

## A Multi-type Scatterer Implementation of the Debye Formula.

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Small Angle Scattering modelling is an established procedure [1] for the study of structural parameters of individual particles such as large biomolecules [2] and of the morphology of particle aggregates [3]. A CPU-efficient implementation of the optimised Debye formula utilising pair distance histograms is described in [1]. For multi-type scatterer systems the Debye formula can be expanded as a sum of partial intensities

$$I(q) = \sum_{i=1}^{N_{types}} N_i F_i^2(q) + 2 \sum_{i=1}^{N_{types}} \sum_{j=i}^{N_{types}} I_{ij}(q) = \sum_{i=1}^{N_{types}} \left[ N_i P_{ii}(q) + 2 \sum_{j=i}^{N_{types}} P_{ij}(q) S_{ij}(q) \right]$$

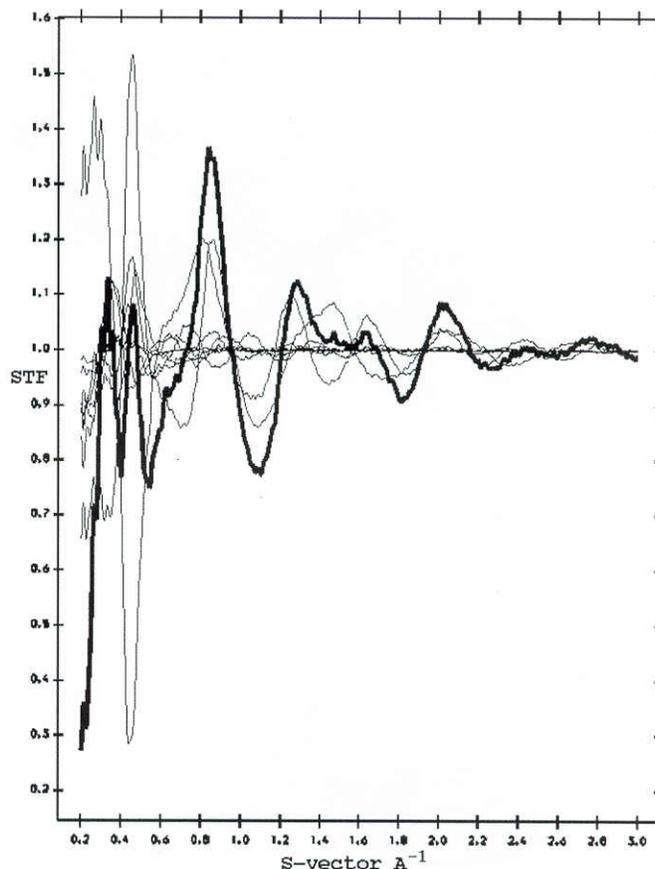
Details and examples of application are given in [4]. The simplicity of the optimised single-type formulation is not lost and the computational efficiency is not compromised seriously for systems containing as many as 10 different types of scatterers, provided the machine on which the code runs is not memory limited. The code permits the use of tabulated atomic structure factors, instead of hard sphere form factors.

Figure 1 shows an example of partial structure factors computed from the coordinates of a model sodium disilicate glass system [5]. The partials permit the assignment of the relative contributions of scattering from different elements or element pairs. Notice that the total intensity can be significantly different from some of the partials.

The Silicon Graphics and SUN Solaris executables together with example input and output data will soon be available through the CCP13 Programme Library.

### References

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**Figure 1.** Simulated Structure Factor for sodium disilicate glass. The thicker line gives the total and the thin lines the partial structure factors.

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### “imageNCIF”: An Initiative to Standardise Image Formats”

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### Introduction

Data formats and in particular image formats are a problem! Most scientists will know the problem of having to find or write a conversion program, to convert a file in a particular format to a format which can

be input to the analysis program which they want to use. Similarly application programmers will know the problem that however many formats they support, there will always be new detectors with new data formats which will be required.

If a sufficiently versatile common format was widely adopted this "Babylon" of formats would at least be limited and maybe the number would eventually decrease. Initiatives to define such formats are not new, but for storage of large quantities of experimental data there has been no commonly adopted format to date. For the task of passing more limited quantities of processed, typically structural, data amongst the crystallographic community, a common standard format has been defined: "CIF" [1]. "CIF" stands for "Crystallographic Information File", and is an ASCII text based, flexible and extensible human readable archive file format.

In March 1995 a computing workshop was organised at the Brookhaven National Laboratory concentrating on the use and design of "Graphical User Interfaces" (GUI's). One subject area which was clearly of general common interest was image formats.

A working group was formed and time was dedicated to an open discussion on the requirements of such a format. It was here (to my knowledge) that the idea of extending the CIF-concept to define the header information for images was first discussed by a sizeable group.

### What is CIF?

CIF is a standard format maintained and "owned" by the International Union of Crystallography (IUCr) for archiving and transporting crystallographic data [1]. One important current use is the submission of papers on structure determinations to Acta Crystallographica Section C.

The format consists of simple ASCII text keyword and keyword value pairs. A large "dictionary" defines the keywords and possible values. There is support for comments, for multi-line character text, and for structuring separate data sections. The keywords are defined from a hierarchical "class" (data name categories) and "sub-class" system. e.g. All keywords which start with `_diffrn` refer to data from diffraction measurements. A typical line from a CIF file, which could also be relevant to an experimental image file is:

```
_diffrn_radiation_wavelength 0.76 #This  
is 16.3 keV
```

Importantly, the dictionary defines the precise meaning of the data names, and the units and valid range when appropriate.

A number of software tools are available from the IUCr to work with CIF's, and libraries are available to help read and write CIF's. CIF is maintained and extended through the IUCr COMCIFS committee. At present there are initiatives to extend CIF (Core dictionary) to cover the extra needs of macromolecular crystallography (mmCIF) and powder diffraction.

For more information on CIF, there is a World-Wide-Web page with links to associated pages (<http://www.iucr.ac.uk/cif/home.html>).

### What is "imageNCIF"?

"imageNCIF" (= image (Not) CIF; cf. mmCIF), is an E-mail based working/discussion group working on the idea of extending the CIF concept to cover the storage of experimental data, and in particular 2-D "image" data. There are presently about 15 members of this group, which contains representatives from commercial detector manufacturers, programmers of data analysis software, members concerned with data acquisition at user facilities, and crystallographers from a variety of different scientific disciplines.

The aim is to standardise the passing of image and associated crystallographic experimental data from one institute to another, one make of computer system to another, and from one computer program (acquisition or analysis) to another. It is desirable that the image file contains the necessary associated experimental information to make data processing as automatic as possible.

The basic aim is essentially the same as that of CIF, but the difference in the quantities of data involved mean that there is agreement amongst the members of the group that the ASCII encoding of image data is not appropriate. Given that it is highly desirable that header information and the image data are kept together in the same file this means that the format is binary in nature, and cannot be considered as compliant CIF. Hence the "Not" in "imageNCIF".

Nevertheless, the advantage of using the existing CIF structure for naming and defining data names and

definition is sufficiently important to define an associated format which is closely related to CIF: "CIF-compatible". The header section of such a file would contain CIF keyword / value pairs. This would differ from CIF in the manner in which the "lines" were separated. A simple utility program could extract the header section and write it out in a true CIF form. Similarly, analysis programs might use some of the header information for their own processing, ignore other items, and write an output CIF which contains their results plus most of the original CIF-style items. A number of new CIF data names need to be defined.

The details of this approach are presently being discussed by the working group.

### Joining "imageNCIF"

So far there are no "rules" and "imageNCIF" seems to work reasonably well on an open membership basis. I suggest that appointing one representative per institute, or one per scientific group within an institute is a sensible means by which to channel views whilst making sure that the working group does not get too big. Some input from the CCP13 community would be valuable, either through individuals or through a representative member.

### References

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### Synchrotron Studies of Amyloid Fibrils

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Amyloid fibrils derive from the deposition of abnormal forms of normally soluble proteins in an insoluble, aggregated state in certain disease processes. Amyloid is associated with some of the most widely-publicised of modern diseases, such as Alzheimer's disease, Creutzfeldt-Jakob disease (and its animal versions Scrapie and BSE or Mad Cow's disease), Type II or late-onset diabetes, and some polyneuropathies. In most cases the diseases are chronically progressive leading inevitably to death. Each disease is associated with a specific protein which is deposited as amyloid often as the result of specific amino-acid mutation, and/or abnormal proteolytic cleavage. The amyloid most extensively

studied here is that from Familial Amyloidotic Polyneuropathy (FAP) which is composed of the protein transthyretin (TTR) in which Val30 has been substituted by Met.

In the electron microscope, amyloid fibrils have a characteristic appearance of uniform, straight, unbranched fibres about 100Å in diameter and of indefinite length. X-ray analysis of these fibrils using laboratory X-ray sources gives a relatively weak diffraction pattern, characterised by a sharp 4.8Å meridional reflection, and a more diffuse 10Å equatorial reflection, with few if any other reflections. This diffraction pattern is characteristic of the so-called cross-β fibre structure in which the polypeptide chains in the fibril are arranged in β-sheets, such that the β-strands are perpendicular to the fibre axis, and the sheets parallel to the fibre axis. The meridional reflection then derives from the characteristic 4.8Å hydrogen-bonded separation of adjacent strands in the β-sheet, and the equatorial reflection from the typical β-sheet separation of about 10Å. Use of synchrotron radiation at Daresbury gives much more detailed diffraction patterns which can extend to at least 2.0Å along the fibre axis and to about 3.5Å perpendicular to the fibre axis. However, difficulties with orientation of the long fibrils from disease state amyloid prevents the development of reflections other than meridionals and equatorials. In spite of this limitation 7 clear meridional reflections can be detected on the synchrotron pattern, which can be indexed precisely to an axial repeat of 115.5Å. The tendency for the indices of the stronger meridional reflections to be divisible by 3 indicates the presence of a (pseudo) three-fold screw axis parallel to the fibre axis.

Interpretation of the synchrotron X-ray patterns is based on the observation that the intense 4.8Å meridional is the 24th. order of the 115.5Å cell, indicating that the axial repeat corresponds to a group of 24 β-strands which the three-fold screw axis divides into three sets of eight. As the TTR molecule is composed of four β-sheets each containing eight β-strands it is possible, though not necessary, for the cell to contain three TTR molecules. In any event a "standard" 8-stranded β-sheet will be 38.5Å wide and its β-strands will twist through 120°, and thus when operated on by a three-fold screw axis will generate a 24-stranded β-sheet 115.5Å long whose 0th. and 24th. strands are twisted through 360°. On this basis the repeat unit in the amyloid fibril corresponds to one turn of a β-sheet helix containing 24 β-strands. This new

description of the molecular structure of the amyloid fibril reconciles the classical description of it as a cross- $\beta$  structure with the twisted  $\beta$ -sheet now known to be the lowest energy state, and in doing so generates a new fibrous protein structure in which  $\beta$ -strands can be hydrogen-bonded together over large distances in a stable structure.

#### Time-Resolved SAXS Measurements on Gel Setting and Gel Melting of Triblock Copolymer Gels

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The unique properties and practical applications of triblock copolymer gels are related to the specific morphology. The endblocks of a triblock copolymer, polystyrene-block-(hydrogenated butadiene)-block-polystyrene (SEBS) or polystyrene-block-(hydrogenated isoprene)-block-polystyrene (SEPS), will associate in a solvent selective for the midblock, and result in a three-dimensional organisation. This implies an electron density difference, detectable in a small-angle scattering experiment, if the superstructure has dimensions in the range of 10 - 1000 Å.

The gels were investigated by small-angle scattering of X-rays and neutrons (SAXS and SANS) and exhibit a pronounced superstructure [1] which appears to be a function of the block copolymer concentration, molar mass, end/mid block ratio, deformation [2] and temperature. From the variations in the two interdomain interference maxima and one or more intradomain interferences with varying sample parameters, it follows [3] that the polymer concentration and molar mass influence the degree of organisation in the gel. A certain molar mass corresponds, for a specific polymer concentration, to the most disordered arrangement. Each deviation from these values introduces more order. The scattering patterns of the isotropic samples are fitted with two different models [3]; the Percus-Yevick hard-sphere interacting liquid model and the local coordination model of distorted hexagonal lattice.

Recent time-resolved SAXS experiments [4] reveal changes in the superstructure. Upon heating, the morphological order disappears, in agreement with DSC and rheological experiments. Gels with high molar mass, show a initial increase in order. These gels reach a thermodynamically more favourable morphology. After heating these gels with high block

molar mass above the temperature where disordering occurs, followed by cooling, they again exhibit increased order. Thermo-reversibility appears for these gels after a second heating scan.

Another effect is the temporarily change in structure for gels with relatively low block molar mass from hexagonal to a cubic-like order at a temperature around 120°C. Isothermal SAXS experiments in a temperature region around 100°C, reveal the same changes in superstructure. These morphological changes involve chain mobility which is relatively low in the case of long chain lengths. This explains the temperature difference, related to a time-effect, between dynamic and isothermal SAXS measurements. These morphological changes correspond to a critical gel formation in rheology experiments [5], performed in the same temperature region.

In the case where no improvement of order occurs during heating, cooling the gel from the temperature where the gel is in the disordered state yields the same scattering curve as before heating, indicating their thermo-reversibility.

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