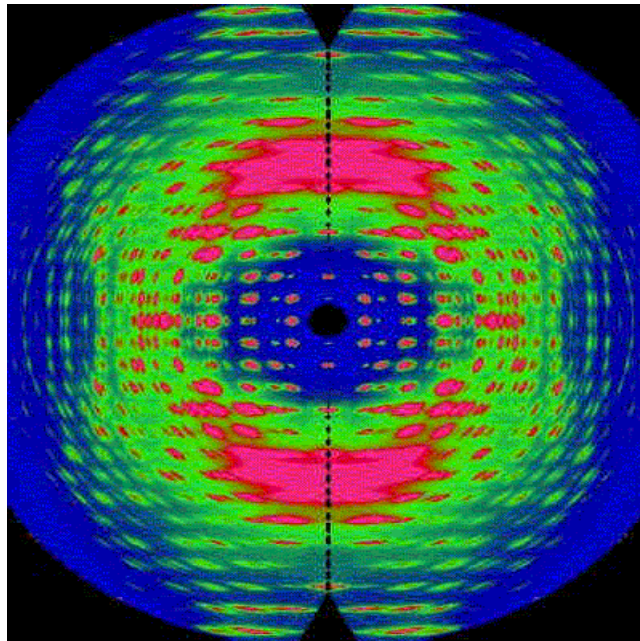


CCP13

A quick look at

XCONV, XFIX and CORFUNC for Windows[®]



Steve King

January 2005

CCP13 is the Collaborative Computational Project for fibre diffraction.

CCP13 is supported by the Biotechnology & Biological Sciences Research Council (BBSRC), the Engineering & Physical Sciences Research Council (EPSRC), the Council for the Central Laboratory of the Research Councils (CCLRC) and the Diamond Light Source Ltd. However, participation is world-wide.

CCP13 exists to focus the development of software used to analyze and model data from the wide variety of samples that give rise to “fibre diffraction”. These samples are characterized by having a structure which is ordered and preferentially aligned along one axis. Typical of such structures are long-chain biological or synthetic polymer molecules with a regular axial repeat but with different degrees of lateral packing regularity between molecules. However several other types of system, such as shear-ordered surfactant mesophases or magnetically-aligned liquid crystals, for example, can give “fibre-like” scattering patterns.

The **CCP13** program suite executes on a variety of platforms running UNIX, LINUX, and increasingly, under Microsoft Windows. The programs are available free of charge to academic users.

CCP13 also publishes a peer-reviewed journal (*Fibre Diffraction Review*), operates an e-mail bulletin board, and holds an Annual “Workshop” (conference). Further details about all of these can be found on the projects website at www.ccp13.ac.uk.

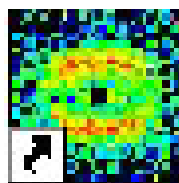
This practical is designed to give you a feel of three different pieces of data analysis software:

- XCONV - a data file format conversion utility
- XFIX - a program for the preliminary analysis of scattering patterns
- CORFUNC - a suite of programs to extract the 1D density correlation function or to generate an interfacial density distribution function

You will need to locate a PC which has these programs installed. There should be icons on the Desktop:



CCP13 ICE

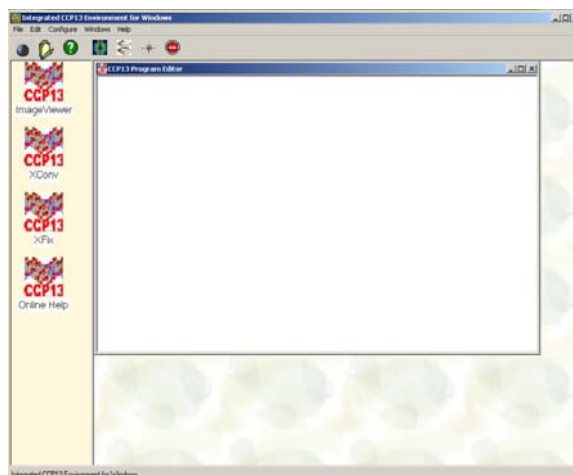


CORFUNC

ICE

ICE stands for Integrated Computing Environment. Basically it is a “shell” program that provides you with a simple way to invoke the other CCP13 Windows® programs, but it also incorporates “hot links” to the CCP13 websites and an application called the “Program Editor”. This can be used to write shell “scripts” (i.e., command files) for some of the programs.

Double click on the **CCP13 ICE** icon...



ICE will appear.

We don't need the Program Editor so close that down.

Double click on the **XCONV** icon...

XCONV

At the present time all of the CCP13 Program Suite is (for historical reasons) standardised around a file format called BSL, though this situation may change in the future. Consequently, in order to use any of the CCP13 programs it is necessary to convert data into the BSL format.

XCONV will read four types of generic binary data file (though you will need to have some idea of how the file is structured), “raw” data files from a number of specific X-ray detector types, or “reduced” data files from the SANS instruments at ISIS or Riso (1D or 2D data) or ILL (2D data only):

FLOAT	- Floating point numbers
INT	- Unsigned integers
SHORT	- Unsigned short
CHAR	- Unsigned characters
SMAR	- Small MAR image plate (1200 x 1200)
BMAR	- Big MAR image plate (2000 x 2000)
FUJI	- Fuji image plate (2048 x 4096)
FUJI2500	- BAS2500 Fuji image plate (2000 x 2500)
RAX2	- R-Axis II image plate
RAX4	- R-Axis IV image plate
PSCI	- Photonics Science CCD
RISO	- RISO file format
TIFF	- TIFF (8,12,16-bit greyscale with all Compression type)
ESRF ID2	- ESRF Data Format
LOQ 1D	- One dimensional ASCII data files recorded at LOQ
LOQ 2D	- Two dimensional ASCII data files recorded at LOQ
SMV	- Used by ADSC CCD detectors (8 bit unsigned, 16 bit unsigned, 32 bit signed integer, 32 bit floating point)
ESRF ID3	- ESRF Data Format

- BRUKER** - Area detector Frame format Siemens/Bruker file format (8 ,16 or 32 bits per pixel)
- MAR345** - MAR345 Image Plate (file extension mar1200, mar1800, mar1600,mar2400, mar2000, mar3000, mar2300, mar3450)
- ILL SANS** - Two dimensional ASCII data files recorded at the ILL (e.g., D11 & D22)

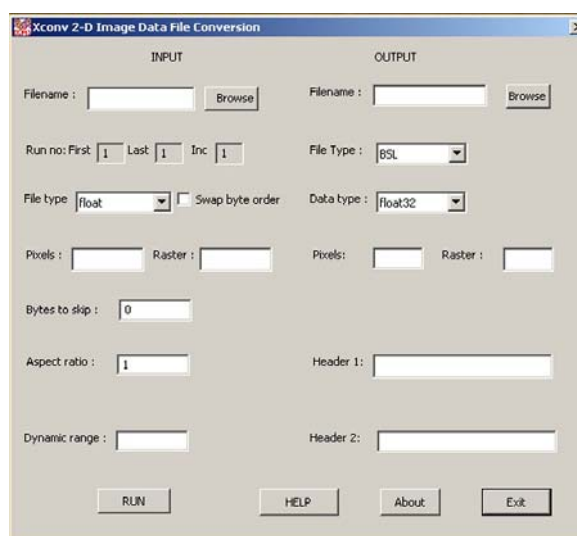
The BSL file format is described in detail in the online BSL Manual that may be found through the XCONV help file or on the CCP13 web site, but essentially each BSL data set consists of a (ASCII) “Header” file and one or more binary files, typically of the form:

- Xnn000.mdd - the header file
 Xnn001.mdd - the (normally small-angle) data
 Xnn002.mdd - any calibration data
 Xnn003.mdd - any wide-angle data

where the first letter (X) and the next two digits (nn) identify the individual experiment and/or data set, and the next three digits refer to the type of information contained in the file as shown above. Traditionally the (mdd) file descriptor gives the date on which the data was recorded/created, the first digit (m) being the duodecimal month and the other digits the date, but you may choose to use your own scheme. For example:

- Xnn000.807 - for 7th August
 Xnn000.A24 - for 24th October
 Xnn000.L1D - for one dimensional LOQ data
 Xnn000.L2D - for two dimensional LOQ data

Conversions from some of the “reduced” 1D formats generate files with the file descriptor QAX. These are the Q-axis data (BSL puts the “intensity” and “Q” data in separate files!).



Under INPUT:

Select the “file type” LOQ2D. Then “browse” your way to the file C:\CCP13 ICE\samples\98940.ZER.

Under OUTPUT:

Enter the “filename” C:\CCP13 ICE\samples\X40000.L2D.
 Enter 100 “pixels” by 100 “rasters”.
 For “Header 1”, enter LOQ 2D 98940.ZER.
 Keep the “file type” as BSL and the “data type” as float32.
 Click on RUN... When conversion is complete, click OK and EXIT.

Image Viewer

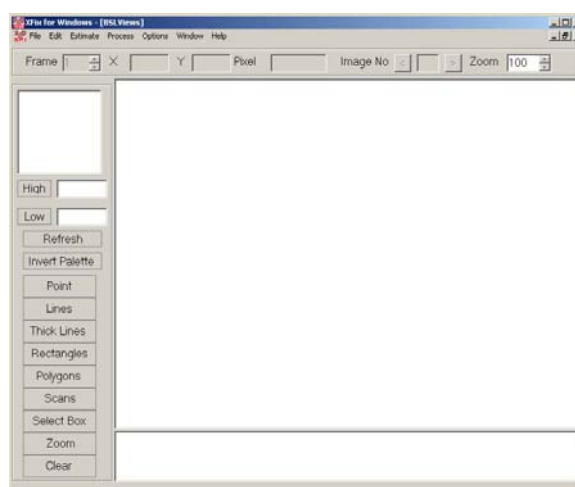
The Image Viewer is exactly that! It is a simple, no frills, application that enables you to view a two dimensional BSL data set. In fact it can be used to view any JPG, TIFF, GIF and BMP images!

XFIX

XFIX is designed to help ascertain some of the important parameters of a fibre diffraction pattern, such as the pattern centre, detector orientation, and fibre tilt. Putative unit cells can be plotted over the pattern, there are a selection of background subtraction algorithms, and there is the facility for the transformation of image space to reciprocal space.

In addition, there are more general functions such as the capability to plot slices or scans through the pattern. It is this functionality that you can experiment with here.

Double click on the **XFIX** icon...



XFIX will appear.

Do “File” → “Open” and navigate to the file `C:\CCP13 ICE\samples\X40000.L2D` that you created earlier. There is only one frame (i.e., time slice) of small-angle scattering data in the file so when the “FileInfo” pop-up window appears, just click OK. (It doesn’t matter that the drop down box says SAXS and not SANS). Notice that the file location in the title bar points to the 001 file and not the 000 file.

An image should have appeared. It is a bit small though. So “Zoom” to 500%.

Do “Options” → “Palette” and choose a false colour scheme.

You can also toggle between a *linear* and *logarithmic* intensity scales by doing “Options” → “LogScale”.

As you move the cursor over the image you will get a “cross-hair” and the image pixel position and the intensity (i.e., differential cross-section) at that location will appear at the top of the screen.

In the upper left corner is a smaller image window. This contains a magnified view about the current location of the cross-hair. You can change the magnification by doing “Options” → “Magnification”.

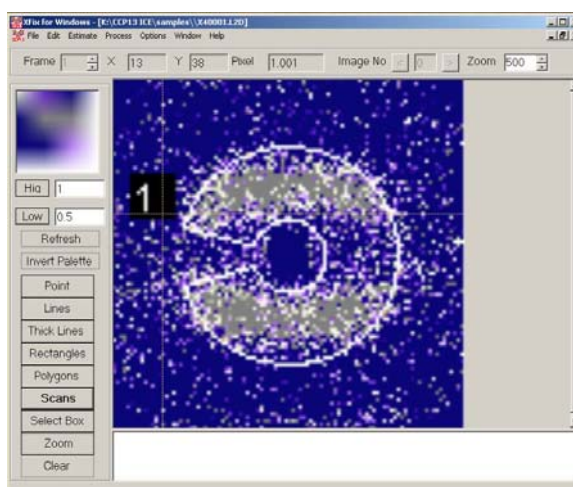
You can change how the range of the colour scale is partitioned by altering the values of “High” and “Low”.

Now let us imagine that you want to know how the intensity varies across the scattering pattern. Click on “Lines” then, using the mouse, draw a vertical line from top-centre to bottom-centre. Right-click on the line you have just drawn. It should change colour.

Do “Process” → “Plot Lines”. A second graphics window will appear displaying the intensity along the line as a function of pixel position.

You can remove the line by doing “Edit” → “Lines”, highlighting the line in the box, and then clicking “Delete” and “OK”.

Now suppose that you want to know how the *integrated* intensity varies around the obvious feature in the data. Click on “Scans” then, using the mouse, position the cursor at the left-centre (9 O’clock) outer edge of the feature, click the left button and whilst keeping it depressed drag the cursor in towards the centre of the pattern. A “doughnut-shaped” ring should appear. When you have selected all of the feature, release the left button and click the right button. The program will number the doughnut.



This may take a little practice, so if you want to start again, delete the existing doughnut by doing “Edit” → “Scans”, highlighting the scan in the box, and then clicking “Delete” and “OK”.

When you are happy with the region you have selected, do “Process” → “Plot Scans”. Another graphics window will appear. The data can be displayed as:

Azimuthal Scan – the scan is integrated radially and the integrated values are plotted as a function of angle.

Radial Scan - the scan is integrated azimuthally and the integrated values are plotted as a function of radius.

CORFUNC

CORFUNC is actually a “wrapper” application that calls and passes information between other programs that prepare and extrapolate an input data set, transform it into a one dimensional density correlation function (assuming an ideal lamellar morphology) or an interfacial density distribution function, and then extract parameters from that function.

A correlation function may be interpreted in terms of an imaginary rod of some finite length moving through the system giving rise to the small-angle scattering data. The correlation function is the probability that the rod has equal electron (for X-rays) or scattering length (for neutrons) density at either end. Hence a frequently occurring spacing within the system will appear as a peak in the one-dimensional correlation function.

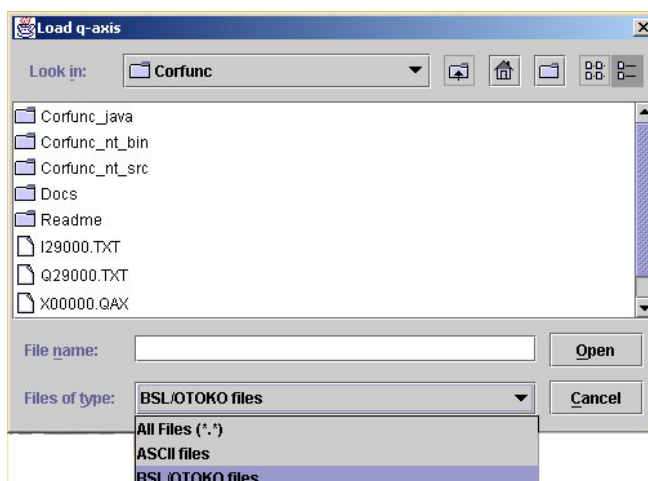
The interfacial density distribution, or volume fraction profile, describes how the number density of an adsorbed species (typically a surfactant or polymer) varies with distance from an interface (such as a particle surface). It may be analysed to obtain parameters describing the extent (thickness) of the adsorbed layer, the amount of material adsorbed, and what proportion is "bound" at the interface.

To start with, we'll generate a correlation function.

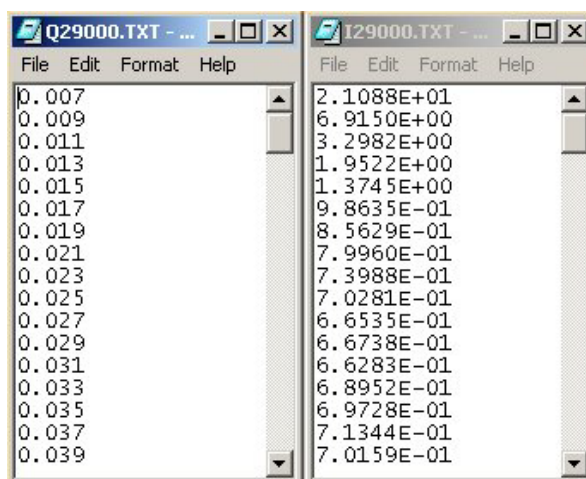
Double click on the **CORFUNC** icon...

An MS-DOS command box will appear (minimise this) followed by the CORFUNC Control Window.

Do "File" → "Load Q-axis". The following dialog box will appear:



CORFUNC will recognise two types of file format; BSL (as output by XCONV) or a plain ASCII "text" format consisting of a single column of numbers...



...however do note that filenames must still conform to the BSL naming convention on page 4.

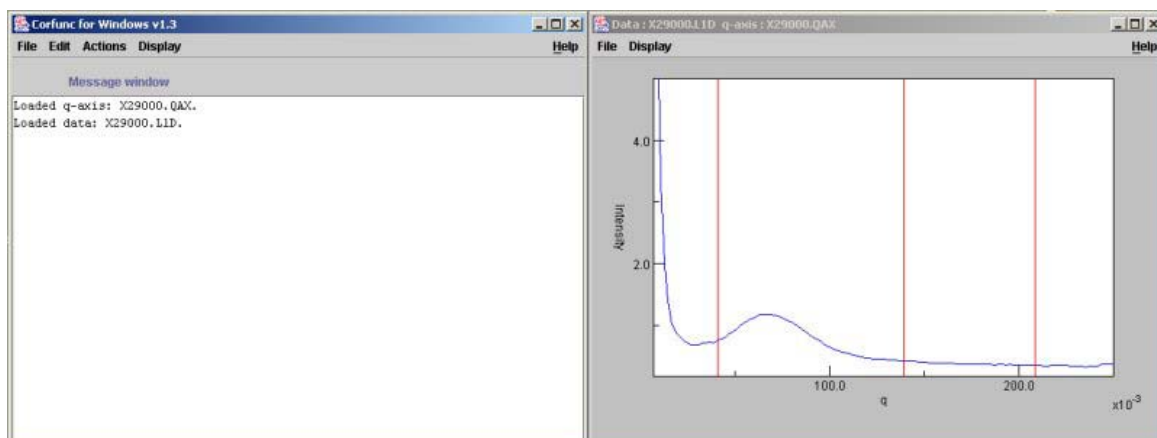
The ASCII input routine is also rather "fussy"; stray delimiters and hidden control or pagination characters will cause it to reject the file. If the data was originally in something like MS Word[®] or eXcel[®] it is advisable to cut-and-paste it into a simple text editor like Notepad[®] before saving it as a ".TXT" file.

Select "BSL/OTOKO" from the drop-down menu, navigate to the file `K:\Corfunc\X29000.QAX` and double-click on the filename.

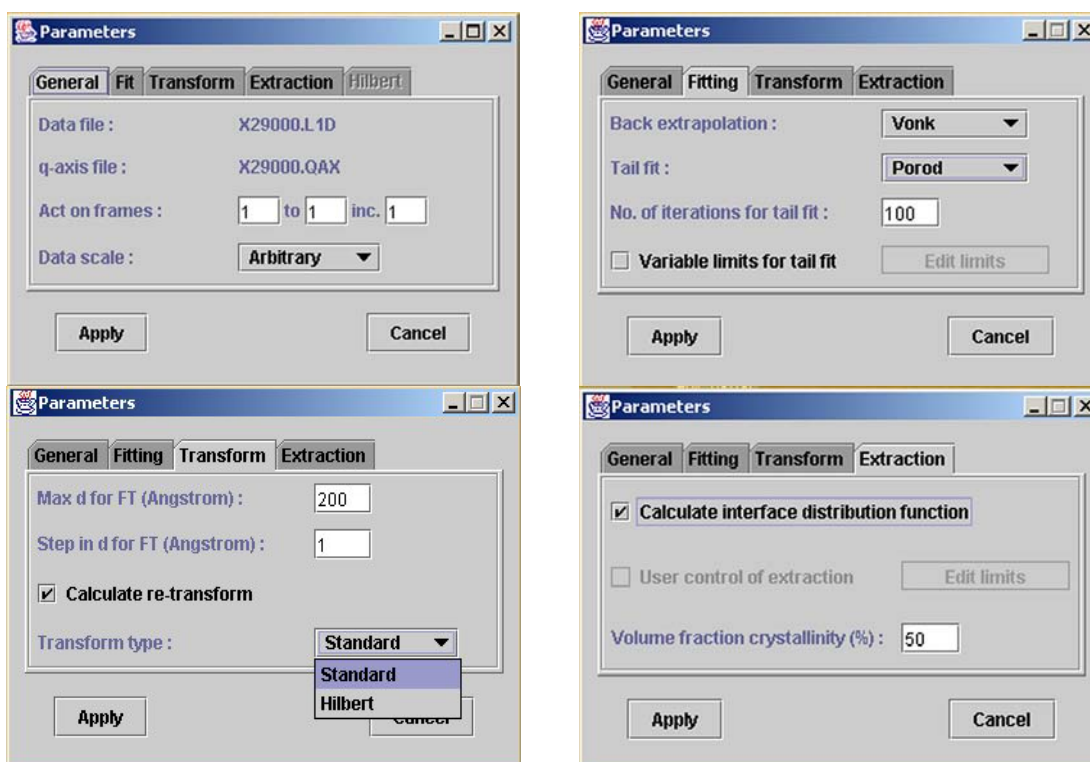
Now do "File" → "Load data" and read in the file `K:\Corfunc\X29000.L1D`.

IMPORTANT! At present the data and Q-axis files to be analysed *must* be located in the top-level of the CORFUNC folder for the program to work properly.

A graphics window will appear displaying the data and three vertical red lines.



Do “Edit” → “Parameters” and set:

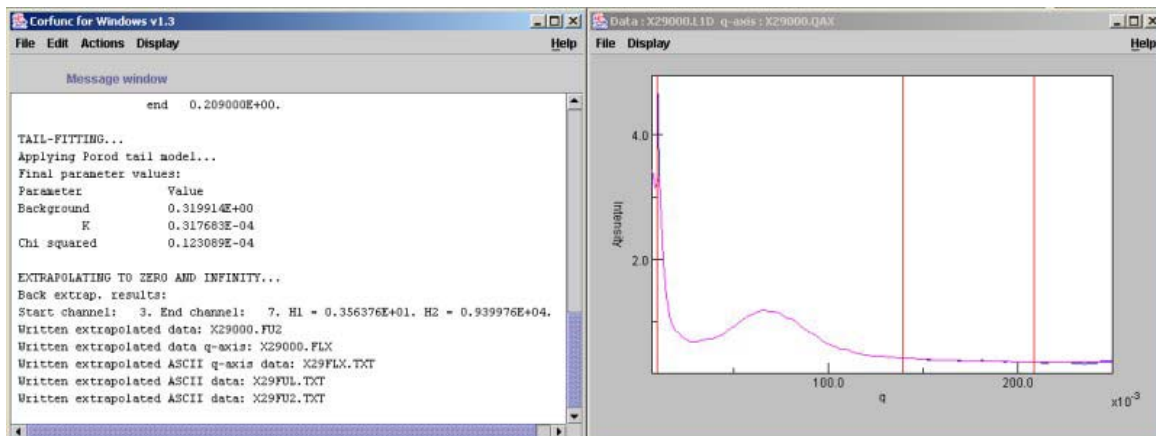


IMPORTANT! The Hilbert transform option, present in more recent versions of the program, *must not* be selected if CORFUNC is being used to generate density correlation functions. This option is provided for the calculation of interfacial density distributions. Using CORFUNC for this type of analysis is discussed later in this document.

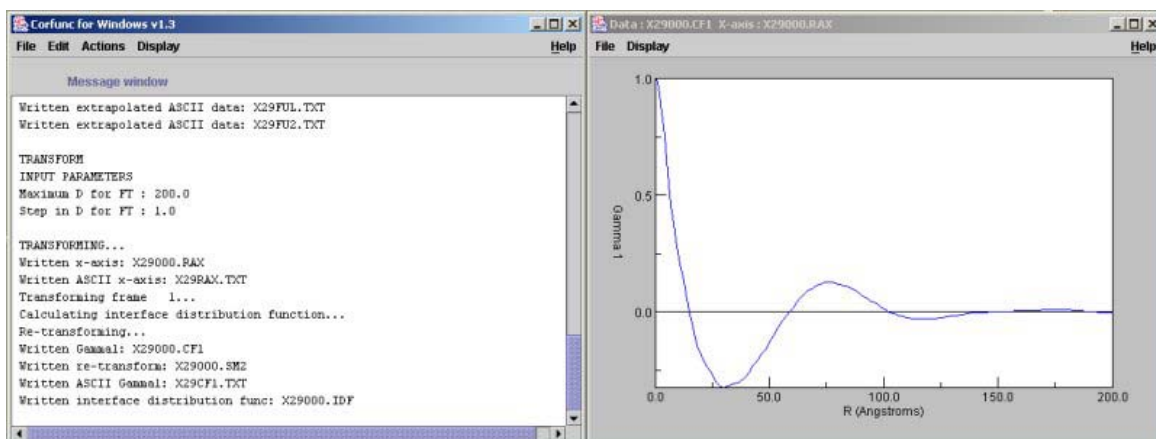
Click “Apply” when done.

Using the mouse, right click on the rightmost red line and, keeping the button depressed, move the line to $q \sim 270 \times 10^{-3}$. CORFUNC will use the region between the middle and rightmost red lines to judge the high- q extrapolation of the data that it will perform. Leave the leftmost red line where it is (which by default should be the first sensible low- q point).

Do “Actions” → “Extrapolate”. CORFUNC will extrapolate the data to low and high q values and display the extrapolation in the graphics window.



Do “Actions” → “Transform”. CORFUNC will transform the extrapolated data and calculate the correlation function which will appear in a second graphics window. The program *also* back-transforms the correlation function into reciprocal space and overplots this on the original and extrapolated data sets.



Do “Actions” → “Extract parameters”. CORFUNC will then calculate various parameters from the correlation function. How this is done is explained in the CORFUNC help file. The parameters are written to the ASCII *.OUT file. Here is some sample output:

```

CORRELATION FUNCTION ANALYSIS OUTPUT
~~~~~

Analysis performed on
Data file: K:\Corfunc\X29000.L1P
Start frame: 1.      End frame: 1.
Frame increment: 1.  Total number of frames: 1.
Intensity units: Absolute (cm^-1).
Q axis filename: K:\Corfunc\X00000.QAX

EXTRAPOLATION PARAMETERS:
Tail model applied: Porod.
Tailfit start channel 67.
End channel 102.
Back extrapolation model: Vonk.
FOURIER TRANSFORM PARAMETERS:
Transform performed up to D = 0.200000E+03 Angstroms.
Steps of 0.100000E+01 Angstroms.

1. MOMENTS RESULTS.
Zeroth moment: 0.693215E+07 cm^-2.

```

Invariant: 0.456331E+21 cm⁻⁴.

2. LAMELLA MORPHOLOGY RESULTS.

Long period [Angstroms]	0.760000E+02.
Average hard block thickness [Angstroms]	0.150709E+02.
Average soft block thickness [Angstroms]	0.609291E+02.
Bulk volume crystallinity [No units]	0.224838E+00.
Local crystallinity [No units]	0.198302E+00.
Average hard block core thickness [Angstroms]	0.400000E+01.
Average interface thickness [Angstroms]	0.300000E+01.
Polydispersity [No units]	0.269384E+01.
Electron density contrast [cm ⁻²]	0.546961E+11.
Specific inner surface [Angstroms ⁻¹]	0.298373E-01.
Non-ideality [No units]	0.443213E-01.

3. POROD RESULTS.

Porod constant [cm ⁻⁵]	0.317685E+28.
Porod chord length [Angstroms]	0.182984E+02.
Crystalline chord length [Angstroms]	0.236059E+02.
Amorphous chord length [Angstroms]	0.813849E+02.
Surface to volume [Angstroms ⁻¹]	0.380986E-01.

END OF CORRELATION FUNCTION RESULTS.

The *real-space* (R) axis and the correlation function itself are written to the ASCII *RAX.TXT and *CF1.TXT files, respectively.

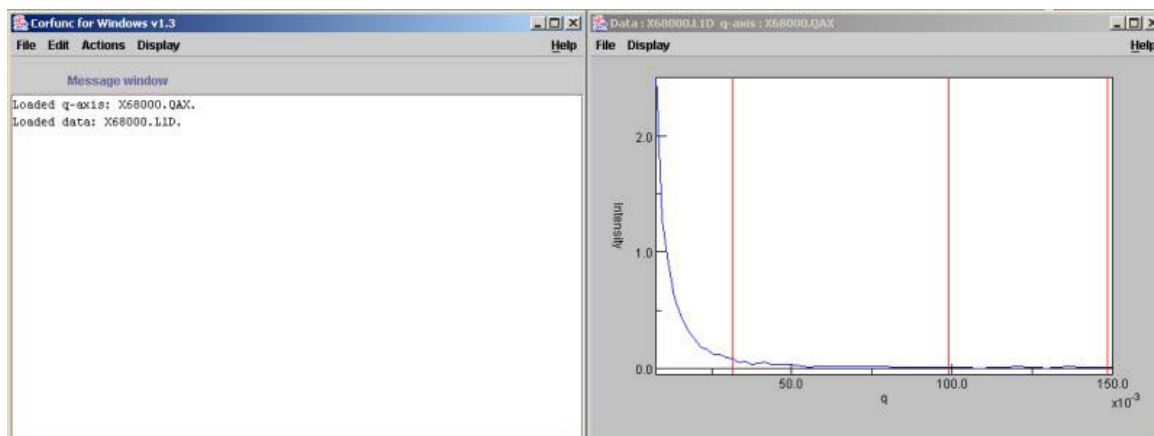
Now we'll generate a volume fraction profile. The principles of this type of analysis are described in "Using SANS to study adsorbed layers in colloidal dispersions", King, S M; Griffiths, P C; Cosgrove, T. Chapter 4 in "Applications of Neutrons in Soft Condensed Matter", Gabrys, B J (editor), Gordon & Breach, 2000.

Do "File" → "Load Q-axis". Select "BSL/OTOKO" from the drop-down menu, navigate to the file K:\Corfunc\X68000.QAX and double-click on the filename.

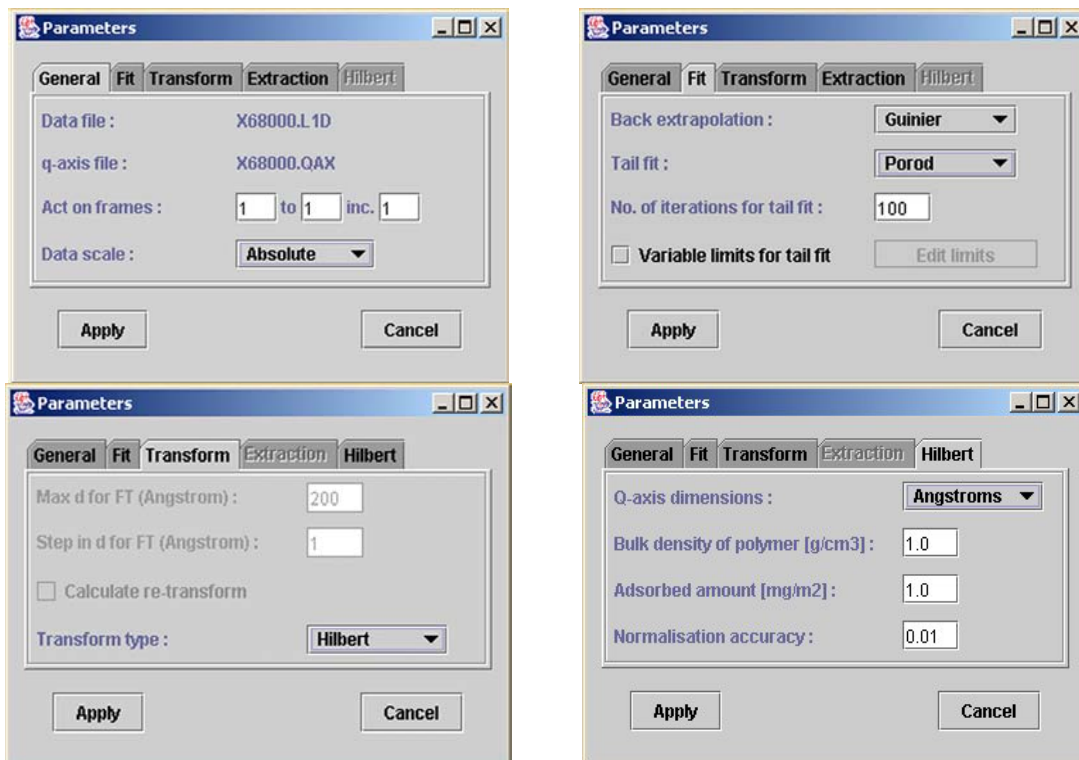
Now do "File" → "Load data" and read in the file K:\Corfunc\X68000.L1D.

IMPORTANT! At present the data and Q-axis files to be analysed *must* be located in the top-level of the CORFUNC folder for the program to work properly.

A graphics window will appear displaying the data and three vertical red lines.



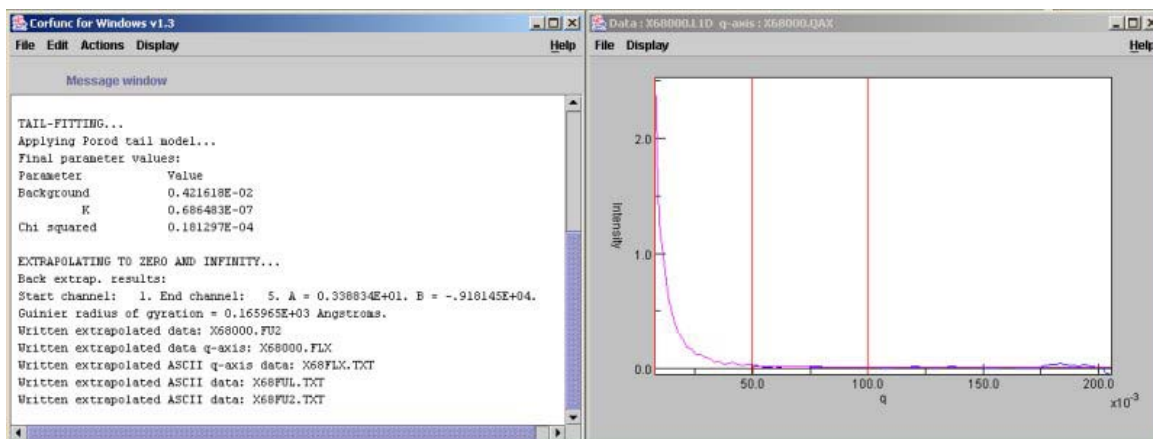
Do "Edit" → "Parameters" and set:



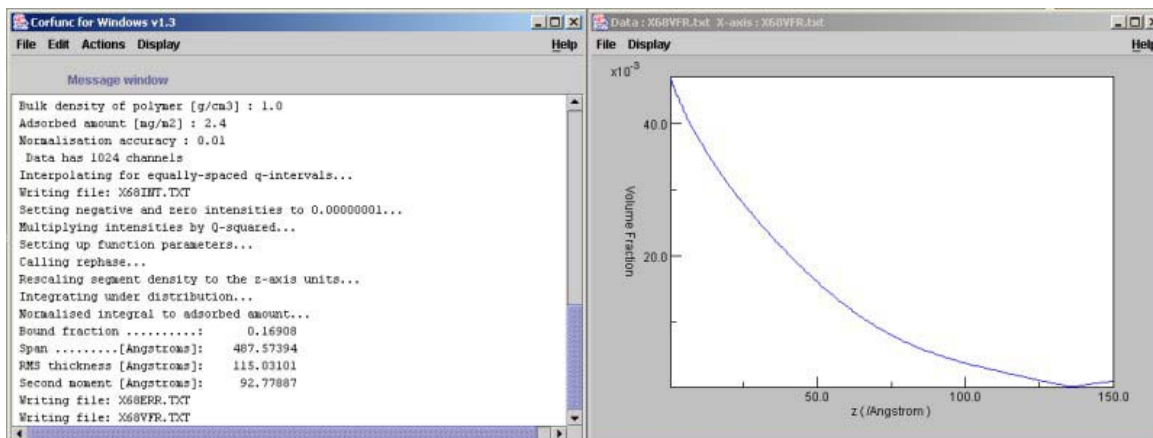
Click "Apply" when done.

Using the mouse, right click on the leftmost red line and, keeping the button depressed, move the line to $q \sim 0$. CORFUNC will use the region between the middle and rightmost red lines to judge the high- q extrapolation of the data that it will perform. Move these to encompass the region $50 \times 10^{-3} < q < 100 \times 10^{-3}$.

Do "Actions" → "Extrapolate". CORFUNC will extrapolate the data to low and high q values and display the extrapolation in the graphics window.



Do "Actions" → "Transform". CORFUNC will transform the extrapolated data and calculate the volume fraction profile which will appear in a second graphics window.



Doing “Actions” → “Extract parameters” has no effect when the “Transform type” is set to “Hilbert” (in fact the program may crash!). However, structural information relating to the profile is displayed in the CORFUNC window and written to the file TROPUS.TXT. Here is some sample output:

```

Reading file: X68FUL.TXT
Reading file: X68FLX.TXT
Y
  1.00000
  2.40000
Normalisation factor is
  0.01000
Data has 1024 channels
Writing file: X68INT.TXT
Integral is:      0.13139
.
. †
.
Integral is:      2.39129
Bound fraction .....: 0.16908
Span .....[Angstroms]: 487.57394
RMS thickness [Angstroms]: 115.03101
Second moment [Angstroms]: 92.77887
Writing file: X68ERR.TXT
Writing file: X68VFR.TXT

END OF VOLUME FRACTION PROFILE ANALYSIS

```

[†] the program rescales the volume fraction profile here until the integral equals the (usually known) adsorbed amount entered \pm the normalisation accuracy parameter.