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A simple temperature monitor for the PW1100 diffractometer

During collection of X-ray intensities at temperatures different from room temperature it is advisable to have some method to monitor the temperature. This recording is essential to check the proper functioning of the cooling apparatus and facilitates the estimation of the long- and short-term stability of the temperature.

It is the purpose of this note to describe a simple way to incorporate the temperature information in the information of the diffraction experiment. For that reason a digital voltmeter has been connected to our diffractometer [Philips PW1100, equipped with a P9205 (8K) computer, PW1115 cassette recorder system and Leyboldt Heraeus NCD1 cooling device].

The voltage of the controlling thermocouple of the cooling device is measured by an Analogic AN2559 digital voltmeter (15 bits) with a parallel BCD output option. The BCD output is connected to the high-speed paper-tape reader interface of the computer, which is not used in our diffractometer. The interface as supplied by the manufacturer is able to handle eight parallel lines. Therefore the BCD output (with the sign information dropped) is split into two seven-bit words to which one bit is added for identification purposes. The temperature information can then be read into the computer by two read instructions. With a copper/constantan thermocouple a resolution of 10 μV can be used from liquid-nitrogen temperature to room temperature. The corresponding temperature resolution is 0.5 K at 100 K. For temperatures above 150 K a resolution of 1 μV can be used.

A routine to handle the temperature data is incorporated in the data collection program. The temperature is read before the reference reflections are measured. The output of the temperature data is either on the teletype or at the magnetic tape recorder. When no low-temperature measurement is made the temperature monitoring can be suppressed by means of one of the sense switches on the computer. The memory locations for the temperature routine have been obtained by removing the balanced-filter subroutine

in this program version. A provision has also been made to record for each reflection measurement the status of the buffer Dewar vessel (empty/filled).

A more detailed description of the hardware and software involved can be obtained from the first author.

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Meeting Report

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Fourth European Crystallographic Meeting

The Fourth European Crystallographic Meeting was held in Oxford from August 30 until September 3, 1977. It attracted 950 participants, more than twice the number expected, but the city and university of Oxford seemed well able to cope with such an influx of scientists. The format of the meeting was very successful. A general lecture was held in the Playhouse from 9 a.m. to 10.15 a.m. each day. The rest of the meeting was held in the Department of Zoology (the home of protein crystallography in Oxford). Simultaneous symposia, that is, lectures on selected topics, were held until lunchtime. Poster sessions, a new one each day, were held in the afternoons, and workshops were run from 4 p.m. to 6 p.m. One early afternoon and two evening sessions were held as open meetings of IUCr Commissions. On the first evening there was a reception in the Examination Schools, and a dinner and party at St. Catherine's College were held on the last evening. Both were lavish and enjoyable affairs. In addition one afternoon was devoted to excursions into the surrounding countryside. There were several exhibits of instruments, and one of books by Blackwells, the notable Oxford bookstore, which made an effort to have copies of books authored by participants in their display.

The four invited lectures set the tone for the symposia and posters for the rest of the day. Each one of these lectures described present technology and experiments but also gave us a glimpse into the future. David Sayre (USA) discussed the experimental problems and advantages of both diffraction and microscopy by X-rays, electrons and neutrons. The use of synchrotron radiation was reviewed by Kenneth Holmes (West Germany). Details of the properties of such radiation, which will be extremely useful in the next few years for studies of biological macromolecules, were outlined. Dorothy Hodgkin (UK) gave a historical account (1935–1977) of studies of insulin from the first X-ray photographs to the three-dimensional structure determined in the last few years. In particular, she was able to explain many early experimental observations in light of our present knowledge of the structure of the hormone. Raymond Weiss (France) reviewed the extensive contribution that X-ray crystallography has made to our understanding of the chemistry and stereochemistry of complexation by macrocyclic ligands and the reasons for the specificities, if any, of these ligands. Workshops were arranged on subjects such as synchrotron radiation, protein structure refinement and the use of the Cambridge structural data file. They gave workers in similar fields an excellent chance to talk about their common experimental or computational problems.

Several topics were discussed at a meeting of the IUCr Commission on Crystallographic Apparatus. These included X-ray safety standards, the status of the current microdensitometry intensity project, the best techniques available for the accurate measurement of intensities from powder samples, and current problems encountered in measuring intensities in small-angle scattering experiments. Programs for minicomputers, computing packages for the solution of small structures on a routine basis, and the use of interactive computer graphics as an electronic Richards box to trace the polypeptide or polynucleotide chain in the electron density maps of macromolecules were described at the meeting of the IUCr Commission on Crystallographic Computing. The IUCr Commission on Crystallographic Teaching reviewed the problems of teaching crystallography to non-specialists and of helping non-crystallographers to understand the crystallographic literature. These two themes were continued in the Summer School in Erice, Sicily, which followed immediately after the Oxford meeting.

In connection with radiation sources, pulsed neutron sources were described in symposia. In X-ray energy-dispersive diffractometry using synchrotron radiation all peaks appear simultaneously while the crystal-diffractometer is in a fixed geometry. The uses of synchrotron radiation sources in studying X-ray topography, phase transitions, domain wall motion and plastic deformation were described, and the advantages of using the polarization properties of the beam were stressed. Experiments in progress to test the feasibility of studying the structures of excited-state species by X-ray analysis were described. Crystal defects and structure building principles were considered in detail and stressed the complementarity of X-ray and electron microscopy techniques. Superstructures and polytypic transformations were also discussed. The symposium on industrial chemistry concentrated on powder film data, particularly its use in identification and structure determination. Texture analysis and the interpretation of multiple diffraction patterns from epitaxial layers was also discussed.

Studies of relevance to pharmaceutical chemistry involved, particularly, work on enkephalins which have a morphine-like activity. It had always been puzzling that there are morphine receptors in the brain, since morphine is not a compound that occurs naturally in the body. It is now believed that the receptors are specific for enkephalins, naturally occurring peptides, and that morphine, which has a more rigid structure, also binds to these same receptors. Similarly a sleep factor may have a natural tranquilizing action and explain why there are receptors for valium and librium in the brain. A three-dimensional study of structure vs activity is of great interest in this area and is now in progress. In addition the conformations of other drugs (anticonvulsants, GABA analogs, and psychoactive drugs) were discussed.

The posters, over three hundred of them, provided a fantastic blaze of information, and all were presented in the light and airy rooms of the Zoology building. I hesitate to write about them because space in this journal is limited, and because so many of the posters deserve detailed mention. All I can do is describe a few, together with symposium topics, list some categories, and hope that, somehow, readers of this report will be able to get hold of the two hefty booklets of abstracts, 738 pages in all, and delve more deeply into each subject.

Many posters were devoted to the study of crystal defects. Several techniques can

be profitably used to approach this problem. For example, as described for natural and synthetic diamonds, X-ray topography can be used together with cathodoluminescence topography and the results may be correlated with data from transmission electron microscopy. Many other posters stressed the use of electron microscopy plus X-ray topography. It was shown that neutron diffraction topography is useful for the study of compounds such as cerusite (PbCO_3) which has a high linear absorption factor for X-rays.

A lively discussion of direct methods occurred in the symposia and continued at the poster display. The neighborhood principle, quartet and quintet relations, magic integers and the new *MULTAN* seemed particularly prominent. The random approach to structure determination was discussed. In this 50–100 sets of random phases for about 80 reflections were refined by a set of linear equations and a figure of merit was used to select the ten best sets and then to develop more phases. Many computer programs and algorithms were described. Direct methods using difference structure factors were advocated, particularly when the structure contains heavy atoms in special positions. The method of structure determination which uses molecular averaging was described. This is an iterative procedure that is useful for compounds, such as viruses, and allosteric enzymes that have non-crystallographic symmetry. In each cycle the electron density map is computed using current phase angles; an averaged electron density for an asymmetric unit is then calculated and subjected to a Fourier inversion to give new calculated structure factors and phases. Constraints may be applied on certain phases determined by isomorphous replacement if necessary. Protein phases may also be determined with resonant neutron scattering in which, as suggested some years ago, Bijvoet intensities are measured at two or more wavelengths.

Other computer-based techniques described involved analyses of screenless rotational photographs, anisotropic scaling of three-dimensional data, analysis of rigid-body motion, the determination of bonding electron densities, the calculation of absorption effects, what to do about 'negative intensities' and how to analyze Bragg reflection profiles.

At one symposium it was shown how structural changes along pathways of chemical reactions can be obtained from a detailed analysis of structural results. The molecule or molecular fragment distorts on interaction with an environment. Host-guest packing and chemistry, for

example the photoaddition of ketones to deoxycholic acid for which the isomer formed is that expected from molecular packing in the crystal, was also discussed. A detailed review of the importance of side chains in protein chemistry was given, especially for acid proteases where the differential reactivities of certain side chains may be correlated with their three-dimensional locations in the protein. In the case of hormones the fact that the molecule has to fold so that hydrophobic groups are forced into solution gives information on the probable binding sites to a receptor. Many interesting posters were given on the subject of structure and reactivity. For example in ketoacid-lactone alcohol equilibria a lone pair on an adjacent oxygen atom antiperiplanar to a C–O bond was considered important in lengthening and weakening this C–O bond. Anthracene was reexamined and a new stress-induced phase transition was found and studied. In this new crystalline phase the distance between C(9) in nearby molecules is reduced over that in the normal anthracene structure. This may be the form that undergoes photodimerization. In several cases initial, intermediate and final structures in a reaction pathway were studied. It was pointed out that the Cambridge data file contains much information for such a study of reaction pathways.

All kinds and sizes of molecules of biological interest were represented at this meeting. In studies of viruses, electron density maps at 2.8 Å resolution were described for tobacco mosaic virus protein disk. This molecule consists of subunits, each of molecular weight 17000, related by 17-fold non-crystallographic symmetry. Views of the protein portion of the molecule were shown, and these, together with results from the 4 Å resolution map of the virus, have given some information on the mechanism of virus assembly. Icosahedral tomato bushy stunt virus, with 180 subunits of molecular weight 40000 each, was also described. Recent work on the two-dimensional crystalline patches in the purple membrane of halobacteria was described with emphasis on trying to find out how the protein, which has been shown to span the membrane as a series of α -helices, is involved in the pumping action. Labeling with ferritin molecules showed which side of the protein is on the outside of the membrane.

Structures reported included those of the lithium salt of NAD^+ which showed that the coenzyme is in an extended form, the potassium salt of phosphoenolpyruvate in which the anion has a different

shape from that found in the cyclohexyl ammonium salt, and a tetradeoxynucleotide of alternating adenine and thymine bases which showed base pairing. The intercalation of flat molecules in DNA was studied by way of complexes with dinucleotides. A 3:2 complex of proflavine and cytidyl (3'-5') guanosine (CpG) was shown and two forms of crystals of CpG complexed with ellipticine were reported on. Steroid-like molecules described included some neuromuscular blocking agents, and some cardioactive steroids including cinobufagin (from the dried venom of the Chinese toad) and the glycoside ouabain. Conformational transmission was analyzed for some progesterone derivatives. The structures of some semi-synthetic penicillins were compared and the structure of an antibiotic, nosiheptide, molecular weight 1222, with a 26-membered macrocyclic ring, was determined. Studies of bile pigments and bilirubin were described. Transport of ions was investigated by studies of some ionophores, such as anniatine B and its rubidium complex and the barium picrate complex of beauvericin.

Many protein structures were displayed in posters. The effect of substrate and inhibitor binding on the conformation of the protein was the subject of much interest since this can give some information on how the protein works. The binding of water, cyanide and carbon monoxide to the insect hemoglobin erythrocrurin was analyzed (1.4 Å resolution) in terms of shifts in the relative positions of the globin, heme, iron and histidine groups. In phosphorylase *b* (3 Å resolution) the active site, a nucleotide-binding site, a glycogen-binding site, and an inhibitor-binding site were identified. Chicken triose phosphate isomerase was studied to 2.5 Å resolution and the effects (at 6 Å resolution) of phosphate, phosphoglycolate (a postulated transition state analog) and dihydroxyacetone phosphate, each of which cause a conformational change in the protein on binding, were discussed. A list of other proteins displayed on posters includes: seal myoglobin (2.5 Å resolution), cat muscle pyruvate kinase (2.6 Å resolution), 6-phosphogluconate dehydrogenase (6 Å resolution), an acid protease from the microorganism *Endothia parasitica* (2.7 Å resolution), actinidin which is a sulfhydryl protease with similar kinetic properties to papain (2.8 Å resolution), penicillinase which inactivates penicillin by converting it to penicilloic acid (4 Å resolution), prealbumin which transports vitamin A and thyroid hormones and which has sites that could be complementary to double-stranded DNA although prealbumin

is not known to bind DNA (1.8 Å resolution), a bacteriochlorophyll-protein from green photosynthetic bacteria containing seven porphyrins per subunit, a bacterial cytochrome C_3 (3 Å resolution), apoferitin (molecular weight 440000, data to 2.8 Å resolution) which is a hollow shell with inner and outer diameters 80 Å and 130 Å containing six channels 10–17 Å across, and the A fragment from bovine prothrombin (156 aminoacids, 20% carbohydrate, 2.5 Å resolution). A comparison of the tertiary structures of hen egg-white lysozyme and baboon lactalbumin was described. Other protein studies of interest include neutron diffraction studies of triclinic hen egg-white lysozyme and a comparison with the X-ray structural data, low-angle neutron diffraction studies of tomato bushy stunt virus using varying $H_2O:D_2O$ mixtures and aimed at distinguishing water from virus, and small-angle neutron and X-ray studies of collagen. In the last case the mineralization of turkey leg tendon was studied and it was shown that there is a definite structural relationship between collagen and hydroxyapatite when they are laid down when tissue is calcified *in vivo*.

Several posters considered the problem of 'bound water', that is, water attached to a protein and which has different properties from bulk water. The characterization of this 'bound water' seems to depend on the physical technique used to detect it, although the fraction of water that cannot be frozen readily seemed to be generally accepted as 'bound water'. Techniques used for analysis include infrared spectroscopy, differential scanning microcalorimetry, proton magnetic resonance spectroscopy and low-angle neutron diffraction. In the area of hydrogen bonding the importance of the structural surroundings of a molecule were stressed. As a result it may not be possible to predict hydrogen bonding from purely geometrical considerations of an isolated molecule.

A multitude of posters were concerned with organic structures. Several salts of the complexing agent, 7,7,8,8-tetracyanoquinodimethane (TCNQ), were studied with respect to structure and electronic and magnetic properties. The extent of molecular overlap and the inter TCNQ distances in crystalline complexes were analyzed at various temperatures in some semiconducting complexes. In addition the extent of charge transfer was inferred from measured bond lengths. Several halogenated hydrocarbons were studied and it was shown that the geometry of such fluorinated molecules differs only slightly from that of the parent hydrocarbon. In

polychlorinated biphenyls the distortions are more noticeable. Some sugars, including compounds with sulfur in place of the ring oxygen atom, compounds prepared by the interaction of sugars with amines, and some unsaturated sugars, were reported on. The geometry of the malonate ion in various compounds showed that chelate formation stabilizes the planar conformation of this anion, but in order to do this the C—C—C angle is deformed. In the nonchelated form this angle is nearly tetrahedral and the oxygen . . . oxygen separation is increased. Other anions such as oxydiacetate and various thiooxalates were also reported on.

The structures of some host-guest compounds, notably a cyclodextrin polyiodide complex which can be considered a model for the blue starch iodide complex, were displayed. In this complex the iodine lies in channels as triiodide and pentiodide. The interactions are hydrophobic with no iodine . . . oxygen interactions. Cycloheptaamylose complexes containing *p*-substituted acetanilides were also described. Compounds which can exist in a variety of phases, some liquid crystalline, were studied in the crystalline phase and the nature and differences of packing in nematogenic and smectogenic crystals were analyzed.

Conformations in some novel compounds were investigated. For example, 1-amino substituted 1*H*-1,2,4,6-thia(IV) triazines consist of a six-membered ring with the sulfur atom 0.3 Å out of the plane of the other five atoms and the substituted amine group linked pseudo-axially to the sulfur atom. Some cyclophanes with donor or acceptor groups substituted into the rings were studied in order to determine the packing arrangements of intramolecular EDA complexes and compare them with similar π complexes between free molecules. Analyses of the effect of substituents on ring bond angles were continued for a series of phenyl derivatives. The angle at the point of substitution may be correlated with the electron-withdrawing or -donating activity of the substituent group.

The high-resolution crystal structure of two forms of a polymer, poly(oxyethylene) was reported. It is unusual that high-quality crystals suitable for such an analysis can be obtained from polymers. The asymmetric unit contains only the $-CH_2-O$ group. Two modifications, one orthorhombic and well determined, and the other described as 'hexagonal', were studied. The 'hexagonal' form is helical and a 38/21 helix was chosen as the first approximate model. The torsion angles differ in the two forms.

Many inorganic structures were also presented. Several could be described by the packing of spheres, such as $A_3B_2X_9$ ($A = \text{Rb, Cs}$; $B = \text{As, Sb}$; $X = \text{Cl, Br, I}$) which consists of hexagonal AX_3 layers with B in octahedral interstices. $\text{LaFe}_2\text{P}_{12}$ is a structure derived from CoAs_3 structures by filling distorted icosahedral and octahedral voids with lanthanum and iron atoms respectively. The trigonal pyramidal nature of As(III)-O stereochemistry was noted. In compounds $\text{MP}_{7-x}\text{As}_x$ ($M = \text{Li, Na}$) the distribution of phosphorus and arsenic is not statistical, but depends upon the formal charge at the position and the electronegativity of the atom filling it. The I_3Cl_2^- ion in I_3SbCl_6 was shown to have a linear arrangement of iodine atoms with a chlorine atom at each end arranged perpendicular to the I_3 group. Silver iodide complexes Ag_2INO_3 and $\text{Ag}_2\text{I}_2\text{F}_5$ were studied and K_2FeF_6 was shown to exist as infinite zigzag *cis* chains of FeF_6 octahedra. The shapes of the phosphorus network in polyphosphides were also investigated.

Other structural studies included ternary phosphorus chalcogenides, the TeO_3^{2-} ion, cadmium germanates and some silicates such as $\text{K}_3\text{NdSi}_6\text{O}_{15}$ and $\text{K}_2\text{CeSi}_6\text{O}_{15}$. Several minerals such as hohmannite, malachite, gibbsite, biotites, and tvalchrelidzeite were studied. In the case of silicates the chains formed by SiO_4 tetrahedra can be linear or branched. The periodicity (number of tetrahedra in the identity period of the chain) depends on the cation present. In such polymers the Si-O-Si angle can deviate appreciably from the mean value of 140° . In the case of some barium silicates, high-resolution electron microscopy was used to show the variation in chain multiplicity.

Magnetic and ferroelectric properties were the object of many studies. The anti-ferromagnetic domain distribution in nickel oxide was studied by magnetic neutron Laue diffraction which allows the arrangement and direction of magnetic moments to be determined. The ferroelectricity associated with the high-temperature phase of ammonium nitrate was investigated and a structural basis involving nitrate groups was suggested to account for this effect. Charge transfer in $\text{M}_x\text{Mo}_3\text{S}_4$ compounds from the interstitial cation M to the closely packed octahedron formed by the molybdenum atom is probably an important factor in this class of high-field superconductors. Also the semiconductor-semiconductor phase transition of V_3O_5 at 155° , which involves a change in crystal structure, was studied by a comparison of both structures. The paraelectric phase of CsH_2PO_4 was also described. Some octa-

hedral complexes of Cu(II) and Mn(III) were studied with respect to Jahn-Teller distortions. Cu(OH)_6^{2+} shows four Cu-O distances of 1.96–1.97 Å and two of 2.81 Å. Other examples of (4+2)-distorted coordination octahedra were shown. The MnF_6 group in the compound $\text{Rh(NH}_3)_6$. MnF_6 is regular, implying a dynamic Jahn-Teller effect.

Many organometallic and coordination compounds were examined. Cubane-like structures were found containing four of each Co(II) and F^- or Ni(II) and OH . A tetrahedral Ru_4 cluster was found to contain two short and four longer Ru-Ru edges. Many chelated metal salts were studied, for example neodymium fumarate, some iron(III) dithiocarbamates and many complexes of Cr(III) , Pt(II) , Cu(II) , Zn(II) and Ni(II) . Some Pt(I) complexes contained Pt-Pt bonds and a Pt(0) complex, $\text{Pt(C}_2\text{H}_4)_2(\text{C}_2\text{F}_4)$ was shown by neutron diffraction to be a π complex and its geometry was described. Some niobium(V), indium(III) and Ni(II) porphyrins were studied. Some very interesting transition-metal hydride coordination complexes were studied by neutron diffraction. A metal-metal (Re-Re) bond bridged by four hydrogen atoms was found. A neutron diffraction study of hydrated $\text{Na}_3\text{Mo}_5\text{PO}_3(\text{OH})_3$ showed an anion structure consisting of a wide channel 6 Å diameter which contains water.

The meeting was also a celebration for Dorothy Hodgkin on the occasion of her retirement, although, of course, she will continue working as ever. Many of her former students, from countries all around the world, made a point of attending the meeting. Gifts were brought for her and presented at the end of the meeting. It was hoped that the excellent scientific caliber of the meeting might serve as an appropriate tribute to her.

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Crystallographers

This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).

Professor **R. A. Cowley**, Professor of Physics at the University of Edinburgh,

Dr **A. Howie**, lecturer in physics at the University of Cambridge, Dr **R. B. Nicholson**, Managing Director of Inco Europe Ltd, London, Dr **D. A. Ramsay**, principal research officer at the National Research Council of Canada, Ottawa, and Professor **J. V. Smith**, Professor of Mineralogy and Crystallography at the University of Chicago, USA, have been elected Fellows of the Royal Society.

Dr **David Harker** has joined the staff of the Medical Foundation of Buffalo, USA, as a Research Scientist Emeritus.

Dr **H. R. Morris**, of the Department of Biochemistry, Imperial College, has been awarded the BDH Award in Analytic Biochemistry for 1978.

International Union of Crystallography

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