009 Å, with the maximum 0·018 (6) Å. The substituent oxygen and two methyl carbon atoms also lie in the plane of the rings. Mean dimensions are shown in Fig. 2. The four nitrate groups are also planar, the N atoms lying 0·006 (5), 0·011 (6),

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, angles subtended at Cd(1) and Cd(2) and least-squares-planes' information have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39356 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0·002 (6) and 0·012 (5) Å out of the planes of the O atoms.

A similar structural arrangement to this has been observed in dinitratotris(pyridine)Cd^{II} (Cameron, Taylor & Nuttall, 1972) and also in aquadinitratobis(quinoline)Cd^{II} (Cameron, Taylor & Nuttall, 1973). The only structural results for 2,6-dimethyl-4-pyrone compounds are for the hydrobromide monohydrate (Hope, 1965) determined with limited X-ray data, dinitratobis(2,6-dimethyl-4-pyrone)zinc (Brown & Lewis, 1984*a*) and dinitratobis(2,6-dimethyl-4-pyrone)-copper (Brown & Lewis, 1984*b*).

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Picric Acid–Naphthalene 1/1 π Complex, $C_6H_3N_3O_7.C_{10}H_8$. A Disordered Structure

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Abstract. $M_r = 357{\cdot}28$, monoclinic, $P2_1/a$, $a = 16{\cdot}248\ (5)$, $b = 6{\cdot}871\ (2)$, $c = 14{\cdot}306\ (5)$ Å, $\beta = 96{\cdot}62\ (5)°$, $V = 1586{\cdot}47$ Å^3, $Z = 4$, $D_m = 1{\cdot}47\ (1)$, $D_x = 1{\cdot}496$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 1{\cdot}16$ mm^{-1}, $F(000) = 736$, $T = 293$ K, $R = 0{\cdot}066$ for 918 observed reflexions ($I > 3\sigma_I$). Molecules lie in (010) parallel to each other, stacking alternately picric acid and naphthalene with hydrogen bonds linking picric acid molecules across alternative symmetry centres and also along **a**. The two arrangements are present in ratio ≈4:1, and it was not feasible to separate the two sets of atomic coordinates.

Introduction. Unit-cell dimensions have been reported (Herbstein & Kaftory, 1975) as have the crystal structure determinations of a number of homologues (Carstensen-Oeser, Göttlicher & Habermehl, 1968; Herbstein & Kaftory, 1976). Physical properties have been reported by Mindovich (1956) and by Westwood (1978). In none of these reports was there any indication of structural disorder, and this work was undertaken at the instigation of Dr Westwood in order to explain some peculiarities observed.

Experimental. Sample prepared by Dr C. V. Westwood of the Chemistry Department, City of London Polytechnic; recrystallized (*ca* $1{\cdot}5 \times 0{\cdot}2 \times 0{\cdot}2$ mm) from ethanol; m.p. 401 K. D_m by flotation in NaI solution. Lattice parameters from rotation and Weissenberg photographs, refined by least-squares fit to 16 selected reflexions measured on a Stoe Stadi-2 diffractometer; intensities from $h0l \rightarrow h6l$ levels on the diffractometer using ω scans, interlayer scaling and $0k0$ intensities from **c** Weissenberg. 1559 measured reflexions, 918 observed with $I > 3\sigma_I$; index range h 0 to 16, k 0 to 6, l −15 to +17; $2\theta_{max} = 110°$. Seven standard reflexions, no variation. Corrections for Lp and an empirical correction for extinction to eight reflexions during refinement. No absorption correction. Structure solved by trial-and-error as direct methods failed. Patterson map indicated layer structure with $b/4$ separation. Refinement by least squares on F using initially B_{iso} and finally B_{ij}. H atoms included at calculated positions with $B_{iso} = 8{\cdot}0$ Å^2, but not refined. Difference Fourier during refinement showed two alternative sites for the O of the picric acid OH; site occupation refinement converged to 0·80 for O(1) position and 0·20 for O(8) and these values used in further SFLS refinement until final shifts all $<0{\cdot}3\sigma$. Final $R = 0{\cdot}066$, $R_w = 0{\cdot}136$, $\sqrt{w} = 1/F_o$. Max. electron density in final difference Fourier map $\pm 0{\cdot}2$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1962). *NRC* programs (Ahmed, Hall, Pippy & Huber, 1970) implemented on our DEC-10 computer.

Discussion. The final atomic coordinates and equivalent isotropic temperature parameters are listed in Table 1.* Bond lengths and interbond angles are in Table 2. The arrangements of the molecules in the unit cell, together with the atom numbering and hydrogen bonds are shown in Fig. 1.

Molecules of both picric acid and naphthalene lie approximately parallel to (010) in layers with $y = \frac{1}{4}, \frac{1}{2}, \frac{3}{4}$ and $\frac{1}{2}$ and π-bonding interactions across the 3·4 Å ($b/2$) spacing; the overlap diagram is shown in Fig. 2 as a normal projection along **b**. C(14) of the naphthalene ring lies almost exactly above the mid-point of the picric acid ring.

* Lists of structure factors, anisotropic thermal parameters and calculated H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39515 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final atomic parameters and e.s.d.'s*

$B_{eq} = \frac{4}{3}[(\beta_{11}/a^{*2}) + (\beta_{22}/b^{*2}) + (\beta_{33}/c^{*2})]$.

	x	y	z	B_{eq}(Å^2)
C(1)	0·9413 (4)	0·1182 (8)	0·1542 (4)	3·30 (14)
C(2)	0·8541 (5)	0·1027 (10)	0·1526 (4)	4·63 (15)
C(3)	0·8145 (3)	0·1105 (7)	0·2347 (3)	3·30 (10)
C(4)	0·8623 (4)	0·1189 (9)	0·3190 (4)	3·50 (14)
C(5)	0·9503 (4)	0·1096 (11)	0·3272 (5)	5·07 (16)
C(6)	0·9872 (3)	0·1123 (9)	0·2456 (5)	4·47 (13)
C(7)	0·6395 (6)	0·1211 (9)	0·5927 (6)	5·56 (17)
C(8)	0·5552 (5)	0·1325 (11)	0·6061 (5)	5·45 (16)
C(9)	0·5347 (4)	0·1231 (8)	0·7002 (4)	2·63 (12)
C(10)	0·4508 (5)	0·1413 (11)	0·7093 (5)	4·89 (17)
C(11)	0·4328 (5)	0·1152 (10)	0·7958 (6)	6·15 (17)
C(12)	0·4875 (7)	0·1229 (11)	0·8743 (6)	7·45 (20)
C(13)	0·5671 (6)	0·1294 (12)	0·8647 (6)	5·73 (20)
C(14)	0·5947 (4)	0·1259 (9)	0·7742 (5)	4·40 (15)
C(15)	0·6795 (5)	0·1116 (10)	0·7592 (6)	6·42 (17)
C(16)	0·6998 (5)	0·1180 (11)	0·6672 (5)	5·61 (17)
N(1)	0·7991 (5)	0·0996 (10)	0·0619 (5)	9·04 (19)
N(2)	0·8264 (4)	0·1093 (8)	0·4111 (4)	5·58 (13)
N(3)	1·0772 (4)	0·1259 (9)	0·2546 (6)	7·36 (17)
O(1)†	0·9799 (5)	0·1131 (10)	0·0775 (5)	7·35 (17)
O(2)	0·8301 (6)	0·1472 (14)	−0·0063 (6)	11·93 (22)
O(3)	0·7262 (5)	0·0744 (13)	0·0678 (5)	12·57 (21)
O(4)	0·7473 (4)	0·0854 (8)	0·3935 (5)	7·95 (15)
O(5)	0·8685 (5)	0·1293 (9)	0·4839 (4)	7·27 (15)
O(6)	1·1150 (4)	0·1389 (10)	0·3398 (6)	9·08 (19)
O(7)	1·1098 (4)	0·1457 (9)	0·1851 (6)	8·22 (18)
O(8)‡	1·0013 (15)	0·1302 (41)	0·4125 (17)	5·99 (51)

† 80% site occupation factor.
‡ 20% site occupation factor.

Table 2. *Bond lengths* (Å) *and interbond angles* (°)

C(1)–C(2)	1·417 (11)	C(9)–C(10)	1·389 (9)
C(1)–C(6)	1·430 (10)	C(9)–C(14)	1·354 (9)
C(1)–O(1)†	1·326 (10)	C(10)–C(11)†	1·316 (11)
C(2)–C(3)	1·404 (8)	C(11)–C(12)	1·351 (12)
C(2)–N(1)	1·489 (10)	C(12)–C(13)	1·317 (15)
C(3)–C(4)	1·359 (8)	C(13)–C(14)	1·419 (10)
C(4)–C(5)	1·423 (10)	C(14)–C(15)	1·422 (10)
C(4)–N(2)	1·503 (9)	C(15)–C(16)	1·395 (11)
C(5)–C(6)	1·373 (10)	N(1)–O(2)	1·195 (11)
C(5)–O(8)‡	1·400 (26)	N(1)–O(3)	1·209 (12)
C(6)–N(3)	1·455 (9)	N(2)–O(4)	1·291 (9)
C(7)–C(8)	1·407 (13)	N(2)–O(5)	1·186 (9)
C(7)–C(16)	1·363 (11)	N(3)–O(6)	1·304 (11)
C(8)–C(9)	1·426 (9)	N(3)–O(7)	1·187 (12)

C(2)–C(1)–C(6)	115·3 (6)	C(10)–C(9)–C(14)	123·5 (6)
C(2)–C(1)–O(1)†	123·5 (6)	C(9)–C(10)–C(11)	113·7 (7)
C(6)–C(1)–O(1)†	120·6 (6)	C(10)–C(11)–C(12)	125·5 (8)
C(1)–C(2)–C(3)	122·5 (6)	C(11)–C(12)–C(13)	118·4 (8)
C(1)–C(2)–N(1)	120·9 (6)	C(12)–C(13)–C(14)	120·8 (8)
C(3)–C(2)–N(1)	116·2 (6)	C(9)–C(14)–C(13)	116·0 (6)
C(2)–C(3)–C(4)	118·3 (5)	C(9)–C(14)–C(15)	120·4 (6)
C(3)–C(4)–C(5)	122·5 (6)	C(13)–C(14)–C(15)	123·5 (7)
C(3)–C(4)–N(2)	122·4 (5)	C(14)–C(15)–C(16)	118·6 (7)
C(5)–C(4)–N(2)	114·5 (6)	C(7)–C(16)–C(15)	120·8 (7)
C(4)–C(5)–C(6)	117·6 (6)	C(2)–N(1)–O(2)	116·1 (7)
C(4)–C(5)–O(8)‡	123·7 (1·2)	C(2)–N(1)–O(3)	115·9 (7)
C(6)–C(5)–O(8)‡	117·9 (1·2)	O(2)–N(1)–O(3)	127·2 (9)
C(1)–C(6)–C(5)	123·0 (6)	C(4)–N(2)–O(4)	108·2 (5)
C(1)–C(6)–N(3)	119·5 (6)	C(4)–N(2)–O(5)	121·5 (6)
C(5)–C(6)–N(3)	117·2 (6)	O(4)–N(2)–O(5)	130·2 (6)
C(8)–C(7)–C(16)	121·2 (7)	C(6)–N(3)–O(6)	116·6 (7)
C(7)–C(8)–C(9)	117·7 (7)	C(6)–N(3)–O(7)	118·3 (7)
C(8)–C(9)–C(10)	115·0 (6)	O(6)–N(3)–O(7)	124·6 (8)
C(8)–C(9)–C(14)	120·7 (6)		

† 80% site occupation.
‡ 20% site occupation.

Hydrogen bonds link pairs of picric acid molecules related by centres of symmetry either through O(1)–O(1^{i}) 2·844 (10) Å across (0,0,0) with 80% site occupation, or through O(8)–O(8^{iii}) 3·082 (36) Å across (0,0,½) with 20% site occupation. These alternative sites can be occupied with only small disturbance to the coordinates of the other picric acid atoms. The OH···HO hydrogen bonds release a spare proton from half the picric acid molecules and this appears to be transferred to either O(4) or O(6) enabling H bonding to take place: O(4)···H···O(6^{iii}) 2·901 (9) Å (Fig. 1). That H bonding does occur between the nitro groups is supported by the distances N(2)–O(4) 1·291 (9) and N(3)–O(6) 1·304 (11) Å which are both appreciably longer than the other four N–O bonds [mean 1·194 (11) Å]. The stabilizing influence of the postulated H bond is also reflected in the values of B_{eq} which are ≈7·5 Å^2 for O(4) and O(5) and ≈8·5 Å^2 for O(6) and O(7) whereas for O(2) and O(3) which are

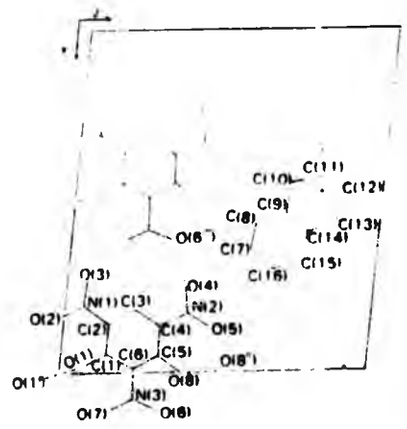

Fig. 1. Arrangement of molecules in part of the unit cell ($y = 0$ to ½) showing atomic numbering and hydrogen bonds (dashed lines). Symmetry code: (i) $-x, -y, -z$; (ii) $-x, -y, 1-z$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, z$.

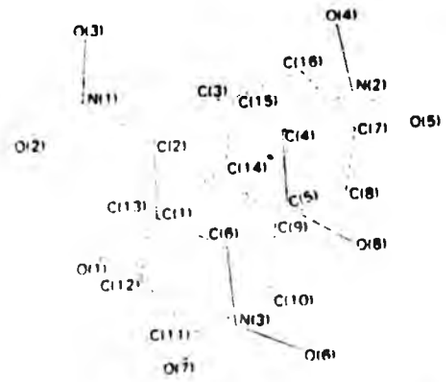

Fig. 2. Overlap of naphthalene and picric acid molecules in sections perpendicular to b.