

User's Manual

PDFgetX

Version 1.1

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Preface

Disclaimer

By downloading the program PDFgetX, you agree to the terms and conditions concerning its use specified in the license agreement that is provided as part of the distribution. End users wishing to make commercial use of the software must contact Libraries, Computing & Technology, Michigan State University, East Lansing, MI 48824; (517)353-0722 prior to any commercial distribution to discuss terms. The Software is provided to End User by MSU on an as is basis. No user support is provided or implied.

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Using PDFgetX

Publications of results totally or partially obtained using the program **PDFgetX** should state that **PDFgetX** was used and contain the following reference:

JEONG, I.-K., THOMPSON, J.,PROFFEN, TH., PEREZ, A. AND BILLINGE, S. J. L.
“PDFgetX, a program for obtaining the atomic Pair Distribution Function from X-ray powder diffraction data”

Acknowledgments

The **PDFgetX** is coded using Yorick language [1]. The atomic scattering factors are calculated using the analytic formula and coefficients developed by D. Waasmaier and A. Kirfel [2]. The mass attenuation coefficient data of elements are obtained from the web at: <http://physics.nist.gov/PhysRefData/FFast/html/form.html> [3].

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Contents

1	Introduction	6
1.1	What is PDFgetX	6
2	Installation	7
2.1	System Requirements	7
2.2	What You Need	7
2.2.1	Yorick	7
2.2.2	PDFgetX	8
2.2.3	Installing and Configuring PDFgetX	8
2.2.4	Report problems and suggestions	9
3	Tutorial: In_{0.33}Ga_{0.67}As Semiconductor	10
3.1	Preliminary Data Analysis	11
3.1.1	Reduction of SPEC file	11
3.1.2	Reduction of Multi-Channel Analyzer(MCA) data	18
3.2	Refine structure function of In _{0.33} Ga _{0.67} As	20
4	Using PDFgetX	27
4.1	Overview of PDFgetX	27
4.1.1	Launching PDFgetX	28
4.1.2	Exiting PDFgetX	28
4.1.3	The Main Menu	28
4.2	Data Analysis Procedure in PDFgetX	30
4.3	History File	32
4.4	Some Yorick Information	32
5	Data Corrections	33
5.1	Dead-Time Correction	33
5.2	Multiple Scattering Correction	35
5.3	Polarization Correction	36
5.4	Absorption Correction	36
5.5	Compton Scattering Correction	36
5.6	Normalization	38
5.7	Laue Scattering Correction	38
5.8	Pair Distribution Function	38

<i>CONTENTS</i>	3
5.9 Error Propagation	39
A SPEC file format	40
B Description of the history file	42
C MCA file format	44

List of Figures

3.1	Comparison of normalized elastic scattering	15
3.2	MCA spectrum of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ at $Q=40\text{\AA}^{-1}$	20
3.3	(a) Effects of corrections on raw data of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$. (b) Comparison between normalized DATA and $\langle f^2 \rangle$	24
3.4	Data corrections in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ alloy	24
3.5	Reduced Structure Function and PDF of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor	25
4.1	Data analysis procedure in PDFgetX	30
4.2	Structure function refinement procedure in PDFgetX	31
5.1	Dead-time correction in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor	34
5.2	Double scattering ratio for nickel	35
5.3	Absorption factors in transmission and reflection geometry	37
5.4	Comparison between Compton and elastic scattering intensities in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$	37
C.1	MCA file format	45

List of Tables

2.1	Known Platforms Supporting PDFgetX	7
3.1	Summary of structure function refinement	25

Chapter 1

Introduction

1.1 What is PDFgetX

PDFgetX is a program to be used to obtain the atomic Pair Distribution Function (PDF) from a measured X-ray powder diffraction data. PDFgetX is written using the Yorick, an interpreted language. This will require users to obtain the Yorick distribution and install it yourself. See Chapter 2 for help in installation.

PDF is the instantaneous atomic number density-density correlation function which describes the atomic arrangement in materials. A useful characteristic of PDF method is that it gives both *local* and *average* structure information because both Bragg peaks and diffuse scattering are used in the analysis. And from the PDF peak width, it's possible to obtain the information about bond-length distribution (static, thermal) [4] and correlated atomic thermal motion [5]. By contrast, an analysis of the Bragg scattered intensities alone, by a Rietveld type analysis for instance, yields the *average* crystal structure only and the extended x-ray absorption fine structure(EXAFS) gives nearest-neighbor and next nearest-neighbor distance information. PDF analysis method has long been used to characterize glasses, liquids and amorphous materials. Recently, however, it has found more application in the study of local structural disorder in crystalline materials, where some deviation from the *average* structure is expected to take place.

Obtaining total scattering structure function (and PDF) from raw diffraction data requires many corrections for experimental effects such as absorption, polarization corrections and removing of Compton and multiple scattering contribution to the elastic scattering. Also it needs proper error propagation to be used in modeling of PDF using either PDFFIT (real-space Rietveld) [6] or a Reverse Monte Carlo approach [7] using e.g. DISCUS [8] to yield structural parameters. PDFgetX allows users to do all these data corrections and error propagation in convenient ways. During the refinement, PDFgetX displays each correction effect to the raw data and saves all the parameters used for refinement. This makes the refinement processes easy to understand and allows reproducible results. PDFgetX supports the following data formats: multi-column ascii file, SPEC and multi-channel analyzer(MCA) files.

To find out about recent updates of PDFgetX or to get further information visit the PDFgetX homepage at the following site:

<http://www.pa.msu.edu/cmp/billinge-group/programs/PDFgetX>

Chapter 2

Installation

2.1 System Requirements

PDFgetX should run on any UNIX/Linux platform supported by Yorick. This includes PC/Unix and SGI. It also runs on Windows NT and 95. For a list of systems on which PDFgetX is known to work, see Table 2.1. If you successfully install PDFgetX on a system not included in this list, please contact us and let us know. If you cannot install PDFgetX on a system, and have studied the documentation thoroughly, please contact us and ask for help. Without access to a similarly configured system, we may not be able to help you with the installation, but see Section 2.2.4 for instructions on how to report your trouble.

Table 2.1: Known Platforms Supporting PDFgetX

Hardware	Operating System
Intel 486	RedHat Linux 6.0
	Windows 95/NT
DEC-ALPHA	Digital Unix
SGI	Irix

2.2 What You Need

2.2.1 Yorick

Before you can run PDFgetX, you will need to install Yorick. PDFgetX is written in the Yorick language, which is an interpreted C-like language (and it's free). The distribution of PDFgetX contains only the source code files for PDFgetX; it does *not* come with Yorick. The latest version of Yorick can be downloaded from the official site:

<ftp://wuarchive.wustl.edu/languages/yorick/yorick-ad.html>

This document provides no information about installing Yorick; see the Yorick readme files for help with the installation and checking that the installation was successful. Before installing PDFgetX, be sure that your installation of Yorick works.

2.2.2 PDFgetX

You may obtain the latest version of PDFgetX from the PDFgetX website:

<http://www.pa.msu.edu/cmp/billinge-group/programs/PDFgetX>

PDFgetX is provided as a compressed file. Use the command

```
tar -xzvf pdfgetx1.1.tar.gz
```

which will extract the files into a new directory called “PDFgetX/”. And you can find the following program files under the directory PDFgetX/.

```
pdfgetx.i, pdfgetxdistribution.i, pdfgetx_custom.i  
ASF.DAT, PERIODIC_TABLE.DAT, MASS_ABS_COEFF.DAT, LICENSE.TXT
```

If the `-z` flag does not work on your system, then use the commands

```
gzip -d pdfgetx1.1.tar.gz, tar -xvf pdfgetx1.1.tar
```

to extract the files.

2.2.3 Installing and Configuring PDFgetX

To customize the PDFgetX installation, you need to modify two files, “custom.i” and “pdfgetxdistribution.i”. If you are new user of “Yorick”, you can simply rename “pdfgetx_custom.i” (included in compressed file) to “custom.i” and give the proper path for the “Actual_PATH” in the following two lines in “pdfgetx_custom.i” file.

```
#include "Actual_PATH/pdfgetx.i"  
#include "Actual_PATH/pdfgetxdistribution.i"
```

And then create a directory “/Yorick” under your home directory and place your own version of “custom.i” there. If you already have your own version of “custom.i”, simply add the above two lines in the “custom.i” file.

For Windows OS, you need to beware of a few things. First, the path should look like the following: “/c/pdfgetx/..”. Second, in windows OS, users can set the size of font and graphic window in “custom.i” file. Therefore it is better to copy the “custom.i” file coming with the Yorick Window version and add the above two lines there than just rename “pdfgetx_custom.i” to “custom.i”. Finally, remember that if the directory name has a space (“ ”) as in “My Directory”, the Yorick couldn’t find the directory.

After configuring the “pdfgetx_custom.i” file, if you open the file “pdfgetxdistribution.i” using a text editor, you will find the following code:

```
asf_file="Actual_PATH/ASF.DAT"  
periodic_table="Actual_PATH/PERIODIC_TABLE.DAT"  
mabscoeff_file ="Actual_PATH/MASS_ABS_COEFF.DAT"
```

again, give the proper path for the “Actual_PATH”. These code set paths for three important data files: Atomic scattering factor, Periodic table, and Mass absorption coefficient. Also, the variable name, e.g. `asf_file`, should not be changed, otherwise PDFgetX couldn’t find these data files.

If you want to print graphs directly from PDFgetX, you need to edit the file “pdfgetxdistribution.i” and change the following line:

```
printer_string="lpr -h -Prm31 __temp.ps"
```

Modify the printer string to reflect your system. When printing, PDFgetX creates a temporary postscript file called “__temp.ps” and makes a system call to print the file. Do not change the name of the file in the printer string or printing will not work. For windows OS, “lpr” command doesn’t work, instead use “print” command. This is a DOS command and it seems it sends the graph to a printer connected via “LPT1” port. If this setting is not working, we would recommend windows OS users to use another method to print graphs. First, save the graph as a postscript (PS) file or window meta file (WMF) using [S] option in the main menu, and open it using ghostview (PS file) or using MS word (WMF file). Then print the graph using print command in the program.

2.2.4 Report problems and suggestions

If you have any problems in installing & running PDFgetX and have any suggestions about the PDFgetX, please send email to the following address:

billinge@pa.msu.edu

<http://www.pa.msu.edu/cmp/billinge-group/programs/PDFgetX.html>

Chapter 3

Tutorial: $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ Semiconductor

Now you might have installed PDFgetX and can start it simply by typing **pdfgetx** at Yorick prompt. In this tutorial, you'll get a chance to analyze $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy data collected at Cornell High Energy Synchrotron source (CHESS) using intense x-rays of 60 KeV ($\lambda = 0.206 \text{ \AA}$). The tutorial files can be downloaded from the PDFgetX homepage. In this experiment, the incident x-ray energy was selected using a Si(111) double-bounce monochromator. The data were collected at 10 K to minimize thermal atomic motion in the sample, and hence increase the sensitivity to static displacement of atoms due to alloying using a closed cycle helium refrigerator mounted on the Huber 6 circle diffractometer. All the signal measured was saved to a file using the system controlling software, SPEC.

The signal measured using the intrinsic Ge solid state detector was processed in two ways. Using single-channel pulse-height analyzer (SCA), the elastic scattering, Compton scattering, and elastic + Compton scattering were collected separately. In the measurements using SCA, the proper energy window setting for the elastic scattering is very important because any error in the window setting could cause an unknown contamination to the elastic scattering thus make data corrections very difficult. At the same time, the signal was fed to multi-channel analyzer (MCA) to record the complete energy spectrum of each value of Q. The elastic and Compton scattered radiation could then be separated using software after measurement. Collecting data using MCA has advantages and disadvantages. In the MCA method, since the entire energy spectrum of the scattered radiation is measured at each value of Q, the error caused by the mis-set of energy window is negligible. The main disadvantage of the MCA method is that it has a larger dead-time, although this can be reliably corrected [9].

This tutorial is composed of two subsections, "Preliminary Data Analysis" and "Refine structure function". The "Preliminary Data Analysis" section is mainly concentrated on how to reduce SPEC and MCA file to build PDFgetX *input file* for structure refinement. In "Refine structure function" section, the step by step procedure of structure function refinement is presented. Users can build the *input file* using tutorial SPEC file in "Preliminary Data Analysis" section or use a tutorial *input file* coming with the program. In this manual, *SPEC file* refers to the data collected using SCA and *MCA file* for the data collected using MCA.

3.1 Preliminary Data Analysis

3.1.1 Reduction of SPEC file

The raw data from x-ray powder diffraction measurements using either a sealed x-ray tube or synchrotron source could have many different file formats and could contain multiple scans that ought to be averaged together. Therefore it is very difficult to use the raw data directly in the structure function refinement. With these things in mind, we limited the “Preliminary Data Analysis” to support only the SPEC file format and N-column ascii file format. For details about SPEC file format, please refer to Appendix A. This section will show you how to reduce the raw SPEC data into the *input file* from which to start analysis. This process includes extracting scans from SPEC file, comparison of different scans, applying dead-time correction and combining different scans.

In general, one SPEC file contains many scans. The following shows a scan header of SPEC file collected at CHESS.

```
#L pmQ ereal elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC
```

During the SPEC file reduction process, we will use *column number* to refer to a specific variable such as *ELASTIC*, *IC2*, *PULSER*, *etc* , so you need to remember which column corresponds to which variable. Follow along with this example terminal output. It will guide users to learn about how the “Reduction of SPEC file” works. The comments in */* ... */* mark are added just for explanation purpose and will not be shown in the real analysis.

```
current directory> yorick
```

```
Copyright (c) 1996. The Regents of the University of California.
All rights reserved. Yorick 1.4 ready. For help type 'help'
```

```
> pdfgetx
```

```
Pair Distribution Function from the X-ray powder diffraction (PDFgetX 1.1)
```

```
0) Preliminary data reduction
1) Build a setup file
2) Background Substraction
3) Reduction of Structure Function: S(Q)
   Input file format: (Q, I, dQ, dI)
4) PDF calculation: G(r)
   Input file format: (Q, S(Q), dQ, dS)
P) Print, S) Save, U) Unzoom, L) Limits windows
Q) Quit
```

```
[0-4 qsulp] 0 /* Enter to the Preliminary data reduction level */
```

```
1) Extract Scan(s) from SPEC file
2) Compare N-column(N>=2) ascii files
```

- 3) Combine N-column($N \geq 2$) ascii files
- 4) Build PDFgetX input format: (Q, I, dQ, dI)
- 5) Convert MCA file to N-column ascii file
- Q) Return to Main

[1-5Q] 1

The Input should be SPEC file format : Continue (y/n)? y

ENTER SPEC FILE NAME TO READ: in33_tutorial.spec

- The following shows scan information in in33_tutorial.spec

```
#S 1 601 pts --> ascan pmQ 1 13 600 1
#S 2 601 pts --> ascan pmQ 1 13 600 1
#S 3 1401 pts --> ascan pmQ 12 40 1400 1
#S 4 1401 pts --> ascan pmQ 12 40 1400 1
#S 5 1401 pts --> ascan pmQ 12 40 1400 1
-----
```

EXTRACT SCANS FROM SPEC FILE:

- Each scan will be saved as an ascii file
- Enter all scans to be read: [Ex: 2 4 5] 1 2 3 4 5
- /* Extract good scans by entering the scan number */

SAVE SCANS TO ASCII FILE:

- Output file name will be 'samplename_scannumber.asc'
- Enter your 'samplename' [Ex. InAs] : in33

Return to Preliminary data reduction

Now each scan is saved as ascii file. As it is mentioned during extracting process, the file name will be "in33_1.asc", "in33_2.asc", and so on. Now we can compare these different scans.

[1-5Q] 2 /* Compare N-column ($N \geq 2$) ascii files */

The Input should be N-column ascii file format :
Plot $y(=\text{data}/\text{norm})$ vs. $x(=q)$. Continue (y/n)? y

ENTER FILE NAMES TO COMPARE, TO QUIT READING, ENTER 'Q':

- File name to compare : in33_1.asc

```
#L pmQ ereal elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC
```

ASSIGN COLUMN NUMBER TO VARIABLES:

- Column # corresponding to X-axis : 1
- Column # corresponding to DATA : 13

- Normalization of data :
 for constant normalization, enter '0'
- Column # corresponding to NORM. : 12

ENTER FILE NAMES TO COMPARE, TO QUIT READING, ENTER 'Q':

- File name to compare : in33_2.asc

.
.

ENTER FILE NAMES TO COMPARE, TO QUIT READING, ENTER 'Q':

- File name to compare : in33_5.asc

ENTER FILE NAMES TO COMPARE, TO QUIT READING, ENTER 'Q':

- File name to compare : q

/* Dead-time correction setting */

READ FILE : in33_1.asc

APPLY DEAD-TIME CORRECTION FOR DATA (y/n)? y

- 1) Dead-time correction using detector dead-time
- 2) Dead-time correction using pulser measurement
- Q) Exit Dead-time correction

[1-2Q] 2

Enter column # containing pulser : 9

APPLY DEAD-TIME CORRECTION FOR MONITOR (y/n)? n

/* For details, refer to CH. 4 Using PDFgetX */

CHECK VARIABLE AND ASSIGNED COLUMN #

- X-axis : 1
- DATA : 13
- Normalization : 12

Detector dead-time Correction using pulser

```

- Pulser column          : 9

No Monitor dead-time Correction

CHANNEL SETTINGS ARE CORRECT (y/n)? y

READ file: in33_2.asc

.
.

READ file: in33_5.asc

Colors in order of reading: magenta, cyan, blue, green, red, and blacks

COMPARE OTHER VARIABLE (y/n)? n

EXIT COMPARING FILES:

Return to Preliminary data reduction
-----

```

Fig. 3.1 shows dead-time corrected elastic scattering after normalization by monitor. It shows that these scans overlap with each other quite nicely. Maybe it's good to check how the comparison looks like if the dead-time correction is not applied. Since the normalized elastic scattering from different scans overlap with each other, we can combine all of them.

```

[1-5Q] 3 /* Combine N-column (N>2=2) ascii file */

The Input should have N-column ascii file format !
The X-axis column should have constant step(dX) !
Combine upto five variables: X-axis(Q, Two-theta)!
DATA, NORM, and Aux1, Aux2 (auxiliary variables) !
Continue (y/n)? y

ENTER FILE NAMES TO COMBINE, TO QUIT READING, ENTER 'Q':
- File name to combine : in33_1.asc

#L pmQ ereal elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC

ASSIGN COLUMN NUMBER TO VARIABLES:

- If the column # is set to '0', the corresponding variable
- will not be propagated !!

```

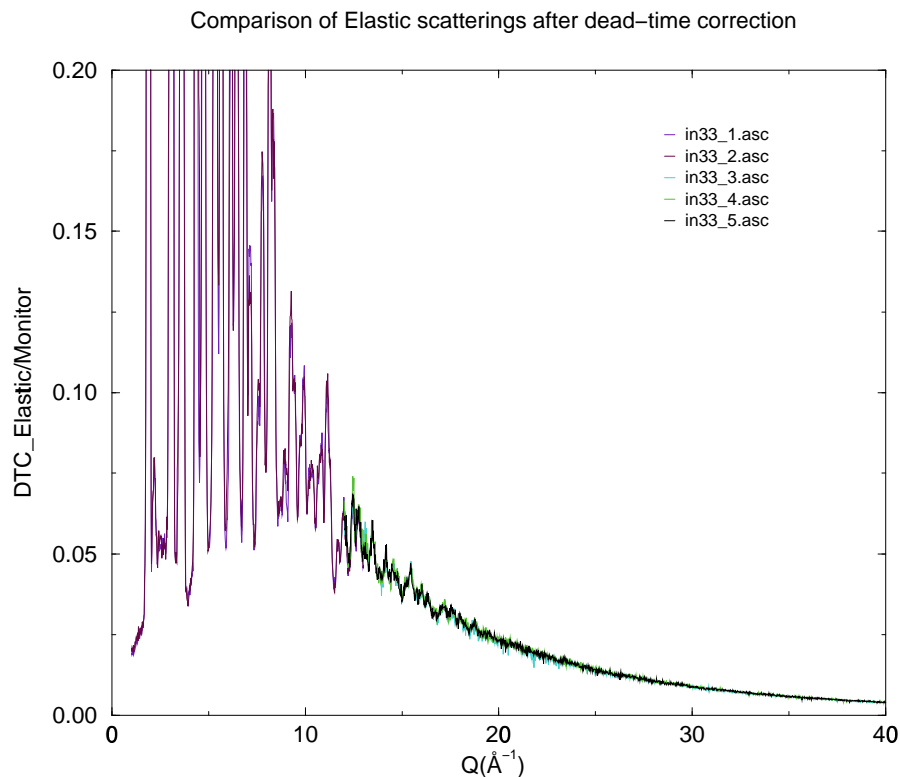


Figure 3.1: Comparison of normalized elastic scattering in five scans after dead-time correction. It shows that the elastic scattering in each scan overlap with each other quite nicely.

```

- Column # corresponding to X-axis : 1
- Column # corresponding to DATA  : 13
- Column # corresponding to NORM.  : 12
- Column # corresponding to Aux1   : 0
- Column # corresponding to Aux2   : 0

```

ENTER FILE NAMES TO COMBINE, TO QUIT READING, ENTER 'Q':

```
- File name to combine : in33_2.asc
```

```
.
.
```

ENTER FILE NAMES TO COMBINE, TO QUIT READING, ENTER 'Q':

```
- File name to combine : in33_5.asc
```

ENTER FILE NAMES TO COMBINE, TO QUIT READING, ENTER 'Q':

```
- File name to combine : q
```

READ file : in33_1.asc

Apply DEAD-TIME CORRECTION FOR DATA (y/n)? y

- 1) Dead-time correction using detector dead-time
- 2) Dead-time correction using pulser measurement
- Q) Exit Dead-time correction

[1-2Q] 2

Enter column # containing pulser : 9

APPLY DEAD-TIME CORRECTION FOR MONITOR (y/n)? n

CHECK VARIABLE AND ASSIGNED COLUMN #

- X-axis	: 1
- DATA	: 13
- Normalization	: 12
- AUX1	: 0
- AUX2	: 0

Detector dead-time Correction using pulser :

- Pulser column	: 9
-----------------	-----

No Monitor dead-time Correction

CHANNEL SETTINGS ARE CORRECT (y/n)? y

READ file : in33_2.asc

.
.

READ file : in33_5.asc

Enter variable name corresponding to X-axis: Q

Enter variable name corresponding to DATA : Elastic

Enter variable name corresponding to NORM. : Monitor

SAVE COMBINED DATA:

- Enter file name for combined data: in33_tutorial.comb

Make sure the X-column and combined variables have the correct value !!

Return to Preliminary data reduction

We just obtained combined N-column ascii file. The next step is to convert it to the PDFgetX input format: (Q, I, dQ, dI). Here Q is the magnitude of scattering vector and defined as $Q = 4\pi \sin(\theta)/\lambda$. I is the intensity of elastic scattering normalized by the monitor, dQ and dI are errors in Q and I. In this tutorial we'll not preprocess the background data. It'll be given as 4-column (Q, I, dQ, dI) format.

```
[1-5Q] 4 /* Build PDFgetX input file */
```

```
The Input should be N-column(N>=2) ascii file
All columns should have same number of lines
Blanks and commas in columns are not permitted
Continue (y/n)? y
```

```
ENTER FILE NAME TO READ: in33_tutorial.comb
```

```
- Enter number of comment(and blank) lines in the data header : 2
```

```
CHECK FILE FORMAT:
```

```
1) Column contains Q/Two-theta?      : 1
   - Data in Q or Two-theta value?    : Q
2) Column contains Intensity?        : 2
3) Column contains Monitor/Time?     : 0
   - Normalization by Monitor or Time?: No Normalization
x) Exit.
```

```
[1-3X] 3
```

```
Column contains Monitor/Time? : 3
```

```
- Normalization by Monitor or Time(M/T)? m
```

```
1) Column contains Q/Two-theta?      : 1
   - Data in Q or Two-theta value?    : Q
2) Column contains Intensity?        : 2
3) Column contains Monitor/Time?     : 3
   - Normalization by Monitor or Time?: Monitor
```

```
x) Exit.
```

```
[1-3X] x
```

```
SAVE PDFgetX INPUT FILE (Q, I, dQ, dI):
```

```
- Enter file name to for input file: in33_tutorial.input
```

```
Return to Preliminary data reduction
```

```
-----
```

Now, you obtained the *input file* for the structure function refinement. If the X-column is 2θ , then the program converts it to Q.

3.1.2 Reduction of Multi-Channel Analyzer(MCA) data

The MCA file format could be different depending on the instruments. Therefore here we assume an MCA file format which we used in the data reduction process. The MCA file format we used is attached in Appendix B. In this file, the scattered intensity at every Q point is distributed to the whole MCA channels. So each block which is separated by blank corresponds to each Q point. In order to convert this file to the normal N-column ascii file, we need to know the following information: Minimum Q or two-theta, number of data points, Q/two-theta step (this should be constant), total channel number of MCA. Many of these information can be obtained using “scan_summary” function which is a part of PDFgetX. We will not provide whole MCA file to do the analysis but just one MCA file to show you how it works. However we can tell that the structure function obtained using SPEC and MCA data are basically same.

```
[1-5Q] 5 /* Convert MCA file to N-column ascii file */
```

```
File should have MCA file format: Continue (y/n)? y
```

```
ENTER MCA FILE NAME TO READ: in33_tutorial.mca
```

```
- Build an X-axis column of corresponding MCA data
```

```
- The X-axis should be either 'Q' or 'Two-theta'
```

```
- The X-axis should have constant step (dX)
```

```
- Enter variable name corresponding to X-axis, [Q/TT] : q
```

```
- Enter minimum Q value : 36
```

```
- Enter number of data points : 251
```

```
- Enter Q step : 0.02
```

```
- Total channel number of MCA : 1024
```

```
READING MCA DATA, BE PATIENT!!
```

```
SET UP THE INTEGRATING REGIONS OF MCA SPECTRUM:
```

```
- Propagate upto four variables:
```

```
Elastic, Elastic+Compton, Aux1 and Aux2
```

- If the starting and ending channel number of a variable
- is same, the corresponding variable will not be propagated

```

- Start of elastic channel           : 639
- Stop of elastic channel           : 657
- Start of elastic_compton channel  : 470
- Stop of elastic_compton channel   : 657
- Start of MCA auxiliary channel 1  : 0
- Stop of MCA auxiliary channel 1   : 0
- Start of MCA auxiliary channel 2  : 0
- Stop of MCA auxiliary channel 2   : 0

```

read-write binary stream: `_mcachannel`

```

In directory: /home/jeong/analysis/gainas/data/in50/mcafile/
/* Open a binary file to save MCA channel setting */

```

CHECK SETTING OF MCA CHANNEL

```

1) Start of elastic channel           : 639
   Stop of elastic channel           : 657
2) Start of elastic_compton channel  : 470
   Stop of elastic_compton channel   : 657
3) Start of MCA auxiliary channel 1  : 0
   Stop of MCA auxiliary channel 1   : 0
4) Start of MCA auxiliary channel 2  : 0
   Stop of MCA auxiliary channel 2   : 0
Q) Exit MCA channel setting

```

Enter to reset channel setting [1-4Q] : q

INTEGRATE REGIONS OF INTEREST!!

SAVE MCA DATA TO ASCII FILE:

```
- Enter file name for MCA-derived data: in33_mca.dat
```

READ ANOTHER MCA FILE(y/n)? n

EXIT MCA FILE READING:

Return to Preliminary data reduction

Fig. 3.2 shows MCA spectrum of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ at $Q=40\text{\AA}^{-1}$. The elastic and Compton scattering are well separated at this value of Q . It also shows some fluorescence peaks in low channel number

side. If you check the output file, you'll find that it contains Q, MCA_Elastic, MCA_ElaCompt, and

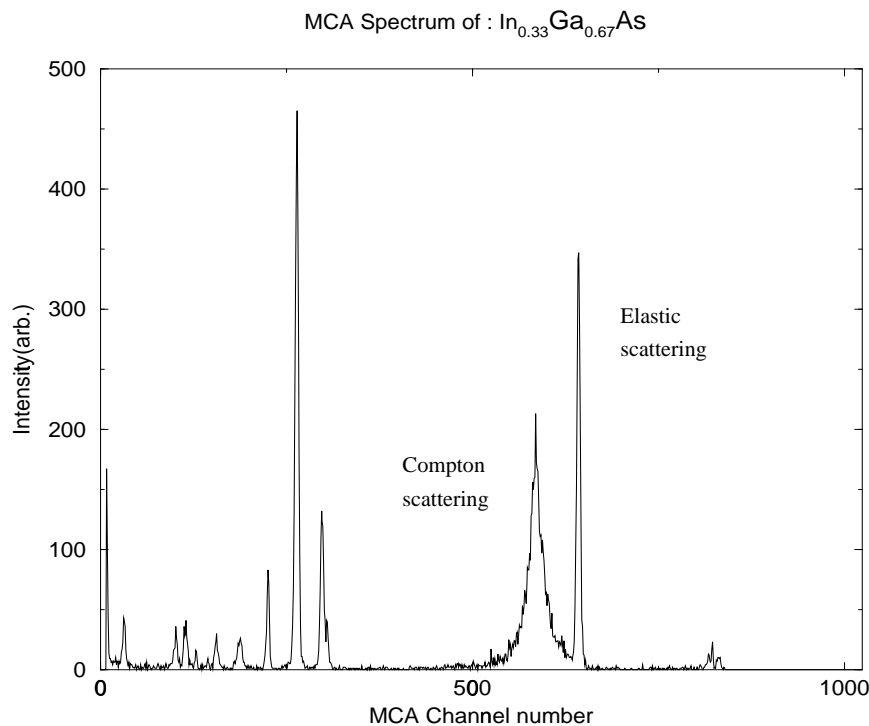


Figure 3.2: MCA spectrum of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ at $Q=40\text{\AA}^{-1}$. It shows that the very sharp elastic scattering around channel number 640 is well separated from the broad Compton scattering. The fluorescence of the alloy are shown below channel number 300.

MCA_Det_Tot. When the program generates X-column, it assumes constant X-step. The whole MCA files can be converted to N-column ascii files in this way and then as we did in “Reduction of SPEC file” these files can be compared and combined. Also it's possible to apply dead-time correction for MCA file.

3.2 Refine structure function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$

Now we are almost ready to refine structure function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy except two more things; building setup file and background subtraction.

```
[0-4 qsulp] 1 /* Enter to Build a setup file in Main */
```

```
BUILD SETUP FILE:
```

```
-----
Note that the setup file is a text file written
using Yorick syntax, and may be modified in emacs without going
through this whole procedure of building a new one.
```

If PDFgetX crashes, check the (text) input file very closely!

ENTER THE TYPE OF INCIDENT X-RAY RADIATION:

- [S]ilver, [M]olybdenum, [C]opper or enter [E]nergy [smce]? e
- Enter Energy of incident X-ray (KeV) : 59.67

SAMPLE INFORMATION:

- Number of elements in the sample[Ex. InGaAs => 3]? 3

- Element #: 1
- Enter the element (ions not yet supported) : in
- Enter fractional composition: 0.33

- Element #: 2
- Enter the element (ions not yet supported) : ga
- Enter fractional composition: 0.67

- Element #: 3
- Enter the element (ions not yet supported) : as
- Enter fractional composition: 1

- Enter absorption coefficient*thickness of sample, mu*t: 1.11

MONOCHROMATOR INFO.:

- 1) d-spacing of monochromator: 3.135
 - 2) Position of monochromator : incident_beam
 - 3) Type of monochromator : perfect_cryst
- Q) Exit

[123Q] Q

SAVE SETUP FILE AS: in33_tutorial.setup

You've finished creating a setup file which contains information about the sample composition and experimental setup. You may take a look at the setup file using a text editor and check what you have there. Also you can add some more comment using Yorick syntax if you want. The final step before starting refinement is to subtract background. Because the sample itself affects magnitude of background, sometimes instrument background (background measured without sample) over estimate the real background. So in background correction, the program allows users can change the magnitude of background by multiplying correction constant in order to make it match data

more nicely in low Q.

```
[0-4 qsulp] 2 /* Enter to Background subtraction in Main */

The Input should be 4-column ascii file(Q, I, dQ, dI)
Continue (y/n)? y

ENTER DATA FILE NAME TO READ      : in33_tutorial.input

ENTER BACKGROUND FILE NAME TO READ : in33_bkg.input

BACKGROUND SUBTRACTION:
- Multiply correction constant to background to make
- it match data more nicely in low Q (y/n)? n
- 30 negative intensities set to 0.000208106.
/* negative value are set to minimum intensity */

ENTER FILE NAME FOR BACKGROUND CORRECTED DATA: in33_cfbg.input
-----

[0-4 qsulp] 3 /* start structure function refinement */

ENTER SETUP FILE NAME: in33_tutorial.setup

READ INPUT FILE: It should be 4-column ascii file(Q, I, dQ, dI)
- Enter data file name to read: in33_cfbg.input

DATA REDUCTION :

read-write binary stream: _history.pdb
In directory: /u24/jeong/PDFgetX/manual/
/* Open binary file to save refinement history. Refer to the Appendix C */

SMOOTH DATA USING SAVITZKY & GOLAY METHOD (y/n)? n

Flat Symmetric [R]eflection or [T]ransmission geometry (r/t)? t
/* Choose either symmetric flat reflection or transmission geometry */

-----
Flat Plate Symmetric Transmission Geometry Data Correction
-----

WINDOW 0: CORRECTION EFFETS ON RAW DATA !
```

```

APPLY MULTIPLE SCATTERING CORRECTION (y/n)? y
  - Does the data contain Compton scattering in high Q (y/n)? n
  - Multiple scattering calculation in transmission geometry

WINDOW 3: MULTIPLE SCATTERING RATIO!

APPLY POLARIZATION CORRECTION (y/n)? n
  => Polarization correction NOT applied!

APPLY ABSORPTION CORRECTION (y/n)? y

NORMALIZATION USING MID-HIGH Q PART OF DATA
  - Enter a mid-range Q value (roughly 26.4): 25
  - 751 points are used for normalization
  - Approximate normalization constant: 1904.38

WINDOW 1: CORRECTED DATA vs. TIS!

ENTER NORMALIZATION CONSTANT: 1920

APPLY COMPTON SCATTERING CORRECTION
  - Apply Compton correction in MID-LOW Q region using 'Ruland' method.
  - Enter integral width 'b' (try 0.008): 0.003
  - For details, please refer to the MANUAL !!

WINDOW 4: <f^2>, Compton, and Modified Compton by the Ruland function

WINDOW 2: REDUCED STRUCTURE FUNCTION, Q*(S(Q)-1)!

CHECK IF F(Q) = (S(Q)-1)*Q IS APPROXIMATELY 0 AT HIGH Q
  - Is F(Q) approximately 0 at high Q (y/n)? y

SAVE STRUCTURE FUNCTION, S(Q), TO ASCII FILE:
  - Enter file name to save data: in33_tutorial.soq
-----

```

Now you've obtained structure function. Before we move on, let's examine the corrections applied during data reduction. First, Fig. 3.3(a) shows all the correction effects on the raw data. It shows step by step changes of raw data after each correction. In high Q, the changes of slope is noticeable. Fig. 3.3(b) shows comparison between normalized data after all correction and total independent scattering(TIS). We can see that TIS lines up with data in high Q region nicely. Finally Fig. 3.5(a) shows reduced structure function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor. The oscillating diffuse scattering is clear in high Q region.

Table 3.1 shows all the inputs used in the refinement. Now let's calculate Pair Distribution Function(PDF) using the structure function just we obtained.

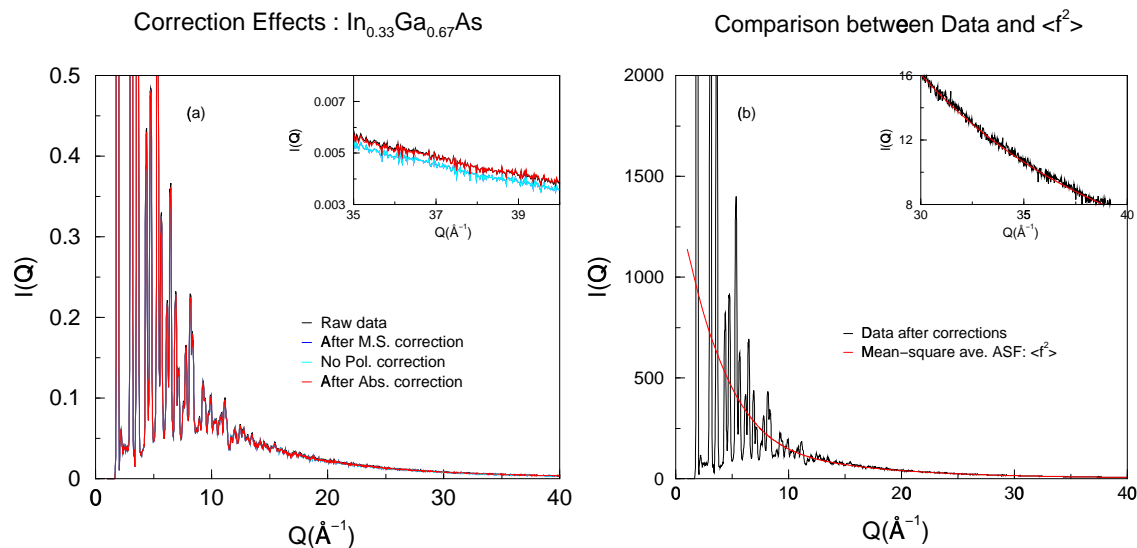


Figure 3.3: (a) Corrections on raw data of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy. In high Q region, after each correction, the change of slope is noticeable. (b) Comparison between normalized data after corrections and mean-square average atomic scattering factor, $\langle f^2 \rangle$. In high Q , those two line up quite nicely.

Data Corrections

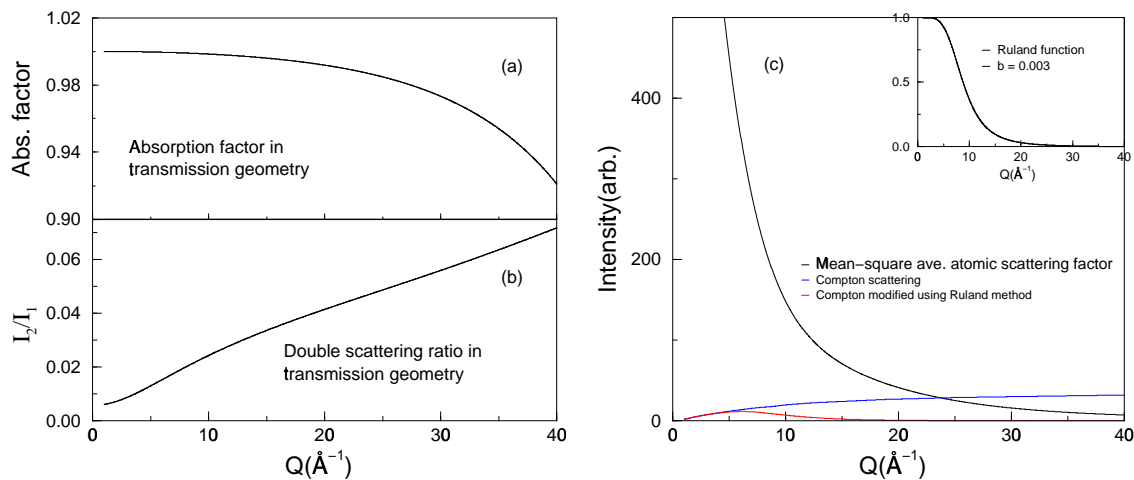


Figure 3.4: Data corrections in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy: (a) Absorption factor ($\mu t = 1.11$). Absorption effect becomes larger as Q increases. (b) Double scattering ratio. (c) Comparison between mean-square average atomic scattering factor, $\langle f^2 \rangle$, Compton, and modified Compton using the Ruland function. Inset shows the Ruland function for the integral width, $b=0.003$.

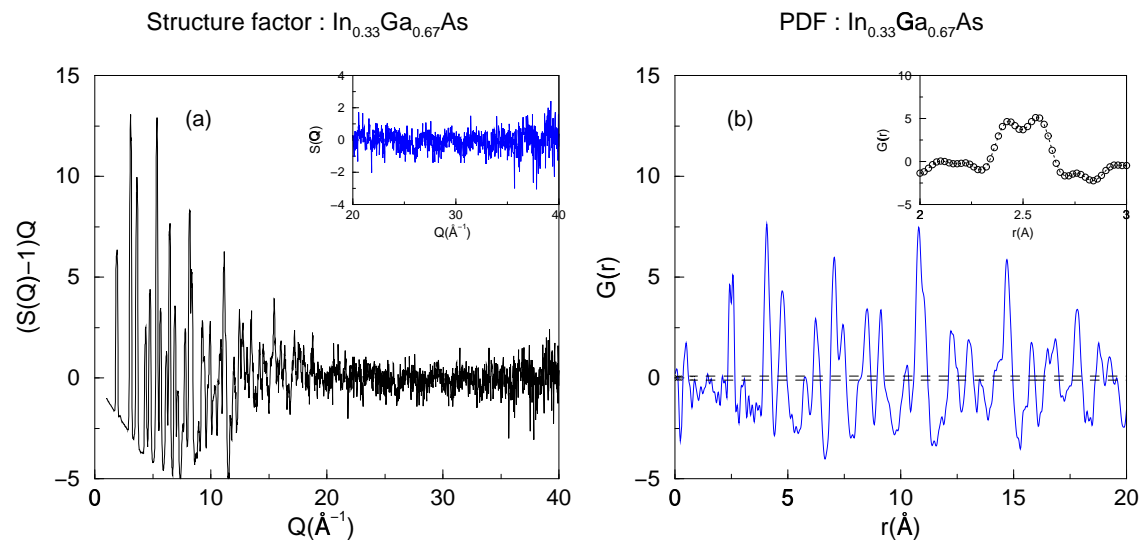


Figure 3.5: (a) Reduced Structure Function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor. The high Q data shows oscillating diffuse scattering. (b) Pair Distribution Function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor. The nearest-neighbor peak is split into a doublet corresponding to shorter Ga-As and longer In-As bonds

Table 3.1: Summary of structure function refinement

Input file	in33_tutorial.input
Background file	in33_bkg.input
Setup file	in33_tutorial.setup
Smoothing	No
Geometry	Transmission
Multiple Scattering Correction	Yes Compton in high Q region is discriminated
Polarization Correction	No
Absorption Correction	Yes
Normalization Constant	1920
Compton Correction	Remove mid-low Q Compton intensity using Ruland method. Integral width, $b = 0.003$

CALCULATE PAIR DISTRIBUTION FUNCTION(PDF): $G(r)$

- Read structure function: $(Q, S(Q), dQ, dS)$

ENTER FILE NAME: in33_tutorial.soq

- Enter Q_{max} at which to cut the data: 40

- Read structure function from $Q = 1$ to $Q = 40$

- Enter maximum range, r (Angstrom) for PDF calculation: 20

- Enter PDF step size(dr): 0.02

Calculating PDF up to $r_{\text{max}}=20$ with $dr=0.02$.

- SAVE PDF: (r, G(r), dr, dG) (y/n)? y
 - Enter file name to save data: in33_tutorial.pdf

 - Recalculate PDF (y/n)? n
-

Congratulations! You've made a PDF. Fig. 3.5(b) shows pair distribution function of $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy. The nearest-neighbor(NN) peak shows well resolved doublet which corresponds to shorter Ga-As and longer In-As bonds. This clearly shows the power of high real-space resolution PDF method to study the local structure of the alloy. It could be instructive to obtain the PDF using different Q_{max} to see how it affects the shape of NN peak. The dotted line shows \pm one standard deviation(σ) of PDF; the error propagated to PDF from the raw data. The ripples around sharp peaks are known as the termination ripple. It is caused by the limited Q value in Sine Fourier transform. And the noise peaks near to $r=0$ are caused by noises in the data.

Chapter 4

Using PDFgetX

This chapter will teach you how to **use** PDFgetX. For this purpose, first we'll give you overview of PDFgetX. And then explain how the program works; we will explain the structure function refinement process.

4.1 Overview of PDFgetX

Before learning the specific commands and procedures to control PDFgetX, it is best to understand how PDFgetX works in a very general way. This section documents the “broad overview” of PDFgetX while the following sections discuss the specifics at length.

The function of PDFgetX is to produce PDFs from x-ray powder diffraction data, whether from an sealed tube x-ray source or from a synchrotron source. Obviously, to begin the analysis one requires the *raw data*. The raw data, however, is in general too “raw” for analytical processing; not only does every facility has a different data file format, but the data file could contain multiple scans that ought to be averaged together. PDFgetX can help reduce the raw data into a more convenient format from which to start the analysis, but ultimately the responsibility for doing so will lie with the end user.

The *input file* from which PDFgetX can start the analysis contains the averaged intensities. Please be aware of the possible name confusion that can occur: the *raw data file* refers to the file that is directly output from the computer (like SPEC file) whereas the *input file* refers to a input data which will be used for the calculation of structure function, $S(Q)$. Some information regarding the experiment (such as the wavelength used) and some information regarding the specimen characteristics (such as the stoichiometry) are required in order to apply proper correction. The experiment and specimen information are contained in a *setup file* that is required at every step of the analysis.

With the *setup file* and the *input file*, the analysis can begin. The first stage of the analysis is to produce $S(Q)$ which is saved as the $S(Q)$ file. However, only the $S(Q)$ file is used in the second stage of the analysis to produce the PDF. Note that each stage of the analysis is independent of the others, so long as the necessary input files are present. That is, to recalculate the PDF of a specimen, you do not have to start the analysis from input file; instead, you can specify the correct $S(Q)$ file and the analysis will immediately create the PDF.

4.1.1 Launching PDFgetX

You can start Yorick from any directory by typing **yorick** at the prompt. At the Yorick prompt, type **pdfgetx** and Yorick should begin executing PDFgetX.

```
current directory: > yorick
Copyright (c) 1996. The Regents of the University of California.
All rights reserved. Yorick 1.4 ready. For help type 'help'
```

```
> pdfgetx
```

Then, this is what you should see:

```
Pair Distribution Function from the X-ray powder diffraction (PDFgetX 1.1)

0) Preliminary data reduction
1) Build a setup file
2) Background Substraction
3) Reduction of Structure Function: S(Q)
   Input file format : (Q, I, dQ, dI)
4) PDF calculation :
   Input file format : (Q, S(Q), dQ, dS)
P) Print, S) Save, U) Unzoom, L) Limits windows
Q) Quit

[0-4 hlqpu]
```

This is the main menu, and Section 4.1.3 will explain the menu in detail.

4.1.2 Exiting PDFgetX

To quit PDFgetX, type “q” at the main menu prompt. This will exit PDFgetX but leave you still in Yorick. Type “quit” to exit Yorick.

```
[0-4 hlqpu] q

Exiting (PDFgetX 1.1):
> quit
```

```
current directory: >
```

4.1.3 The Main Menu

```
Pair Distribution Function from the X-ray powder diffraction (PDFgetX 1.1)

0) Preliminary data reduction
1) Build a setup file
```

- 2) Background Substraction
- 3) Reduction of Structure Function: $S(Q)$
Input file format : (Q, I, dQ, dI)
- 4) PDF calculation :
Input file format : (Q, $S(Q)$, dQ, dS)
- P) Print, S) Save, U) Unzoom, L) Limits windows
- Q) Quit

[0-4 hlqpu]

The main menu provides you with several options. Simply type the number or letter of the option you want and hit “Enter”.

Option 0: This will access an interactive routine that can extract scan(s) from raw SPEC data and MCA data acquired from x-ray powder diffraction experiments. Correction for detector and monitor dead-time correction can be applied. You can also compare variables(e.g. elastic) in each scan and combine scans to get average value.

Option 1: This will access an interactive routine used to create a setup file describing the conditions of your experiment. The setup file is needed at several stages in the analysis; PDFgetX will prompt for the name of the setup file at the appropriate points.

Option 2: This will access an interactive routine that can subtract a background from a PDFgetX input file.

Option 3: This will access an interactive routine that applies most of the corrections to the data and produces $S(Q)$. Those corrections requiring feedback from the user will prompt for the necessary information.

Option 4: This will access an interactive routine that calculates the PDF from $S(Q)$.

Option P: When there is a Yorick window present on your screen, you may select this option to print the contents of the window. When prompted, specify the number of the window (this number should be visible in the title bar of the window. “Yorick 3” would indicate a window number of 3.). This option is only available from the main menu, which means that printing is not possible while doing the analysis.

Option S: This option save the specified window as postscript (PS) file or windows meta file (WMF) in your directory instead of sending figure to printer.

Option U: This UN-zooms a window. Yorick permits zooming on a data window (left-button zooms, right-button UN-zooms, and middle-button drags. You may also click on one axis only to zoom or unzoom that axis.) but sometimes it is difficult to make the window look the way it did before the zooming. In that case, select this option and, when prompted, specify the window number to unzoom the window. Unzooming only returns the window to the state it was in before the

mouse-based zooms; manually-specified axis limits (option L) supersede the effects of this option.

Option L: This allows you to manually specify the axis limits for a given window. Enter the window number when prompted.

4.2 Data Analysis Procedure in PDFgetX

The Fig. 4.1 shows flow chart of data analysis procedure in PDFgetX. As is shown, the analysis procedure is composed of four main blocks, “Preliminary Data Reduction”, “Build PDFgetX input file”, “Refine Structure Function”, and “PDF Calculation”. Since most processes in the main

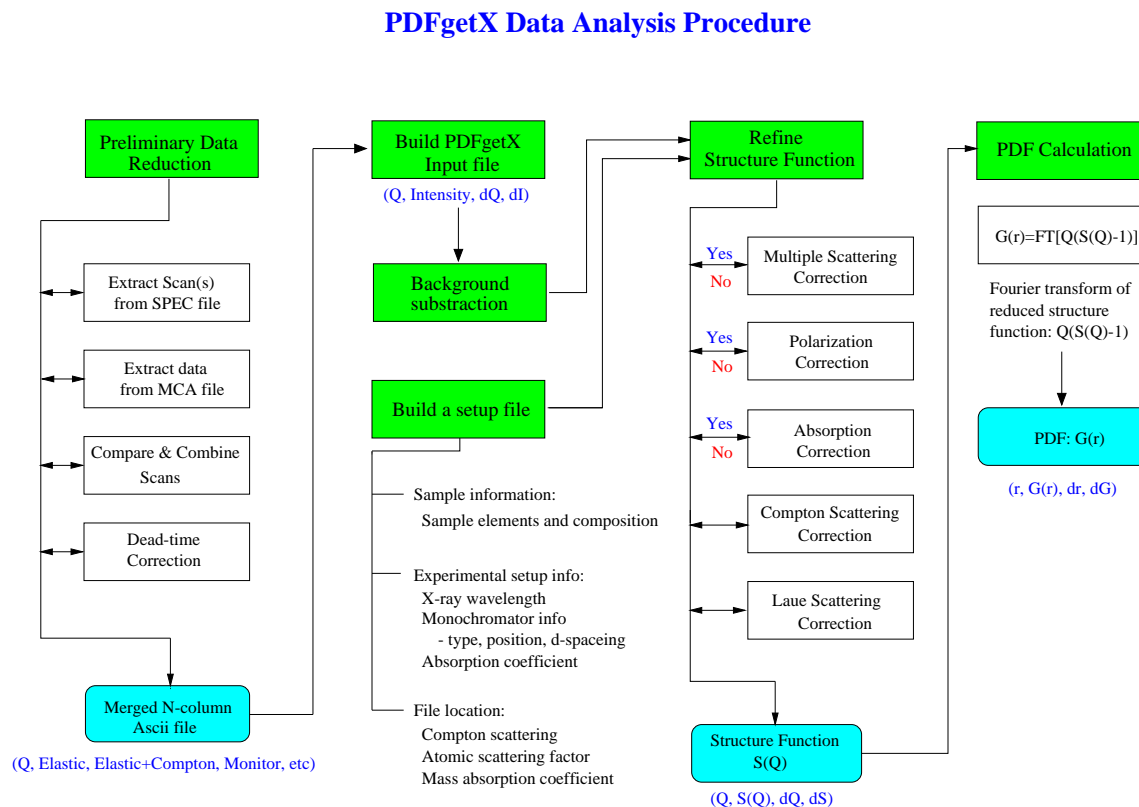


Figure 4.1: Data analysis procedure in PDFgetX

blocks are already explained in the tutorial chapter, we will not repeat the explanation for the whole process. Instead, we’ll give explanation for the refinement process in detail.

In order to refine the structure function, we need to apply five major corrections; dead-time, multiple scattering, polarization, absorption, and Compton scattering corrections. In these corrections, dead-time correction will be applied in the preliminary data reduction. All other corrections will be applied during structure function refinement. The Fig. 4.2 shows flow chart of structure

function refinement process. During the refinement process, the program asks input if it is necessary so please beware of the messages on the screen.

4.3 History File

During data reduction, PDFgetX records all the experimental information, parameters used for corrections, intermediate correction results to a binary file. The default file name is “_history.pdb”. You can look at the content of this file using Yorick command.

```
> o = openb("_history.pdb")
> show, o
37 non-record variables:
      R           date           nc
      Z           deg_pol        pf
absflag      dis_mono      polflag
      aft         dstran        pos_mono
      aw          elementsname   q
coh_data     f2ave          soq_process
      compo       fave2         rn_data
      compton     fpara        rn_data_cfcompt
compton_hiq  geometry         smflag
      data_cfbg   lambda          soq
      data_cfbgms mabscoeff      typ_mono
      data_cfbgmspf mscflag
      data_cfbgmspfabs mut
```

For example, you can simply check your experimental geometry by typing

```
> o.geometry
"r" /* "r" means reflection geometry */
```

For a complete description of history file see Appendix C.

4.4 Some Yorick Information

This section describes some miscellaneous information regarding the operation of Yorick, within the context of PDFgetX.

At any time, you may stop the execution of PDFgetX by entering control-C. You may restart PDFgetX at any time. If PDFgetX, for some reason, crashes, you can simply restart PDFgetX from within Yorick. It will not usually be necessary to exit Yorick before restarting PDFgetX. You may zoom any Yorick window using a mouse. The left mouse button zooms in, the right mouse button zooms out, and the middle button can be used to drag (if you have a two-button mouse, use both the right and left buttons at the same time). Click on an individual axis to affect only that axis.

Chapter 5

Data Corrections

The measured X-ray diffraction intensity may be expressed [10] by

$$I^{mea}(Q) = PA[N(I_{eu}^{coh} + I_{eu}^{inc} + I_{eu}^{mul})] \quad (5.1)$$

where P is the polarization factor, A the absorption factor, N normalization constant, and I_{eu}^{coh} , I_{eu}^{inc} , I_{eu}^{mul} are the coherent, incoherent(Compton), and multiple scattering intensities, respectively, in electron units. We can define the structure function($S(Q)$) in the following form.

$$S(Q) = [I_{eu}^{coh} - (\langle f^2 \rangle - \langle f \rangle^2)] / \langle f \rangle^2 \quad (5.2)$$

where $\langle f \rangle$ is the sample average scattering factor. Therefore to get a structure function, we have to do the following corrections [11] step by step on raw data.

- 0) Dead-time correction
- 1) Multiple scattering correction
- 2) Polarization correction
- 3) Absorption correction
- 4) Normalization
- 5) Compton scattering correction
- 6) Laue diffuse correction

5.1 Dead-Time Correction

In high-energy, high-intensity synchrotron x-ray diffraction experiments, the detector and monitor dead-time effect on the measured experimental data is rather lager. Therefore in these experiments, proper dead-time correction should be applied before applying standard corrections.

In the PDFgetX, the dead-time effect can be corrected by measuring detector dead-time and using the following Eq.(5.3).

$$N_{dtc} = \frac{N_m}{\left(1 - \frac{\tau * N_{tot}}{t_o}\right)} \quad (5.3)$$

where τ is dead-time of detector or monitor, N_{dtc} the dead time corrected counts, N_m the measured counts, N_{tot} the total counts of detector or monitor and t_o the measuring time for each data point. Or dead-time effect can be corrected using the pulser method. [9] A pulser-train from an electronic pulser of known frequency can be fed into the detector preamp. The measured counts in the pulser signal in an SCA window set on the pulser signal is then recorded for each data point. The data dead-time correction is then obtained by scaling the raw data by the ratio of the known pulser frequency and the measured pulser counts. The Fig. (5.1) shows a comparison of dead-time correction using these two methods.

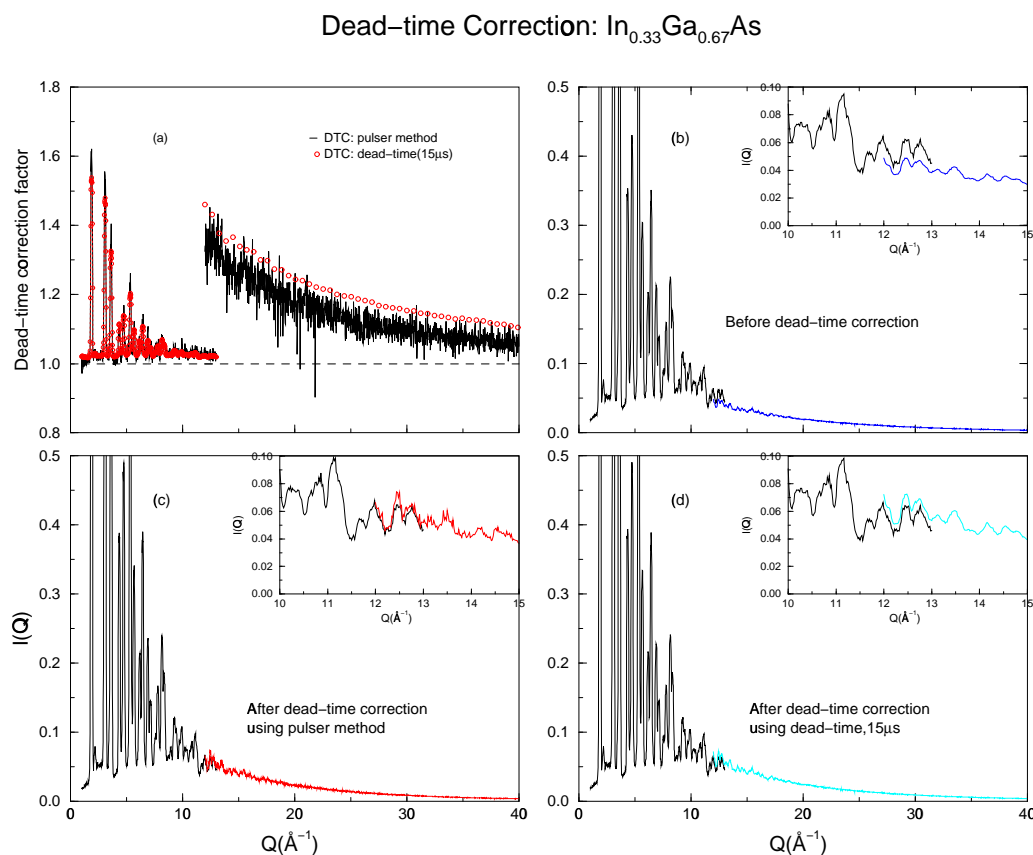


Figure 5.1: Dead-time correction in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$ semiconductor alloy: (a) Comparison between the dead-time correction using the pulser method and dead-time ($15 \mu\text{s}$) measurement. Comparison between low Q and high Q elastic scattering: (b) before dead-time correction. Low Q data don't overlap with the high Q data at $Q=12-13 \text{\AA}^{-1}$, (c) after dead-time correction using the pulser method, (d) after dead-time correction using the dead-time measurement. After dead-time correction in both cases, the low Q and high Q data overlaps with each other quite well.

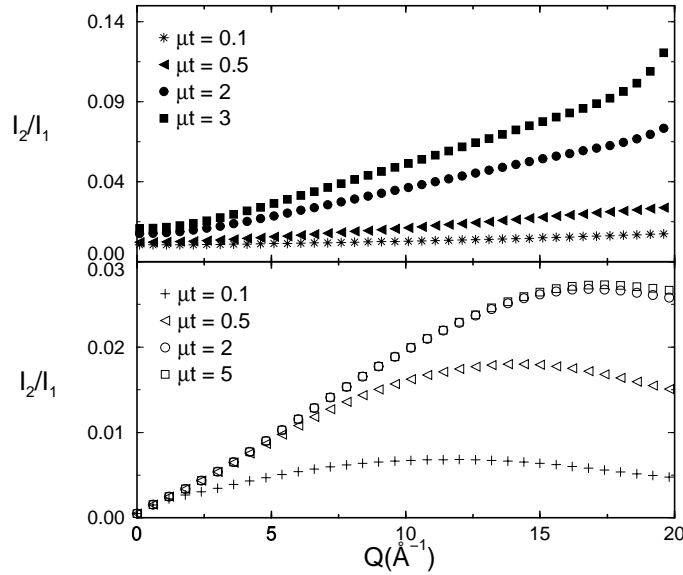


Figure 5.2: Double Scattering Ratio in Ni, upper panel: transmission geometry, lower panel: reflection geometry, experimental data includes Compton scattering, wavelength of x-ray: 0.7107\AA

5.2 Multiple Scattering Correction

We'll consider here only the double scattering process since it represents the major part of the multiple scattering. To calculate double scattering ratio, we followed the method suggested by Warren and Mozzi [12]. According to Warren and Mozzi, the double scattering ratio is given by Eq. (5.4)

$$\frac{I(2)}{I(1)} = \frac{B^2 Q_M(2\theta, a, b, \mu t)}{J(2\theta) \sum_{i=1} A_i \mu_i(m)} \quad (5.4)$$

where, $B = \sum_i Z_i^2$ and $A_i, \mu_i(m)$ are the atomic weights and mass absorption coefficients of the atoms. And $J(2\theta)$ is an approximate representation for independent scattering, $\sum_i f_i^2$ or $\sum_i [f_i^2 + i(M)]_i$ depending on whether the measurements include only the coherent scattering or both the coherent and incoherent scattering and given in Eq. (5.5).

$$J(2\theta) = B \left(a + \frac{1-a}{1+b \sin^2 \theta} \right) \quad (5.5)$$

where, a, b are parameters and can be obtained by fitting $J(2\theta)$ to either $\sum_i f_i^2$ or $\sum_i [f_i^2 + i(M)]_i$. Q_M is a complicated function depending on $Q, \mu t$, fitting parameters a and b and geometry. For details, refer to the papers by Dwiggins Jr. [13, 14]. As you can see in Fig. (5.2), the multiple scattering depends on absorption coefficient and geometry. In transmission geometry it becomes larger as Q increases. In reflection geometry, however, it increases up to maximum point and decrease a little bit after that. We can see that smaller the absorption coefficient, smaller double scattering ratio, in both cases [15].

5.3 Polarization Correction

Polarization factor P is given by the following Eqs. [10]:

(A) Using a filter

$$P = (1 + \cos^2 2\theta)/2 \quad (5.6)$$

(B) Using a crystal monochromator

$$P = (1 + x \cos^2 2\theta)/(1 + y) \quad (5.7)$$

where 2θ is the scattering angle, $x = \cos^2 2\alpha_c$ for a mosaic monochromator crystal or $x = \cos 2\alpha_c$ for a perfect monochromator crystal where $2\alpha_c$ is twice the Bragg angle of the monochromatic crystal. In Eq. (5.7) $x = y$ when the monochromator is located in the incident beam, and $y = 1$ when the monochromator is set in the diffracted beam.

In the case of the sealed tube X-ray diffractometer, incident beam is unpolarized, so the full polarization correction should be applied. However, the Synchrotron X-ray radiation (e.g. CHSS) is almost perpendicularly polarized to the detector plane therefore only partial polarization correction is necessary, usually less than 5%.

5.4 Absorption Correction

Absorption factor A is given by the following Eqs. :

(A) Flat plate reflection geometry

$$A_{refl} = [1 - \exp(-2\mu t/\sin \theta)]/2\mu \quad (5.8)$$

(B) Flat plate transmission geometry

$$A_{tran} = t \exp(-\mu t/\cos \theta)/\cos \theta \quad (5.9)$$

Fig. (5.3) shows the absorption factor as function of angle and absorption coefficient. In reflection geometry, if the absorption coefficient is large enough ($\mu t \geq 4$), there's almost no angle dependence of absorption factor. In transmission geometry, however, when absorption coefficient is around 1, the angle dependence is minimal.

5.5 Compton Scattering Correction

Compton scattering correction is very important and difficult in X-ray diffraction data analysis. Fig. (5.4) shows elastic and Compton scattering. We can see that Compton scattering becomes much larger than coherent scattering in high Q. So even the small error in Compton correction causes big error in determining coherent scattering. Therefore it's better to discriminate Compton scattering from elastic scattering than to correct it theoretically. Compton scattering can be removed experimentally, particularly at large scattering angles, using an analyzer crystal in the diffracted beam, or using a solid state detector with a very narrow energy window setup. When

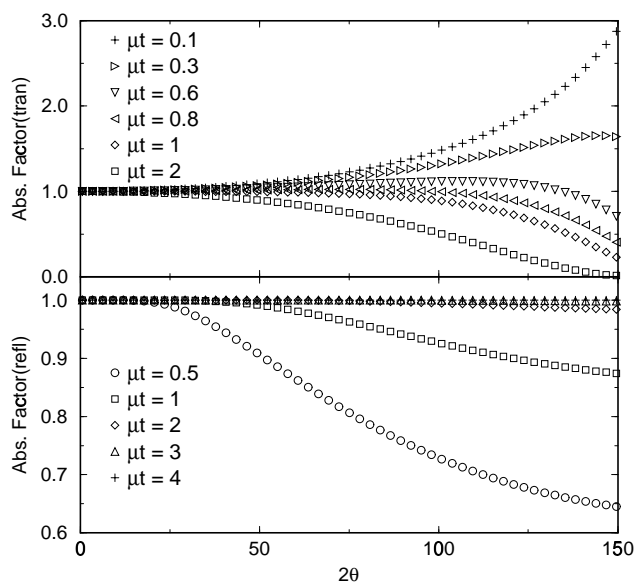


Figure 5.3: Absorption Factor in transmission geometry(upper panel) and in reflection geometry(lower panel)

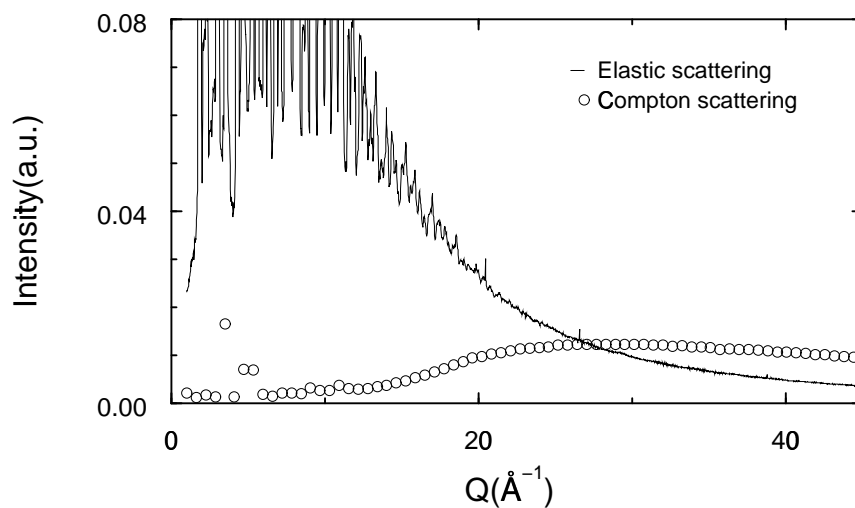


Figure 5.4: Comparison between Compton and elastic scattering intensities measured in $\text{In}_{0.33}\text{Ga}_{0.67}\text{As}$. Above $Q=30\text{\AA}^{-1}$, the Compton becomes larger than the elastic scattering.

the Compton scattering is not discriminated, we have to use theoretical Compton profiles to apply correction. In this case we have to take into account the ‘Breit-Dirac’ recoil factor, R [16]. Formerly, R was usually set equal to unity, which is still an acceptable approximation for elements of high atomic number. For light elements and for present-day high-precision diffractometric measurement, however, it is essential that R be numerically evaluated if the maximum amount of information inherent in the experimental data is to be extracted. According to Ergun [16], the following Eq. (5.10) should be applied when the *number of photons per unit area per unit time* is measured, as with counters.

$$R = \left(\frac{\lambda}{\lambda'}\right)^2 = \frac{1}{\left(1 + \frac{2h}{mc} \frac{\sin^2\theta}{\lambda}\right)^2} \quad (5.10)$$

where λ and λ' are the wavelength of incident and Compton scattered beam.

In this program, we use analytical Compton scattering formula [18] to calculate Compton profile. One can compare this results with theoretical Compton scattering data from the ‘International tables for crystallography C’ [17] and find the difference between these two are very small. Even when the Compton scattering in high Q is discriminated, the data still contains Compton in mid-low Q region. In order to remove the Compton in mid-low Q region, we use the method suggested by Ruland [19]. In this method, the Compton intensity in the data is smoothly attenuated with increasing Q as is shown in Fig. 3.4(c).

5.6 Normalization

The measured x-ray intensity is arbitrary value. The intensity should be normalized properly to get physical meaning. To determine normalization constant, N , we use high Q part of data. In this method, the normalization constant, N is defined in the following way.

$$N = \frac{\int_{Q_{mid}}^{Q_{max}} [\langle f^2 \rangle + I_{eu}^{inc}(Q)] dQ}{\int_{Q_{mid}}^{Q_{max}} [I^{cor}(Q)] dQ} \quad (5.11)$$

In Eq. 5.12, I^{cor} corresponds to the data after corrections for background, multiple scattering, polarization, and absorption. The theoretical atomic scattering factor is calculated using the analytical formula suggested by D. Waasmaier & A. Kirfel [2].

5.7 Laue Scattering Correction

Laue term is defined as $\langle f^2 \rangle - \langle f \rangle^2$. The Laue scattering occurs when there is no short-range order and the atoms are distributed randomly and it decreases monotonically with increasing scattering angle [12].

5.8 Pair Distribution Function

The atomic Pair Distribution Function(PDF), $G(r)$, can be obtained from powder diffraction data through a sine Fourier transform:

$$G(r) = 4\pi r [\rho(r) - \rho_o] = \frac{2}{\pi} \int_0^\infty Q [S(Q) - 1] \sin(Qr) dQ \quad (5.12)$$

where $\rho(r)$ is the microscopic pair density, ρ_o is the average number density, and Q is the magnitude of the scattering vector. The PDF is a measure of the probability of finding an atom at a distance r from another atom and gives information about both average and the local structure of materials. For more about PDF analysis method, look up the papers by Egami, Toby and Billinge [20, 21, 22].

5.9 Error Propagation

In most diffraction experiments, the measured diffraction intensities are subject to statistical fluctuations. It is known that the detection process is well represented by the Poisson distribution. According to Poisson distribution, the standard deviation of statistical fluctuations is given by \sqrt{N} for the measured N counts. This error in measured intensities will be propagated to the error in a function (e.g. PDF) determined from these measured intensities. The estimated error in PDF will be used to test the quality of modeling. In general, an error in function $f(x_1, \dots, x_n)$ can be calculated by the following Eq.

$$\delta f = \sqrt{\left(\frac{\partial f}{\partial x_1} \delta x_1\right)^2 + \dots + \left(\frac{\partial f}{\partial x_n} \delta x_n\right)^2} \quad (5.13)$$

The error in the Structure function, $S(Q)$, is estimated by propagating error in the measured intensities through each correction step. For the calculation of error in $G(r)$, the following Eq. is used [21].

$$\sigma_{G(r)} = \frac{2}{\pi} \sum_k \sigma_{S(Q_k)} Q_k \Delta Q_k \sin Q_k r \quad (5.14)$$

Appendix A

SPEC file format

In this appendix, the SPEC file format used in the data analysis is presented. The following shows sample SPEC file.

```
#F in33_tutorial.spec
#S 1  ascan  pmQ 1 13  600 1
#D Fri Sep 18 16:13:55 1998
#T 1  (Seconds)
#L pmQ  ereal  elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC
1  2.07 1.967 75931 2.11758 556914 396634 394.395 416 2866 233 31718 606
1.02  2.07 1.968 75934 2.11849 558523 396548 394.159 432 3000 217 31791 610
1.04  2.06 1.962 75936 2.10892 555188 394768 392.324 414 3030 253 31569 591
1.06  2.07 1.969 75939 2.11886 558933 396616 394.023 417 3138 240 31776 647
1.08  2.07 1.977 75942 2.1189 559126 396636 393.919 419 2923 246 31839 639

#S 2  ascan  pmQ 1 13  600 1
#D Fri Sep 18 16:40:55 1998
#L pmQ  ereal  elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC
1  2.07 1.999 77606 2.11876 490517 396566 353.616 418 2397 186 27129 533
1.02  2.069 1.997 77609 2.11807 490872 396438 353.319 415 2486 194 27167 558
1.04  2.07 1.989 77612 2.11884 489377 396583 353.419 416 2672 177 27045 536
1.06  2.07 1.996 77614 2.11884 492200 396585 353.414 428 2551 195 27218 551
1.08  2.06 1.989 77617 2.10866 488500 394682 351.707 419 2458 199 26993 550

#S 3  ascan  pmQ 12 40  1400 1
#L pmQ  ereal  elive Epoch Seconds IC1 IC3 I_CESR PULSER TOTAL COMPTON IC2 ELASTIC
12  2.07 1.115 88417 2.11861 633382 399154 451.721 317 44519 7243 634757 29557
12.02  2.06 1.137 88419 2.1088 628412 397322 449.505 313 43218 7290 630504 28163
12.04  2.07 1.166 88422 2.11877 625842 399215 451.612 327 42631 7336 630216 27395
12.06  2.07 1.185 88425 2.11884 624286 399227 451.478 299 41732 7166 629057 26469
```

As is shown in the sample SPEC file, all the comments and characters start with # mark. To specify scan number #S is used and for the scan header, #L and so on. To separate scans blank line is used. Except these things the SPEC file is the same as the multi-column ascii file.

Appendix B

Description of the history file

In this appendix, the content of the history file (“_history.pdb”) is described. The history file contains all the experimental information, parameters used for corrections, intermediate correction results.

Parameter	Description
elementsname	Name of sample elements ; Ex. [“In”, “Ga”, “As”]
Z	Atomic number of sample elements ; Ex. [49,31,33]
compo	Composition of sample ; Ex. [0.33,0.67,1]
aw	Atomic weight of sample elements ; Ex. [114.82, 69.72, 74.92]
mabscoeff	Mass absorption coefficient of sample elements at wavelength λ Ex. [6.36, 1.88, 2.23] for $\lambda=0.2078 \text{ \AA}$
lambda	Wavelength of incident X-ray Ex. [0.2078] for E = 60 KeV X-ray radiation
mut(μt)	Absorption coefficient*sample thickness ;
geometry	Configuration of diffractometer r=Reflection geometry t=Transmission geometry
mscflag	0=No multiple scattering correction 1=Multiple scattering correction
mscParam	mscflag = 1: fpara, dsrefl, dstran fpara=parameters used to approximate scattering in function J(Warren & Mozzi, 1996); dsrefl=double scattering ratio, I2/I1 in reflection geometry dstran=double scattering ratio, I2/I1 in transmission geometry
polflag	0=No polarization correction 1=Polarization correction
polParam	polflag = 1: pos_mono, typ_mono, dis_mono, deg_pol, pf pos_mon : Position of Monochromator inc=Primary beam Monochromator, ref=Diffracted beam Monochromator typ_mono : Type of Monochromator pc=Perfect crystal monochromator mc=Perfect crystal monochromator dis_mono : Distance between crystal plane

	Ex. Graphite(002), $d = 3.3570 \text{ \AA}$, Si(111), $d = 3.135 \text{ \AA}$
	deg_pol : Degree of polarization of incident X-ray beam
	Synchrotron source $\Rightarrow 1$, X-ray tube $\Rightarrow 0$
	pf = polarization factor
absflag	0=No absorption correction 1=Absorption correction
absParam	absflag = 1: afr, aft, mut(μt) aft=absorption factor in transmission geometry afr=absorption factor in reflection geometry
smflag	0=No smoothing of data 1=Smoothing using the Savitzky-Golay filter
smoothParam	smflag = 1: q_s, num_ps q_s = starting point of smoothing num_ps = number of point used in Savitzky-Golay filter
compton_hiq	Y= contain Compton scattering in high Q region of data N= Compton in high Q is discriminated; no Compton in high Q
comptonParam	compton_hiq = N : integral_width, wf integral_width = control parameter for a width of window function wf = Ruland window function
nc	normalization constant
R	Breit-Dirac Recoil Factor
soq_process	0=S(Q) reduction process incomplete, no S(Q) obtained 1=S(Q) reduction process completed, S(Q) obtained
date	Date of refinement
f2ave	$\langle f^2 \rangle$, sample average of square of scattering factor
fave2	$\langle f \rangle^2$, square of sample average of scattering factor
compton	Theoretical Compton scattering
q	$Q(4\pi \sin(\theta)/\lambda)$ array
data_cfbg	Data after background correction
data_cfbgms	Data after multiple scattering correction
data_cfbgmspf	Data after multiple scattering & polarization correction
data_cfbgmspfabs	Data after multiple scattering, polarization & absorption correction
rn_data	Normalized data after all necessary corrections
rn_data_cfcompt	Data corrected for Compton scattering after normalization
coh_data	Coherent scattering data
soq	Structure factor

Appendix C

MCA file format

In this appendix, the MCA file format used in the data analysis is presented. The MCA file format used in this manual is two column ascii file as shown in the following Fig. C.1. The correspond MCA spectrum is shown in Fig. 3.2. The first column corresponds to the MCA channel number which starts from 0 to 1023 in this case (so total MCA channel # = 1024). And the second column is the intensity detected at each channel. Each 1024 lines corresponds to one Q value and separated by the blank line. Therefore to convert MCA file to N-column ascii file, the following information is needed; total MCA channel number and the corresponding “Q” column. You can get the “Q” column from the corresponding scan (saved in SPEC file) or you can generate “Q” column if it has constant step. In this case you need Q_{min} of your scan, total number of points in your scan, and Q step.

MCA channel		Intensity	
0		0	Q(i)
1		0	
2		0	
.		.	
.		.	
600		23	
601		34	
602		15	
603		65	
604		34	
605		22	
.		.	
.		.	
1020		0	
1021		0	
1023		0	
	blank line		
0		0	Q(i+1)
1		0	
2		0	
.		.	
.		.	
600		23	
601		34	
602		15	
603		65	
604		34	
605		22	
.		.	
.		.	
1020		0	
1021		0	
1023		0	
	blank line		
0		0	Q(i+2)
1		0	
2		0	
.		.	
.		.	
600		23	
601		34	
602		15	
603		65	
604		34	
605		22	
.		.	
.		.	
1020		0	
1021		0	
1023		0	

Figure C.1: MCA file format

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