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CONFORMATION OF BIOLOGICAL MOLECULES**

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COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH  
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## 11. DISCREPANCY INDICES FOR USE IN CRYSTAL STRUCTURE ANALYSIS

S. PARTHASARATHY, Centre for Advanced Study in Physics, University of Madras, Guindy Campus, Madras 600 025.

The uses of discrepancy indices (i. e. R-indices) in crystal structure analysis as an index for the correctness of trial structures in the structure completion stage and as an index indicating the extent of refinement in the refinement stage are well known. A number of R-indices have been suggested by various workers for this purpose. A systematic comparative study of these in their normalised form will be made to assess their relative efficiency for the following three crystallographic situations, namely, (i) structure completion stage, (ii) refinement of an incomplete model and (iii) refinement of a complete model. A comparative study of the normalised and unnormalised forms of Booth type index carried out for a variety of situations to assess the advantage of the normalisation procedure during the structure completion stage will be described. The evaluation of the R-indices for crystals containing heavy atoms and for crystals containing atoms of widely different scattering powers will be dealt with. The necessity of taking into account the space group symmetry of the crystal in such evaluations will be pointed out. Expressions needed in the presence of random errors in the observed intensity will also be deduced.

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## 12. DEFECTS IN METALLIC STRUCTURES

P. RAMA RAO, Department of Metallurgical Engineering, Banaras Hindu University, Varanasi 221 005.

The concept of defects has its origin in the attempt to explain the observed weakness compared to the theoretical strength of crystalline solids and to this day the importance of this subject stems essentially from its significance in the explanation of the mechanical-behaviour of metallic materials. Classification of the various types of defects is readily possible in terms of their zero, one, two and three dimensions and these are accordingly labelled point, line, planar and volume defects respectively. Vacancies, interstitial atoms, impurity atoms and combinations of these represent point defects. Dislocations, categorised as line defects, constitute the central element on the study of defect

structures in metals. The basic orderliness of a crystalline solid is also manifested in the interesting fact that a regular structure can go wrong in a limited number of precisely definable ways. The example of stacking faults, an important class of planar defects involving crystal surfaces bounded by imperfect dislocations, illustrates this pleasing principle. Other examples of planar defects are antiphase boundaries in ordered alloys and grain boundaries in polycrystalline aggregates. Clusters of vacancies, interstitials and solute atoms often occur in crystals and may condense to form volume defects such as voids and precipitates with a variety of morphological features once again controlled by the structure of the solid material. This talk will describe the features of the various defects and aim to bring out the utility of modern metallographic techniques in not only observing but for working with lattice defects. Interplay between the defects and their importance in not only understanding but predicting the strength and mechanical behaviour leading to design of alloys will be pointed out in conclusion.

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### 13. CRYSTAL GROWTH: REACTED FLUX AND GEL METHOD

A. R. PATEL, Department of Physics, Sardar Patel University, Vallabh Vidyanagar (Gujarat)

There exists a wide variety of methods each having its own importance, for growing single crystals. The present talk concerns with the techniques of crystal growth which has been developed in our laboratory.

A potential method of growing good quality single crystals, which possess high melting point, is the 'chemical reacted flux method' developed in this laboratory. It consists essentially in allowing two substances to react together in their molten phase with the result that the insoluble product crystallizes out. The method, in effect, is a genuine modification over the normal flux technique.

Another interesting and very simple and inexpensive method to grow single crystals is the 'gel method' which has been largely used by us growing single crystals of different materials. A modified experimental set-up for the growth technique which has the basic advantage of providing greater lateral degree of freedom to the mobile ions to approach the growth sites is suggested. The set-up is very simple and inexpensive and hence it offers an easy possibility to grow crystals on seeds in gels.

Both the methods of crystal growth will be fully described and illustrated by the crystals grown in this laboratory. Chemical etching studies carried out on the crystals grown will be described and how it helped us to reveal relative crystalline perfection of crystals grown by different methods will be discussed.

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#### 14. AUTOMATION IN SINGLE CRYSTAL DIFFRACTOMETRY

R. CHIDAMBARAM and A. SEQUEIRA, Neutron Physics Section, Bhabha Atomic Research Centre, Bombay 400 085.

The evolution of automatic single crystal x-ray and neutron diffractometers over the last decade will be reviewed with particular reference to the developments in this field at Trombay. Starting with the early instruments for collecting two-dimensional data, the various stages leading to an indigenous design for a four-circle neutron/x-ray diffractometer will be discussed. The necessary hardware for automating this diffractometer has also been designed and two types of automation systems have been developed. One uses an 'off-line' punched paper-tape control and the other an 'on-line' computer. The latter is based on third generation TDC-312 computer with 4K memory, an ASR 35 teletype and an x-y plotter. The instantaneous positions of the four shafts as well as the signal and monitor counts are visually displayed. A system of programs that have been developed for the collection of three-dimensional single crystal intensity data as well as the interface associated with the computer control will be described in detail. There are possibilities of optimising the efficiency of the computer usage by time-sharing and by utilising the idling time for data processing and computer graphics.

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#### 15. CRYSTALLOGRAPHY OF TOPOTACTIC REACTIONS

H. MANOHAR, Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore 560 012.

Some reactions in solids are known to be governed purely by structural considerations. In these reactions one finds that a single

crystal of the starting material is converted into pseudomorph containing one or more products in a definite crystallographic orientation and the conversion takes place throughout the volume of the crystal. These observations can be made with the help of suitable diffraction techniques. The reason why there is no breakup of the lattice in such cases is because the crystal structures are to a large extent preserved. These reactions, known as Topotactic reactions, can therefore be adequately explained only if the relevant structures are taken into account. Topotaxy has been observed in a variety of reactions, such as polymorphic transformations, oxidations, dehydrations, decompositions, isomerisations etc. both in organic and inorganic systems. The interest in these studies, the experimental techniques used and some examples of work in this area will be presented in the talk.

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## A1. INTERPRETATION OF THE INITIAL SLOPE OF THE FOURIER COEFFICIENT AGAINST ORDER CURVE FOR X-RAY DIFFRACTION LINE PROFILES

G.B. MITRA and B.K. MATHUR, Department of Physics, Indian Institute of Technology, Kharagpur 721 302.

The Fourier coefficient for a given order of a pure diffraction line profile is the product of the Fourier coefficients for the same order of its constituent profiles like particle size, strain, stacking and twinning faults, variable interlayer spacing etc. The Fourier coefficient for particle size effect is known to be  $AP(n) = (\langle N \rangle - |n|)$  where  $\langle N \rangle$  is the average particle size and  $n$  the order of the Fourier coefficient. According to Warren (1958) in the presence of a stacking fault, the Fourier coefficient derivative at zero order, i.e.,  $A'(0)/A(0) = -f(a)/N$  where  $f(a)$  is a function -- generally a linear combination of stacking and twinning faults. Thus, the initial slope of  $A(n)$  against  $n$  curve gives an "effective" particle size as well as stacking and twinning faults. It has been shown here that Warren's calculation involves some approximations which are not valid and that rigorous calculation of the Fourier coefficient for stacking and twinning faults as well as variable interlayer spacing is of the type  $\phi^n$  where  $\phi$  is a function of these fault parameters. The strain dependent Fourier coefficient is shown by Mitra and Chaudhuri (1974) to be of the type  $\exp(-n^2 \epsilon^2)$  where  $\epsilon$  is a function of the r.m.s. strain and its derivatives. Combining all these, it has been shown that for all cases

$$\frac{A'(0)}{A(0)} = - \frac{1}{\langle N \rangle} .$$

## A2. EFFECT OF TRUNCATION ON X-RAY LINE PROFILE ANALYSIS

G.B. MITRA and A.K. CHAUDHURI, Indian Institute of Technology, Kharagpur, India.

The effect of truncation on the x-ray line profile analysis has been discussed with reference to Fourier coefficient and variance method. Fourier coefficients of the pure diffraction profile will be unaffected if both the observed and geometrical profile are terminated at the same finite limits. For symmetrical profiles the second moment will give the

correct results whereas in the case of unsymmetrical profile, a correction has to be made. Hence the importance of expressing the second moment as a function of the Fourier Coefficient and its various derivatives at zero order is stressed.

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### A3. ON THE DETERMINATION OF ORDERING PARAMETERS FROM THE DEBYE CHARACTERISTIC TEMPERATURE OF BINARY ORDERED ALLOYS

B.K. SAMANTARAY and G.D. NIGAM, Department of Physics, Indian Institute of Technology, Kharagpur 721 302.

One often faces difficulties while determining the order parameters from x-ray measurements because of the effects of static atomic displacements, thermal vibration of atoms, Compton scattering etc. on the diffuse scattering of the x-rays. It becomes almost impossible to determine the same specially when the constituents of the alloy have nearly same scattering factor. However Mitra and Chaudhuri (*Acta Cryst.* A30, 385, 388) have suggested an alternative method for determining the ordering parameter from the Debye temperature for the cases when alloy and the constituent metals have the same type of structure and have assumed an A (or B) type of atoms to be surrounded by a B (or A) type of atom only. The above theory has been generalised in the present case such that it is valid for all cases such that the alloy and the constituent metals can be fcc, bcc or simple cubic and is valid for all types of super lattices in the cubic system. The above theory has been applied for several alloys and the predicted values agree quite well with the experimental data.

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### A4. ON THE ADDITIVITY OF ELASTIC CONSTANTS AND DEBYE CHARACTERISTIC TEMPERATURES OF MIXED CRYSTALS

G.B. MITRA and B.K. SAMANTARAY, Department of Physics, Indian Institute of Technology, Kharagpur 2.

Mixed crystals of carbides, oxides and halides etc. are of great interest to material scientists because of their varied applications and

their physical properties show large variations with concentration. Moreover some of the crystals show local ordering which is one of the principal factors determining the properties. In the present investigation expressions have been derived connecting the elastic constants, bulk modulus of elasticity and the Debye characteristic temperature with that for the component crystals and the extent of local ordering. The results have been applied in case of the Debye characteristic temperature of Tungsten Carbide-Vanadium Carbide system and good agreement with the experimental values have been obtained.

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#### A5. X-RAY DIFFRACTION FROM A MODEL OF CYLINDRICAL CRYSTALLINE WITH ELLIPTICAL CROSS-SECTION

S. BHATTACHERJEE and G.D. NIGAM, Physics Department, Indian Institute of Technology, Kharagpur 2, W.B.

It is well established that many minerals and high polymers of medical and industrial importance possess curved lattices with cylindrical symmetry. It is quite likely that under natural condition of growth and ordinary stress and strain due to mechanical handling the curvature may not be uniform all along. In such cases cylindrical crystallites will possess non-uniform curvature - or in other words a more realistic model will be a cylindrical crystallite with elliptical cross-section.

X-Ray diffraction intensities from such a model has been worked out following a method similar to that of Mitra and Bhattacharjee [Acta Cryst.(1968) A22, 266; Acta Cryst.(1971) A27, 22]. The final expression derived is very general in nature and is easy to handle numerically. Attempts are being made to explain some experimental data in the light of the expression derived.

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A6. A PROCEDURE TO REDUCE TERMINATION ERRORS IN THE COMPUTATION OF RADIAL DISTRIBUTION FUNCTIONS

RAMESH NARAYAN and S. RAMASESHAN, Materials Science Division, National Aeronautical Laboratory, Bangalore 560 017.

In the Fourier transformation of the X-ray scattering data of liquids and amorphous solids to obtain their radial distribution function (RDF), errors are introduced because the intensity function is available only for a finite range of  $\mu (= 4\pi \sin \theta/\lambda)$ . An iterative procedure is proposed to reduce these errors, based on the knowledge that the RDF cannot be negative. The method consists of repeatedly Fourier transforming and cutting off the negative portions of the calculated RDFs. Computer calculations made on model systems indicate a two-fold improvement. Firstly, the unphysical oscillations at small  $r$ -values of the RDF are reduced. Secondly, the positions and amplitudes of peaks and valleys in the RDF show improvement in the right directions.

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A7. PROBABILITY DISTRIBUTION CONNECTED WITH STRUCTURE AMPLITUDES OF TWO RELATED CRYSTALS. PART VII - THE CASE OF AN APPROXIMATELY CENTRO-SYMMETRIC STRUCTURE

P. SWAMINATHAN and R. SRINIVASAN, Centre of Advanced Study in Physics, University of Madras, Guindy Campus, Madras 600 025.

This reports an extension of the studies in the earlier parts of this series on probability distributions, to two new cases. The results are applicable to the case of a pair of structure factors one corresponding to a "trial model" which is truly centrosymmetric while the "true model" is approximately centrosymmetric (distortion type). Statistical parameters and other tests derivable from the basic distributions will be discussed and their application to actual situation illustrated.

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## A8. PROBABILITY DISTRIBUTION OF BIJVOET DIFFERENCES

P. SWAMINATHAN and R. SRINIVASAN, Centre of Advanced Study in Physics, University of Madras, Guindy Campus, Madras 600 025.

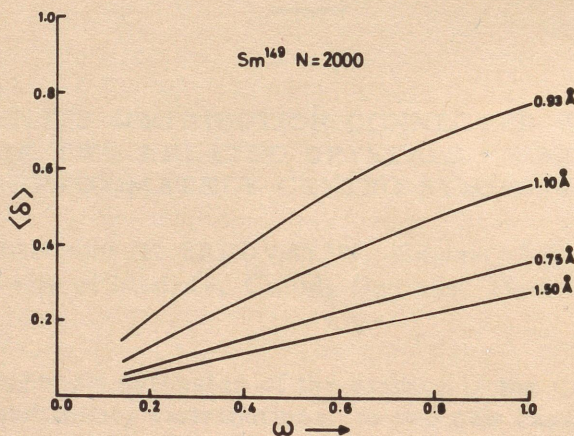
The earlier theoretical treatment of the probability distribution of Bijvoet differences [Parthasarathy & Srinivasan (1964). *Acta Cryst.* 17, 1400-1407], has been extended to four new situations, namely, when the nonanomalous scatterers (Q) take up centro-symmetric configuration with the anomalous scatterers (P) corresponding to  $P = \text{one}$ ,  $P = \text{two}$ ,  $P = \text{many atoms with centrosymmetric (MC)}$  and  $P = \text{many atoms with non-centro-symmetric (MNC)}$  configuration. The theoretical distributions have been verified with hypothetical models.

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## B1. EXPECTATION VALUE OF THE BIJVOET RATIO IN NEUTRON DIFFRACTION

S. K. SIKKA, Neutron Physics Section, Bhabha Atomic Research Centre, Trombay, Bombay 400 085.

Parthasarathy [Acta Cryst. (1967) 22, 98] has carried out a theoretical analysis of the expectation value of the Bijvoet ratio [ $\delta = |\Delta I| / \frac{1}{2}(I + \bar{I})$ ] for the X-ray case with the assumption  $|F_A''| \ll |\bar{F}_N'|$ . This assumption is not strictly applicable in neutron diffraction and consequently his expressions predict for  $\langle \delta \rangle$ , values greater than 2 for some cases. We have carried out the calculations for the general case and studied the dependence of  $\langle \delta \rangle$  on the wavelength ( $\lambda$ ) of the incident neutron beam and concentration ( $\omega$ ) of the resonant isotopes  $\text{Cd}^{113}$  and  $\text{Sm}^{149}$ . It has been found (see Fig.) that with proper choice of  $\lambda$  and  $\omega$ , large  $\langle \delta \rangle$  values can be experimentally achieved even for complex crystals like proteins.



## B2. ON THE METHOD OF ASSESSING THE NUMBER OF ATOMS IN THE UNIT CELL OF AN ORGANIC CRYSTAL

S. S. RAJAN, Centre of Advanced Study in Physics, University of Madras, Guindy Campus, Madras 600 025.

It is shown that the rough method of arriving at the number of non-hydrogen atoms from the volume of the unit cell of an organic

crystal (Kempster and Lipson, 1972, Acta Cryst., B28, 3674) can be very much improved upon by including hydrogens also. The empirical relation  $V = 8.9N$  where  $V$  is the volume of the unit cell and  $N$  = Number of atoms including hydrogens, is arrived at by least squares fit of data from 114 aliphatic compounds.

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### B3. $L_p$ FACTOR CORRECTION IN THE STRUCTURE DETERMINATION OF $CdI_2$ CRYSTALS

P.C. JAIN and G.C. TRIGUNAYAT, Department of Physics and Astrophysics, University of Delhi, Delhi 110 007.

A hitherto unnoticed snag in the structure determination of  $CdI_2$  polytypes, involving Lorentz and polarization correction has been pointed out. Attention has been drawn to the fact that the intensities for  $10.l$  spots should be calculated for the same range as the range of the observed spots, viz.  $l = 2n$  to  $4n$  instead of the range  $l = 0$  to  $2n$ , which has been the common practice uptill now, because the  $L_p$  factor has quite different variation in the two ranges. The choice of a wrong range may possibly lead, in some particular case, to a wrong structure.

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### B4. ABSORPTION CORRECTION IN PLATE-SHAPED CRYSTALS OF HIGH ABSORPTION

P.C. JAIN and G.C. TRIGUNAYAT, Department of Physics and Astrophysics, University of Delhi, Delhi 110 007.

A simple expression for absorption correction, derived for an infinite plate intercepting the whole beam, has been shown to be applicable to the structure analysis of the plate-shaped crystals of cadmium iodide and lead iodide, of usual sizes. The expression holds almost exactly for crystals which have slightly bigger cross-section than the X-ray beam, while for the crystals completely bathed in the X-rays, the expression, with slight modification, holds approximately. This work now makes it possible to apply the necessary correction for absorption in these crystals, which uptill now has always been ignored in the want of a suitable formulation. The expression is applicable to the plate-shaped crystals of other substances, too, having appreciably high absorption.

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B5. SOLUTION OF CRYSTAL STRUCTURES FROM CONSIDERATIONS OF MOLECULAR PACKING

N. SHAMALA, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012.

The principle of this method is to calculate the intermolecular van der Waals contacts for different orientations ( $\theta$ ,  $\phi$  and  $\psi$ ) and positions (X, Y, Z) of the molecule in the unit cell. The acceptable solution is one in which no predominant short contacts are present and the hydrogen bonds are satisfactory.

Thus, in practice, we place one molecule of assumed shape, or stereochemistry, with a suitable position in the unit cell, to start with. The coordinates of all translated molecules and symmetry related molecules are generated. The intermolecular contacts are then calculated, and, if there are short contacts, the molecule is given a rotation  $\theta$ , keeping  $\phi$  and  $\psi$  constant. The intermolecular contacts are then calculated and the process repeated. In this fashion, varying ( $\phi$ ,  $\theta$ ,  $\psi$ ) as well as (X, Y, Z), we try to find out the position in the unit cell for which no short contacts are present and the hydrogen bonds are also well formed.

A general program has been written which can do the calculations on an IBM 360/44 computer. The program gives a very good result for the crystal structure of L-alanine, which is known. Details will be presented.

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B6. ORIENTATION DETERMINATION OF MOLECULES USING COSINE SEMINVARIANTS

SRINIVASA NATARAJAN, Department of Physics, Madurai University, Madurai 625 021.

By definition the structure invariants are those linear combination of phases which have the same value (determined only by the crystal structure) for all choices of origin. Therefore the values of  $\text{Cos}(\phi_1 + \phi_2 + \phi_3)$  for  $h_1 + h_2 + h_3 = 0$  depends only on the orientation of the structure w. r. t. crystallographic axes. Using this fact we can determine the orientation of the molecule, when part of its configuration is known. Assuming that the known molecular fragment controls most of the cosine seminvariant values.

Rotation search method is used to find out the good match between the values of observed and calculated cosine seminvariants. Two to three hundred sigma 2 triplets with high A value will be sufficient to get the orientation of the molecule. Computer programme has been written to do all the calculation the output of which will give  $C_{ij}$  matrix for a good match.

Above method is being tried for a known crystal structure of Estradiol urea (1:1) Acta Cryst. 1972, B28, 1864.

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C1. THE CRYSTAL STRUCTURE OF 2-AMINO  $\Delta^{3,3a}$  HEXAHYDRO-BENZOTHAZOLE-7-ONE

N. SHAMALA\* and P.S. NAGANATHAN\*\*, Centre of Advanced Study in Physics, University of Madras, Madras 600 025.

The crystal structure of 2-amino  $\Delta^{3,3a}$  hexahydrobenzothiazole-7-one, has been determined using three-dimensional photographic data. The crystals are monoclinic, space group  $P2_1/c$ , with cell dimensions  $a=6.49$ ,  $b=14.92$ ,  $c=8.17 \text{ \AA}$  and  $\beta=94.9$ . The structure was solved by heavy atom method and refined by full-matrix least squares method to an R-factor of 0.109 for 1412 observed reflections. The crystal structure is stabilized by a three dimensional network of hydrogen bonds of the type N-H...O and N-H...N.

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C2. STRUCTURE AND CONFORMATION OF SULPHUR HETERO-CYCLIC AMINO ACIDS

K.K. CHACKO and S.K. BHATTACHARJEE, Centre of Advanced Study in Physics, University of Madras, Madras 600 025.

The structure and conformational features of the following compounds will be dealt with - namely, (a) L-thioprolin, (b) 4-amino 4-carboxyl thiapyran and (c) 4-amino 4-carboxyl thiapyran hydrobromide. A comparative study will be made on the structures (a), (b) and (c) with the corresponding sulphur free amino acids. Although L-proline and L-thioprolin (where the  $C^\alpha H_2$  group of proline is replaced by sulphur atom) crystallise in the same Space Group  $P2_12_12_1$ , the two structures are completely non-isomorphous. However, structure (b) has similarity in packing with the corresponding sulphur free amino acid - namely, the structure of 1-amino cyclohexane carboxylic acid. A similar feature is observed in the case of structure (c) and 1-amino cyclohexane carboxylic acid hydrochloride.

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### C3. ARRANGEMENT OF PLANAR MOLECULES IN ORGANIC STRUCTURES

Y. T. THATHACHARI (Biomedical Engineering Division, Indian Institute of Technology, Madras 600 036); M. R. NARASIMHAMURTHY and K. VENKATESAN, Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012.

The arrangements of planar molecules in known structures were studied systematically. From the published crystal structures data the mutual separations and orientations of adjacent layers were calculated, and correlated with the size, shape and structure of the layers. A preliminary survey of the results indicates that the presence of planar molecules in a structure does not necessarily lead to their parallel alignment: e.g. the unsubstituted hydrocarbons like Benzene, Biphenyl and Naphthalene - i.e. small layers or layers with extension in only one dimension - the nearest neighbours are inclined and not parallel. Some of the conditions favouring the parallel alignment, an interlayer separation close to 3.4 Å and increasing overlap of adjacent layers are: the extension of the layer in two dimensions as in Coronene and Perylene; introduction of hetero atoms in the ring even when the extension is in only one dimension: e.g. in anthracene the nearest neighbours are inclined; in Phenazine which differs from it only in the substitution of two nitrogen atoms in the middle ring, the nearest neighbours are parallel and have considerable overlap. In substituted hydrocarbons the adjacent layers tend to be parallel; but the extent of overlap still seems to depend on the layer size: e.g. in Benzoquinone there is little overlap between adjacent parallel layers while in Naphthoquinone there is significant overlap.

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### C4. THE CRYSTAL AND MOLECULAR STRUCTURE OF THE MONOETHYLENETHIOKETAL OF TRANS-METHYL 3,5-DIKETOCYCLOPENTANE-1,2-DICARBOXYLATE

T. N. GURU ROW, K. VENKATESAN, D. K. BANERJEE, J. ALEXANDER and A. V. ABRAHAM, Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012.

It appeared to us that the monothioketal of trans, methyl 3,5-diketocyclopentane-1,2-dicarboxylate (I) would be an ideal intermediate for the synthesis of Prostaglandins, because of its amenability to pro-

gressive alkylation with suitable sidechains followed by successive decarbomethoxylation under the neutral conditions of Krapcho, the two keto groups, one protected as its thioketal, being in the positions corresponding to those of oxygen functions present in prostaglandins. The title compound was prepared by Dieckmann cyclization of the thioketal of methyl-2-keto-butane-1,3,4-tricarboxylate where another isometric compound, the monothioketal of 1,3-diketocyclopentane-2,4-carboxylate, could also have been formed. In order to determine the molecular structure of this compound X-Ray crystallographic investigation was undertaken. The crystals are monoclinic with cell dimensions  $a = 14.49 \text{ \AA}$ ,  $b = 5.90 \text{ \AA}$ ,  $c = 14.69 \text{ \AA}$ ,  $\beta = 86.0^\circ$ , space group  $P2_1/c$ , with  $Z = 4$ . The structure was solved by direct methods. The final R index with all the non-hydrogen atoms anisotropically refined is 0.117. X-ray crystallographic studies confirmed that the crystalline monothioketal has the structure (I). The carbomethoxy groups in the cyclopentane ring are trans with respect to each other. The conformation of the molecule and the packing in the crystal structure will be discussed.

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#### C5. AN X-RAY CRYSTALLOGRAPHIC STUDY OF 3-NITRO 4-HYDROXY PHENYL ARSONIC ACID

A. CHATTERJEE and S. P. SEN GUPTA, Department of General Physics and X-Rays, Indian Association for the Cultivation of Science, Calcutta 32.

The present work forms a part of our programme of systematic X-ray crystallographic studies on the important organic analytical reagent arsonic acid and its derivatives on which very little structural work has so far been made. The compound  $\text{NO}_2\text{-OH-C}_6\text{H}_3\text{-ASO(OH)}_2$  crystallizes in the triclinic space group  $P\bar{1}$  (confirmed from the  $N(z)$  test) with two molecules in the unit cell having dimensions  $a = 5.89 \text{ \AA}$ ,  $b = 8.32 \text{ \AA}$ ,  $c = 12.09 \text{ \AA}$  and  $\alpha = 97^\circ 45'$ ,  $\beta = 107^\circ 22'$ ,  $\gamma = 125^\circ 59'$ , which have been determined from the Weissenberg oscillation photographs along all the three axes and which differ slightly from the earlier reported ones taken along two axes only (Goswami & Dutta, 1963). The 2D-Patterson maps along the three axes (zero layer projection) have revealed the  $(x, y, z)$  position of heavy atom As and using the heavy atom method the trial structure of the molecule with 14 atoms excepting the hydrogen atoms has been obtained from the successive 2D-Fourier synthesis along the shortest axis of projection with a R value  $\sim 15\%$ . Details of the structure refinement from a 3D-analysis will be presented.

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C6. THE CRYSTAL AND MOLECULAR STRUCTURE OF 7a-METHYL-4,7-DIOXO-CYCLOPENTATHIANE-1,1-DIOXIDE

V.K. WADHAWAN, Neutron Physics Section, Bhabha Atomic Research Centre, Bombay 400 085.

Total synthesis of heterocyclic steroid compounds is of great current interest, particularly because of the interesting pharmacological properties exhibited by some of them. The title compound, with molecular formula  $C_9H_{12}O_4S$ , was synthesised<sup>1</sup> so as to constitute the C and D rings of the corresponding sulphur-containing steroid. Its crystal and molecular structure has been determined from three-dimensional Weissenberg data, with visual estimation of intensities. Crystallisation of the compound from a warm solution of chloroform-petroleum ether mixture gave needle-shaped crystals belonging to the monoclinic system, space group  $P2_1/n$ ,  $a = 14.301(6)$ ,  $b = 9.962(5)$ ,  $c = 6.969(4)$  Å,  $\beta = 95.01^\circ(2)$ ,  $Z = 4$ , and  $\rho_x = 1.452(2)$  g cm<sup>-3</sup>. A total of 1918 independent reflections were recorded. The structure was solved by the heavy atom method. The hydrogen atoms could also be located from a difference Fourier synthesis. Full-matrix least squares refinement gave a final R value of 0.072 for 1326 non-zero reflections.

The S-C bond lengths are 1.76(2) and 1.81(1) Å, the S=O bond lengths 1.43(1) and 1.45(1) Å, and both the C=O bond lengths are 1.21(1) Å. The structure is held together essentially by van der Waals forces. The study has conclusively shown that the molecule exists in the cis configuration, thus indicating the need for its isomerisation before further attempts can be made for synthesising the complete heterocyclic steroid.

1. G. V. Bhide and P. S. Jogdeo (to be published). The author is indebted to them for kindly supplying crystals of the title compound.

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C7. X-RAY STUDIES ON SOME MESOGENIC COMPOUNDS

G. V. VANI, Raman Research Institute, Bangalore 560 006 and KALYANI VIJAYAN, Materials Science Division, National Aeronautical Laboratory, Bangalore 560 017.

As part of a programme of X-ray structural studies on the solid phase of compounds which exhibit liquid crystalline behaviour, N-p-

methoxybenzylidene-p-phenylazoaniline,  $C_{20}H_{17}ON_3$ (I), Cholesteryl Chloride,  $C_{27}H_{45}Cl$  (II) and p-cyanobenzylidene-p'-octyloxyaniline,  $C_{22}H_{27}ON_2$  (III) were crystallized. All the three compounds belong to the monoclinic system with the following crystal data:

- I. Space group:  $P2_1/c$ ;  $a = 9.79 \pm 0.04$ ,  $b = 23.75 \pm 0.04$ ,  $c = 15.67 \pm 0.03 \text{ \AA}$  and  $\beta = 112^\circ 26'$ ;  $Z = 8$ .
- II. Space group:  $P2_1$ ;  $a = 10.83 \pm 0.02$ ,  $b = 7.61 \pm 0.02$ ,  $c = 16.44 \pm 0.02 \text{ \AA}$  and  $\beta = 103^\circ 18' \pm 1^\circ$ ;  $Z = 2$ .
- III. Space group:  $P2_1/a$ ;  $a = 7.66 \pm 0.02$ ,  $b = 15.91 \pm 0.2$ ,  $c = 15.97 \pm 0.04 \text{ \AA}$  and  $\beta = 91^\circ 46' \pm 1^\circ$ ;  $Z = 4$ .

Of these, N-p-methoxybenzylidene-p-phenylazoaniline was taken up for complete structure determination. The possible orientations of the rod-like molecules in the unit cell could be deduced from an examination of the three dimensional Patterson map and trial calculations using a few strong low angle reflections. Solution of the structure by direct methods presents difficulties, presumably on account of the extreme paucity of intensity data at high Bragg angles and the unfavourable reflection to parameter ratio, viz., 1074 independent observed reflections in the copper sphere for 48 non-hydrogen atoms in the asymmetric part. Attempts are now in progress to solve the structure by a combination of direct methods, packing considerations and trial and error calculations. Some of the problems encountered in the solution of such light atom structures will be discussed.

#### C8. REFINEMENT OF THE CRYSTAL STRUCTURE OF ZINC ACETATE DIHYDRATE

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and L. CARTZ<sup>+</sup>, <sup>\*</sup>Physics Department, Madurai University, Madurai 625 021, <sup>+</sup>Materials Science Division, Marquette University, Milwaukee, Wisconsin, USA, <sup>+O</sup>presently at the Madurai University.

As part of the structural studies on inorganic acetates, the refinement of the crystal structure of zinc acetate dihydrate, solved by van Niekerk, Schoening and Talbot (Acta Cryst. 1953, 6, 720) using only projection data, was taken up. Three-dimensional data for this compound were collected on a GE-XRD6 quarter circle diffractometer using  $CuK\alpha$  radiation with balanced filters upto an angle of about  $150^\circ$  for  $2\theta$ . Starting from the atomic parameters of the earlier work, the structure

was refined to an R factor of 0.094 for 532 reflections. A difference Fourier synthesis computed at this stage revealed at the expected positions four of the five hydrogen atoms, two belonging to the water molecule and two to the methyl group. The coordination polyhedron for the divalent zinc ion, lying on the 2-fold axis in the structure, is a highly distorted octahedron. The average Zn-O distance is 2.17 Å and the O-Zn-O angles vary from 59° to 157°. A notable feature of the coordination is the bidentate bridging of the acetate group, which is rather rare. The C-O distances of the planar acetate group are 1.25 and 1.29 Å, and the C-C distance is 1.53 Å. The water molecule forms two hydrogen bonds with the oxygen atoms of different acetate groups. These hydrogen bonds run along the crystallographic b and c axes, the respective O<sub>w</sub>-O distances being 2.68 and 2.73 Å.

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#### C9. SOME ASPECTS OF COORDINATION AND HYDROGEN BONDING IN THE CRYSTAL STRUCTURE OF LEAD ACETATE TRIHYDRATE

R.K. RAJARAM and J.K. MOHANA RAO, Physics Department, Madurai University, Madurai 625 021.

As a part of the crystallographic investigations on inorganic acetates, the crystal structure of lead acetate trihydrate was taken up. This compound, one of the few water-soluble lead compounds, crystallizes in a tetramolecular unit cell of dimensions  $a = 15.80$ ,  $b = 7.31$ ,  $c = 9.07$  Å,  $\beta = 109.8^\circ$ . From systematic absences, the space group can be C2, Cm or C2/m. The crystal structure, solved by the heavy atom technique in the space group C2/m, has been refined to an R value of 0.13 for 796 observed CuK $\alpha$  reflections. The lead atom and an acetate group lie on the mirror plane, and the second acetate group has its C-C bond on the mirror plane. Lead is coordinated to either oxygen atoms dodecahedrally, the minimum and the maximum Pb-O distances being 2.31 and 3.10 Å. A feature of this coordination is that both the oxygens of the two acetate groups coordinate to the same lead ion. The nature of coordination for the plumbous ion will be discussed.

All the oxygen atoms belonging to the water molecules and the acetate groups participate in a complicated network of hydrogen bonding with the possible existence of H<sub>3</sub>O<sup>+</sup>, OH<sup>-</sup> ions and neutral H<sub>2</sub>O molecules. These hydrogen bonds are approximately perpendicular to the b axis.

It is recorded in literature that lead acetate is piezo- and pyroelectric, and it is not clear whether it is the trihydrate, the common form. Preliminary dielectric measurements indicate that this could be possibly a ferroelectric up to its melting point, in which case the space group has to be noncentric. Anyway, the departure from the centric structure will be slight and difficult to detect using X-ray techniques.

Financial assistance by the Department of Atomic Energy, Bombay, under the scheme "X-ray Diffraction and other Physical Studies on Materials showing Phase Transitions" is gratefully acknowledged.

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#### C10. THE CRYSTAL STRUCTURE OF A SECOND MODIFICATION OF *m*-NITROBENZOIC ACID

N. N. DHANESHWAR, A. G. KULKARNI, S. S. TAVALE and L. M. PANT, National Chemical Laboratory, Poona 8.

The crystals are monoclinic, space group  $P2_1/n$  with  $a = 7.78(1)$ ,  $b = 11.15(1)$ ,  $c = 17.15(1)$  Å;  $\beta = 93.4(3)^\circ$ ;  $\rho_o = 1.498$  g. cm<sup>-3</sup>;  $\rho_c$  for  $Z = 8$  is 1.495 g. cm<sup>-3</sup>. The structure, solved by direct methods with visually estimated data refined to a  $R$  0.099 for 1485  $F_{obs}$ . As in the first modification, the structure consists of dimeric units, each consisting of crystallographically independent molecules A and B; however, the nitro groups of the dimer are in the cis configuration in this structure. The only significant difference between the molecular dimensions in the two crystal modifications is that the C-O bonds of the carboxylic group in both A and B are nearly equal in this structure whereas they are about 0.04 Å different in both A and B in the first modification.

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#### C11. THE CRYSTAL AND MOLECULAR STRUCTURE OF 12-ETHOXY-2,3-BENZO-6,5-NAPHTHO(b)(7,12)-THIAPHOSPHORIN-7,7,12-TRIOXIDE

N. N. DHANESHWAR, A. G. KULKARNI, S. S. TAVALE and L. M. PANT, National Chemical Laboratory, Poona 8.

Crystals are monoclinic, space group  $P2_1/n$  with  $a = 14.71(1)$ ,  $b = 8.07(2)$ ,  $c = 14.30(1)$  Å;  $\beta = 94.0(2)^\circ$ ;  $\rho_o = 1.406$  g. cm<sup>-3</sup>;  $\rho_c = 1.405$  g. cm<sup>-3</sup> for  $Z = 4$

The structure was solved by direct methods using visually estimated data and refined by the least squares method to an R value of 0.115 for 1906 reflections.

The dihedral angle between the plane of the naphthalene ring and that of the benzene ring on either side of the sulphur and phosphorous atoms is  $133.8^\circ$ ; P lies in the naphthalene plane and S is  $0.153 \text{ \AA}$  away from it whereas S lies in the benzene plane and P is  $0.159 \text{ \AA}$  away from it. The S=O and S-C bonds around S have a slightly distorted tetrahedral configuration suggesting greater s character of the S=O bonds than of the S-C bonds; the distortion in tetrahedral configuration around P is more severe. The ethyl group undergoes large anisotropic thermal vibrations due to loose van der Waals contacts with atoms of neighbouring molecules.

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#### C12. STRUCTURE REFINEMENT OF ANTHRAQUINONE: A CORRECTION

S. N. SRIVASTAVA, Physics Department, University of Allahabad, Allahabad.

Structure of anthraquinone was refined three dimensionally first by Murty (1960) and later on by Lonsdale, Milledge, and El Sayed (1966). Murty refined the structure to R-value of 19.6% using isotropic thermal parameters whereas Lonsdale et al using anisotropic thermal parameters ended up with R-value of 12.0% for their data at  $20.5^\circ\text{C}$ . Upon comparison of the observed and calculated values of the structure amplitudes in the two sets it was found that large differences occur for every value. A number of structure factors were calculated and no error was detected in the set given by Murty, but the values calculated by Lonsdale et al were found to be smaller by a factor of four.

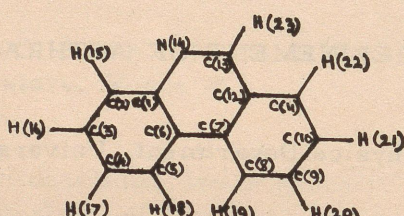
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#### C13. CRYSTAL & MOLECULAR STRUCTURE OF PHENANTHRIDINE

P. ROY CHOWDHURY, X-ray Laboratory, Presidency College, Calcutta.

Phenanthridine  $\text{C}_{13}\text{H}_9\text{N}$  forms orthorhombic crystals with four molecules in the unit cell of dimensions  $a = 11.72$ ,  $b = 16.41$ ,  $c = 4.97 \text{ \AA}$  and has space group  $P2_12_12_1$ . Approximate positional parameters were

obtained by combination of weighted reciprocal lattice plot for the hko reflections and trial and error method. Least squares refinement led to the solution of the structure having the final residual value of 7.63% with 491 reflections intensities obtained photographically with  $\text{CuK}_\alpha$  radiation. Visual method of intensity measurement was adopted. Electron density sections and difference synthesis for the confirmation of hydrogen positions were computed. The remarkable feature of the structure is the hyper shortening of the C(13)-N(14) and the lengthening of C(6)-C(7) and C(12)-C(13) bond distances. Theoretical calculations of the bond lengths on the basis of SCFMO were carried out. Even though the experimental and theoretical bond lengths differ, yet qualitatively there is a close resemblance between the trends of the bond lengths. C(6)-C(7) bond being contact bond linking the two benzenoid rings is longer than the normal aromatic C-C distance (1.396) - a characteristic feature of aromatic molecules containing phenanthrenoid arrangement of rings.



C14. THE CRYSTAL STRUCTURE OF 3-HYDROXY, 2-NAPHTHOIC ACID,  $\text{C}_{11}\text{H}_8\text{O}_3$

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The crystal structure of title compound has been determined by X-ray diffraction using 653 independent reflections, data refined by full-matrix least squares to an R of 0.094. The crystals are monoclinic with  $a = 10.13$ ,  $b = 11.845$ ,  $c = 7.88 \text{ \AA}$ ,  $\beta = 105.5^\circ$ ,  $Z = 4$ , space group  $\text{P2}_1/\text{c}$ . In the crystal, the molecules form dimers across centres of symmetry (hydrogen bond  $2.66 \text{ \AA}$ ), the hydrogen bonds being linear. There is also an intramolecular hydrogen bond ( $2.66 \text{ \AA}$ ) between the oxygen atoms of the hydroxy and carboxyl groups in the molecule. Other inter-atomic distances are of the van der Waals type. The carboxyl group is twisted out of the main aromatic ring by  $4.5^\circ$ .

C15. THE CRYSTAL STRUCTURE OF 2-ETHOXY, 1-NAPHTHYL  
ETHYL METHYL ETHER,  $C_{15}O_2H_{18}$

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of Ranchi, Ranchi 834 008.

The crystal structure of title compound has been determined by  
X-ray diffraction. The crystals are orthorhombic with  $a = 15.66$ ,  $b =$   
 $11.25$ ,  $c = 7.24 \text{ \AA}$ ,  $Z = 4$ , space group  $Pna2_1$ . In the crystal the molecules  
are stacked roughly in the (001) plane, forming a spiralling assembly  
around 001 due to the operation of screw axis symmetry. They are held  
together by van der Waals forces. The ethoxy group is nearly in the  
same plane as the naphthyl group but the ethyl methyl ether group is  
twisted out of this plane by large amount.

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C16. THE CRYSTAL STRUCTURE OF ZINC SALT OF ACETYLENE  
DICARBOXYLIC ACID  $ZnC_4O_4, 3H_2O$

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Physics, Ranchi University, Ranchi 834 008.

The crystal structure of title compound has been determined by  
X-ray diffraction using 920 independent X-ray reflections. The crystals  
are triclinic with  $a = 6.46$ ,  $b = 5.96$ ,  $c = 11.15 \text{ \AA}$ ,  $\alpha = 92.5^\circ$ ,  $\beta = 105.0^\circ$ ,  
 $\gamma = 91.3^\circ$ ,  $Z = 2$ , space group  $P1$ . The zinc atoms occupy special posi-  
tions. The acetylene dicarboxylate groups are hydrogen bonded via the  
three water molecules some of which are donors as well as acceptors in  
hydrogen bond formation. The zinc atoms has a four-fold co-ordination.

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C17. THE CRYSTAL STRUCTURE OF CALCIUM HOMOPHTHALATE  
 $CaC_9H_6O_4, 2H_2O$

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sity, Ranchi 834 008.

Crystals of calcium homophthalate dihydrate are triclinic with  
 $a = 6.57$ ,  $b = 14.01$ ,  $c = 6.02 \text{ \AA}$ ,  $\alpha = 92.9^\circ$ ,  $\beta = 108.2^\circ$ ,  $\gamma = 89.9^\circ$ ,  $Z = 2$ , space

group  $P\bar{1}$ . The structure has been determined by X-ray diffraction using 600 reflections. In the crystal the calcium ion has six-fold co-ordination. The homophthalate groups are hydrogen-bonded via the water molecules, along a. The geometry of the homophthalate group is as reported by earlier workers [Gupta & Sahu, Acta Cryst. (1971), B27, 2469].

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#### C18. THE CRYSTAL AND MOLECULAR STRUCTURE OF GUANIDINE DIPICRYL AMINATE

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The crystal structure of guanidine dipicryl aminate,  $(\text{NH}_2)_2\text{CNH} \cdot \text{C}_6\text{H}_2\text{N}_3\text{O}_6 \cdot \text{NH} \cdot \text{C}_6\text{H}_2\text{N}_3\text{O}_6$ , a charge-transfer complex, has been determined by the direct method MULTAN using 1300 X-ray reflexions, the data refined by full-matrix least squares to an R of 0.091. The crystals are orthorhombic with  $a = 19.46$ ,  $b = 20.56$ ,  $c = 9.88 \text{ \AA}$ , space group Pbca,  $Z = 8$ . In the crystal, dipicryl aminate and guanidine moieties co-exist linked by weak hydrogen bonds, coulombic and van der Waals forces. The aromatic rings in the dipicryl aminate anion are in planes normal to each other and the  $\text{NO}_2$  groups are rotated out of these planes by angles ranging from  $3.6$  to  $28.6^\circ$ . The guanidine group is planar.

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#### C19. THE CRYSTAL AND MOLECULAR STRUCTURE OF p-DIMETHYLAMINO BENZOIC ACID

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A study of the structure of the p-Dimethylamine Benzoic Acid (PDBA) has been carried out in an attempt to determine the characteristics of the interactions between para substituents in benzoic acid moiety. The crystals of PDBA were obtained from alcohol-water solution. The crystal system is triclinic with space group  $P\bar{1}$ ,  $Z = 2$ ,  $\rho_o = 1.292$  and  $\rho_c = 1.305$ . The unit cell dimensions are  $a = 8.60$ ,  $b = 8.06$  and  $c = 6.65 \text{ \AA}$ ,  $\alpha = 66.09^\circ$ ,  $\beta = 94.62^\circ$  and  $\gamma = 90.97^\circ$ , volume =  $420.02 \text{ \AA}^3$ . PDBA molecules form dimers centrosymmetrically. The benzene ring and consti-

tuents were assumed to be coplanar. Some of the strong reflections proved to be helpful while fixing the molecule with the help of stereochemical evidences. The structure has been determined from a study of projection of  $\ell_o$  and  $(\ell_o - \ell_c)$  along a and b axes, and has been refined by Fourier and Least squares methods. Corrections have been made for temperature factor variations and thermal anisotropy. The benzene ring is found to be planar. Bond lengths and bond angles show reasonable agreement with the values reported for related molecules.

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C20. AN X-RAY STUDY OF 3, 3'-DIBENZOYL 4, 4-DIHYDROXY BIPHENYL

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3, 3'-dibenzoyl 4, 4'-dihydroxyl biphenyl has been synthesised ( $C_{26}H_{18}O_4$ ) and the crystals are monoclinic with the space group  $P2_1/c$  having cell dimensions  $a = 19.80$ ,  $b = 29.32$ ,  $c = 35.22 \text{ \AA}$ ,  $\beta = 110^\circ$ ,  $\rho_o = 1.352$ ,  $\rho_c = 1.362 \text{ g/cm}^3$ ,  $M = 394$ ,  $Z = 4$ .

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C21. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIBENZOTHIOPHEN,  $C_{12}H_8S$

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This investigation was undertaken as a part of a programme of research on heterocyclic compounds in our laboratory because of their importance in drugs and dyes and specially that most cancer arresting substances are heterocyclic. The object of the investigation was to augment the information currently available for C-O, C-S bond lengths which are constituents of many heterocyclic compounds.

Dibenzothiophen is monoclinic,  $P2_1/c$  with four molecules per unit cell. The structure has been solved by heavy atom technique and

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refined by full matrix least squares using three dimensional intensity data to a final R-index of 0.091. The C-S bond length has been found to be  $1.753 \pm .014 \text{ \AA}$ . The analysis revealed some interesting aspects that the contact bond (C<sub>11</sub>-C<sub>12</sub>) in the molecule was lengthened like those of carbazole and dibenzofuran. To account for these sort of lengthening in phenanthrenoid arrangement of rings, a theoretical calculation considering  $\pi$ -electron delocalisation has been done which fairly agree with those of the experimental values.

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DI. AN X-RAY STUDY OF SILVER AMALGAM AND ITS STOICHIOMETRY

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Single crystals of silver amalgam grown from N/50 AgNO<sub>3</sub> solution and a globule of mercury are used in the present study. Long, needle-like, six-faced single crystals, grayish in colour, grow on the surface of the mercury drop placed in N/50 AgNO<sub>3</sub> solution. The conditions obtained from Weissenberg patterns taken with the crystal mounted about the needle (c-axis) and the perpendicular axes indicate the Hexagonal space group P6<sub>2</sub> or P6<sub>4</sub> with the lattice parameters  $a = 8.147(2) \text{ \AA}$  and  $c = 8.855(3) \text{ \AA}$ . The measured density is 12.11 gm/cc. Accepting the most probable stoichiometry for the compound as Ag<sub>2</sub>Hg<sub>2</sub>, the theoretical value for the percentage of silver present is 34.99 and the calculated density is 12.07 gm/cc. The chemical analysis shows that the percentage of silver present in the compound is 33.61 with a 3.9 percent error.

The d-spacings were obtained from X-ray diffraction powder photographs taken with Debye Scherrer method using nickel filtered copper K $\alpha$  radiation. The powder pattern was indexed by analytical method and was further checked by graphical method using Bjurstrom chart for Hexagonal crystals and Bjurstrom fan diagram. The relative intensities were measured using a Photovolt Multiplier Photometer.

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D2. THE CRYSTAL STRUCTURE OF POTASSIUM DIURANYL TRISOXALATE TETRAHYDRATE

N. C. JAYADEVAN, K. D. SINGH MUDHER and D. M. CHACKRABURTTY, Radiochemistry Division, Bhabha Atomic Research Centre, Bombay 400 085.

The crystal and molecular structure of the title compound K<sub>2</sub>(UO<sub>2</sub>)<sub>2</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub>.4H<sub>2</sub>O was determined by Fourier and least-squares methods. Two formula units are present in a monoclinic cell of  $a = 8.85 \text{ \AA}$ ,  $b = 19.67 \text{ \AA}$  and  $c = 5.37 \text{ \AA}$  and  $\beta = 91.5^\circ$  belonging to space group P2<sub>1</sub>/n. The final weighted R factor for the visually measured single crystal photographic data of 920 absorption correction reflections is

0.107. The structure consists of polymeric network of  $[(\text{UO}_2)_2(\text{C}_2\text{O}_4)_3]_n^{2n-}$  anions, and  $\text{K}^+$  ions. The water molecules are associated with the cation. The linear uranyl ions ( $\text{U}-\text{O} = 1.75 \text{ \AA}$  and angle  $\text{O}-\text{U}-\text{O} = 176^\circ$ ) have six secondary oxalate oxygen atoms at an average distance of  $2.49 \text{ \AA}$ . The oxalate groups are all tetradentate and planar and bridges two uranyl ions forming five membered rings. The structure differs from that of the anhydrous ammonium salt (1) in which only one third of the oxalate groups are tetradentate and planar while the rest are tridentate and folded about the C-C axis. This leads to a pentagonal coordination around the uranyl ion as in uranyl oxalate itself (2). However, the structure of the potassium salt is more in line with the presence of dimeric ions in solution from which it is crystallised out.

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### D3. AN X-RAY STUDY OF InSe

K. C. NAGPAL and S. Z. ALI, National Physical Laboratory, New Delhi 110 012.

Complete X-ray powder pattern of InSe, so far not available in literature has been given. The layer structure is based on a rhombohedral lattice cell, with space group  $R\bar{3}m$ ,  $a_0 = 4.0045 \pm 0.0005$ ,  $c_0 = 25.957 \pm 0.0012 \text{ \AA}$ , and atomic positions in 3(a) of the space group:

$$Z_{\text{In}} = \pm 0.0555, \quad Z_{\text{Se}} = 2/3 \pm 0.106$$

Extreme softness and initial observe-reverse orientation of the rhombohedral cell in adjacent parts of a crystal or in thin lamellae parallel to the basal cleavage planes appear to be the main factors responsible for the characteristic sets of sharp and diffuse lines in the powder patterns normally obtained. These and Gondolfi camera powder patterns for several crystals have been given.

Heat treatment of powders obtained through cold work has indicated that comparatively short duration of 'annealing' at  $600^\circ\text{C}$  improves the powder patterns to some extent, but longer periods give products consisting of other phases of the In-Se system with small amount of InSe. The relative proportion of free space over the InSe powders in capsules as well as the amount of cold work seem to be the two main factors affecting the decomposition of InSe and appearance of other phases.

Large platy crystals, on the other hand, withstand the heat treatment for a longer time. Even they undergo drastic changes in structure on prolonged heating at  $600^{\circ}\text{C}$ , yielding hexagonal phases with a common  $c_0 = 19.26$  and  $a_0 = 4.0$  and  $7.11 \text{ \AA}$  respectively.

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#### D4. THE DEBYE-WALLER FACTORS, AMPLITUDES OF VIBRATION AND DEBYE TEMPERATURES OF AMMONIUM CHLORIDE AND AMMONIUM BROMIDE

D. B. SIRDESHMUKH and K. G. SUBHADRA, Physics Department, Post-Graduate Centre, Warangal 506 001.

Using X-ray powder diffractometric technique, the Debye-Waller factors, amplitudes of vibration, Debye temperatures and structure factors of ammonium chloride and ammonium bromide have been determined.

The results are discussed vis-a-vis data available from other methods.

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#### D5. GENERATION OF QUASI-STATIONARY STATES IN LATTICES OF METALLIC COBALT

S. RAI, B. G. GOKHALE, S. D. RAI and B. SINGH, Physics Department, Allahabad University, Physics Department, Lucknow University Chemistry Department, Allahabad University

In the last few years a vast and, indeed, promising field of investigation regarding structural details of metallic crystals has sprung from their studies while interacting with x-rays. The challenge in this field has been to select the fundamentally new and interesting phenomena and to relate these to a consistent theory which throws light on crystallographic structures. In the process of K-excitation by x-irradiation of solid crystals, the specification of the final state requires that the wave function shall have a two fold axis of symmetry. The propagation sphere in the reciprocal lattice must pass through two points so located that the sum of the propagation vector vanishes. Accordingly, during the K-

absorption of x-rays the fate of the ejected electron would be to undergo a total reflection ( $\theta = 90^\circ$ ). This gives rise to a standing wave pattern in the vicinity of the parent atom which leads to the existence of a number of allowed energy states called quasi-stationary states (QSS). Considering total reflections of ejected K electron from (110), (203), (130) and (232) planes of metallic cobalt the energy positions of QSS A, B, C and D have been freshly computed. Results obtained from emission experiments have been used to attest the validity of the existence of QSS A and B for the first time. Significantly enough, absorption-experiment-data do go in favour of the existence of all the aforesaid QSS. Emphasis is laid on better resolution absorption experiment to get more insight in to the problem.

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D6. CRYSTAL STRUCTURE OF BIS(N-CYCLOHEXYLSALICYLALDIMINATO) COPPER(II)

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The crystal structure of the title compound has been determined using three-dimensional X-ray diffraction data from single crystals. It has a transplanar configuration around copper atom and the packing is governed by van der Waals forces. The angle of tilt between the mean planes of benzene ring and salicylaldimine residue is  $1.2^\circ$ .

The crystals of the compound are triclinic with one molecule per unit cell of dimensions  $a = 7.91 \text{ \AA}$ ,  $b = 12.57 \text{ \AA}$ ,  $c = 6.51 \text{ \AA}$ ,  $\alpha = 107^\circ$ ,  $\beta = 79^\circ$ , and  $\gamma = 101^\circ$ . The structure was solved by the heavy atom method using 1599 observed reflections. A final R value of 0.096 was reached.

Details of the crystal and molecular structure will be presented.

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D7. A COMPARISON OF THE THERMAL EXPANSION BEHAVIOUR OF SOME SCHEELITE TYPE COMPOUNDS

S. V. SURYANARAYANA and V. T. DESHPANDE, Physics Department, Osmania University, Hyderabad 7.

The data on the lattice thermal expansion of ten scheelite type tungstates, molybdates and periodates are presented. Though all these compounds are isotypic, there are variations in the individual coefficients. The observed anisotropies in the expansion behaviour are explained in terms of the ionic size, structure and shapes of the  $\text{BO}_4$  tetrahedra. For some of the compounds, from the available elastic data, the Debye characteristic temperatures are evaluated and correlated with the linear coefficients of expansion.

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D8. STRUCTURE OF  $\text{As}_{45}\text{Te}_{55}$  GLASS

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Experimental data obtained by X-ray diffraction techniques will be presented for the semiconducting glass  $\text{As}_{45}\text{Te}_{55}$ . The selection of the material satisfies the criterion that the anomalous scattering factor for one atom (Te) increases considerably from  $\text{MoK}\alpha$  through  $\text{CuK}\alpha$  to  $\text{CrK}\alpha$  radiation whereas the increase is very small for the other atom. Therefore, the method suggested by S. Ramaseshan and T. G. Ramesh (J. Phys. C: Solid State Physics, 4, 3029, 1971) for separating the partial structure factors could be employed. The experimental problems in doing this will be discussed.

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D9. ON THE RELIABILITY OF ANHARMONIC COUPLING PARAMETERS DEDUCED FROM DEBYE WALLER FACTOR STUDIES IN POTASSIUM CHLORIDE

T.M. HARIDASAN, Physics Department, Madurai University, Madurai 625 021.

Recently Solt et al (Report from International Centre for Theoretical Physics, 1972) have analysed the intensity data of 200 and 400 reflections at different parameters, from the study of Mossbauer gamma ray diffraction of KCl and estimated the anharmonic coupling parameters to be  $-6.2 \times 10^{13}$  erg/cm<sup>3</sup>. This value differs by a factor of about 6 from that given earlier by Leibfried and Ludwig. So in order to check the reliability of this recent value, a calculation of thermal expansion of KCl using this anharmonic coupling parameter was undertaken.

The theory of Timmesfeld and Elliott employing the lattice green functions has been used in the calculation. A direct comparison with very reliable experimental data on thermal expansion favours the value of Solt et al, even though their value seems to be the upperbound. The true value lies inbetween the recent value and that given by Leibfried and Ludwig. The present calculations also confirm the expectation of Solt et al that inclusions of higher order reflection data, such as from 600 would improve the value of the anharmonic parameter, deduced from Debye Waller factor studies.

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D10. THERMAL EXPANSION OF CALCIUM OXIDE

REVATHI RAGHAVAN and T.M. HARIDASAN, Physics Department, Madurai University, Madurai 625 021.

There has been very little study on the thermal expansion of alkaline earth oxides, but for the experimental work by Smith and Leider (J. appl. cryst. 1, 246, 1968) on CaO by X-ray diffraction. Recently Ruppin made a lattice dynamical calculation on a pressure dependent shell model. His results on thermal expansion, apart from being consistently higher than the above experimental results, also did not give directly any information on the anharmonic coupling parameter.

In this investigation, we have calculated the thermal expansion, apart from being consistently higher than the above experimental results,

also did not give directly any information on the anharmonic coupling parameter.

In this investigation, we have calculated the thermal expansion, explicitly using an anharmonic coupling parameter. The method of Timmesfeld and Elliott using the lattice green functions has been employed, by fitting the third order coupling parameter, first to the experimental result of thermal expansion at 300°K and then using this parameter to evaluate the thermal expansion over a range of temperatures. Our results show much better agreement with the experimental results.

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#### D11. X-RAY STUDY OF AN UNUSUAL SYNTHETIC DIAMOND POWDER

R.H. BHAWALKAR, U. DHAWAN and S.Z. ALI, National Physical Laboratory, New Delhi 110 012.

An unusual sample of imported synthetic diamond powder giving diffuse broad bands, instead of sharp Debye-Scherrer lines, has been investigated. Copper radiation with scintillation counter, pulse height discrimination and step scanning was used to record the diffraction profiles of the 111, 220 and 311 reflections. The centre of gravity of each profile was determined and corrections for transparency and flatness of the specimen, axial divergence, polarization and dispersion, were applied. No significant shift of the lines was found as compared with standard diamond pattern and thus, the observed line broadening was wholly ascribed to small crystallite size.

Half widths of the profiles, corrected for instrumental broadening by using suitable reflections of silicon powder (5-10 $\mu$ m size range), and for  $\alpha_1\alpha_2$  separation were used to find the crystallite size using the formula:

$$L_{hkl} = 0.9\lambda/\beta \cos \theta_{hkl}$$

X-ray fluorescence analysis showed presence of copper in the sample. The diamond 111 profile has not been used for crystallite size determination due to superimposition of the copper 111 profile and the proximity of another line at about 2.17 Å Bragg spacing.

The diamond crystallite size as determined from 220 and 311 profiles is of the order of 90 Å.

D12. A NEUTRON DIFFRACTION STUDY OF AMMONIUM TARTRATE

V.S. YADAVA and V.M. PADMANABHAN, Neutron Physics Section, Bhabha Atomic Research Centre, Trombay, Bombay 400 085.

A single crystal neutron diffraction study of the structure of ammonium tartrate has been carried out. The structure is monoclinic with  $a=7.083$ ,  $b=6.128$ ,  $c=8.808 \text{ \AA}$ ,  $\beta=92.42^\circ$  and  $Z=2$ . Neutron intensities of 725 reflections were measured using the diffractometer 3D-FAD. The positions of 12 hydrogen atoms were located from the nuclear scattering density map obtained using the phases from our x-ray study. The structure has been refined to an R index of 0.063. The hydrogen positions are in conformity with the x-ray study.

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D13. A NEUTRON DIFFRACTION STUDY OF AMMONIUM SULPHATE

V.M. PADMANABHAN and V.K. WADHAWAN, Neutron Physics Section, Bhabha Atomic Research Centre, Trombay, Bombay 400 085.

A single crystal neutron diffraction study of ammonium sulphate has been made in which 340 reflections were measured. The structure was refined by least squares technique. The non hydrogen atom positions obtained agree well with the x-ray parameters reported earlier by the authors. The position of the sulphamate hydrogen atoms confirm the  $sp^3$  configuration. The study also reveals the details of the hydrogen bond scheme.

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D 14. CRYSTAL AND MOLECULAR STRUCTURE OF  $N_4P_4Cl_6(NMePh)_2$

K. KRISHNA BHANDARY, Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore 560 012.

The tetrameric phosphazene  $N_4P_4Cl_6(NMePh)_2$  crystallises in the monoclinic space group  $P2_1/n$  with two formula units per unit cell of dimensions  $a=12.35$ ,  $b=16.59$ ,  $c=6.08 \text{ \AA}$  and  $\beta=93.23^\circ$ . The structure

has been solved by direct method and refined by three-dimensional least-squares procedures to a reliability index of 0.082 with anisotropic temperature factors for all the non-hydrogen atoms. The eight-membered phosphazene ring possesses a chair conformation. The details of the molecular structure with emphasis on the conformation of the (P-N) ring will be presented.

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#### D15. EVALUATION OF SATELLITE INTENSITIES IN A SPINODALLY DECOMPOSED Al-Zn ALLOY

K.G. SATYANARAYANA\* S. IELE and T.R. ANANTHARAMAN, Department of Metallurgical Engineering, Banaras Hindu University, Varanasi 221 005.

It is generally accepted that Al-Zn system transforms spinodally in the early stages of their decomposition. Such an alloy exhibits a modulated structure and side-bands will appear in its X-ray and Electron Diffraction patterns. Following an earlier approach, intensities of side-bands and main reflections of a spinodally decomposed Al-Zn alloy have been evaluated. These are in agreement with a qualitative intensity data reported for side-bands in an earlier work on Al-30 at % Zn alloy.

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#### D16. NON-DESTRUCTIVE ANALYSIS OF METAL COMPONENTS FOR Cr, W AND V BY X-RAY FLUORESCENCE TECHNIQUE

A.K. ROY, K.C. SINGHAL and B.K. BANERJEE, Planning & Development Division, Fertilizer Corporation of India Limited, Sindri, Bihar.

Non-destructive analysis of certain metal components of catalyst pelleting machine has been done by X-ray fluorescence technique. The method has been standardized for estimation of Cr, W and V with an accuracy of 2-3%. Instead of sample preparation, samples are accommodated in the sample chamber by using specially designed adaptors and irradiated by X-rays using suitable diaphragms.

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D17. TEMPERATURE VARIATION OF THE LATTICE CONSTANT  
AND THE COEFFICIENT OF THERMAL EXPANSION OF AMMONIUM  
CHLORIDE

V.K. SRINIVASA and B.K. BANERJEE, Planning & Development Divi-  
sion, Fertilizer Corporation of India Limited, Sindri, Bihar.

Using an extrapolation technique as well as Cohens least square  
method, precision determination of the lattice constant of Ammonium  
chloride has been made at different temperatures between 20 - 245°C.  
From these, by a graphical treatment, the coefficients of thermal ex-  
pansion at various temperatures have been evaluated. The transforma-  
tion of  $\alpha \rightarrow \beta$  form of Ammonium chloride around 185°C has been con-  
firmed by Guinier-Lenne high temperature X-ray diffraction technique.

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## F1. DISORDER IN THE CRYSTAL STRUCTURE OF ZINC PERCHLORATE HEXAHYDRATE

MINAKSHI GHOSH and SIDDHARTHA RAY, Department of Magnetism, Indian Association for the Cultivation of Science, Calcutta 700 032.

Crystal structure of zinc perchlorate hexahydrate resembles that of lithium perchlorate trihydrate (space group  $P6_3mc$ ) as far as the perchlorate-water arrangement is concerned. Distribution of the zinc atoms among twice their number of available sites gives rise to orthorhombic symmetry (space group  $Pmn21$ ), and crystals of this salt grow as peculiar three-component twins, where the perchlorate-water arrangement is continuous throughout a crystal, the only discontinuity being in the distribution of zinc atoms. Moreover, odd layer Weissenberg photographs taken about the  $c$ -axis contain some diffuse spots, streaking out to form a hexagonal network, so that each regular sharp spot is surrounded by a hexagon formed by the streaks. This suggests mistakes in repetition in the plane normal to the unique axis. The nature of this disorder has been worked out.

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## F2. AN X-RAY DIFFRACTION STUDY OF LATTICE IMPERFECTIONS IN COLD-WORKED SILVER-GALLIUM ( $\alpha$ -PHASE) ALLOYS

S.K. CHATTERJEE, S.K. HALDER and S.P. SEN GUPTA, Department of General Physics and X-rays, Indian Association for the Cultivation of Science, Calcutta 32.

X-ray diffraction profiles from the four compositions of  $\alpha$ -phase Ag-Ga alloys containing 3.55, 9.45, 13.05 and 15.90 atomic percentages of solute gallium have been recorded in a Geiger counter X-ray diffractometer for both the cold-worked and fully annealed states of the samples, and a detailed analysis of the line shifts, line asymmetry and Fourier line shapes have been performed considering recent improvements made in such studies. The line shift analysis from 111, 200, 220 and 311 reflections has shown the prominent effect of net stacking faults  $\alpha = \alpha' - \alpha''$  ( $\alpha'$ ,  $\alpha''$  being intrinsic and extrinsic faults respectively) over the residual stresses and this has also been observed from the shifts in the lattice parameter values ( $\Delta a_{hkl}$ ). The logarithmic dependence of  $\alpha$  with the solute concentration has been examined from the semi-empirical expressions with a satisfactory agreement. Combining the results of all

the methods of analysis, the individual concentrations of  $\alpha'$ ,  $\alpha''$  and  $\beta$  (twin faults) have been evaluated which indicates the existences of intrinsic as well as extrinsic faults with the absence of twin faults in the deformed structures.

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### F3. THE APPLICATION OF THE CONVOLUTION RELATIONS IN THE X-RAY LINE BREADTH ANALYSIS

R.K. NANDI and S.P. SEN GUPTA, Department of General Physics & X-rays, Indian Association for the Cultivation of Science, Calcutta 32.

X-ray diffraction profile resulting from microstructural changes in the materials concerned is a convolution of the instrumental and the true diffraction profile. The present series of investigations concern the simultaneous convolutions of the functional forms representing the respective profiles. For the present case, the convolution of the Schoening's true diffraction profile originating from Cauchy particle size profile and gaussian strain profile with the instrumental profile having several functional forms namely, the gaussian, Cauchy and an intermediate exponential one has been examined and applied to profiles of several cold-worked alloys and vapour-deposited silver films. A fairly good agreement for the calculated and experimental integral breadths of the profiles has been observed for the case of a gaussian instrumental functions. The analysis gives a confirmation on the functional forms assumed and also on the Schoening's particle size and strain parameters.

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### F4. ON THE SIZE-FACTOR DEPENDENCE OF STACKING FAULT DENSITY IN COPPER- AND SILVER-BASE ALLOYS

M. DE and S.P. SEN GUPTA, Department of General Physics and X-rays, Indian Association for the Cultivation of Science, Calcutta 32.

A critical analysis of the size-effect (i.e., the effect of size difference between solvent and solute atoms) has been made in order to see its influence on the microstructural change (namely, concentrations of stacking faults) in deformed binary alloys of copper and silver. Size-

factor values, SF have been evaluated from several considerations of atomic sizes for fairly large number of copper- and silver-base binary alloys. Plots of these SF values against the stacking fault density,  $\alpha$  of these alloys at different electron/atom ratios showed, in general, a linear dependence with increasing slopes at higher  $e/a$ . From these observations it may be inferred that size-effect plays an important role in controlling the substructure of these deformed alloys.

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#### F5. AN X-RAY LINE SHIFT ANALYSIS IN VACUUM-EVAPORATED COPPER FILMS

SUCHITRA SEN, S.K. HALDER and S.P. SEN GUPTA, Department of General Physics & X-rays, Indian Association for the Cultivation of Science, Calcutta 700 032.

Thin films of spectroscopically pure copper having thicknesses 1431 Å, 1884 Å, 2112 Å, 2491 Å and 3174 Å were prepared on glass substrates using an eccentric source in a vacuum coating unit at a residual pressure of  $\sim 10^{-5}$  Torr. X-ray diffraction profiles for the reflections 111, 200, 220, 311 and 222 were recorded by a point counting method in the Philips Counter X-ray diffractometer. The peak positions of the first four reflections were determined by a parabolic fitting method and from the measurements of the relative peak shifts of adjacent pairs of reflections, the contributing factors namely the lattice parameter changes, the residual stresses and stacking fault (intrinsic and extrinsic) concentrations were evaluated. It has been observed, unlike the already reported silver films, that for relatively thin films the residual stresses  $\sigma$  ( $\sim 24 \times 10^4$  psi) in the plane of the films are tensile in nature. The net stacking fault probability  $\alpha = \alpha' - \alpha''$  ( $\sim 20 \times 10^{-3}$ ) indicates the predominance of intrinsic over the extrinsic ones. There is also an increase in the lattice parameter values for the thin films which approach the bulk value with increasing thickness. Details of the analysis will be presented

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F6. EFFECT OF LATTICE PERTURBATIONS ON PSEUDO-KIKUCHI PATTERNS IN THE SCANNING ELECTRON MICROSCOPE

S.K. BHAMBRI, Materials Science Division, National Aeronautical Laboratory, Bangalore 560 017.

The effect of mechanical damage and surface oxides on lattice perfection has been studied by utilizing orientation dependent Pseudo-Kikuchi patterns from bulk single crystals in the Scanning Electron Microscope. 99% purity aluminium sheets were strain annealed to obtain large grains. Specimens containing single grains were cut by abrasive cutter and by spark erosion separately. Stereoscan Mark II was used to obtain Kikuchi patterns. Large damage caused by abrasive cutting process is evident from quality of patterns. In another series of experiments, a chemically polished single crystal specimen was annealed at different temperatures for an equal period of time. The Kikuchi patterns as observed at room temperature and by intensity measurements from a particular band indicate deterioration in quality with increased layer of surface oxides. The quality of Pseudo-Kikuchi patterns permits rapid evaluation of the extent of lattice perturbations.

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F7. THE GROWTH AND CHARACTERISATION OF STRONTIUM FLUOROCHLORIDE SINGLE CRYSTALS

H.L. BHAT and P.S. NARAYANAN, Department of Physics, Indian Institute of Science, Bangalore 560 012.

A preliminary report on the growth of single crystals of strontium fluorochloride is given in this paper. The growth was accomplished by heating a stoichiometric mixture of analytical grade  $\text{SrCl}_2$  and  $\text{NaF}$  to a temperature of  $850^\circ\text{C}$  and allowing them to react in the molten state to form  $\text{SrFCl}$  and  $\text{NaCl}$ . The slow cooling of the molten salt solution resulted in the crystallization of  $\text{SrFCl}$ , the only stable solid phase under the prevailing growth conditions.

The X-ray analysis of the crystal shows that it belongs to the tetragonal space group  $P4/nmm$  with the following unit cell dimensions  $a = 4.079 \pm 0.008 \text{ \AA}$  and  $b = 6.914 \pm 0.01 \text{ \AA}$ . The crystal is likely to be isostructural with  $\text{BaFCl}$ . The calculated density for  $\text{SrFCl}$  with two molecules in its unit cell ( $4.102 \text{ g cm}^{-3}$ ) agrees with the reported value ( $4.18 \text{ g cm}^{-3}$ ).

CHI The synthetic crystals are of tabular habit parallel to (001) and  
where of average size 6 mm x 3 mm x 0.2 mm. The microscopic exami-  
bo- nation of the habit faces has revealed the presence of dendritic growth  
patterns indicating the attainment of high degree of supersaturation in  
the last stages of growth as is often the case of flux growth. The crystal  
exhibits profound cleavage along (001). The quality of the crystals was  
assessed by the technique of selective etching of the cleaved surfaces.

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## F8. VARIATION OF THE SIZE EFFECT WITH COMPOSITION IN MIXED CRYSTALS

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It is well known that in mixed crystal systems there will be a dis-  
s- tortion of the electron density, giving rise to a size effect ( $\exp(-2M')$ ),  
which reduces the x-ray diffraction intensities without broadening them,  
analogous to the Debye Waller factor ( $\exp(-2M)$ ). For an ideally disor-  
dered binary system AB

$$2M' \approx 48 \frac{x}{1-x} (V_A^{1/3} - V_{AB}^{1/3})^2 \frac{\sin^2 \theta}{\lambda^2} \quad \dots \text{Weiss, 1966}$$

where x is the fractional compositions of A in AB and V is the atomic volume.

For such a system, it is readily deduced from the above relation that the size effect will be a maximum for the intermediate composition,  $x = 0.5$  and fall off parabolically. This has been studied for the system  $\text{KBr}_x \text{Cl}_{1-x}$ , for different values of x. Single crystal measurements were obtained on a spectrometer (Mohanlal, 1971) while the measurements on polycrystalline samples were carried out on a GE XRD-6 diffractometer (Marquette University, Wisconsin, USA). The analyses of the independent measurements confirm the above expectation.

A 'virtual crystal model' for the Debye Waller factors in such mixed systems has been worked out in our group (Haridasan and Revathi Raghavan) and attempts are in progress to take the size effect into such a model.

F9. X-RAY DEBYE WALLER FACTORS IN MIXED CRYSTALS

VISPY D. DOCTOR, REVATHI RAGHAVAN, T.M. HARIDASAN and K.S. CHANDRASEKARAN, Department of Physics, Madurai University, Madurai 625 021.

The lattice spectra of some mixed crystals,  $A_xB_{1-x}$  show a "one mode", with a single peak which shifts with the  $x$  value, while, for certain other systems, a "two mode" is observed with 2 peaks which vary in height but without shift in their positions. The criteria have been reviewed (Mitra), and compared with experimental work for a wide variety of cases.

We have been interested in the behaviour of the X-ray Debye Waller factors in systems showing a one mode ( $KBr_xCl_{1-x}$ ) and two mode ( $K_xRb_{1-x}I$ ). On the theoretical side, utilising the Green's functions, computer calculations have been made for the Debye Waller factors of the end members and using further a virtual crystal model for the mixed systems. These calculations are compared with data already available (Mohanlal et al) and other measurements in progress.

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F10. STUDIES ON THE VAPOUR PHASE DEPOSITS OF NICKEL SULPHIDE

A. GOSWAMI and G.D. TALELE, National Chemical Laboratory, Poona 411 008.

A detailed study has been made on the vapour phase deposits of nickel sulphide formed on (100), (110) and (111) faces of NaCl and also on polycrystalline sodium chloride tablets at different temperatures (25°-500°C). These deposits, however, showed phase transitions at different temperatures and a ZnS type of structure (a 5.4 Å) along with its hcp form was observed between 150° to 350°C. But at a higher temperature region another new phase presumably of spinel structure (a 8.90 Å) appears to form. All these deposits however grew epitaxially with appropriate orientations at suitable temperature of the substrates.

## F11. KINETICS OF GROWTH OF POTASSIUM DIHYDROGEN PHOSPHATE (KDP) CRYSTALS

M.S. JOSHI and BABY K. PAUL, Department of Physics, Sardar Patel University, Vallabh Vidyanagar (Gujarat).

Mechanism of growth of KDP crystals was studied from measurements on the kinetics of growth of crystals grown from pure aqueous solutions, at a constant temperature ( $30^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ ), in static and dynamic systems, at different supersaturations ( $3.7 \times 10^{-2}$  to  $35 \times 10^{-2}$ ). In the dynamic system mother liquor was stirred at a constant rate (80 r.p.m.) and in the static system it was not stirred. Seed crystals were prepared from a supersaturated solution ( $\sigma = 11.1 \times 10^{-2}$ ) by slow solvent evaporation. Measurements on kinetics of growth of [010] and [011] faces were carried out on selected seed crystals ( $3 \times 3 \times 5$  mm). Three methods were employed for kinetics measurements: (i) linear growth rate ( $dl/dt$ ) measurements by a cathetometer, (ii) linear growth rate measurements from photographs of crystals and (iii) overall growth rate ( $dm/dt$ ) measurements by weighing crystals. For each method, graphs of growth rate (R) against supersaturation ( $\sigma$ ) were plotted, for each system. R was found to be a parabolic function of  $\sigma$  at low  $\sigma$  values ( $< 7.5 \times 10^{-2}$ ) and linear at high  $\sigma$  values ( $> 7.5 \times 10^{-2}$ ) for both the systems. Results are explained on the basis of surface diffusion theory by Burton, Cabrera and Frank.

Influence of agitation of the mother liquor (shear) on the growth rate was also studied. Growth rates of [010] and [011] faces were found to be several magnitudes greater in the dynamic system than that in the static one, for identical conditions of temperature and supersaturations.

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## F12. STUDIES OF DISLOCATIONS IN MOLYBDENUM DISELENIDE USING TRANSMISSION ELECTRON MICROSCOPE

M.K. AGARWAL and BABU JOSEPH, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

The use of transmission electron microscopy for the direct observation of dislocations in thin foils was developed by Hirsch and co-workers. Since then a large number of substances have been studied using this technique. We report here some observations of dislocations

in molybdenum diselenide.

MoSe<sub>2</sub> has been selected because it has a pronounced cleavage along the basal plane. This has made it possible to prepare foils which will be thin enough to transmit electron beam and exhibit dislocation configuration in the plane of observation.

The specimens were examined in the electron-optical plant EF(4) manufactured by Carl Zeiss Jena at 65 K. V.

It is observed that dislocations occur as (i) two fold ribbons having different stacking fault widths (ii) extended nodes (iii) undissociated dislocations.

Since ' $\mu$ ' for MoSe<sub>2</sub> was not known a quantity ' $\gamma/\mu$ ' which is proportional to the stacking fault energy ' $\gamma$ ' has been obtained from the width of the dislocation ribbons and from the radii of the extended nodes. From these values the dislocations are classified into three different types:

Type	' $\gamma/\mu$ '
I	' $\gamma/\mu$ ' $\geq 10^{-11}$ cm.
II	$4 \times 10^{-12}$ cm $< \gamma/\mu < 5.6 \times 10^{-12}$ cm.
III	$12 \times 10^{-12}$ cm $< \gamma/\mu < 21 \times 10^{-12}$ cm.

A possible explanation for the existence of three different types of dislocations has been suggested.

### F13. ETCHING OF CUBIC ZnS

S.M. PATEL and VERGHESE GEORGE, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

Natural and flux-grown crystals of cubic ZnS were subjected to the etching studies. Using etchants composed of mineral acids and alkalis, it is shown that the etch pits develop at the sites of dislocations. By etching matched pairs, and isolated cleavages successively for different periods, the configuration of dislocations has been worked out. The stepped nature of many of the etch pits has been attributed to the

jogging of dislocations. By subjecting the [111] faces of the flux grown ZnS crystals to the above etchants, slip traces and volume indentation has been revealed. The slip is formed due to the complex state of thermal stress introduced during cooling whereas the volume indentation, due to the thermal stress arising from the difference in thermal expansion between that of the crystal and the impurity. Twinning in natural sphalerite crystals has been revealed by etching. By heating the crystal cleavages in vacuum at about 800°C for 1 hour, thermal etch pits have been produced. It has been found that thermal etch pits are not formed at all types of dislocation sites. The implications are discussed.

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#### F14. DISLOCATIONS IN FORSTERITE ( $Mg_2SiO_4$ ) CRYSTALS

A.R. PATEL, C.C. DESAI and JOHN V., Physics Department, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

Single crystals of Forsterite have been obtained from Dr. C. B. - Finch of Oak Ridge National Laboratory, USA. They are cleaved along (010) planes. The cleavage faces are etched in hydrochloric acid, nitric acid, chromic acid and hydrogen peroxide. The etch patterns obtained are studied optically. The etch pattern consists of flat and point bottomed rectangular pits. Symmetric as well as asymmetric pits are observed. Correspondence in the etch pattern has been established on matched cleavage faces. It has been observed that the shape of the pits differ when they are etched in acids and in hydrogen peroxide. By etching a cleavage face successively for different periods it is shown that individual isolated pits are produced at the sites of dislocations. When cleavage faces are thermally etched at about 1700°C in vacuum, square shaped pits are produced. Rows of pits along with the normal etch patterns have been observed. Correspondence of the rows of pits is established on the matched faces. The implications are discussed.

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F15. EXPITAXIL GROWTH OF AlSb ON NaCl

A.R. PATEL and S. MOHANA, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

AlSb is the only III-V semiconductor containing Al which has been investigated. Though it is a good rectifier, it generally forms unstable alloy, especially in thin film form. The film has been grown on glass and Ge at elevated temperature by different workers which is highly fragile and decomposes in humid air. After a series of experiments, the authors are successful not only in growing strong and stable AlSb alloy film, but also the alloy film has been grown epitaxially on NaCl cleavage, by successive evaporation of Sb and Al at room temperature. Other alkali halides and mica have also yielded epitaxial films when used as substrates. The effect of substrate temperature, changing the sequence of evaporation, velocity of vapour atoms, annealing and ageing of the alloy film etc. on the nature and structure of the alloy film has been investigated by electron diffraction. [110], [111], [211] orientations are frequently observed and rarely [100] is also met with. Implications are discussed.

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F16. GROWTH OF SINGLE CRYSTALS OF CADMIUM TUNGSTATE MAGNESIUM TUNGSTATE AND ZINC TUNGSTATE BY DOUBLE DECOMPOSITION IN MELT

A.R. PATEL and K.M. SUTHAR, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

The suitability of double decomposition reaction in molten phase for the growth of good quality single crystals of cadmium tungstate, magnesium tungstate and zinc tungstate has been investigated. The crystals grown have been identified by X-ray analysis and chemical analysis. While the crystals of cadmium tungstate are of yellow colour, bounded by smooth and well-developed (010) and (100) prism faces with C-axis as the needle axis, the crystals of magnesium tungstate and zinc tungstate are colourless transparent and grey respectively. A suitable etchant has been developed for revealing the dislocation sites. Studies made on chemical etching of the cleavages have revealed that the crystals are relatively perfect. The detailed microtopographical, cleavage and etching studies made on cadmium tungstate are described. The implications are discussed.

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## F17. GROWTH OF SINGLE CRYSTALS OF CADMIUM TUNGSTATE IN GEL

A.R. PATEL and K.M. SUTHAR, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

Single crystals of cadmium tungstate upto 3 mm in length have been grown in silica gels and identified. While 'impure' gel has been found to produce large many small crystals, good quality single crystals have been obtained by using 'pure' gel. The amount of gel inclusion is found to increase with the increasing size of the crystal producing opaque and poorly faceted crystals. Some aspects, such as effect of pH value, temperature and concentration changes in reactants, have been studied and described. The detailed microtopographical studies made on cadmium tungstate are described. The implications are discussed.

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## F18. COMPARATIVE STUDY OF FLUX-AND GEL-GROWN CRYSTALS OF BARIUM AND STRONTIUM TUNGSTATES

A.R. PATEL and S.K. ARORA, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat State.

Isothermal flux-evaporation method employing a polycomponent flux ( $\text{NaCl-LiCl}$ ) and  $\text{Na}_2\text{WO}_4$  alone as flux has been successfully applied by the authors to grow  $\text{BaWO}_4$  and  $\text{SrWO}_4$  crystals which have also been grown by using the gel technique. It is the primary purpose of this paper to present a comparative study of the crystals grown by these two different methods.

### Flux growth:

Studies on the microstructures of the finished faces reveal two-dimensional layer mechanism of crystal growth. Dendrities are observed on the faces of the crystals grown between 800-900°C. The plastic deformation observed in some crystals has been studied and described.

### Gel growth:

The crystals grown in 'impure' gels showed the development of spherulites which grow larger and opaque when the diffusion of feed

solutions was faster. The crystals grown in 'pure' gels, however, were devoid of any surface structures.

Distinctive conclusion:

While the flux-grown crystals showed on their habit faces several features, such as hopper-formation, dendrites, etch-pittings, voids, cavities, over-growths, etc., the faces of gel-grown specimens were flat and smooth.

Further, the gel-grown crystals were found to be impressively transparent as against flux-grown ones which were less translucent. The dislocation density, as determined by the etch-pit count method, indicated that the flux-grown crystals contained more dislocations.

Thus the paper concludes that the gel-grown crystals are far better than and hence superior in quality to those grown using flux methods in so far as the crystal-size and perfection both are concerned.

#### F19. LATTICE ENERGIES OF KCl-KBr SOLID SOLUTIONS

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The lattice energies of different compositions of KCl-KBr mixed crystals grown in our laboratory were calculated using the expression

$$U/N = -\frac{ae^2}{r_0} (1 - \rho/r_0) \quad (1)$$

where  $a$  is Madelung constant,  $\rho$  is the repulsive range parameter and  $r_0$  is the interionic distance.

The lattice parameters for the above mixed crystals were determined by X-ray powder method using least square fitting. The repulsive range parameter  $\rho$  was calculated using the equation

$$\rho = ae^2/18(B r_0^3 + ae^2/9 r_0)$$

where  $B$ , the bulk modulus is calculated from the expression  $B = 1/3 (C_{11} + 2C_{12})$ .

re The elastic constants  $C_{11}$  and  $C_{12}$  were determined by ultrasonic pulse technique.

The lattice energy of the mixed crystal can be expressed as  $U = C_1U_1 + C_2U_2 \dots$  (2) where  $C_1$  and  $C_2$  are mole fractions,  $U_1$  and  $U_2$  are lattice energies of KCl and KBr respectively. The values of lattice energy calculated on the basis of equations (1) and (2) were found to be in good agreement for all the compositions. The lattice energy is found to be varying linearly with composition of KBr in KCl.

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## F20. DETERMINATION OF STRAIN DERIVATIVES IN COPPER-ZINC ALLOYS AND THIN FILMS OF SILVER

G. B. MITRA and A. K. CHAUDHURI, Department of Physics, Indian Institute of Technology, Kharagpur 721 302.

The method of Fourier coefficients of x-ray diffraction line profile developed by Mitra and Chaudhuri (J. Appl. Cryst. 7(1974), 350), has been applied for determining the average strain derivatives  $\langle e.e' \rangle$  in case of Copper-Zinc alloys containing 70% by weight of copper, coldworked and annealed at various temperatures. A single line technique of determining the strain gradients has also been developed for the cases of thin films of different thicknesses prepared both by vapour and chemical deposition method. The different results so obtained have been discussed. It is observed that the strain gradients, so long neglected, give considerable contribution to line broadening. It has also been shown that this parameter helps in distinguishing between edge and screw distribution.

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## F21. STUDY OF AN ASBESTOS MINERAL

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A specimen of fibrous asbestos from Jhirkpani mines of Singhbhum district in Bihar, was selected for the present work. The sample was subjected to X-ray and electron microscopic studies for the proper identification of the particular group of mineral comprising the asbestos

and its structural characteristics under different tensions.

For X-ray study, a simple Laue type camera with a convenient sample holder suitable for fibre photograph was designed and fabricated. The sample holder had provisions for stretching the fibre to different extent as well as for inclining the fibre axis with respect to the incident X-ray beam to different degree which could be measured precisely.

Results of electron microscopic study reveals that the sample consists of long cylindrical particles of uniform diameter of average length  $2 \times 10^{-4}$  cm. and average diameter  $5 \times 10^{-5}$  cm. X-ray study shows that the mineral belongs to the amphibole group. However, the major component is tremolite amphibole with traces of Nephrite. The measurement from fibre photograph gives the following cell dimensions  $a = 9.17 \text{ \AA}$ ,  $b = 17.18 \text{ \AA}$ ,  $c = 5.055 \text{ \AA}$  (fibre axis)  $\beta = 106^\circ$ . The effect of stretching and other characteristics have also been discussed.

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## F22. X-RAY STUDIES OF LATTICE DEFECTS AND THERMAL VIBRATIONS OF TELLURIUM

N.K. MISRA and A.K. CHAUDHURI, Department of Physics, Indian Institute of Technology, Kharagpur, India.

The study of structural defects and Thermal vibrations of Tellurium is of great importance for its semiconducting property. The useful technique of X-ray diffraction analysis has been employed to determine these effects. The geiger counter diffractometer has been used to obtain the X-ray diffraction profiles from tellurium powders by point counting method. The composite line broadening has been attributed to effective particle size and lattice strain. Line shape analysis has been carried out to determine the defects along (100) and (101) directions only because along other directions multiple reflections could not be obtained very precisely. From integrated intensities of the reflections root mean square amplitudes of the atoms along A and C-axes have been determined for both coldworked and annealed powders.

F23. A FAULTED-MATRIX MODEL FOR THE SPIRAL GROWTH OF POLYTYPE STRUCTURES. I CADMIUM IODIDE POLYTYPES

DHANANJAI PANDEY and P. KRISHNA, Department of Physics, Banaras Hindu University, Varanasi 221 005.

The origin of polytype structure has been attributed to spiral growth round suitable screw dislocations created in a basic structure. In deducing the different polytype structures that can result from such a mechanism, the basic structure has been assumed to be perfect. It is shown that it is necessary to consider the possibility that the parent matrix may contain stacking faults near its surface at the time of the origin of the screw dislocation ledge. This can drastically affect the structure of the resulting polytype.

The most probable fault configurations that can occur in a parent 4H or 2H structure of  $\text{CdI}_2$  have been deduced from a calculation of the stacking fault energies. Polytype structures originating from single screw dislocations created in a faulted 2H or 4H matrix containing one of the more probable fault configurations near the surface have been deduced. It is shown that all the observed polytype structures of  $\text{CdI}_2$  can result from such a mechanism and represent structures with minimum or close to minimum stacking fault energy. There is an excellent agreement between the structures deduced on the basis of the faulted matrix model and those actually observed. It has been possible to explain the origin of the observed structure series in  $\text{CdI}_2$  and the limitation of the Zhdanov numbers to 1, 2 and 3. It is possible to predict theoretically the more probable structures for a polytype which greatly simplifies its structure determination.

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F24. A FAULTED-MATRIX MODEL FOR THE SPIRAL GROWTH OF POLYTYPE STRUCTURES. II SILICON CARBIDE POLYTYPES

DHANANJAI PANDEY and P. KRISHNA, Department of Physics, Banaras Hindu University, Varanasi 221 005.

The faulted matrix model of polytypism suggested in the previous paper has been applied to the deduction of  $\text{SiC}$  polytypes. It is shown that all the  $\text{SiC}$  polytypes so far regarded as 'anomalous' can result from an appropriate screw dislocation ledge exposed in a 6H, 15R or 4H matrix. The probable fault configuration in each of these parent structures

have been determined from a calculation of stacking fault energy. The most frequently occurring structure series in SiC are those which have the lowest stacking fault energy. Thus, there is an excellent agreement between the polytype structures expected on the basis of the faulted matrix model and those actually observed. The limitation of the Zhdanov numbers to 2, 3 and 4 in a polytype structure is explained. It is possible to predict the more probable structures for a polytype on the basis of the above model for determining structure.

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#### F25. CHEMICAL ETCHING AND X-RAY DIFFRACTION STUDIES OF SILICON CARBIDE CRYSTALS

U. S. RAM and G. SINGH, Department of Physics, Banaras Hindu University, Varanasi 5.

Silicon carbide crystals have been successively etched for different durations. The (0001) face of the crystals were examined optically and photographed at each stage of etching. Similarly x-ray diffraction Laue patterns were recorded at every stage. Growth characteristics and defect structure of Silicon carbide polytypes have been studied from these observations.

Usually initial Laue photograph from an unetched crystal contains spots characteristic of more than one polytypic structures. After gradual etching, spots due to individual structure disappear one by one. In another category of crystals whose Laue photographs initially contain sharp spots due to syntactic structures superimposed by continuous streaks, it was found at a later stage that streaks disappear leaving the characteristic spots unchanged. It is finally concluded that these streaks are not actually due to one dimensional disorder present in polytypic structures, rather they arise because of thin disordered layers sandwiched between two ordered structures.

The defect structures revealed by etch pits has been discussed.

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F26. ROLE OF ISLAND FORMATION ON THE GROWTH OF SILICON CARBIDE POLYTYPES

U.S. RAM and G. SINGH, Department of Physics, Banaras Hindu University, Varanasi 5.

Silicon carbide is known to crystallize in several crystallographic modifications. The screw dislocation mechanism of polytype formation explains most of the structural features of SiC polytypes satisfactorily. However, there are certain experimental observations that cannot be accounted for in terms of a purely screw dislocation mechanism. The feasibility of periodic slip process which plays an important role in the growth of ZnS polytypes, as a possible mode of growth of SiC polytypes has been examined. It is shown that it does not seem to be an appropriate mode of growth for SiC polytypes. It is proposed that the nucleation of islands on the helicoidal growth front created due to a screw dislocation, is responsible for the growth of most of the unusual SiC polytypes. According to this proposition, most of the unusual features of SiC polytypes are explained satisfactorily.

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G1. A HIGH TEMPERATURE WEISSENBERG CAMERA FOR SINGLE CRYSTAL X-RAY DIFFRACTION STUDIES

A.M. SHAIKH and M.A. VISWAMITRA, Department of Physics, Indian Institute of Science, Bangalore 560 012.

A high temperature Weissenberg camera for X-ray diffraction analysis of single crystals in air, vacuum or inert atmosphere, constructed in the laboratory has been described. Details are given of a vacuum chamber, heating attachment, film cassette, translational gear system. The apparatus enables back reflection oscillation and Weissenberg photographs up to an equi-inclination angle of  $30^\circ$ . It is especially suitable for high precision lattice constant and structure determination. At present it can be used for single crystal studies up to  $1000^\circ\text{C}$  in air atmosphere and up to  $600^\circ\text{C}$  in vacuum.

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G2. HIGH TEMPERATURE X-RAY CRYSTALLOGRAPHY DEVICE FOR SINGLE CRYSTAL ANALYSIS

M.A. VISWAMITRA, A.M. SHAIKH and K. JAYALAKSHMI RAMANUJA, Department of Physics, Indian Institute of Science, Bangalore 560 012.

Various single crystal heaters for X-ray diffraction work developed since 1964 have been compiled in this review article, in the form of a bibliography. A general outline of various heating methods, temperature measurement, calibration and control, specimen holders and adhesives is given as a background to the bibliography.

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G3. CHOICE OF SCANS IN NEUTRON DIFFRACTION

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The conditions under which unbiased measurements of neutron Bragg intensities can be carried out with the use of various modes of scan including Werner's optimum scan (Acta Cryst. A27, 665, 1971) have been examined. General expressions for the scan widths, the divergences

of the integrated intensity distributions sensed by the detector, and the fractional loss in the measured integrated intensity due to the finite size of the detector aperture for different modes of equatorial scans have been derived. It is shown that in the conventional  $\theta:2\theta$  mode of scan undistorted estimates of Bragg intensities can be obtained with any fixed angular aperture in front of the detector whereas in Werner's optimum scanning mode, although the required detector aperture is a minimum it changes from reflection to reflection. The fact that the relative intensities of Bragg reflections in the  $\theta:2\theta$  mode are independent of the angular aperture in front of the detector has been experimentally verified. Simplified expressions for the optimum scanning ratio  $g$ , the width of the crystal rocking curve  $\sigma$  and the width of the Bragg scattered beam  $\zeta$  have been derived. They have also been experimentally evaluated for a typical neutron diffractometer and found to be in good agreement with theoretical predictions.

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#### G4. METHOD OF INCREASING THE DETECTED INTENSITY IN X-RAY DIFFRACTOMETERS

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A method is proposed to increase the intensity detected in a powder diffractometer by increasing the width of the detector slit. This method will be of use in line profile analysis and in the study of liquids and amorphous solids. The error introduced in the measured intensity by increasing the slit width can be corrected for by expanding the intensity as a Taylor series in the scattering angle. To the second order, the correction is proportional to the curvature of the measured intensity. For higher order corrections, the higher Taylor coefficients of the measured intensity should be determined. Alternately, it is shown that with two different slit widths, a correction to fourth order can be made by determining only up to the second Taylor coefficient at each width. An error analysis has been made and it indicates that in most practical cases the statistical fluctuations in the corrected intensity is less than that in the measured intensity. The measured intensity can be still further increased by removing the Soller slits in the detector assembly. The full analysis of this geometry has been carried out and here the correction depends on both the slope and the curvature of the measured intensity. This last modification is most useful in the study of liquids and amorphous solids. The above methods have been applied with success to the study of a binary glass.

G5. LASER OPTICAL TRANSFORMS OF ELECTRON MICRO-  
GRAPHS AND CONTACT MICRO-RADIOGRAPHS OF TOBACCO  
LEAVES INFECTED BY VIRUSES

G. B. MITRA, B. K. SAMANTARAY and H. BHATTACHARYA, Depart-  
ment of Physics, Indian Institute of Technology, Kharagpur 721 302.

For studying the nature and distribution of components in biological specimens in situ, a new technique has been developed. It consists of obtaining CMR's of the samples by scanning it with x-ray microbeams of different wavelengths. For this purpose a new type of x-ray micro-radiograph apparatus capable of selecting beams of the necessary wavelength has been constructed. The CMR's so obtained are now scanned with the beam from a He-Ne laser and optical transforms of the different parts of the CMR's are recorded. Since the CMR's can be considered to be proportional to the electron distribution over the areas studied, the optical transforms will be equivalent to low angle diffraction patterns of the components of this sample. Thus a nondestructive scanning diffraction study of the sample is achieved. Theoretical investigation of the information thus obtained has been made. This technique has been applied to tobacco leaves unaffected and affected by TMV. In the regions where TMV concentration is high, characteristic diffraction patterns have been observed. Similar studies with transmission electron micrographs have also been made and the results are found to be similar.

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G6. ON THE OPTICAL TRANSFORMS OF THE ELECTRON-  
MICROGRAPHS OF BIO-MOLECULES

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The importance of small angle x-ray scattering and fibre photographs for obtaining informations regarding the structure of bio-molecules is well known. However, sometimes one faces difficulties in separating good fibers and does not obtain the desired results. Selective area small angle electron diffraction can be used for obtaining the same information but it has not been used extensively because very few laboratories can afford the same. In the present investigation the optical transforms of the electron micrographs of the biomolecules

have been used as an alternative tool for obtaining the same information. The optical transforms were obtained using an optical diffractometer with a He-Ne laser as the source, the detail of the construction being same as that presented earlier (Mohanty et.al., conference on non-metallic solids, Madras 1974). The obtained results agree fairly with the known structures. The effect of the various aberrations on the electron microrgraphs and hence on the optical transforms shall also be presented.

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G7. PERIODIC VARIATION WITH ANGLE OF SCATTERING OF T.D.S. FROM PIEZOELECTRIC CRYSTALS PLACED IN OSCILLATING ELECATRIC FIELDS

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It is well known that T.D.S. depends upon elastic constants of a crystal and it is equally well known that for piezoelectric crystals the elastic constants vary with applied electric fields. To study this variation in quartz crystals TDS from (10T) face of a quartz crystal subject to and free from an applied electrical field of strength 10 volts, frequency  $6.87 \times 10^5$  c.p.s. has been studied. While the field free sample gave the expected type of T.D.S., the crystal, when subject to the oscillating electric field gave rise to T.D.S. varying periodically with the angle of scattering. This effect cannot be explained on the basis of the usual kinematic theory. By using the dynamical theory of diffraction, this effect has been explained. Thus this effect is of the same category as Borrmann effect, and Pendulum effect which can be explained only on the basis of the dynamical theory of diffraction.

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G8. A NOTE ON THE ACCURATE DETERMINATION OF CELL DIMENSIONS

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Lattice constants can be determined to a high degree of precision with equatorial Weissenberg pictures taken about different crystallographic axes. The measured  $\sin^2\theta$  values for the common reflections from different pictures do not, generally, agree well in spite of the best precautions. To bypass this difficulty it is suggested that all the patterns for various axes be recorded on the same film. This can conveniently be carried out on a Weissenberg camera provided with two film cassettes. On a single film contained in one cassette are recorded the patterns for the various axes. The second cassette is used for aligning the crystal specimens about other axes as soon as the exposure about one axis is complete on the first cassette. The common spots can be made either to appear side by side or to coincide exactly by adjusting the drum readings of the camera. Finally, a powder pattern of some standard material is recorded at one end of the film for obtaining the film radius accurately. Even though the spots on the film thus obtained will, no doubt, be overcrowded, measurements of  $\sin^2\theta$  can easily be made because separate indexed Weissenberg pictures about all the axes are available. In this way uncertainty in the agreement of the measured  $\sin^2\theta$  values can be overcome. Errors due to shrinkage would be common for all the patterns since they appear on the same film. Where two cassettes are not available, the samples can be aligned on separate goniometer heads. Later the patterns can be recorded on the same film by replacing the goniometer head, immediately after the exposure about one axis is complete, by another one containing an aligned specimen about a different axis. The cell dimensions of Lead Acetate Trihydrate were determined using the principle mentioned above.

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G9. EQUIPMENT FOR THE GROWTH OF SINGLE CRYSTALS OF ORGANIC COMPOUNDS

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Three apparatuses have been designed and constructed on the principle of zone melting technique for purification and growth of single

crystals of organic compounds. During the growth of a single crystal, mechanical shocks, erratic container movement and other effects (due to traversal of the crystal container) which tend to disturb the liquid-solid interface of the growing crystal, may be prevented by keeping the crystal container fixed. Based on this principle, in the case of first apparatus which has a single heating zone the sample container remains fixed horizontally and only the heating zone moves, traversing coaxially with the sample tube. In the other two apparatuses having ten heating zones each but varying in heating elements, the sample container traverses through the heating zones and in this case heating zones remain stationary. The multi-heating zone method employed in these two apparatuses is superior to the single zone method because it speeds up the processes of purification and the growth of single crystals. Furthermore, the chance phenomenon to initiate the growth of single crystal increases. By making use of these apparatuses single crystals of a number of dihalogenated benzenes have been grown and the largest single crystal grown so far is 25 cm. in length and 14 mm. in diameter.

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#### G10. ELASTIC CONSTANTS OF SILICON CRYSTALS USING A HIGH RESOLUTION X-RAY DIFFUSE REFLECTION TECHNIQUE

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We have used essentially a transmission X-ray topographic set up for measurement of intensities of diffuse X-ray reflections to determine elastic constants. In the conventional method developed by Ramachandran and Wooster the spread of diffraction spot or the width of the diffraction curves is in degrees and diffuse reflection measurements are carried out at angular separation of several degrees away from the peak position. In the present method the half width of the diffraction curve is about one minute of arc and the rotation of few seconds of arc can be given to the crystal. Diffraction peaks due to  $K\alpha_1$  and  $K\alpha_2$  components of the characteristic radiations are well resolved and measurements can be carried out around any one of the peaks. For diffuse reflection measurements successive rotations of 10-20" of arc are given to the crystal. Corresponding angular shift for detector for measurement along a particular rekha has been calculated analytically. Results of measurements made on [001] silicon single crystal wafers will be reported.

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## H1 FERROELASTIC PHASE TRANSITIONS

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The role of ferroelasticity in crystallographic phase transitions in solids has acquired considerable significance. A prototype "species" 422F4 has been examined from first principles, for ferroelastic or non-ferroelastic behaviour. After giving Aizu's definition of ferroelasticity in crystals, using Landau's theory and second order phase transitions, the stringent condition for ferroelasticity in a crystal has been derived. The orientational states, coercive stress and the principal feature which makes a crystal ferroelastic have been written down for all the known ferroelastics. An examination of the point groups of paraelastic and ferroelastic phases with respect to their centricity and non-centricity (Cracknell, 1972) shows for example, that a ferroelastic phase derived from the centrosymmetric point group has necessarily to be centrosymmetric. Other factors like the movement of the domains by external stresses alone, spontaneous birefringence (anomalous), a soft mode at the transition temperature have been taken into account and ferroelasticity has been predicted in a few crystals. Finally, it is shown that like "improper ferroelectrics", 'improper ferroelastics' also exist (Tolenado, Pateau 1974) and the much debated displacive phase transition in  $\text{SrTiO}_3$  at  $106^\circ\text{K}$  is probably nothing but "weak ferroelasticity" of faintness index two.

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45, 4, p 1611.

## H2. RECRYSTALLIZATION AND PHASE TRANSFORMATION IN THIN FILMS OF SiC

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Thin films of SiC have been prepared by electron beam evaporation and examined under Electron Microscope. A single crystal (6H) of SiC was evaporated to prepare different films on formvar coated glass slides. As grown films give a uniform features in the electron micrograph and the corresponding electron diffraction patterns indicate non-crystalline nature of films. However, after pulse annealing the films at different pulse currents, crystallization starts showing the following features.

1 The annealing behaviour of SiC films deposited on a Formvar layer indicates the simultaneous nucleation of 2H and 3C phases. These nuclei grow into bigger grains as the annealing temperature is raised by increasing the pulse current in the electron microscope.

2 Initial crystallization into 2H and 3C phases ultimately transform to a twinned 6H structure.

The mechanism of polytype formation is not yet fully understood. Therefore the growth of SiC polytypes by the recrystallization of this film of SiC is expected to prove useful in this respect.

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## H3. STUDY OF STRUCTURAL TRANSFORMATIONS DURING GROWTH OF LEAD IODIDE POLYTYPES

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For understanding the suspected role of the stacking faults in the formation of polytypes, the growth history of the polytypic crystals of lead iodide grown by silica gel method, has been revealed by successively cleaving them and examining the crystal structure by X-ray diffraction, after each cleavage. Characteristic structural changes have been found to be similar to cadmium iodide, with the significant difference that here, owing to free suspension of the crystal platelet in the growth medium, growth is found to take place on both the faces

from the centre outwards, after the initial nucleation. The results have been discussed in terms of the stacking faults produced during crystal growth.

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#### H4. IDENTIFICATION OF RHOMBOHEDRAL LATTICE IN CdI<sub>2</sub> CRYSTALS

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Hitherto, a distinction between rhombohedral and hexagonal lattices in CdI<sub>2</sub> crystals has been done by comparing a Weissenberg or an oscillation photograph of a polytype to a corresponding photograph of a basic polytype 2H or 4H. This involves two photographs having different shrinkage and different central spots and therefore makes the method impracticable for higher polytypes. In the present paper a new method, in which two rows of spots 10.l and 20.l of a polytype are recorded side by side in the same photograph, is used for identification. If  $\sin \theta_{10.l_1} = \sin \theta_{20.l_2}$ ,  $l_1$  spot in 10.l row should coincide with  $l_2$  spot in 20.l row. Indexing is done assuming the polytype to be hexagonal and if  $l_1$  coincides  $l_2$ , the lattice is hexagonal and if it does not, it is rhombohedral.

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#### H5. EVALUATION OF STACKING FAULT ENERGY OF CADMIUM IODIDE STRUCTURES - ITS RELEVANCE IN GROWTH AND TRANSFORMATION

R.S. TIWARI, A.K. RAI and O.N. SRIVASTAVA, Department of Physics, Banaras Hindu University, Varanasi 221 005.

It is now generally believed that the growth of polytypes is germane in the creation and ordering of stacking faults in the parent structure. If this is so, there should exist a correlation between the stacking fault energy and the atomic structure of the polytypes. Evidence for the existence of such correlation was first obtained by Prasad and Srivastava (1971) who employed pair interaction method for evaluation of stacking fault energy. However, the calculation of stacking fault energy in terms of bond energies using central force model has not been carried out for

the polytypic structures, since the estimation of stacking fault energy in terms of bond energies brings out more facts and is also more rigorous (Hirth and Lothe, 1968), a model has been developed for the calculation of stacking fault energy of polytypic structures of cadmium iodide in the present investigation. The model has been applied for the calculation of stacking fault energies of various polytypes of cadmium iodide and silicon carbide. The relevance of these calculations in regard to the growth and transformation of polytypes has been outlined.

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#### H6. ON THE STRUCTURAL TRANSFORMATIONS IN CADMIUM IODIDE AND LEAD IODIDE POLYTYPIC CRYSTALS

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Structural transformations amongst high periodicity polytypes were noticed for the first time in cadmium iodide crystals by Tiwari and Srivastava (1970). Two prominent modes of transformations in cadmium iodide crystals consequent to annealing in vacuum correspond to (i) transformation of a long period polytype  $nH$  or  $(nR)$  to a  $4H$  parent phase with superimposed disorder and (ii) transformation of a hexagonal or rhombohedral polytype to another hexagonal or rhombohedral polytype having the same number of layers but with a different arrangement of stacking sequence. In cadmium iodide unlike long periodicity polytypes, low identity period parent structure  $4H$  does not transform on annealing. In the case of lead iodide polytypic crystals which is isostructural with cadmium iodide, the transformation modes are remarkably different than those observed in cadmium iodide. The high periodicity polytypes of lead iodide do not transform at all even on repeated annealing runs. On the contrary when a low period parent structure such as  $2H$  or  $6R$  is annealed, it transforms to a long period polytypic structure together with some amount of superimposed disorder. The differences in the transformation modes of cadmium iodide and lead iodide polytypes can be broadly understood in terms of differences in the impurity content of the two crystals.

J1. SIFOR : A CRYSTALLOGRAPHIC FOURIER SUMMATION  
PROGRAM FOR IBM 1130

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SIFOR is a crystallographic Fourier summation program for the 16K IBM 1130 computer, designed to run with a single disk drive. It is written in Basic Fortran IV. The program uses the Beever-Lipson factorisation and accommodates all space groups in the triclinic, monoclinic and orthorhombic systems, except the two space groups (Nos. 43 and 70) having diamond glides. Floating-point arithmetic, a software feature in the IBM 1130, has been kept to a minimum and trigonometric functions are obtained through table look-up. The Fourier summation may be carried out in any desired order, the necessary permutation of the Miller indices being under program control. The grid spacing is restricted to integral multiples of 1/120th of all the three cell edges. The reduction of the Fourier expression for the individual space groups and the application of the appropriate multiplicity corrections are automatic once the symmetry elements necessary to define the space groups concerned are specified. For maximum efficiency of the program, the input structure factor data should be properly sorted.

## IS1. THE THREE-DIMENSIONAL STRUCTURE OF YEAST PHENYLALANINE TRANSFER RNA

A. RICH, Biology Department, Massachusetts Institute of Technology, Cambridge, Massachusetts, USA.

An x-ray diffraction analysis has been carried out on orthorhombic crystals of yeast phenylalanine transfer RNA and the electron density map has been analyzed at 5.5, 4.0 and more recently 3.0 Å. The folding of polynucleotide the chain was reported almost two years ago [Kim, et al, *Science* 179, 285 (1973)]. The molecule was found to exist in an L shaped conformation with secondary structure similar to that found in the familiar cloverleaf diagram for tRNA sequences. The CCA acceptor stem was more or less parallel to the T $\psi$ C stem while the dihydrouracil and anticodon stems were organized at right angles to this to form an L shaped molecule. The anticodon is at one end of the L, the terminal CCA acceptor is at the other end and the coiling of the dihydrouracil and T $\psi$ C loops is at the corner of the L. At 3 Å resolution the tertiary interactions have been analyzed [Kim, et al, *Science* 185, 435 (1974)]. These show that nine different types of tertiary hydrogen bonding, many of which involve the use of nucleotides which are constant to all transfer RNA's. Several types of hydrogen bonding are found in the tertiary interactions in addition to the familiar Watson-Crick pairing. The molecule is stabilized considerably by the hydrophobic stacking interaction along the two branches of the L shaped molecule. We are currently concentrating on extending the analysis to a 2.5 Å resolution in order to work out the finer details of the interactions within the molecule.

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## IS2. X-RAY CRYSTALLOGRAPHIC VISUALIZATION OF DRUG-NUCLEIC ACID INTERACTIONS

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Several years ago, we successfully co-crystallized the antibiotic, actinomycin D, with deoxyguanosine and solved the structure by X-ray crystallography. This enabled us to construct a model of how actinomycin binds to DNA. In the model, the phenoxazone ring system on actinomycin intercalates between the base-paired dinucleotide sequence, dG-dC, while the peptide subunits lie in the narrow groove of the DNA

helix and interact with deoxyguanosine residues on opposite chains through specific hydrogen bonds. The binding of actinomycin to DNA has demonstrated a general symmetry principle that several classes of probe disteins utilize in recognizing symmetrically arranged nucleotide sequences on the DNA helix.

We have now co-crystallized the drug, ethidium bromide, with the dinucleoside monophosphates, cytidyl(3'-5') guanosine (CpG) and uridylyl (3'-5')adenosine (UpA), as well as with iodinated derivatives of these dinucleotides. These structures are 2:2 complexes and crystallize in different lattices. We have now solved the first of these structures (ethidium: 5-iodoUpA) and this has allowed us to directly visualize intercalative binding by this drug to a fragment of a nucleic acid double helix. 5-iodoUpA molecules form a small Watson-Crick like double helix. One ethidium molecule intercalates, the other stacks between adjacent dinucleotides. Symmetry is utilized in the intercalative interaction, the approximate 2-fold axis of symmetry of the ethidium molecule coinciding with the 2-fold axis of the base-paired dinucleotide. I shall describe the structure in detail at the time of the meeting.

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### IS3. CYCLIC PEPTIDES STRUCTURE AND CONFORMATION

GOPINATH KARTHA, Center for Crystallographic Research, Roswell Park Memorial Institute, Buffalo, N. Y. 14203 and Department of Biophysical Sciences, S. U. N. Y. A. B., Buffalo, N. Y. 14226.

Cyclic peptides have been the subject of extensive investigations by various physico-chemical methods. Not only are these molecules of great importance as model compounds in examining various aspects of peptide and protein chemistry, but many of them are also of great importance in their own right because of their biological activity. Many hormones, antibiotics and ion transport molecules belong to this class. In our laboratory, we have been investigating the structure and conformation of a number of these cyclic peptides, some of them also containing unusual non-protein amino acids.

The three-dimensional structures of some of these have been determined in our laboratory by single crystal x-ray diffraction techniques. Though many of these cyclic peptides do form single crystals, in general the quality and size of these crystals are not ideal in yielding high precision diffraction data. Further, few of these crystals contain heavy atoms to facilitate solution of the phase problem. Hence, in many

cases, the crystallographic solution of these structures bring challenging problems. Some of the techniques used in solving these structures will be discussed along with the results obtained from crystallographic and other studies.

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es of IS4. SOME PROBLEMS IN OUR UNDERSTANDING OF PROTEIN  
STRUCTURE

W. KAUZMANN, Department of Chemistry, Princeton University, N.J.  
08540, USA.

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IS5. NUCLEIC ACIDS AND NUCLEOTIDE CONFORMATION -  
FURANOSE RING PUCKERING

V. SASISEKHARAN, Molecular Biophysics Unit, Indian Institute of  
Science, Bangalore 560 012, India.

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Recently, conformational studies on nucleotides have drawn the attention of many investigators. As early as in 1967, we reported allowed conformations of a monomer unit of a nucleic acid chain and stressed that the monomer unit can have only a very limited range of conformations as possible owing to enormous restrictions being imposed by nonbonded interactions. Recently, it has been suggested from analysis of crystal structure data on nucleotides that the 5'-nucleotide is a rigid unit. This has been supported by a few conformational energy calculations. In this talk, results of the classical energy calculations made on different segments of nucleic acid chain will be presented. These calculations essentially stress the importance of the role of the furanose ring puckering on the conformation of nucleic acid chains. The flexibility or otherwise of the 5'-nucleotide unit is discussed. Finally, conformation of di-nucleoside monophosphate, dinucleoside diphosphate and nucleic acid chains will be discussed.

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IS6. ORGANIZATION OF PHOSPHOLIPIDS IN BIOMEMBRANES

GIRJESH GOVIL, Tata Institute of Fundamental Research, Bombay  
400 005.

Cell Membranes are responsible for the biological organisation within their boundary at the expense of disorganisation in their environment. This problem is intimately connected with the structure of the cell walls. Phospholipids, proteins and cholesterol are major constituents of the cell membrane and an organised arrangement is achieved through intra and intermolecular forces between the constituents.

Phospholipids consist of a short polar group ( $\alpha$ ) and two comparatively long hydrocarbon chains ( $\beta$  and  $\gamma$ ) connected to a glycerol residue. Molecular orbital calculations indicate that the possible conformations of phospholipids are highly restricted. When considering conformations relevant to structures in cell membranes, a further selection is possible because of the fact that in aqueous solutions hydrophobic interactions stabilise an arrangement where the  $\beta$  and  $\gamma$  chains are packed parallel to one another leading to a bilayer structure. Various models which satisfy these conditions have been compared and it has been found that only four are favoured by energy considerations. These arrangements differ from one another in the orientation of  $\beta$  and  $\gamma$  chains, close to its linkage with the glycerol group. A low energy pathway connects these conformations and thus a phospholipid molecule can easily flip from one preferred conformer to the other.

The proposed model provide explanations to a number of dynamic and static properties of cell membranes in addition to a theoretical basis for lipid bilayers. Such properties will be discussed in the light of proposed model.

## S1. TRIPLE HELICAL STRUCTURE OF (GLY-PRO-HYP)<sub>n</sub>

MANJU BANSAL and G.N. RAMACHANDRAN, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012.

Synthetic regular polytripeptides of the type (Gly-R<sub>2</sub>-R<sub>3</sub>)<sub>n</sub> where R<sub>2</sub>, or R<sub>3</sub>, or both, are imino acids have been widely studied as model compounds for obtaining information about the molecular structure of collagen. One such polytripeptide is poly(Gly-Pro-Hyp), since triplets with this sequence constitute about 10% of collagen. Recently, a new model has been proposed for this polytripeptide, in which the peptide bond between the glycine and proline residues is in the cis conformation and the  $\gamma$ -hydroxyl group of hydroxyproline forms a direct interchain hydrogen bond within the triple-helix. We have confirmed this structure by model building using computer techniques, and the helical parameters obtained are found to occur over the range 2.75Å to 2.80Å for the unit height and -112° to -114° for the unit twist. These will be discussed along with the fibre pattern of the polymer.

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## S2. ROLE OF HYDROXYPROLINE IN THE STABILITY OF THE COLLAGEN STRUCTURE

MANJU BANSAL, C. RAMAKRISHNAN and G.N. RAMACHANDRAN, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012.

The molecular structure of collagen is now accepted to be based on a triple-stranded coiled-coil, in which the three strands are held together, predominantly, by hydrogen bonds. The controversy about the exact number of interchain hydrogen bonds has been only recently resolved in our laboratory and it has been suggested that there are two interchain hydrogen bonds per tripeptide unit, one of which is a direct interchain bond involving the amino group of the glycine residue, while the second hydrogen bond can be formed with a water molecule as an intermediary between neighbouring chains. It has now been shown that the same water molecule can also form a hydrogen bond with the oxygen of the  $\gamma$ -hydroxyl group of hydroxyproline residues in position 3 in the sequence (Gly-R<sub>2</sub>-R<sub>3</sub>). This hydroxyl group can also take part in an inter-triple-helix hydrogen bond and help in the formation of fibrils from the protofibrils. Hydroxyproline is an amino acid that occurs only in collagen and our recent studies have brought out the role played by it in the structure and stability of collagen.

S3. EXPLANATION OF RARITY OF CIS- PEPTIDE UNITS IN PROTEIN AND POLYPEPTIDE CHAINS

G.N. RAMACHANDRAN, ALOK K. MITRA and A.S. KOLASKAR, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560007, India.

The results of the quantum chemical calculations on simple amides are unable to explain the occurrence of only trans peptide units in polypeptide and protein structures. In order to get an insight into this problem, the total potential energies of tripeptide units, trans - trans - trans and trans - cis - trans, having alanyl side chains, have been calculated using empirical potential energy functions over the whole range of  $(\phi_1, \psi_1)$  and  $(\phi_2, \psi_2)$  at intervals of  $30^\circ$ . The relative probability of occurrence of trans - cis - trans, as compared with trans - trans - trans, was found to be approximately 20%, when the energy difference between trans- and cis peptide units is considered to be zero. However, when the energy difference between trans and cis peptide units, is also included to have a value of 1.0 kcal/mole, the relative probability of occurrence of trans - cis - trans as compared with trans - trans - trans is found to be as low as 3.6%.

These calculations indicate that the reason for the non-occurrence of cis peptide units in open polypeptide structures lies not only in the energy difference between cis and trans peptide units, but also in the fact that the non-bonded interactions are favourable for the trans peptide unit in a polymer chain.

S4. NON-PLANAR DISTORTIONS OF THE CIS- PEPTIDE UNIT

A.S. KOLASKAR<sup>+</sup>, S. RAMAPRASAD<sup>+</sup>, K.P. SARATHY\* and G.N. RAMACHANDRAN\*<sup>+</sup>

<sup>+</sup> Molecular Biophysics Unit, Indian Institute of Science, Bangalore, India, and

\* Department of Biophysics and Theoretical Biology, University of Chicago, Chicago, Illinois 60637 USA.

The possible non-planar distortions for the N-methyl acetamide molecule in its cis-configuration has been studied using CNDO/2 and INDO methods. Available crystal structure data on cyclic peptides having the peptide unit in the cis- configuration, suggest that the dihedral

angle  $\theta_N$  and  $\Delta\omega$  are correlated, while  $\theta_C$  is small and is uncorrelated with  $\Delta\omega$ , exactly as are found in the case of the trans peptide unit. The observed points in the  $(\theta_N, \Delta\omega)$ -plane for various cyclic-peptides containing the cis peptide unit lies in the low-energy region of the iso-energy contours drawn using CNDO/2 and INDO methods for the cis N-methyl acetamide molecule. The results regarding energy contours obtained for the possible non-planar distortions of the cis peptide units are very similar to those obtained previously in our studies for the trans peptide unit. These studies suggest that the out-of-plane distortions at the nitrogen end of the peptide unit are most likely to be present in all actual structures.

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##### S5. THE PYRROLIDINE RING CONFORMATION AND ITS ROLE IN THE FOLDING OF PROTEINS.

U.V. PANDYA and V. SASISEKHARAN, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India.

An analysis of the available crystal structure data in terms of two pseudo-rotational coordinates indicated the nature of the flexibility of the pyrrolidine ring in proline and prolyl residues having both cis and trans units. The data could be classified into a few typical types of puckering following a notation suggested here for the description of the conformation of five-membered rings in general. Also it was observed that there were definite correlations between puckering and the conformational angles  $\phi$  and  $\psi$ . These will be discussed.

Using the average dimensions obtained from the crystal structure analysis, for both trans and cis prolyl residues, classical conformational energy calculations have been made to find out the effect of the puckering of the pyrrolidine ring on the backbone conformations of a polypeptide chain. The important feature of the energy plot is the appearance of a double minima separated by a barrier of approximately 3 to 4 kcal/mole. The relationship between the puckering and the folding of the proteins in terms of the dihedral angles  $\phi$  and  $\psi$ , clearly brought out from the present study, will be discussed.

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S6. CONFORMATIONAL FEATURES OF SOME CYCLIC  
DIPEPTIDES (DKP)

R. RAMANI and V. SASISEKHARAN, Molecular Biophysics Unit,  
Indian Institute of Science, Bangalore 560 012, India.

Crystal structures of cyclic-dipeptides with different side chains are being analysed by single crystal x-ray diffraction technique. As a part of this investigation a literature survey of similar compounds, analysed by x-ray diffraction methods and NMR technique was made. The conformational flexibilities of the DKP ring under different conditions were examined. A general trend observed with respect to the DKP ring is that it can take up mainly three different conformations generally known as (i) planar (or extremely flat chair), (ii) bowsprit boat and (iii) flag-pole boat.

Theoretical investigations of the conformational aspects of the DKP ring, using semiempirical potential functions, are compatible with the experimental observations. The theoretical studies of the possible conformations of the side chains of these compounds also show a good agreement with the experimental observations, though there are some exceptions in the solid state. These will be discussed in detail.

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S7. CONFORMATIONAL STUDIES ON SOME DIPEPTIDES IN  
SOLUTION USING HIGH RESOLUTION NMR

K.R.K. EASWARAN and P. MOHANAKRISHNAN, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India.

The proton NMR studies on cyclic dipeptides, (Tyr-Tyr), (Trypt-Trypt), (Leu-Leu) and (Gly-Leu) have been made and the results will be discussed in terms of the conformation in these. The use of <sup>13</sup>C NMR relaxation studies in conformational analysis of peptides will also be discussed with few examples.

Also the preliminary results on the proton NMR studies on the compound, N-Acetyl-L-Pro-D-Ala N-Methylamide will be presented.

S8. THE NATIVE AND UNFOLDED CONFORMATIONS OF  
 $\beta$ -LACTOGLOBULIN

V. S. ANANTHANARAYANAN and F. AHMAD, Molecular Biophysics  
Unit, Indian Institute of Science, Bangalore 560 012, India.

$\beta$ -Lactoglobulin has been found to undergo a transition from the compact native conformation to an unfolded state in aqueous solution by increasing either the temperature of the solution or the concentration of guanidine hydrochloride (GuHCl) in the solution. The thermally-denatured  $\beta$ -lactoglobulin seems to be not as completely unfolded as the protein in concentrated (e.g. 6M) GuHCl solution, where it has been shown to be devoid of all non-covalent interactions. This is corroborated by the observation of a further co-operative transition undergone by the thermally denatured protein on addition of GuHCl. An insight into the nature of the native and thermally denatured states of the protein has been obtained by analysing the far-ultraviolet optical rotatory behaviour of the protein. Contrary to the normally observed decrease in the magnitude of the reduced mean residue rotation at 232 nm,  $[m']_{232}$ , on an increase in  $[m']_{232}$  in the thermal and GuHCl denaturation of  $\beta$ -lactoglobulin. An interpretation of this anomalous rotatory behaviour of the protein has been made in terms of (a) the native protein possessing a considerable amount of  $\beta$ -structure, and (b) the thermally-denatured protein retaining some intact  $\alpha$ -helical regions which are converted to the random coil state by the addition of GuHCl. These conclusions are supported by our viscosity data on the native and denatured protein.

S9. CONFORMATIONAL STUDIES OF HEXOPYRANURONIC ACIDS

M. NAGARAJAN and V. S. R. RAO, Molecular Biophysics Unit, Indian  
Institute of Science, Bangalore 560 012, India.

The conformation of hexopyranuronic acid is of utmost importance in the chain building of mucopolysaccharides for which the former is one of the building blocks. Hence the favoured conformations has been assigned for hexopyranuronic acids by computing the potential energies. The energy calculations showed that all uronic acids studied favour C1(D) conformation except for  $\alpha$ -D-altouronic acid and  $\alpha$ -iduronic acid which may exist in C1  $\rightleftharpoons$  1C equilibrium in solution. The present study predict the favoured conformations for  $\alpha$ -D-glucuronic acid and  $\beta$ -D-galacturonic acid, in agreement with experimental observations.

The present study also suggests that  $\alpha$ -L-iduronic acid and  $\beta$ -D-glucuronic acid residues in heparin may exist in 1C and C1 conformations respectively

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S10. CONFORMATIONAL STUDIES ON CYCLIC TETRAPEPTIDE WITH CIS AND TRANS PEPTIDE RESIDUES

G. MANJULA and C. RAMAKRISHNAN, Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India.

Conformational analysis of cyclic tetrapeptides consisting of alternating cis and trans peptide units has been made using contact criteria and energy considerations. The study has been restricted to N-substituted amino acids, such as N-methyl glycine and N-methyl alanine, so that comparison with experimental observation is feasible. The results indicate that such a structure with alternating cis and trans peptide units is easily formed with planar units, unlike the earlier all trans structure which requires deviation from planarity of the order of  $12^\circ$ . Only those structures having a symmetry element such as a two-fold axis or a centre of inversion have been considered. It is interesting to note that the residue proline is more favourably incorporated at a cis-trans junction than at a trans-cis junction. The detailed results and comparison with observation will be presented.

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S11. STATISTICAL TREATMENT OF THE NUCLEOTIDE BACKBONE AND TIME AVERAGE PROPERTIES OF POLYRIBOURIDYLIC ACID

R.K. NANDA, R. TEWARI and G. GOVIL, Tata Institute of Fundamental Research, Homi Bhabha Road, Bombay 400 005.

Energy calculations based on EHT, CNDO, PCILO and CPF methods have been used to calculate the probability distribution of the torsional angles ( $\phi'$ ,  $\omega'$ ,  $\omega$ ,  $\phi$  and  $\psi$ ) which fix the structure of nucleotide backbone. Statistical average values of the vicinal  $^1\text{H} - ^1\text{H}$ ,  $^1\text{H} - ^{31}\text{P}$  and  $^{13}\text{C} - ^{31}\text{P}$  NMR coupling constants ( $J$ ) have been calculated from the generalized Karplus relations using the probability distribution in the  $\phi'$ ,  $\phi$  and  $\psi$  space. Experimental  $\langle J \rangle$  values for polyribouridylic acid (Poly U) support theoretical values for the torsional angles ( $\phi' \sim 240^\circ$ ,  $\phi \sim 180^\circ$  and  $\psi \sim 60^\circ$ ). Using Monte Carlo technique random coils of single stranded polynucleotide

have been simulated and the mean square end to end distance  $\langle r^2 \rangle$  has been calculated. The molecular orbital methods suggest considerable flexibility around O-P bonds, leading to fairly small values for the characteristic ratio  $C_\infty \sim 4$ . Observed values of the unperturbed characteristic ratio for polynucleotides are quite large (e.g.  $C_\infty \sim 18$  for poly U) suggesting a fairly rigid nucleotide backbone. The results based on molecular orbital calculations can be reconciled with the experimental values by introducing an additional stabilization of  $\sim 2$  k. cal mole<sup>-1</sup> for the predicted minimum energy region ( $\phi' \sim 240^\circ$ ,  $\omega \sim 290^\circ$ ,  $\omega' \sim 290^\circ$ ,  $\phi \sim 180^\circ$  and  $\psi \sim 60^\circ$ ). Such a stabilization may arise from the association of water molecules and metal ions with the phosphate group. Coulomb interaction between neighbouring phosphate groups may also provide extra stabilization. These calculations enable a semi-quantitative estimate of torsional rigidity in the nucleotide backbone.

## S12. POSSIBLE CONFORMATIONS OF THE ANTICODON LOOP OF YEAST tRNA<sup>phe</sup>

K. SUNDARAM<sup>+</sup> and M. SUNDARALINGAM, Department of Biochemistry, College of Agricultural and Life Sciences, University of Wisconsin, Madison, Wisconsin 53706.

This paper describes the experiences of an attempt to generate stereochemically allowed conformations for the anticodon loop of yeast t-RNA<sup>phe</sup>. A flexible model of the anticodon loop segment of the t-RNA was simulated in a UNIVAC 1110 computer with facilities for graphic display and interactive computation. Pairing of the terminal bases was achieved by choosing a consistent set of internal parameters as determined by a damped least squares procedure. In this process it was found expedient to use the results available on the preferred conformation of a nucleotide unit of a polynucleotide resulting from research on monomer and dimer conformations. Accordingly, the torsion angles  $\omega$ ,  $\phi$ ,  $\psi$ ,  $\phi'$  and  $\omega'$  all along with anticodon loop backbone and the glycosyl torsion angles  $\chi$  of the terminal nucleotides were varied within limited bounds so that each monomer was essentially in the proposed C(3')-endo rigid nucleotide conformation and the phosphodiester conformations were in one of the allowed regions of the  $\omega, \omega'$  map for dinucleotides. For convenience in maintaining ribose ring closure the  $\psi'$  torsion angles were not varied. Despite the restrictions in monomer and dimer conformations, a large number of conformations are possible for an open polymer of nine nucleotides. However, constraining the backbone phosphodiester of the anticodon triplet to the

helical  $g^-g^-$  conformation greatly reduces the number of possible structures for the anticodon loop and technical details of the simulation technique will be discussed.

Research supported by NIH grants GM-17378 and GM-18455.  
+ Work performed while author was on leave of absence from the Department of Biophysics, A.I.I.M.S., New Delhi 110 016.

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S13. AN X-RAY INVESTIGATION OF THE LAMELLAR STRUCTURE OF SPHINGOMYELIN

R.S. KHARE and R.K. MISHRA, Department of Biophysics, All India Institute of Medical Sciences, New Delhi 110 016.

Low and wide angle x-ray data from anhydrous and hydrated (33% by weight) sphingomyelin (SM) were obtained. These diffracted intensities are a continuous function of the diffraction angle and were considered to arise from randomly oriented single bimolecular leaflets. Electron density (ed) profiles after Rand & Luzzati (1) and Small (2) were computed, it shows two peaks of high relative ed and a central trough, these regions are ascribed to the layer of polar groups, mainly phosphorous and nitrogen atoms, and to the methylene and methyl region respectively. By hydration, the peaks of ed profile moves inwards with out appreciable change in its shape. It is observed that the hydrophobic chains of SM bilayer shrink in length from 60.56 Å to 58.10 Å, however the polar hydrophilic portion becomes parallel to the hydrocarbon chain increasing the spacing of 31.12 Å in anhydrous sample to 39.92 Å in the hydrated sample. In both cases a strong 4.2 Å reflection was observed, this represents the average inter-chain distance in close packed order. With 93% hydration only two reflections, 83.26 and 42.51 Å were observed. Changes in lipid bilayer thickness for SM with hydration seems to be of different nature than of glycerophospholipid. We wish to thank Prof. P.H. Geil for supporting these studies which were performed in his laboratory at CWRU, Cleveland, Ohio.

- 1 Rand, R.P. & Luzzati, V., Biophys, J. 8(1968) 125
- 2 Small, D.M., J. Lipid Res. 8(1967) 551.

S14. ROLE OF METAL ION IN ENZYMATIC ACTION OF ATP.  
CONFORMATION STUDY OF METAL ION ATP COMPLEX

V. KOTHEKAR and R.K. MISHRA, Department of Biophysics, All India Institute of Medical Sciences, Ansari Nagar, New Delhi 110 016.

Metal ion plays an important role in activity of enzyme molecules. Their action is highly specific. Quantum chemical evaluation of metal ion ATP interaction has shown considerable dependence of metal ion action on geometrical disposition of the metal ion. Present work reports the study of conformation of metal ion ATP complex using monopole-monopole, monopole-dipole, and Lennard-Jones type interaction. Most probable site for the metal ion interaction is established and interaction of different metal ions is compared. Possible correlation between biological activity and conformation of Metal ion ATP complex is sought.

S15. CONFORMATION OF GLYCEROL AND 2-AMINOPROPANE-1,3-DIOL: A COMPARATIVE STUDY USING EMPIRICAL POTENTIAL FUNCTIONS

RAM S. KHARE, Department of Biophysics, All India Institute of Medical Sciences, Ansari Nagar, New Delhi 110 016.

Complete conformational energy maps (Ramachandran diagrams) for glycerol ( $\text{CH}_2\text{OH} \cdot \text{CHOH} \cdot \text{CH}_2\text{OH}$ ) and 2-Aminopropane-1,3-diol ( $\text{CH}_2\text{OH} \cdot \text{CHNH}_2 \cdot \text{CH}_2\text{OH}$ ) have been obtained by empirical potential functions method (1). Possibility of intra-molecular hydrogen bonding and the free energy of hydration were also studied and relative probability of occurrence of various conformations was estimated (2). Co-operativity of rotation of similar groups were found to reduce the steric requirements. The most stable conformation in both cases was when  $\theta_1$ ,  $\theta_2$ ,  $\theta_3$ ,  $\theta_4$  and  $\theta_5$  were 300, 240, 300, 240 and 300 degrees respectively. Relative probability for this conformation for 2-aminopropane-1,3-diol was 51% and for glycerol it was 22%. Minimum energy conformations for both cases remains almost unchanged in hydrated state, although their probabilities are now much lower, as compared to their probabilities in vacuum, being 40% and 13% respectively. These observations may be consistent with the fact that sphingolipids where 2-aminopropane-1,3-diol forms the backbone are much more stable than glycerolipids, where glycerol forms the backbone.

- 1 Ramachandran, G.N. & Sasisekharan, V. (1968) :  
Adv. Protein Chem. 23, 283.
  - 2 Hopfinger, A.J. (1973) : Conformational properties of  
macromolecules, Acad. Press, N Y.
- 

#### S16. MOLECULAR CONFORMATION IN LIQUID CRYSTALLINE PHASES

N.K. ROPER and R.K. MISHRA, Department of Biophysics, All India Institute of Medical Sciences, Ansari Nagar, New Delhi 110 016.

Conformational study using Classical Potential Functions has been performed on Cholesteryl Caprate molecule by rotating caprate fragment with respect to the cholesteryl moiety. Two minima are obtained. Only the higher energy minimum is found to be consistent with the consideration of packing of cholesteryl caprate molecules in crystallographic configurations of both smectic and cholesteric liquid crystalline phases.

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#### S17. ARRANGEMENT OF PLANAR MONOMERS IN MELANINS

V. T. THATHACHARI, Indian Institute of Technology, Madras 600 036.

Melanins are believed to be random polymers made up of several different types of monomers with little regularity either in their sequence or in the way they are linked. The monomers are, however, either planar molecules or contain planar subgroups. X-ray diffraction and molecular simulation studies have shown that in all melanins the adjacent non-bonded layers tend to be parallel and 3.4 Å apart. This short range spatial order, in turn, suggests some regularity in their chemical structure as well : e. g. preference of certain monomer sequences and bonding sites ; conjugation of adjacent bonded layers ;  $\pi$  complexes between adjacent nonbonded layers etc. The mere presence of planar units in a structure does not necessarily lead to their parallel alignment unless additional criteria are met. This is evident when the environments of planar units in known structures are analyzed e. g. Catechol, L DOPA, Benzoquinone, Naphthalene, Coronene, Biphenyl, Quarterphenyl, Quinhydrone, Polyaromatics like Polystyrene, Polynucleotides like DNA and Carbons like coal and graphite. In carbons and Coronene

where the layers extend in two dimensions, in DNA with a regular structure due to complementary pairs and in Quinhydrone, a charge transfer complex, the adjacent layers are parallel, 3.4 Å apart and have considerable overlap. In the other structures the adjacent layers tend to be at considerable inclinations and where parallel have little or no overlap. A systematic study of the arrangement of planar molecules in known structures has been carried out in collaboration with Narasimha Murthi and Venkatesan and the results will be presented in a separate paper.

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#### S18. METAL ION COORDINATION IN NUCLEIC ACIDS AND THEIR CONSTITUENTS

T.P. SESHADRI and B. SWAMINATHA REDDY, Department of Physics, Indian Institute of Science, Bangalore 560 012, India.

One of the major achievements in the modern biology has been the establishments of the structures of DNA and RNA, and their primary role in the biological reproduction and growth. Single crystal X-ray diffraction analysis of the nucleic acid constituents reveal that the conformational features are influenced to a considerable extent by the metal ions present in the structure. The biological function of the nucleic acids such as replication, transcription and translation of the genetic code generally require participation of metal ions. It has been shown that metal ions can bind to many electron donor sites and in doing so dramatically influence the structure of the polymers. A few review articles discussing these results have appeared already based on the spectroscopic and biological studies. We wish to report some of the observations from various studies of X-ray diffraction analysis of the nucleic acid constituents.

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S19. CRYSTAL DATA OF 2' - DEOXYADENOSINE - 5' - MONOPHOSPHATE - DISODIUM SALT

M. V. HOSUR and M. A. VISWAMITRA, Department of Physics, Indian Institute of Science, Bangalore 560 012, India.

Long platy crystals of 2' - deoxyadenosine - 5' - monophosphate disodium salt were grown by slow diffusion of acetone into water solution of the sample. All the crystals are twinned. The crystal data was obtained from rotation, Weissenberg and Precession photographs. The crystals belong to triclinic system with the following cell parameters.

$$a = 22.66 \text{ \AA}, b = 6.98 \text{ \AA}, c = 6.84 \text{ \AA}, \alpha = 96^\circ 56', \beta = 93^\circ 20', \gamma = 78^\circ 30', d_{\text{obs}} = 1.608, d_{\text{cal}} = 1.607.$$

Complete three dimensional data is being collected for structure analysis.

S20. STRUCTURE AND CONFORMATION OF CRYSTALLINE AMINO ACIDS STUDIED BY NEUTRON DIFFRACTION

M. RAMANADHAM and R. CHIDAMBARAM, Neutron Physics Section, Bhabha Atomic Research Centre, Bombay 400 085.

Data from crystal structures of 18 amino acids (6 studied at BARC) and 2 dipeptides are analysed to find out the systematics of structure, conformation and hydrogen bonding in them. A weighted average structure for the amino acid residue and the  $\alpha$ -amino group has been computed from 15 amino acids, resulting in the following values:  $C-O^1 = 1.246$ ,  $C-O^2 = 1.255$ ,  $C-C^\alpha = 1.531$ ,  $C^\alpha-N^+ = 1.488$ ,  $C^\alpha-C^\beta = 1.524$ ,  $C^\alpha-H^\alpha = 1.096$ ,  $\langle N^+-H^i \rangle = 1.039$  (all in  $\text{\AA}$ ),  $O^1-C-C^\alpha = 117.6$ ,  $O^2-C-C^\alpha = 116.6$ ,  $O^1-C-O^2 = 125.7$ ,  $C-C^\alpha-C^\beta = 110.8$ ,  $C-C^\alpha-N^+ = 110.2$ ,  $C-C^\alpha-H^\alpha = 108.5$ ,  $N^+-C^\alpha-C^\beta = 110.3$ ,  $N^+-C^\alpha-H^\alpha = 107.5$ ,  $C^\beta-C^\alpha-H^\alpha = 108.9$ ,  $\langle C^\alpha-N^+-H^1 \rangle = 110.6$ , and  $\langle H^i-N^+-H^j \rangle = 108.5$  (all in degrees). The conformation of the amino group is influenced by hydrogen bonding and non-bonding electrostatic interaction with  $-\text{COO}^-$  group. Conformation of amide (asparagine, glutamine) and guanidium (arginine) groups are not significantly influenced by hydrogen bonding. There are 102 distinct hydrogen bonds in these structures which are categorised according to the donor and acceptor groups. Some of them are:  $N^+-H \dots O$  (46),  $N-H \dots O$  (10),  $O-H \dots O$  (23),  $N^+-H \dots Cl^-$  (12) and  $O-H \dots Cl^-$  (6).

Various parameters are defined for these bonds, which are then subjected to a systematic linear regression and correlation analysis. Correlations between linear and angular parameters are, in general weak for  $N^+-H---O$  bonds but are strong for the  $N-H---O$  bonds, from side groups.

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## S21. X-RAY INVESTIGATIONS OF SOME CRYSTALLINE COMPLEXES BETWEEN AMINO ACIDS

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Non-covalent interactions among amino acid residues play a crucial role in the structure and assembly of proteins. In order to study the atomic details of these interactions, a programme of x-ray investigations of crystalline complexes involving amino acids and short peptides has been initiated. As part of this programme, the crystallization and preliminary x-ray studies of L-lysine L-aspartate, L-arginine L-glutamate and L-histidine L-aspartate have been carried out. Of these, the crystal structure of lysine aspartate has been determined by direct methods followed by conventional Fourier techniques. The atomic parameters have been refined to an R value of 0.077 for 1134 observed reflections.

The dimensions of the positively charged zwitter ionic lysine molecules in the structure is similar to those found in the crystals of L-lysine monohydrochloride dihydrate. The aspartic acid molecule is also zwitterionic, but, unlike in the structures of L-aspartic acid and DL-aspartic acid, the side chain carboxylate group is deprotonated and carries a negative charge. The bond lengths and bond angles in the molecule, except those in the side chain carboxylate group, are comparable to those in the structures of L-aspartic acid and DL-aspartic acid. Considerable differences, however, exist in the conformation of the molecule in these structures. The crystal structure of lysine aspartate is stabilized by ionic interactions and hydrogen bonds involving amino and carboxylate groups.

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S22. THE CRYSTAL AND MOLECULAR STRUCTURE OF  
N-ACETYL-L-GLUTAMINE

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The crystals of the title compound are orthorhombic with cell parameters  $a = 13.811$ ,  $b = 5.095$ ,  $c = 12.914$  Å, space group  $P2_1 2_1 2_1$  with  $Z = 4$ . The structure was solved by symbolic addition procedure for noncentrosymmetric structures. The final R-index is 3.31% for 898 reflections. The details regarding the non-planarity of the peptide linkage will be discussed. The side chain conformation of the title compound is similar to that of L-glutamine, HCl. The dihedral angle  $\chi_1$  and  $\chi_2$  are  $-56.9^\circ$  and  $-176.5^\circ$ . The dimensions of the amide group are in agreement with those observed in the structure of acetamide.

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S23 THE CRYSTAL AND MOLECULAR STRUCTURE OF L-CYSTINE  
DIMETHYL ESTER DIHYDROCHLORIDE MONOHYDRATE

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L-cystine dimethyl ester is known to be an enzyme substrate and undergoes enzymatic hydrolysis, one of the most important biochemical transformations. The hydrochloride derivative of the compound crystallises in the monoclinic space group  $P2_1$  with cell dimensions  $a = 14.80$ ;  $b = 9.34$ ,  $c = 5.85$  Å,  $\beta = 91.47^\circ$ . The structure was solved by the heavy atom method using three-dimensional x-ray intensity data and refined by the block-diagonal method to an R-index of 0.103. The structure is stabilized by a three-dimensional network of hydrogen bonds. The disulphide dihedral angle is found to be  $84.4^\circ$ . The molecular conformation has been studied. The helical sense of the cystinyl group in the molecule is 'left'.

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S24. THE CRYSTAL AND MOLECULAR STRUCTURE OF DL-TRYPTOPHAN ETHYL ESTER HYDROCHLORIDE

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The crystal structure of DL-tryptophan ethyl ester hydrochloride has been determined using three-dimensional x-ray intensity data. The compound crystallises in the triclinic space group  $P\bar{1}$  with  $a = 15.85$ ,  $b = 8.73$ ,  $c = 5.35\text{\AA}$ ,  $\alpha = 82.73$ ,  $\beta = 88.51$  and  $\gamma = 106.33^\circ$ . The structure was solved by the heavy atom method and refined by the block-diagonal method to an R-factor of 0.089 for 2,012 reflections. The indole ring plane forms a dihedral angle of  $61.5^\circ$  with the carboxylate plane. The D and L molecules are linked by N-H...Cl hydrogen bonds around the centre of symmetry. The hydrogen bonds sandwich the molecules into a double layered system parallel to the bc plane.

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S25. ON THE CONFORMATION OF THE DISULFIDE IN CYSTINE AND ITS DERIVATIVES

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The conformation of the disulfide group in L-cystine is of considerable interest. X-ray analysis of L-cystine and its derivatives has shown that the helical sense of the disulfide group is right in hexagonal L-cystine and tetragonal L-cystine while in four other derivatives the sense is left helical. Energy calculations using semi-empirical methods are therefore expected to throw light on this problem. We have made preliminary calculations using simple non-bonded interactions and simple Pauling type potential for the disulfide group. The results will be discussed in the light of the observed experimental data.

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S26. STUDIES ON SOME ISOMERS OF ISOLEUCINE

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As a part of our studies on molecular structure, symmetry and conformation, the following three isoleucine structures were investigated. (I) D-alloisoleucine (II) D-alloisoleucine HCl H<sub>2</sub>O (III) L-isoleucine HCl H<sub>2</sub>O form II. (I) was found to be non-isomorphous with L-isoleucine [(Torii and Iitaka, Acta Cryst. B27, 2237 (1971)] inspite of the closeness of cell dimensions and identity of space group and was solved by trial and error methods by packing the molecule in a different manner, but satisfying the same type of hydrogen bonding scheme as in L-isoleucine. (II) was solved by heavy atom method. (III) was found to be structurally and conformationally different from D-isoleucine HCl H<sub>2</sub>O [(Trommel and Bijvoet, Acta Cryst. 7, 703 (1954)]. The conformational aspects will be discussed.

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S27. THE CRYSTAL AND MOLECULAR STRUCTURE OF GLYCYL DL THREONINE MONOHYDRATE

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The crystal belongs to the orthorhombic space group  $Pca2_1$  with cell dimensions  $a = 16.26$  (1),  $b = 4.87$  (1) and  $c = 23.24$  (1) Å. The structure was solved by direct methods using MULTAN. The structure has been refined to an R-index of 10.7%. There are two molecules in the asymmetric unit and they are related by an approximate centre of inversion. This is also reflected in the distribution of x-ray intensities.

S28. THE CRYSTAL AND MOLECULAR STRUCTURE OF DL-ALLOTHREONINE HYDROBROMIDE

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The structure was determined using 3-dimensional visual x-ray data by the heavy atom method. The crystal belongs to the monoclinic space group  $P2_1/c$  with cell dimensions  $a = 8.91 (1)$ ,  $b = 7.79 (1)$ ,  $c = 18.91 (1)$  and  $\beta = 92.0^\circ$ . The structure has been refined to an R-index of 10.5%. There are two molecules in the asymmetric unit and one of the molecules exists as cation with its carboxyl group is unionised. The C=O of this carboxyl group is trans to the amino nitrogen as in the case of Aspartic acid hydrochloride. The amino nitrogen of this molecule takes part in a bifurcated hydrogen bond.

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S29. CRYSTAL AND MOLECULAR STRUCTURE OF A MIXED CRYSTAL OF L-THREONINE, L-ALLOTHREONINE

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The compound was crystallised from an aqueous solution containing equal weights of DL-threonine and DL-allothreonine mixture. The structure belongs to the orthorhombic space group  $P2_12_12_1$  with cell dimensions  $a = 13.60 (1)$ ,  $b = 7.87 (1)$  and  $c = 5.16 (1) \text{ \AA}$ . The structure is closely isomorphous with that of L-threonine as well as L-allothreonine and was solved using this information. The unit cell contains the molecule in both L- and L-allo configuration and hence it is spatially disordered. The structure has been refined to an R-index of 12.2%.

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S30. THE CRYSTAL STRUCTURE OF SARCOSINE HYDROCHLORIDE

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Crystals of sarcosine hydrochloride, ( $C_3H_7O_2N.HCl$ ), belong to the monoclinic space group  $P2_1$  with cell dimensions,  $a = 9.00 \text{ \AA}$ ,  $b = 5.93 \text{ \AA}$ ,  $c = 5.41 \text{ \AA}$  and  $\beta = 96^\circ$ . The number of molecules per unit cell is two. The structure has been solved by 3-D heavy atom technique. The hydrogen positions have been located from a three dimensional difference Fourier synthesis. The positional coordinates of all the atoms, together with their temperature factors (anisotropic for non-hydrogen atoms and isotropic for hydrogen atoms), have been refined by full matrix least squares method. The final R value is 0.12. The sarcosine molecule is not a zwitterion, the two C - O distances being  $C(1) - O(1) = 1.330 \text{ \AA}$  and  $C(1) - O(2) = 1.184 \text{ \AA}$ . The molecules are held together by a three dimensional network of hydrogen bonds of the type  $N-H \dots Cl^-$  and  $O-H \dots Cl^-$  with lengths 3.04, 3.34 and 2.96  $\text{ \AA}$  respectively. The protonated aminonitrogen ( $-N^+H_2$ ) forms two hydrogen bonds with the two chlorine ions and assumes a tetrahedral configuration.

S31. THE CRYSTAL AND MOLECULAR STRUCTURE OF DISARCOSINE HYDROBROMIDE

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Disarcosine hydrobromide ( $C_3H_7NO_2$ )<sub>2</sub>HBr crystallizes in orthorhombic space group  $Pca2_1$  with eight molecules in the unit cell. Unit cell dimensions are  $a = 23.79 \text{ \AA}$ ,  $b = 6.18 \text{ \AA}$  and  $c = 15.40 \text{ \AA}$ . The bromine atoms in the asymmetric unit containing two crystallographically independent disarcosine hydrobromide units, ( $C_3H_7NO_2$ )<sub>2</sub>HBr were located from 3-D Patterson synthesis. Light atoms were located from the heavy atom phased 3-D Fourier synthesis. The structure was refined anisotropically by block-diagonal least squares method. The final R value is 0.11. One of sarcosine molecules of the asymmetric unit exists in ionised and the other in non-ionised form. The molecules are

DE held together by a three dimensional network of hydrogen bonds of the type N - H ... Br, N - H ... O and O - H ... O. The protonated amino nitrogen assumes a tetrahedral configuration.

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S32. THE CRYSTAL AND MOLECULAR STRUCTURE OF SILVER COMPLEX OF o-AMINOBENZENESULPHONYL GLYCINE

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The crystal and molecular structure of silver complex of o-aminobenzensulphonyl glycine has been determined by three dimensional heavy atom X-ray crystallographic method. The crystals belong to monoclinic space group  $P2_1/n$  with cell dimensions  $a = 5.73 \text{ \AA}$ ,  $b = 12.16 \text{ \AA}$ ,  $c = 13.94 \text{ \AA}$  and  $\beta = 95^\circ$ . The unit cell contains two formula units of  $HAg[C_6H_4(NH_2)SO_2NHCH_2COO]_2$ . The positional co-ordinates of the heavy and light atoms were refined by a full matrix least squares method using anisotropic temperature factors, the final value of R being 0.11. The molecule has been found to be centrosymmetric with center of inversion at silver atom. The benzene ring with amino nitrogen attached to it is almost planar. The bond distances and angles are satisfactory. The molecules in the crystal are held together by a three dimensional network of hydrogen bonds of the type N - H ... O. Both the nitrogen atoms take part in forming hydrogen bonds with the oxygen atoms of the adjacent molecule.

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S33. CRYSTAL AND MOLECULAR STRUCTURE OF L-PHENYL-EPHRINE HYDROCHLORIDE

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The crystal and molecular structure of L-Phenylephrine Hydrochloride, ( $C_9H_{12}NO_2 \cdot HCl$ ), a sympathomimetic compound was determined by three dimensional heavy atom X-ray technique. L-Phenylephrine hydrochloride crystallizes in monoclinic space group  $P2_1$  with lattice parameters,  $a = 14.64 \text{ \AA}$ ,  $b = 6.85 \text{ \AA}$ ,  $c = 11.72 \text{ \AA}$  and  $\beta = 102.5^\circ$ .

There are four molecules per unit cell. The two molecules of the asymmetric unit are related by a pseudo-glide symmetry. Refinement was carried out first by full matrix least squares method with isotropic thermal parameters and finally by block matrix method using anisotropic thermal parameters. The final R-value was found to be 0.11. The molecule consists of a six membered planar ring and an almost-planar extended side chain containing the protonated nitrogen atom. The dihedral angles between these two planes in the two molecules are  $61^\circ$  and  $107^\circ$ . The side chain is maximally extended and the nitrogen atom assumes a tetrahedral configuration. The molecules are held together by a three dimensional network of hydrogen bonds of the type N - H... Cl, O - H ... Cl and O - H ... O.

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#### S34. SOME CRYSTALLOGRAPHIC STUDIES ON THE ADDITION COMPOUNDS OF GLYCINE WITH CALCIUM HALIDES

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As a part of crystallographic investigations on the addition compounds and complexes of amino acids, a systematic study of the addition compounds of glycine with calcium halides is taken up. Diglycine calcium bromide tetrahydrate, grown from aqueous solution, crystallises in an octamolecular orthorhombic cell of dimensions  $a = 8.97$ ,  $b = 14.74$ ,  $c = 20.30$  Å. From systematic absences, the space group can be either  $Pbc2_1$  or  $Pbcm$ . In the same crop of the above crystals, another morphologically different form was found, which is monoclinic and possibly a hemihydrate. The crystallographic data of diglycine calcium chloride tetrahydrate determined by Jayalakshmi and Vijayan (*Acta Cryst.* 1967, 23, 669) are confirmed. It is interesting to note that the chloride and the bromide compounds, even though they have the same chemical formula, are not isomorphous. Crystals of diglycine  $CaClBr \cdot 4H_2O$  were also grown and these are isomorphous with the bromide compound.

Absence of (O, O, w) peaks in the three-dimensional Patterson synthesis, computed with about 1500 observed reflections, confirmed that the space group of the bromide compound is  $Pbc2_1$ . The four bromine atoms in the asymmetric unit were located and efforts are under way to elucidate the rest of the structure. The structure of the chloride compound is being solved with the three projection data and simultaneously the three-dimensional data are being collected. The results will be discussed.

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S35. CRYSTAL STRUCTURE OF METAL COORDINATION COMPLEXES OF AMINO ACIDS AND PEPTIDES

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As a part of the programme of crystal structure analysis of metal complexes of amino acids and peptides the following two compounds are investigated.

Zn-L-Threonine complex: After completing the work on Copper-L-Threonine complex, (Amirthalingam & Muralidharan, 1974) the corresponding zinc complex was investigated. The colourless prismatic crystals belong to the orthorhombic class with space group  $C222_1$ . The cell parameters are  $a = 5.91$ ,  $b = 10.32$  and  $c = 22.03 \text{ \AA}$  with  $Z = 4$  (1:2 complex with 2  $H_2O$ ). This means the Zn atoms occupy the two fold special position and only one ligand is in the asymmetric unit. The 2-D data (Ok1 and h01) have been collected and Patterson projections were computed to locate the Zn atoms. Later the lighter atoms are located in the projections. The results obtained will be discussed.

Copper complex of L-Valine-L-Tyrosine: The blue crystals are prismatic plates and belong to the orthorhombic class with space group  $P2_12_12_1$ . The cell parameters are  $a = 8.33$ ,  $b = 14.28$  and  $c = 16.23 \text{ \AA}$ ;  $Z = 4$  (of the 1:1 complex with 5  $H_2O$ ).

Patterson projections down 'a' and 'c' axes were computed and Cu was located. The other lighter atoms are located in the projection down 'a' axis. The results obtained will be described.

Reference:-

V. Amirthalingam & K.V. Muralidharan, (1974) Pramana (in press).

S36. CRYSTAL AND MOLECULAR STRUCTURE OF COPPER  
IMINO DIACETATE DIHYDRATE

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Iminodiacetic acid forms the functional group of a very useful chelating ionexchange resin used for the detection, estimation and removal of trace metals from biological systems. In particular, the copper chelated resin has been found to be extremely useful for the rapid separation of the nucleic acid components using ligand exchange chromatography. Copper iminodiacetate dihydrate  $[\text{CuN}(\text{CH}_2\text{COO})_2\text{H}_2\text{O}]\text{H}_2\text{O}$  crystallizes in orthorhombic space group  $Pbca$  with 8 molecules per unit cell of dimensions  $a = 10.27 \text{ \AA}$ ,  $b = 10.43 \text{ \AA}$  and  $c = 13.67 \text{ \AA}$ . The structure has been determined by 3-D heavy atom technique and was refined by full matrix least squares method using isotropic temperature factors for individual atoms. The co-ordination configuration of Cu ion is square pyramidal, involving two carboxyl oxygens, the nitrogen atom and the oxygen of a water molecule. The co-ordination bond distances are  $\text{Cu-O}(1) = 1.98 \text{ \AA}$ ,  $\text{Cu-O}(2) = 1.96 \text{ \AA}$ ,  $\text{Cu-N} = 1.95 \text{ \AA}$  and  $\text{Cu-W}(1) = 1.96 \text{ \AA}$ . The oxygen of the other water molecule forming the apex of the pyramid is at a distance of  $2.5 \text{ \AA}$  from Cu ion, indicating that this water molecule is weakly bound. The molecules are held together by a three dimensional network of hydrogen bonds. Further refinement is in progress.

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S37. THE CRYSTAL AND MOLECULAR STRUCTURE OF AMIDO-  
PYRINE

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As part of a programme of structural studies on pain-relieving medicines and their interactions, the crystal structure of amidopyrine, a dimethylamino derivative of antipyrine, has been determined. The compound crystallizes in the triclinic system, space group  $P\bar{1}$ , with unit cell dimensions,  $a = 7.458$ ,  $b = 10.744$ ,  $c = 17.486 \text{ \AA}$ ,  $\alpha = 98.6$ ,  $\beta = 85.6$  and  $\gamma = 108.6^\circ$ , with four molecules in the unit cell. The structure has been solved by direct methods using three-dimensional photographic data. The solution was aided by packing considerations. The structure has been refined by the least squares method to an R value of 0.098 for 3706 observed reflections.

The structure contains two molecules in the asymmetric part. The two crystallographically non-equivalent molecules have comparable bond lengths and angles. The dimensions of the pyrazolene ring in the molecules are broadly similar to those observed in the structure of anti-pyrine. The hetero nitrogen atoms in the ring are pyramidal. The methyl group attached to one of them is trans about N-N bond with respect to the phenyl ring attached to the other.

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S38. THE CRYSTAL AND MOLECULAR STRUCTURE OF A  
RADIOPROTECTANT 2-AMINOETHYLISOTHIO-URONIUM BROMIDE  
HYDROBROMIDE,  $C_3N_3H_{10}SBr.HBr$ .

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2-Aminoethyl isothiuronium bromide hydrobromide, an important radioprotectant has been found to crystallize in the triclinic system with two molecules in a unit cell of dimensions  $a = 7.23$ ,  $b = 8.32$ ,  $c = 8.66 \pm 0.02 \text{ \AA}$ ,  $\alpha = 71^\circ$ ,  $\beta = 79^\circ$  and  $\gamma = 83^\circ \pm 2^\circ$ . The distribution of bromine and sulphur atom positions deduced from the three dimensional Patterson map indicates that the structure is centrosymmetric. The complete determination of the structure using the heavy atom technique is in progress.

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S39. THE CRYSTAL AND MOLECULAR STRUCTURE OF 2,4-  
DICHLORO PHENOXY ACETIC ACID AND OTHER RELATED  
COMPOUNDS

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The crystal and molecular structures of two series of hypoglycemic agents - the aryloxy acetic acids and the N-aryloxy acetyl ureas - are being studied in our laboratory. 2,4-dichloro phenoxy acetic acid,

whose structure has been completed, crystallizes in the Triclinic system with

$$a = 7.75 \text{ \AA}, \quad b = 8.54 \text{ \AA} \quad \text{and} \quad c = 9.12 \text{ \AA} ; \\ \alpha = 103^{\circ}54', \quad \beta = 81^{\circ}39' \quad \text{and} \quad \gamma = 127^{\circ}38'.$$

There are two molecules per unit cell and the space group is  $P\bar{1}$ . The structure was solved by Patterson sections and the final R value is 0.124. The molecules form strong dimers linked together by O-H ... O hydrogen bonds across a centre of symmetry.

The paper discusses the common features in the structures of the various hypoglycemic agents whose structures are nearing completion.

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#### S40. THE CRYSTAL STRUCTURE OF NICOTINIC ACID : A RE-DETERMINATION

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The crystal structure of nicotinic acid,  $C_6H_5NO_2$ , first determined by Wright & King [Acta Cryst. (1953), 6, 305] has been refined using new X-ray data to an R value of 0.074. Some of the bond lengths, corrected for rigid body motion, have changed significantly from their values reported earlier by as much as 0.05 Å. Basically, the structure proposed by Wright & King (ibid) is correct. The hydrogen bond is of 'bent' type. The carboxyl group is rotated out of the pyridine ring by  $6.8^{\circ}$ .

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#### S41. THE CRYSTAL AND MOLECULAR STRUCTURE OF 1-DIPHENYLMETHYL AZETIDIN-3-OL

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In connection with the studies of the cholinergic receptor using rigid or less flexible molecules the crystal and molecular structure of the title compound has been investigated. The crystals are triclinic with  $a = 8.479 \pm 0.002$ ,  $b = 17.294 \pm 0.004$ ,  $c = 10.606 \pm 0.003$  Å,  $\alpha = 118.59 \pm 0.02^{\circ}$ ,  $\beta = 100.30 \pm 0.02^{\circ}$ ,  $\gamma = 89.63 \pm 0.02^{\circ}$ , space group  $P\bar{1}$  with  $Z = 4$ .

The crystal structure was determined by the symbolic addition method as applied to centric space groups. All the 36 non-hydrogen atoms were located in the E-map. The R factor at the present stage of refinement is 6.6%. The conformation of the two independent molecules in relation to molecular packing and also the details of hydrogen bonding will be discussed.

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S42. ON THE FORM OF TORSIONAL POTENTIAL IN ACETYL-GLYCINE N-METHYLAMIDE (AGMA)

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Vibrational spectra were calculated for AGMA using the method outlined by Dakhis et al. [1] with the following exceptions: a two-fold potential was used to represent barrier to rotation around the C—C bond:

$$V_{\psi} = \frac{1}{2} K_{\psi} (1 + \cos 2\psi)$$

The values of  $K_{\phi}$  and  $K_{\psi}$  chosen were the ones that are being used in our unit and are 1.0 and 0.6 kcal/mole, respectively. The trend of the isofrequency regions obtained was found to be very similar to the ones obtained by using a three-fold potential and extremely high barriers to rotation by Dakhis et al. [1]: the low frequency regions appeared smooth and the high frequency maps revealed behaviour very similar to the one reported by Dakhis et al. [1]. The  $(\phi, \psi)$  angles were found to be  $(90^{\circ}, 180^{\circ})$  for form A and  $(90^{\circ}, 0^{\circ})$  for form B. Dakhis et al. [1] reported the following values  $(90^{\circ}, 180^{\circ})$  for form A and  $(90^{\circ}, 15^{\circ})$  for form B.

Thus it seems doubtful if the present method [1] will be able to shed any light on the form of the torsional potential in AGMA in particular.

Reference

- 1 M. I. Dakhis, V. G. Dashevsky, and A. I. Kaitaigorodski, *Biopolymers* 12, 1763 (1973).

S43. CHARACTERIZATION AND CRYSTALLIZATION OF YEAST  
tRNA NUCLEOTIDYL TRANSFERASE

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tRNA nucleotidyl transferase purified 6000-fold from baker's yeast by the method of Cramer, but largely modified by us in our laboratory, has been characterized before crystallization. The ultraviolet spectrum of the purified enzyme shows a maximum at 278nm and a minimum at 250nm (ratio  $A_{278}/A_{250} = 4.00$ ). The ratio of absorbance at 280nm to 260nm is on the average 2.10.

The enzyme is homogeneous on equilibrium ultracentrifugation and by Sodium dodecylsulphate (SDS) gel electrophoresis. The molecular weight of the enzyme is  $5.2 \times 10^4$  as determined by SDS gel electrophoresis and  $10.80 \times 10^4$  as determined by equilibrium ultracentrifugation using Yphantis technique, thus leading to the conclusion that the enzyme has subunit structure which differs considerably from Cramer's model.

Purified tRNA nucleotidyl transferase has been crystallized for the first time in our laboratory, at pH's near its isoelectric point from sodium citrate solution using vapor diffusion technique of Hampel. The crystals consists of long needles with well formed faces and edges. But they are too thin to get good diffraction patterns, when one could determine the unit cell dimensions and assign the space group. However, still precession photographs taken at  $300^\circ$  for 12 hrs with Cuk radiation from Elliott rotating anode source indicate regular patterns.

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