DARESBURY LABORATORY

INFORMATION QUARTERLY for PROTEIN CRYSTALLOGRAPHY

An Informal Newsletter associated with Collaborative Computational Project No. 4 on Protein Crystallography

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Editor: Sue Bailey

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EDITORIAL

Thanks are due to the contributers to this edition of the newsletter and to Don Akrigg for organising the collection of contributions. A copy of the papers in this newsletter have been sent to Keith Wilson for inclusion in the EACBM (European Association for Crystallography of Biological Macromolecules) newsletter.

Apologies are made for the late arrival of this edition of the newsletter, this is mainly due to a shortage of contributions, but is also caused by a lack of personnel to look after CCP4 since my resignation. Two posts have been advertised and it is hoped that appointments will be made in the near future. In the meantime, John Campbell can handle any urgent enquiries.

Please note that this is not a formal publication and permission to refer to this document should be sought from the authors.

Sue Bailey

Postscript. I'm sure that all the protein crystallography community will wish to extend their thanks to Sue Bailey for her conscientious efforts on behalf of CCP4. This includes the work she has voluntarily put in since finishing at Daresbury. We wish her all the best in her new appointment.

Colin Nave

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DEPARTMENT OF CHEMISTRY
Professor G. G. Dodson

4 January 1988

Dear colleagues

The issue of depositing atomic coordinates when macromolecular crystal structures are reported in the literature needs to be resolved. In order to get the views of the crystallographic community a draft policy document is being circulated throughout the world. The circulation in North America is being managed by an ad-hoc committee, chaired by Charlie Bugg; in Europe the circulation is being carried out via the EACMB. It seems sensible however to include the draft in the CCP4 newsletter to be quite sure the UK community has its own opportunity to decide what it believes is a desirable course of action by the Commission of Macromolecular Crystallography.

There is a parallel initiative by Fred Richards (Yale University) who with a group of crystallographers is circulating a letter to laboratories for supporting signatures. The proposals in Fred Richards letter are more stringent than those in the draft document. A copy of Fred Richards letter is also included for information and for you to respond to (to Fred Richards) if you wish.

I would be grateful, if you have not already done so, to let me have your views on the proposals in the draft document and Fred Richards circular letter. I would also be grateful if you could send copies of your replies to Jan Drenth, (Laboratory for Physical Chemistry, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands)

G G Dodson

DRAFT

POLICY ON THE DEPOSITION OF DATA AND RESULTS FROM CRYSTALLOGRAPHIC STUDIES OF BIOLOGICAL MACROMOLECULES

PREAMBLE

Crystallographic analyses of protein, nucleic-acid and virus structures produce an extraordinary amount of information, and these results are widely recognized as having unique scientific value. Available information transcends that which can be recorded in usual scientific publications, and the Protein Data Bank is used as a supplementary repository for such results. It is also imperative that, as in all science, sufficient information be published so that the structural results can be reproduced and verified. While the crystal itself is the ultimate repository of crystallographic information, the diffraction data recorded in an investigation have especial value in this regard. These data are so voluminous that they too must be deposited as supplementary information, and the Protein Data Bank is used for this purpose as well.

The importance of preserving the fundamental data and results from diffraction studies is recognizedby producers and users of this information alike. There are, however, concerns that results from the early stages of analysis will be inaccurate in detail and that investigators should have the opportunity to complete the analysis and interpretation of their hard-won data without opportunistic competition. On the other hand, an open-ended protection of producer interests tends to conflict with the general scientific good and it creates the risk that valuable information will be lost forever when well-intentioned continuations are never completed. Accordingly, the deposition policy promulgated below takes a strong stance on immediate deposition of atomic coordinates and diffraction data supporting publications in this area, but it provides for the possibility of a specified delay in the release of this information to public use.

POLICY

The Commission on Biological Macromolecules of the International Union of Crystallography endorses a deposition policy for crystallographic studies to permit independent verification of the results and to preserve the primary data for future use. Scientific publications reporting results from crystallographic determinations of macromolecular structure should be accompanied by a deposition of atomic coordinates and structure factor information at a level appropriate to the description given in the paper. This policy is commended to investigators who publish crystallographic results and to all journals that accept publications of this kind. Its provisions should also be respected by users of these results. Specific provisions of the policy are elaborated below.

Provisions for Atomic Coordinates. Two different levels of description arise with respect to coordinates in typical situations. In the case of chain-tracing descriptions, then the alpha-carbon coordinates for proteins or phosphorous positions for nucleic acids would be appropriate for deposition. If the interpretation presented depends on atomic details as shown in figures of side chains or numbers derived from atomic coordinates, then the full coordinate list should be supplied. Atomic displacement parameters (B-values) and occupancy factors that are part of a model should also be deposited. It is essential that coordinates be deposited even when the atomic model has not been refined to completion. *In lieu* of appropriate error estimates, investigators might flag particularly unreliable regions as judged by prior stereochemistry or exceptionally high B values.

Provisions for diffraction data. With respect to the diffraction data, native structure factor magnitudes should be deposited out to whatever limit of Bragg spacings is stated

in the paper. The deposition of additional data used in the phase determination (heavy-atom isomorph data, Bijvoet mates, multiple wavelength measurements, etc.) is encouraged. In the case of structure reports that do not involve atomic models (e.g. low resolution studies) both structure amplitudes and phases used in Fourier syntheses that are reported should be deposited.

Provisions for publications in methodology. The policy applies to reports on structural results. Those papers that purely describe advances in methodology are exempt from this policy even if diffraction data or structural results were required for the development.

Provision for manner of deposition. The Protein Data Bank at Brookhaven National Laboratory is recognized by the Commission on Biological Macromolecules of the International Union of Crystallography as the appropriate repository for results from macromolecular crystallography. Accordingly data should be deposited in machine readable form as instructed by the Protein Data Bank.

Provision for delayed release. The preliminary nature of initially reported results from such studies is recognized as is the need to protect these data for the investigators themselves to complete the analysis made possible by their experimental investigation. Provisions for deposition of diffraction data from studies reported in Acta Crystallographica already allow investigators to hold structure factor depositions from release for up to four years. Similarly it should be possible to hold preliminary atomic coordinates from release for up to N=2, 3, 4 years from date of publication. Depositions of atomic coordinates from reports of completed refinements should continue, as in present policy for several journals, to be released immediately upon verification by the authors.

ENFORCEMENT

The provisions of this policy will be satisfied by a required inclusion in the publication of a statement to the effect that "the atomic coordinates and structure factor data described here have been deposited in the Protein Data Bank". Journals should notify authors that such a statement will be required and the Protein Data Bank should expect cooperation from authors in meeting this obligation of publication.

USER OBLIGATIONS In addition, the provisions for deposition of coordinates and primary data from crystallography should also apply in an analogous way to results of like kind from NMR spectroscopy and from theoretical studies. Users of deposited data should recognize an obligation to cite the primary references rather than the Protein Data Bank when making use of data.

Yale University

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28 October 1987

Dear Colleague,

We are writing to you because of our increasing concern with the preservation of and access to macromolecular structure data and the derived molecular models. It is our intent to send the enclosed letter to the editors of a number of major journals in which such studies are usually published. The content of the letter is self explanatory. The details of the proposal, which will certainly evolve, are less important than the general thrust. We hope very much that you would be willing to cosign this letter.

Since we are trying to get as much support as possible, it is unrealistic to attempt to get all of the original signatures on one letter. If you will join us, we ask that you sign and date the second copy of the final page and return it to F.M.Richards in the enclosed envelope.

Sincerely yours,

Frederic M. Richards
Frederic M. Richards

Richard E. Dickerson
Richard E. Dickerson

Jane S. Richardson
Jane S. Richardson

Michael G. Rossmann
Michael G. Rossmann

David C. Richardson
David C. Richardson

D R Davies
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Joseph Kraut
Joseph Kraut

Don C Wiley
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Dear (Editor):

The undersigned have a long standing concern with the problem of public access to the results of single crystal X-ray diffraction studies on biological macromolecules. The actual data from such research are the measured X-ray intensities, and the primary results are the lists of atomic coordinates derived from those data. While many papers on various proteins and nucleic acids have been published describing and interpreting the results of such a structure analysis, the actual data and results are often not made easily accessible, if at all. We are requesting that the journals which play a major role in the publication of such structural studies adopt and enforce rules for documentation similar to those which apply in all other areas of scientific research. Studies where the structural information has not been made available must be considered incomplete, as would any other piece of research where the data were not provided in published or deposited form.

In the closely related field of small molecule crystallography there is no such problem. The actual X-ray data and the derived structural model are reported directly in the original article in hard copy form. Even for a small protein the amount of data is so large that hard copy publication has occurred only in one or two instances in the early years, but computer readable storage and distribution as currently carried out so effectively by the Protein Data Bank at Brookhaven and its international associated groups is quite practical, and is certainly the preferred form of access today. We note also that a policy requiring coordinate and structure factor deposition would ensure the preservation of important and expensively determined data which otherwise are likely to be lost with the ever-changing personnel and computer installations in numerous laboratories around the world.

There are standard procedures now for the refinement of X-ray structures. Preparation of the data and results for the Protein Data Bank does not constitute a significant burden to authors. The remarks section of the Data Bank file provides plenty of space for any qualifying or expanding remarks that the authors may wish to make. The standard author comment that a structure is not "finished" or not "ready" for filing is just nonsense. If a structure is ready to be discussed in a paper, by definition it is ready for filing. Data and coordinate files can always be updated on the basis of future work, as has already happened many times.

We request that the (Journal) include in its Notice to Authors a paragraph such as the following or one of equivalent intent.

> "Authors of papers describing new structure determinations must be prepared to submit to the Protein Data Bank all of the structural data required to validate the discussion, including both x-ray amplitudes and phases and the derived atomic coordinates. If the paper discusses a protein structure only at the level of the main chain alpha carbon atoms, then only alpha carbon coordinates need be deposited. If the discussion involves higher resolution data, for example all atoms in the active site of an enzyme, then the full set of X-ray data and the coordinate list must be deposited. Following completion of the editorial process and acceptance of the paper, the manuscript will not be sent to the printer until confirmation has been received from the author, if not initially supplied, that the required information has been sent to the Protein Data Bank.
>
> If requested by the authors, the editors will ask the Data Bank not to distribute the information until a specified date. For coordinate lists this date may not be more than one year beyond the acceptance date of the manuscript. For the full structure amplitude and phase data the time interval before distribution may not exceed four years. The release date specified by the author will appear in a footnote to the paper along with the statement that the information has been submitted to the Protein Data Bank. In the absence of a specified release date, it will be assumed that the information is available immediately on appearance of the publication."

Comparable letters have been sent to a number of journals concerned with macromolecular structure. We are aware that several editorial boards are considering this matter, and that a major effort on policy development is underway by the International Union of Crystallography and by certain of its adhering national bodies. We hope that all will act positively (and, if possible, uniformly) on this issue.

A list of the signers of this letter follows. The original signatures are on file in office of the undersigned.

Sincerely yours,

Frederic M. Richards
Corresponding cosignatory
on behalf of the following individuals

STRUCTURAL STUDIES ON AN NADP$^+$ DEPENDENT MALIC ENZYME FROM RAT LIVER

A.Rob, D.W.Rice, P.J.Baker, D.H.Thomas, H.F.Rodgers & E.Bailey

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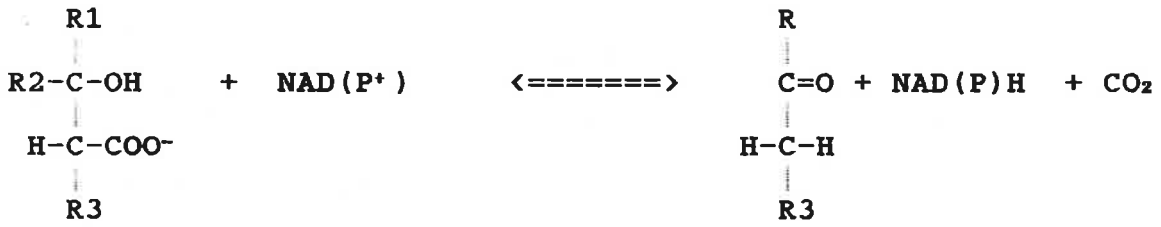
Rat liver malic enzyme (EC 1.1.1.40) catalyses the conversion of malate into pyruvate (Fig 1) and is one of the major NADPH generating enzymes of the lipogenic pathway . Therefore it has been grouped with other enzymes important in lipogenesis such as ATP-citrate lyase,fatty-acid synthetase and the pentose-phosphate pathway dehydrogenase.This enzyme was first reported in 1947 (Ochoa *et al*) and since then it has been isolated from a variety of sources eg micro-organisms,plants and animals (rat,ox,chicken and pigeon).In all , three related enzymic activities have been reported : two NAD$^+$-linked (one of which additionally catalyses the decarboxylation of oxaloacetate) and one NADP$^+$ - linked .The NADP$^+$ linked enzyme has been the subject of much study and has been shown to be composed of 4 identical polypeptide chains, each of MW 65000 Daltons. Recently a complete amino sequence for this enzyme has been determined from 3 overlapping cDNA clones (Magnusson *et al* ., 1986) .

Fig 1. Generalised catalytic scheme for NADP$^+$ dependent oxidative decarboxylases



R2=H,R=R1=COO^- , R3=H : MALIC ENZYME

R1=H,R=R2=$(CHOH)_2 CH_2 OPO_3^{2-}$, R3=OH :6-PHOSPHOGLUCONATE DEHYDROGENASE

R1=H,R=R2=COO^- , R3=$CH_2 COO^-$: ISOCITRATE DEHYDROGENASE

The reaction catalysed by malic enzyme is an example of an oxidative decarboxylation . Thus in terms of the chemical transformation that it brings about ,it shows a strong resemblance to 6-phosphogluconate dehydrogenase (6PGDH) and isocitrate dehydrogenase (ICDH).Comparision of the amino acid sequence for malic enzyme and 6PGDH have failed to reveal any extensive sequence homology between these two enzymes.Therefore it is unlikely that a detailed structural relationship exists between them.In fact differences must exist since the catalytic reaction of malic enzyme shows a requirement for a divalent metal not shown by 6PGDH.Nevertheless the possibility remains that their catalytically active groups may be in some way related and this in itself represent a compelling reason for the structural analysis of malic enzyme.Furthermore there exists a wealth of information on the enzymological properties of malic enzyme eg negative co-operativity (Koshland concept),substrate inhibition at high malate concentration etc (Hsu.R.Y, 1982).A complete understanding of these properties at the molecular level is impossible without the detailed information which only a crystallographic analysis can provide.

Initially extensive crystallization trials were carried out using the hanging-drop method of vapour diffusion for the native enzyme and a wide range of enzyme-substrate complexes. To date , crystals for the native enzyme have not yet been obtained.However two morphologically different crystal forms were grown from enzyme-substrate complexes : a needle shaped morphology for crystals grown in the presence of malate and pyruvate and a distorted tetrahedral morphology from NADPH or $NADP^+$.The latter crystals appeared the most suitable for immediate X-ray analysis and optimization of the crystallization condition yielded crystals with dimensions of 0.70mm x 0.70mm x 0.60mm.

The crystals were stabilised by transferring them to 65% saturated ammonium sulphate in 0.1M sodium phosphate buffer at PH 7.0 with 1mM $NADP^+$,1mM β-mercaptoethanol and 1mM EDTA.X-ray analysis of these crystals showed that they belong to the orthorhombic system with space group F222 and cell dimensions a=263 Å ,b=264Å and c=166Å.Measurements of the crystal density indicated that the asymmetric unit of the cell contains a complete tetramer (Baker *et al* ., 1987) and thus the crystals offer the exiting prospects of utilising this geometrical redundancy in solving the X-ray structure.

If an initial handle can be established on the structure by the use of isomorphous replacement then the application of molecular averaging techniques should ensure that relatively rapid progress can be made despite the proteins high molecular weight.However ,the initial problem will be to produce a derivative with a relatively small number of metal binding site to ensure a relatively straight forward solution of the heavy atom Patterson .One approach to this might be to exploit the iherent metal binding potential of malic enzyme based on its requirement of a metal cofactor (Mg^{2+}or Mn^{2+}ions) for catalysis.Since the crystals were grown in the absence of these ions,experiments were performed to see if their addition triggered a

conformational change which might be a contra-indication for using this approach to find a suitable heavy metal derivative.The crystals remained intact under this treatment and so a range of heavy atom compounds with atomic radii similar to Mg^{2+} or Mn^{2+} ions are now being explored as potential derivatives.

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MEASUREMENT AND INTERPRETATION OF DIFFUSE SCATTERING

by David Moss & Gillian Harris
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In protein crystal diffraction much or most of the diffracted energy goes in diffuse scattering. In conjunction with John Helliwell at York and Ian Glover at Daresbury, we are writing software for the interpretation of diffuse X-ray scattering from protein and nucleic acid crystals.

The static or dynamic displacement of atoms in crystals causes a breakdown of translational symmetry and this leads to the reduction of Bragg intensities at higher resolutions, and the concomitant appearance of diffuse scattering at and between reciprocal lattice points.

As with Bragg reflections, the diffuse scattering depends on atomic positions, occupancies and mean-square atomic displacements. However it also depends on the way in which the displacements are correlated. The diffuse intensity at the terminus of the reciprocal lattice vector $\underset{\sim}{Q}$ can be expressed as

$$I(\underset{\sim}{Q}) = \varepsilon(\underset{\sim}{q})\{| F(\underset{\sim}{Q}) - \langle F(\underset{\sim}{Q})\rangle |^2\rangle\}$$

where $\underset{\sim}{q}$ is a Brillouin zone vector, $\langle F(Q)\rangle$ is the familiar structure factor and the angle brackets denote either a time or ensemble average. The term $\varepsilon(\underset{\sim}{q})$ accounts for the correlation between unit cells while the expression between curly brackets is the structure factor variance and accounts for the correlation within unit cells.

The software development needed to handle diffuse scattering consists of two parts :

a) modification of data processing programs
b) extension of refinements programs to handle the extra parameters needed to account for diffuse scattering.

In data processing from film or area detector, shoeboxes of density must be collected at and between reciprocal lattice points and background corrections must not be carried out (diffuse scattering is the background!). The Lorentz correction depends not only on the experimental geometry but also on the type of diffuse feature being measured.

In refinement against total intensity, parameters must be included to account for solvent scattering, Compton scattering and interatomic correlation. We are interpreting the latter in terms of correlation between secondary structure features such as α-helices, whose main chain atoms are being treated as pseudo-rigid bodies. We have developed and are programming the theory of correlated rigid body kinematics within the harmonic approximation.

All protein crystals give rise to acoustic scattering. This manifests itself as halos of diffuse scattering at reciprocal lattice points and is due to ultrasonic waves. We have studied this in ribonuclease crystals and have related it to the velocity of sound which we have measured in crystals with ultrasonic experiments. The parameter $\varepsilon(o)$ in our refinement allows for this source of diffuse intensity.

We shall be measuring film data from 6-phosphogluconate dehydrogenase (courtesy of Margaret Adams) in order to study the correlation between the movements of its α-helices and its relevance to catalytic activity. In order to interpret rigid body displacement parameters, a preprocessor will have to be written for a graphics program so that the correlated motions can be displayed.

Progress towards the structure of cytochrome c_4

S.A. Rule, I.P.I. Chemistry, Liverpool University and Daresbury Laboratory. (currently Physics, Keele University)
M.M. Harding, I.P.I. Chemistry, Liverpool University
L. Sawyer, Biochemistry, Edinburgh University

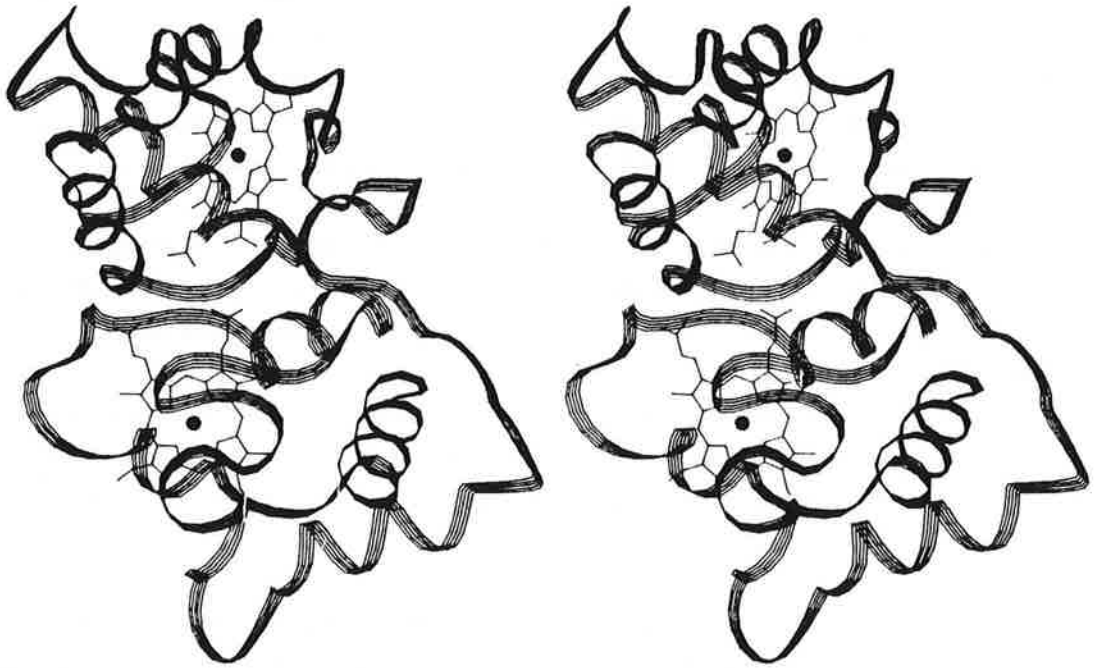
Cytochrome c_4 is a high potential, dihaem cytochrome of 19000 daltons molecular weight, consisting of two short cytochrome folds joined covalently. Crystals of c_4 from the nitrate respiring *pseudomonus aeruginosa*, with one dihaem per asymmetric unit of the $P6_122$ unit cell, have been under study for some time now, although the 3-D structure has remained elusive. Efforts to phase the reflections using two wavelength anomalous scattering information from the iron atoms in conjuction with a uranyl derivative have failed to generate an interpretable electron density map.

More recently, data for a second heavy atom derivative ($K_2Pt[NO_2]_4$) have been measured on film at a wavelength of 0.88 Å on station 9.6 at the SRS. Although the platinum was located in three low occupancy sites, the contribution to the phase information was useful, with a final figure of merit for 5550 reflections to 2.7 Å of 0.70. The resulting electron density map showed notable improvement over earlier maps, enabling a partial polypeptide model to be built into the region of one of the two cytochrome folds. However, the electron density representing the second fold remained noisy and ambiguous.

At this stage we decided to seek further improvement in the map before expending extensive effort on the model-building, and therefore resorted to the automated density modification procedure of B.C. Wang, as implemented by Andrew Leslie (see the March 86 issue of this newsletter). Although the c_4 crystals contain 50% high salt solvent, a mean shift in the protein phases of 23 degrees was obtained after four cycles. Inspection of the electron density map at 2.7 Å resolution revealed a general improvement in clarity and definition, most notably in the previously poorly resolved cytochrome fold.

An initial model of 160 residues (out of 182), including about 50 sidechains, has been built into the map using FRODO running on a PS300. The figure shows a stereo view of the protein backbone. The structure consists of two short cytochrome folds

separated by a length of extended random coil, as was expected from analysis of the amino acid sequence. Each fold displays the major characteristics of other short cytochromes, namely the overall topology, the arrangement of the ligands to the iron atoms, and the principle helices. The 15 residue polypeptide connecting the two folds, which had proved impossible to locate in earlier maps, can be seen on the right of the figure. It would be premature at this stage to draw any conclusions regarding the interaction of the two haem groups and the mechanism of electron transfer. Work is in hand to improve the model using the partial structure and we expect to be able to refine the structure of both the oxidised and reduced c_4 at better than 2 Å resolution.



Some Experiences with The FAST

by Pierre J. Rizkallah, Liverpool University/ Daresbury Laboratory

Introduction

The FAST crystallographic data collection system, at Daresbury, has been in use for around two years so far. Several data sets have been collected with this efficient system, and the software is under constant development, though its current status is quite satisfactory.

The current project, subject of this report, is concerned with testing the feasibility of structural analysis of micro-crystals of small molecule compounds.It has to be emphasised, to start with, that none of this work had been possible with the traditional data collection techniques. The main reasons are the small size of the sample, the relatively high beam dispersion from sealed tubes and rotating anode, and the lower beam intensity compared with synchrotron radiation. But perhaps the worst problem of all is the high mosaic spread of these samples, which causes a large rocking width, and is probably the underlying reason for the absence of larger samples. At a given rocking width, there is a limiting attainable resolution dependent on cell dimensions. Table 1 contains a listing of the diffracted spot size as a function of rocking width and resolution, where the values are in dimensionless reciprocal lattice units, in mm and in detector pixels. With the current FAST pixel size of around 100 microns, the limit of resolution drops rapidly as the rocking width or the unit-cell dimensions increase. The spot separation from the two unit-cells dealt with so far, are also included in Table 1. It is readily obvious that the maximum tolerable rocking width may be a few degrees, which is unthinkable in terms of protein crystallography.

Comparison Between the Daresbury Set-up and Traditional Techniques

The above discussion points to a theoretical limit that is independent of the data collection technique. However, the large rocking width creates a logistical problem of a different kind. As mentioned earlier, traditional diffractometry yielded no useable data, even after a great deal of effort. With synchrotron radiation, the intensity problem is alleviated, but oscillation photographs are usually prone to being swamped with background noise. The current software package is geared towards large unit cells, and subsequently fails to deal with the few spots from a small unit-cell. Laue photography too suffers from the same

bias towards large unit-cell work, but a more severe limitation there, is the streaky pattern associated with large rocking widths. The techniques discussed above could not be used.

The philosophy of data collection with the FAST is totally different from film techniques. The discrimination between fulls and partials does not exist with the former, since all spots are partial, by virtue of the fine sampling range in the oscillation direction, Phi. Consequently, the spatial resolution between spots projected on the detector is greatly improved, since only fewer reflections are in diffracting position simultaneously. Each reflection would then span a few frames, and by sampling each, the intensity reading may be accumulated and integrated.

Structural Studies of Two Compounds

Two structures, falling into the above category, have been tackled recently:

1) EU20 is a silicate that has been obtained by baking EU19 crystallites at 500°C, for 48 hrs. EU19 is a silicate-organic complex, whose structure was solved by S. Andrews and coworkers (see Acta Cryst., **B44**, 73, 1988). On baking, the organic component is burnt off leaving the silicate. Not surprisingly, the resultant crystallite used for data collection had a rocking width of 6.9°. The cell dimensions reveal a contraction in one direction, compared with EU19, which accounts for the burnt off component. Fig. 2 shows a comparison between the two cells. The diffraction pattern was recorded over a Phi range of 180°, with the detector 40 mm away from the sample, and tilted at 18° w.r.t the horizontal. This allows a maximum resolution of 0.9 $\AA$. The new auto-indexing routine in the MADNES software was used to calculate the unit-cell and an associate orientation matrix to fit the diffraction pattern. The statistics of data collection are shown in Table 2, and they compare very well with EU19 statistics. Considering that the rocking width is twice as large, the quality of data processing is very good. Structure solving has not been achieved yet. With reference to Table 1, it may be seen that the highest attainable resolution along the b-axis is only 1.75 $\AA$, which is not sufficient for small molecule structures. Patterson and Direct methods gave similar solutions, but refinement could not improve the R-factor below 45%. Any progress awaits collecting data from a crystallite with a smaller rocking width. The size of the sample used for the above data, was 80x30x20 microns.

2) NICLUS is a Ni-Cl-Organic cluster complex with BF_4. It could be grown only as very small crystallites. Diffractometer data collection yielded only a few reflections and a possible space group (tetragonal). The diffraction pattern was recorded in two stages, first with the detector untilted at 40 mm from the sample, and then with the detector tilted at 18°. The first data set provided intensity measurements up to 1.3 $\AA$ resolution, with a rocking width of 4°. With the measured cell (Table 1), this implies that the highest attainable resolution could only be 1.5 $\AA$ along the b-axis (note that the systematic absences in this body-centered cell augment the resolution, in reciprocal space, between observed reflections). Thus, when the higher resolution data set was tackled, it was impossible to process. The unit-cell dimensions and the space group caused additional problems. Although a tetragonal cell could be fitted to the pattern, the true cell was only slightly different: The two non-unique axes were 0.56 $\AA$ different in length, and the unique angle was 90.58°. The cell is, therefore, only monoclinic, and the body-centered setting is not conventional. It was chosen, however, in order to avoid high correlation between coordinates along two axes. This situation is explained in Fig. 3. Statistics resultant from data processing are shown in Table 3.

Patterson and Direct methods gave an identical solution, with the Ni lying on a 2-fold axis at x=.25,z=0. Possible sites for the other heavier atoms (Cl, P) were deduced. the best R-factor on refinement was only 41%. The resultant electron density map has broad features in the positions expected for the organic component. A model would have to be fitted to the map in the same manner applicable for protein structures. This is currently in hand. A better chance might be to collect high resolution data from a crystallite with a smaller rocking width.

Table 1: Spot Size vs. Rocking Width and Resolution

In Dimensionless Reciprocal Lattice Units

	$\eta(°)$	0.5	2	4	6
	(rad)	.0087	.0349	.0698	.1047
Res ($\AA$)	d*				
3.00	.298	.0026	.0104	.0208	.0312
2.50	.358	.0031	.0125	.0250	.0375
2.00	.448	.0039	.0156	.0313	.0469
1.75	.511	.0045	.0178	.0357	.0535
1.50	.597	.0052	.0208	.0417	.0625
1.25	.716	.0063	.0250	.0500	.0750

In millimeters (Xtal to Detector Dist = 40m.m.)

Res ($\AA$)	d*				
3.00	.298	0.104	0.416	0.832	1.248
2.50	.358	0.124	0.500	1.000	1.500
2.00	.448	0.156	0.624	1.252	1.876
1.75	.511	0.180	0.712	1.428	2.140
1.50	.597	0.208	0.832	1.668	2.500
1.25	.716	0.252	1.000	2.000	3.000

In Detector Pixels

Res ($\AA$)	d*				
3.00	.298	1	4	8	13
2.50	.358	2	5	10	15
2.00	.448	2	6	13	19
1.75	.511	2	7	15	22
1.50	.597	2	8	17	25
1.25	.716	2	10	20	30

Table 1 (Continued)

Cell Dimensions and Spot Separation at 40 m.m.

NICLUS							
a =	16.44	a* =	.0544	Sep in mm =	2.18	Sep in pix. =	22
b =	37.10	b* =	.0241	Sep in mm =	0.96	Sep in pix. =	10
c =	17.02	c* =	.0526	Sep in mm =	2.10	Sep in pix. =	21
EU20							
a =	7.576	a* =	.118	Sep in mm =	4.73	Sep in pix. =	47
b =	17.187	b* =	.052	Sep in mm =	2.08	Sep in pix. =	21
c =	5.149	c* =	.174	Sep in mm =	6.95	Sep in pix. =	70

Fig. 1. Rocking Width vs. Resolution

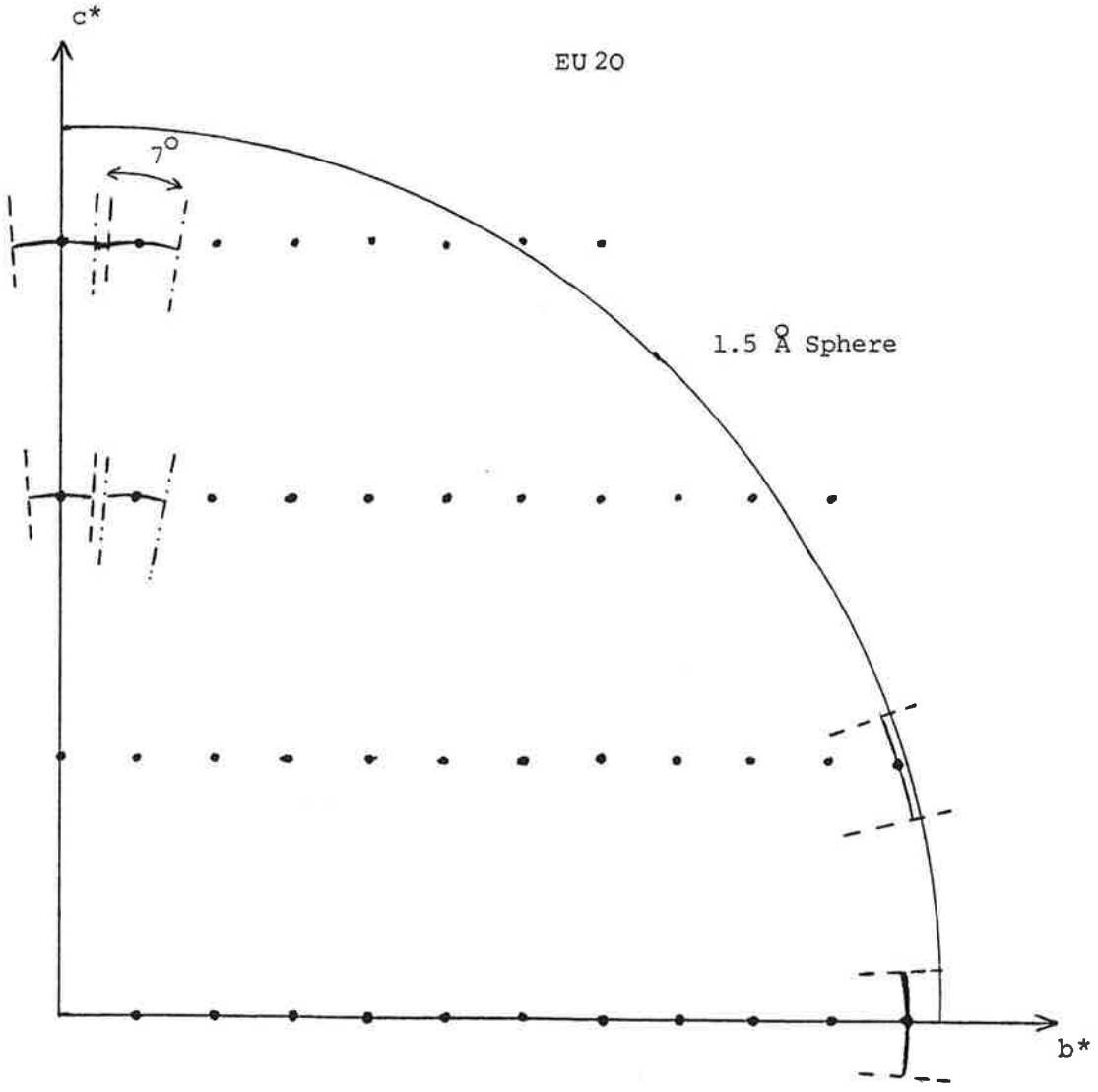


Table 2. Data Statistics for EU20

Number of Reflections Measured

515	Flag Ø	GOOD	
224	Flag 1	WEAK	No Negatives
124	Flag >1	BAD	

Unique Data

Total	Flag	% > 3sd's	R_{merge}
286	Ø	100	9.3%
423	Ø,1	93.1	10.6%
429	Øto 15	93.2	24.7%

Fig. 2. Correlation between EU19 and EU20 Cells

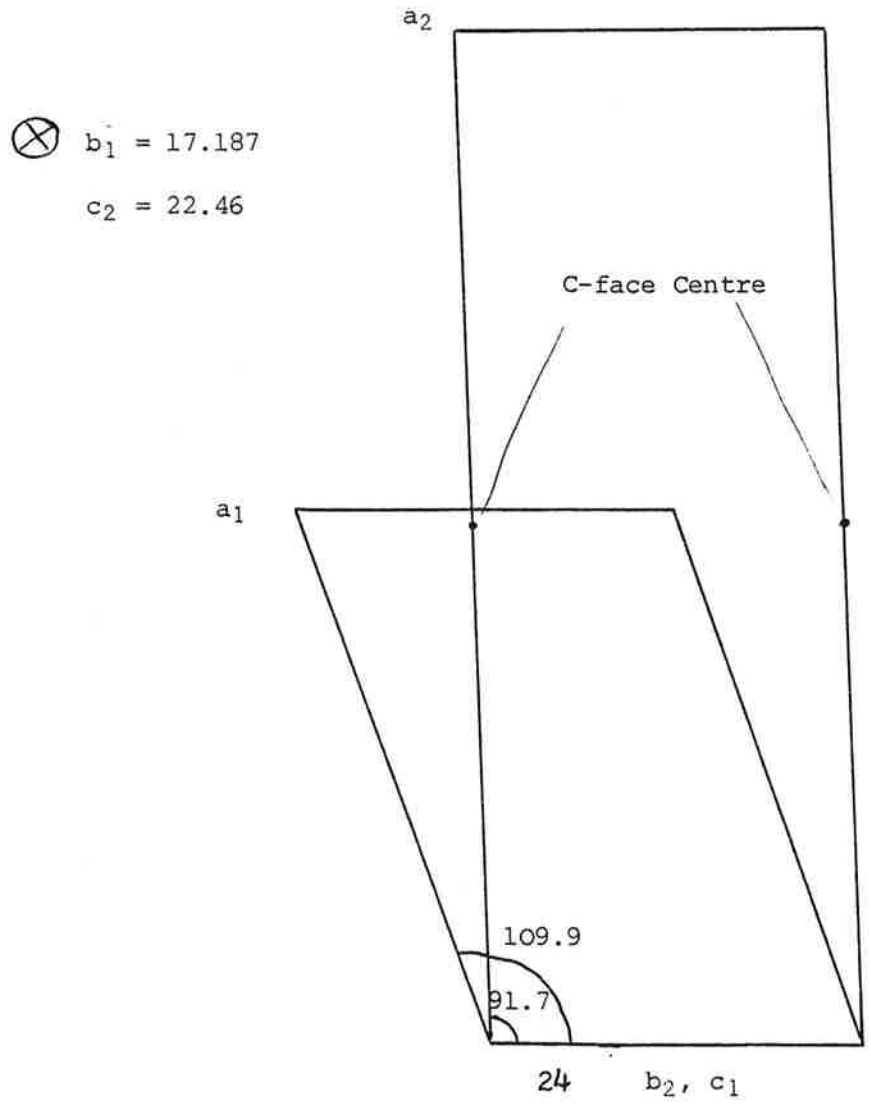


Table 3. Data Statistics for NICLUS

Number of Reflections Measured

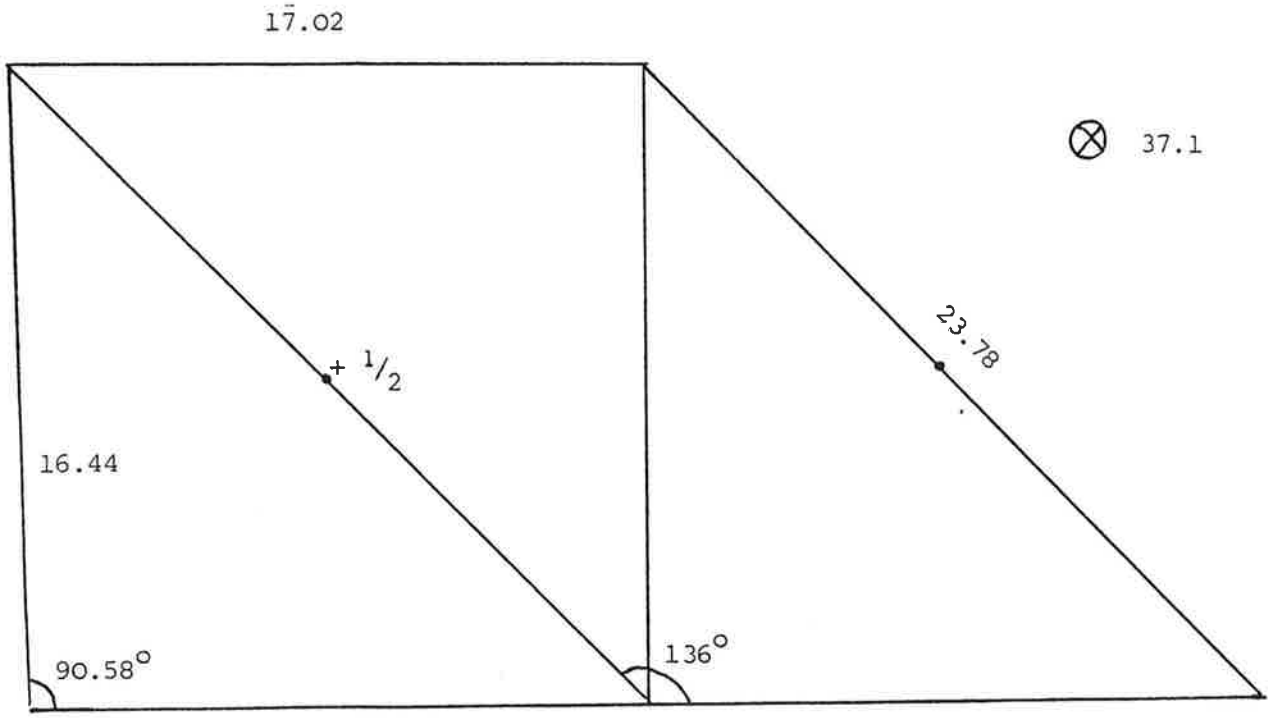
2692	Flag Ø	GOOD	A significant number
1800	Flag 1	WEAK	of measurements were
5231	Flag >1	BAD	duplicated.

Unique Data

Total	Flag	% > 3sd's	R_{merge}
931	Ø	100	6.6%

Only GOOD data were used from this data set.

Fig. 3. Correlation between Conventional and Chosen NICLUS Cells



Oblique Cell is the conventional C-face centered cell

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