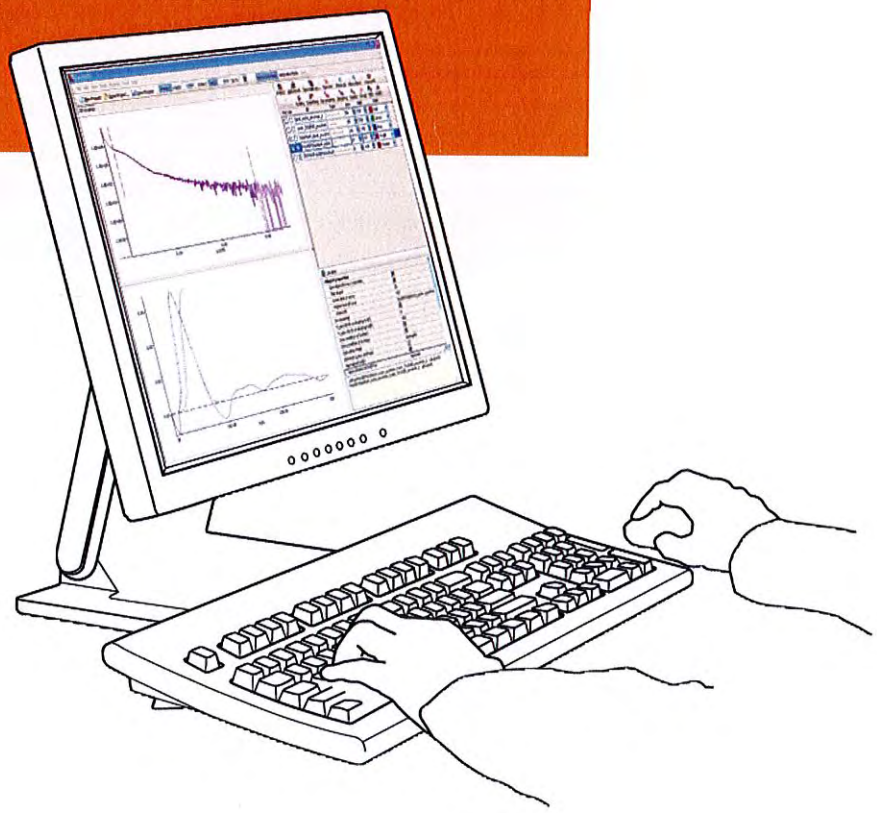
PANalytical

EasySAXS

Quick Start Guide



PANalytical

EasySAXS

Quick Start Guide

EDITION NOTICE: 4022 339 16083, March 2014
This is the original publication of Edition 3 of this document, to be used with EasySAXS version 2.1 or higher.

ACKNOWLEDGMENTS

All registered and unregistered trademarks, domain names and copyrights herein are the property of their respective owners.

DISCLAIMER

The information provided herein is supplied under a non-exclusive license authorizing its use solely for and in conjunction with PANalytical's products. Although diligent care has been used to ensure that the information is accurate, nothing contained herein can be construed to imply any representation or warranty as to the accuracy, currency or completeness of this information.

COPYRIGHT NOTICE

© 2014, PANalytical B.V., Almelo, The Netherlands

PANalytical B.V.
Lelyweg 1
7602 EA, Almelo
The Netherlands
Tel: +31 546 534444
Fax: +31 546 534598
Internet www.PANalytical.com

Contents

Chapter 1	Introduction
Chapter 2	Using the Automatic Mode
Chapter 3	Using the Interactive Mode
Appendix A	Classification of Scattering Curves into Different Types
Appendix B	Template Files
Appendix C	Project Files
Appendix D	Example Measurement Files
Appendix E	Configuration Files

PANalytical

Chapter 1

Introduction

1.1 INTRODUCTION ..... 1 - 3
1.1.1 Examples ..... 1 - 3
1.1.2 Template files ..... 1 - 3
1.2 ABOUT EASYSAXS ..... 1 - 4
1.3 CONTENTS OF THE QUICK START GUIDE ..... 1 - 6
1.4 TERMS AND CONVENTIONS USED ..... 1 - 7
1.4.1 Terms Used to Denote an Action ..... 1 - 7
1.4.2 Instructions and Descriptive Text ..... 1 - 7
1.4.3 Pushbuttons and Fields ..... 1 - 8
1.4.4 Menu Items and Keys ..... 1 - 8
1.5 EASYSAXS MAIN SCREEN ..... 1 - 9
1.5.1 Main Screen in the Interactive Mode ..... 1 - 9
1.5.2 Main Screen in the Automatic Mode ..... 1 - 12

EasySAXS Quick Start Guide

1.1 INTRODUCTION

This Quick Start Guide is intended to help you to use the EasySAXS software quickly and efficiently. It introduces the basic usage of the software, but it does not show or explain all the possibilities of the software.

For detailed information about the EasySAXS software, refer to the on-line Help.

NOTE: File locations are given for **Windows 7**. Other operating systems are not officially supported.

NOTE: There may be differences between the example screens given in this Quick Start Guide and what you see on your screen. In all cases, where there is a difference, follow what you see on your screen.

1.1.1 Examples

The examples show you how to start and use the software to perform fairly simple tasks.

In order to follow these examples you must start with Chapter 2 and then follow the examples through the rest of the chapters.

To do these tasks we will use the **example measurement files (*.xrdml)** that were supplied with EasySAXS (for details see Appendix D). After installation and first start of the program, they are located at:

C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Example Measurement Files.

1.1.2 Template files

Furthermore, we will use the **template files (*.saxsprt)** that were supplied with EasySAXS (for details see Appendix B). After installation and first start of the program, these are located at:

C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Template Files

1.2 ABOUT EASYSAXS

EasySAXS is an advanced, user-friendly software package for the analysis of small-angle X-ray scattering (SAXS) data. It provides information on nanoscale structures and dimensions, nanoparticle shapes and surface areas. EasySAXS offers easily accessible advanced algorithms, a complete data analysis toolbox, automation options and reporting.

The software has a complete toolbox that includes data reduction, Guinier and Porod analyses, least-squares fitting and model simulations.

All parameter settings are accessible in the graphical user interface.

EasySAXS supports data files in the PANalytical XRDML format, as well as ASCII files of background-corrected data.

EasySAXS was developed in cooperation with the EMBL (Hamburg, Germany). It is partly based on the original ATSAS package written by the EMBL ATSAS team. Information about this package can be found in the following reference:

P. V. Konarev, M. V. Petoukhov, V. V. Volkov and D. I. Svergun
ATSAS 2.1, a program package for small-angle scattering data analysis
J. Appl. Cryst. (2006). 39, 277–286

1.2.1 Automatic and Interactive modes

EasySAXS can be run in Interactive mode or in Automatic mode:

The **Interactive mode** gives advanced users and experts easy access to a wide variety of data treatment and analysis options via the respective toolbars. All parameter settings for these operations can be controlled in the Object Inspection Pane. Each treatment or analysis operation results in the creation of a new data file (in the following referred to as "object"). Each object is assigned a specific ID and Type descriptor. In the Object List Pane all objects are listed and conveniently accessible to be selected for plotting or further analysis. Two zoomable graphics panes allow to view and compare scattering curves, and to display analysis results. Objects may also be exported as ASCII files. All the details of the various evaluations made can be saved in a project file. The interactive mode can also be used to create analysis templates that can then be applied for automated particle size distribution analysis in the Automatic mode.

The user interface of the Interactive mode is explained in section 1.5.1. Usage instructions are given in chapter 3.

The **Automatic mode** has been developed for less experienced users who want to perform automated nanoparticle or pore size distribution analysis without having to control the sequence of calculations and the various analysis parameters. This can be accomplished with the help of the Automatic Mode Parameters window, by selecting and applying a suitable analysis template, in which the sequence of optimized analysis steps is pre-programmed. For different types of samples specific analysis templates are available.

In the automatic mode the software can be configured to analyze single or a series of already measured experimental data sets, and it can also function in a standby mode to automatically initiate evaluation of incoming data during a batch measurement. For each analyzed data file a size distribution analysis report is created and the size distribution curve is exported as an ASCII file. In contrast to the Interactive mode, the Automatic mode is restricted to the size distribution analysis functionality.

The user interface of the Automatic mode is explained in section 1.5.2. Usage instructions are given in chapter 2.

1.3 CONTENTS OF THE QUICK START GUIDE

Chapter 2: **Using the Automatic Mode**

This Chapter explains step-by-step how to analyze experimental SAXS data in the Automatic mode, to determine the size distribution of nanoparticles (or pores). Firstly we will see how to perform analysis of single and multiple data files and explain the contents of the analysis report. Then we will learn how to perform the same type of analysis in the Standby Mode.

Chapter 3: **Using the Interactive Mode**

In this Chapter we explain step-by-step how to determine the particle/pore size distribution from experimental SAXS data in the Interactive mode. We will save the sequence of analysis in a project file (*.saxsprj) and also create a template file (*.saxsprt) that can be then used for automatic analysis.

Appendix A: **Classification of Scattering Curves into Different Types**

This Appendix defines various types of scattering curves and gives recommendations for analysis.

Appendix B: **Template Files**

This Appendix gives information about the template files supplied with EasySAXS.

Appendix C: **Project Files**

This Appendix gives information about the project files supplied with EasySAXS.

Appendix D: **Example Measurement Files**

This Appendix gives information about the example measurement files supplied with EasySAXS.

Appendix E: **Configuration Files**

This Appendix gives information about the configuration files supplied with EasySAXS.

1.4 TERMS AND CONVENTIONS USED

In this section, we describe the terms and conventions used in this Quick Start Guide and how they relate to the user interface.

1.4.1 Terms Used to Denote an Action

In this Guide there are several terms that indicate an action.

Check (✔)	Also referred to as a tick mark.
Click	Press the mouse button and quickly release it.
Double-click	Press the mouse button twice (quickly) on an icon, item, file or program.
Drag	Press and hold down the mouse button and move the pointer to define an area or move an object
Enter	Type in information. This can be either text or numerical data.
Press	A key on the keyboard, or a push-button in a window.
Right-click	Press the right mouse button and quickly release it.
Select	Move the mouse cursor to the option you want and click the left mouse button.
Tick (✔)	Also referred to as a check mark.
Toggle	Switch between parameters or states (for example: On-Off-On).

1.4.2 Instructions and Descriptive Text

An instruction is preceded by a bullet "•". Any descriptive text relating to an instruction is given directly after the instruction.

Generally, screen captures are preceded by an instruction and intend to reproduce what you will see on your screen. However, if there are any differences, follow what you see on your screen.

1.4.3 Pushbuttons and Fields

All pushbuttons on a window are normally shown as the actual pushbutton (for example: [OK]), they may occasionally be described in bold text (for example: **Apply** or **Cancel**).

All fields are shown between "quotation marks".

1.4.4 Menu Items and Keys

All menu items are printed in italics, for example: *File - Open* etc.

All keys are shown bold in an italic font. For example: ***Enter, Ctrl, Alt, Del*** etc.

1.5 EASYSAXS MAIN SCREEN

NOTE: The User Interfaces are described in detail in the on-line Help. To access the Help, select *Help - Contents*, or press ***F1***. When you press ***F1*** you will normally see the Help section related to the current screen or activity.

1.5.1 Main Screen in the Interactive Mode

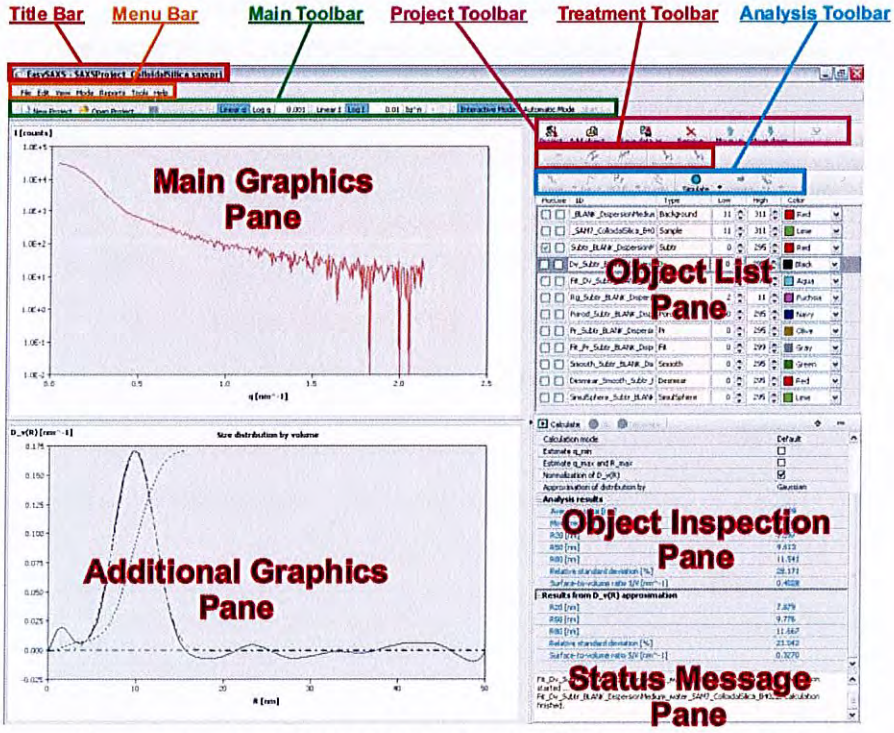


Figure 1.1 : EasySAXS Screen Appearance in Interactive Mode

In the Interactive mode the User Interface includes the following elements:

1.5.1.1 Title Bar

Displays the names of the analysis software and the current project file.

1.5.1.2 Menu Bar

The menu bar contains various menus for handling project files, editing text, setting viewing options, switching between automatic and interactive modes, viewing the analysis report, setting preferences, and accessing help.

1.5.1.3 Main Toolbar

A project file or a template file can be opened in this bar.

Various display options may be set for the Main Graphics pane.

You can also switch between automatic and interactive modes, and start an automated analysis.

1.5.1.4 Main Graphics Pane

Objects that were selected in the object list pane are displayed in this pane.

1.5.1.5 Additional Graphics Pane

Here the results from various analysis procedures (such as Dv(R), PDDF, Guinier, Porod, Smoothen) can be displayed.

1.5.1.6 Project Toolbar

This toolbar enables you to display the properties of a project or template file, to add objects to a project, export data, remove objects, or move objects up and down in the Object list pane.

1.5.1.7 Treatment Toolbar

The Object Treatment Toolbar is used for primary data handling and for basic data treatment, such as background correction, smoothing, and smearing / desmearing operations.

1.5.1.8 Analysis Toolbar

This toolbar contains the major functionalities for SAXS data analysis, such as particle size distribution determination, Guinier and PDDF analyses, Porod analysis as well as simulation and modeling tools.

1.5.1.9 Object List Pane

All the loaded objects as well as objects resulting from data treatment or analysis are listed in this pane. There is a **Plot** check box for each object enabling its display in the Main Graphics Pane. There are also **Use** check boxes that enable an object to be selected for subsequent operation.

1.5.1.10 Object Inspection Pane

Here, the properties of an object highlighted within the object list pane can be inspected. All the settings for the data analysis as well as the analysis results are listed here. The "Calculate" button at the top of this pane allows calculations to be performed on the selected object(s).

1.5.1.11 Status Message Pane

Status messages and warnings are displayed in this window during the data analysis.

1.5.2 Main Screen in the Automatic Mode

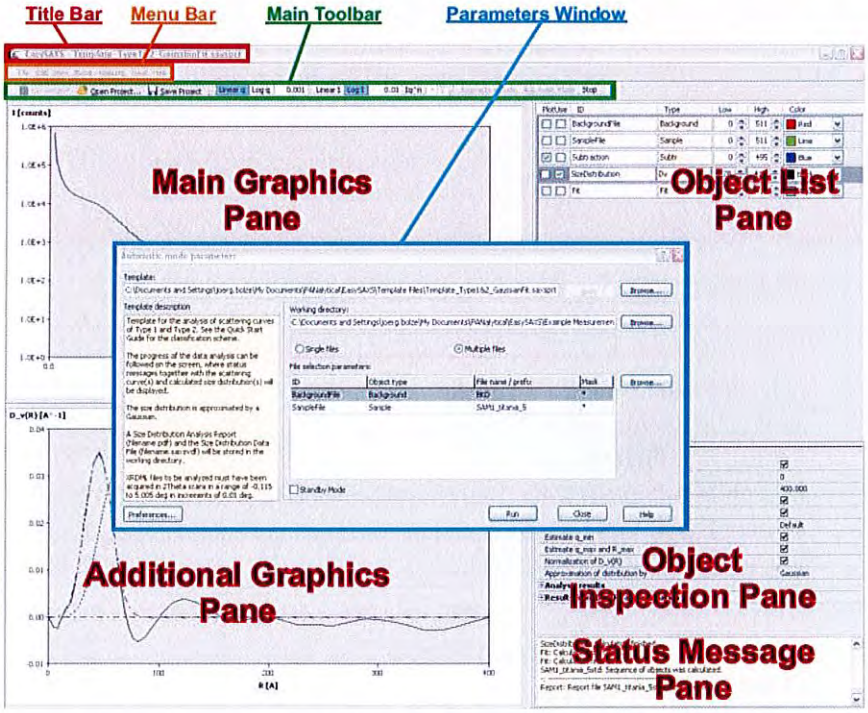


Figure 1.2 : EasySAXS Screen Appearance in Automatic Mode

In the Automatic mode the user interface contains the same elements as in the Interactive mode, with the exception that the Project, Treatment and Analysis toolbars are not visible.

In addition, a Parameters window is used to specify the relevant parameters for automated particle size distribution analysis.

PANalytical

Chapter 2

Using the Automatic Mode

2.1 INTRODUCTION ..... 2 - 3

2.2 AUTOMATIC ANALYSIS OF SINGLE DATA FILES ..... 2 - 3

2.2.1 Selecting and Loading a Template ..... 2 - 4

2.2.2 Setting up the Automatic Analysis ..... 2 - 5

2.2.3 Setting your Preferences ..... 2 - 8

2.2.4 Executing Automatic Data Analysis on Single Data Files ..... 2 - 10

2.2.5 Closing EasySAXS ..... 2 - 11

2.2.6 Inspecting the Analysis Results ..... 2 - 11

2.3 AUTOMATIC ANALYSIS OF MULTIPLE DATA FILES . 2 - 16

2.3.1 Setting Up and Executing an Automatic Data Analysis ..... 2 - 16

2.3.2 Closing EasySAXS ..... 2 - 20

2.3.3 Inspecting the Analysis Results ..... 2 - 21

2.4 AUTOMATIC STANDBY ANALYSIS OF MULTIPLE FILES ..... 2 - 25

2.4.1 Setting Up Data Collector and Initiating SAXS Measurements ..... 2 - 25

2.4.2 Selecting and Loading a Template that Runs in Standby Mode and Applies a Gaussian Fitting on the Size Distribution ..... 2 - 26

2.4.3 Setting Up and Executing the Automatic Data Analysis ..... 2 - 27

2.4.4 Inspecting the Analysis Results ..... 2 - 29

2.1 INTRODUCTION

In this Chapter we will explain step-by-step how to use the Automatic mode of EasySAXS to determine the nanoparticle size distribution from measured SAXS data.

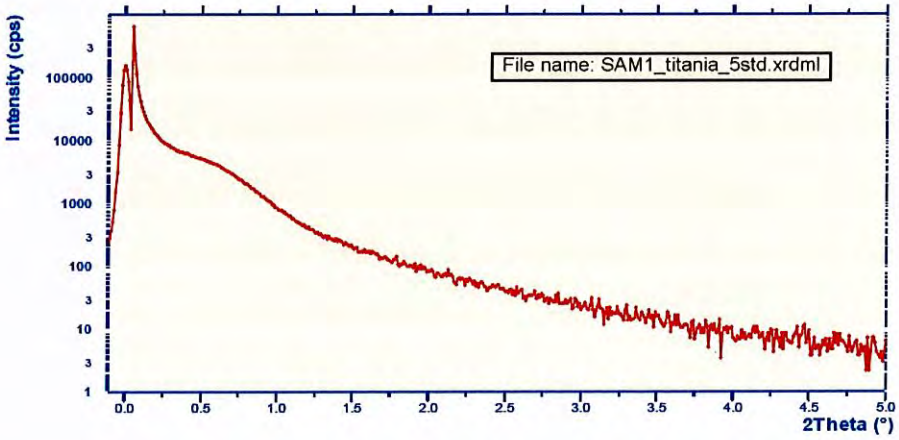
We will see how to perform analysis of single data files (Section 2.2) and multiple data files (Section 2.3), and how we can approximate the size distribution by a Gaussian or log-normal function.

In Section 2.4 we will explain how to use the standby mode.

2.2 AUTOMATIC ANALYSIS OF SINGLE DATA FILES

In this section we will show you how to automatically analyze the particle size distribution from single data files.

We are going to analyze the measurement file "SAM1_titania_5std.xrdml" from a nano-titania powder sample. When plotting this data file in Data Viewer we see a scattering curve with characteristic features that are clearly attributed to a "Type 1 scattering curve" according to the classification scheme detailed in Appendix A.



2.2.1 Selecting and Loading a Template

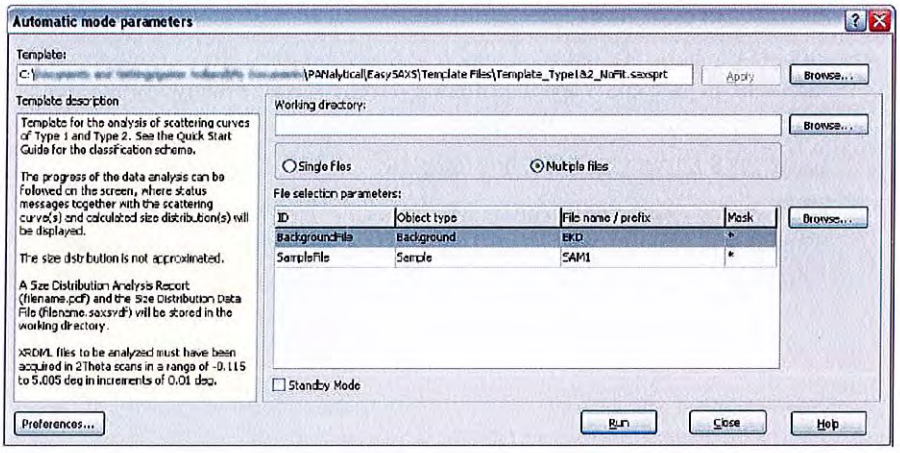
In order to analyze this type of data file we have to choose an analysis template (*.saxsprt) that was made for **Type 1 scattering curves**.

- In Windows Explorer, browse to "*C:\Users\ <currentuser>\Documents\PANalytical\EasySAXS\Template Files*".
- Double-click on "Template_Type1&2_NoFit.saxsprt". This template was made for Type 1 and Type 2 scattering curves; the size distribution curve is not approximated by a function.

  EasySAXS is launched with this template.
- Click on the Start button in the Main toolbar. The "Automatic mode parameters" window will open. This window already has some default entries.

2.2.2 Setting up the Automatic Analysis

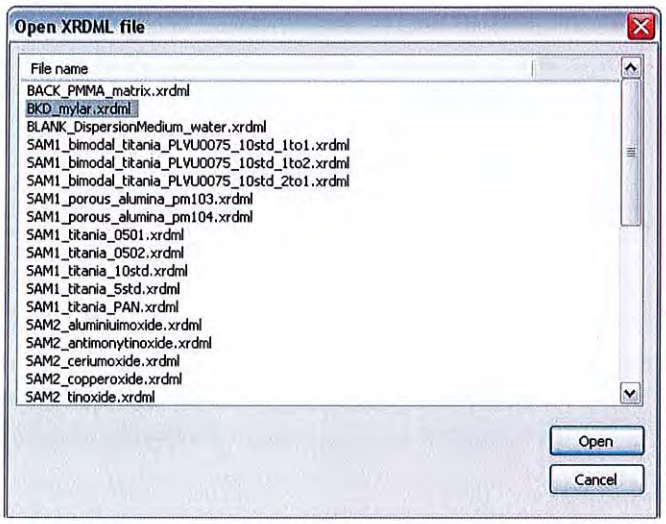
In the template description field you can read the important information about the applicability of the selected template, to confirm that you have selected the appropriate template.



- Enter the working directory. This is the directory in which the to be analyzed data file and its corresponding background file are located. You can select the required directory by clicking on the [Browse...] button next to this text box. In this example "*C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Example Measurement Files*".
- Because we are only going to analyze a single sample file, select the "Single files" option button.

  Now select the sample file and the corresponding background file to be analyzed as follows:
- Under "File selection parameters" select the background file by clicking on the top line ("BackgroundFile"). Then click on [Browse...] next to this text box.

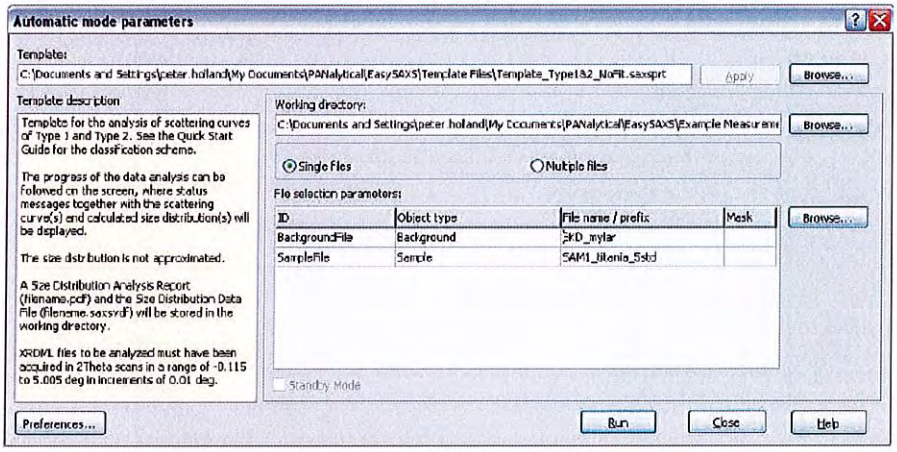
A new window which shows all of the xrdml files located in the working directory opens.



- Select the background file, in our example "BKD_mylar.xrdml", and click [Open]. The file name of the selected background will then be displayed in the column "File name/prefix".

  To select the measurement file of the sample to be analyzed, under "File selection parameters" click on the second line ("SampleFile"). Then click on [Browse...] next to this text box. Select the sample file, in our example "SAM1_titania_5std.xrdml", and click [Open]. The file name of the selected background will then be displayed in the column "File name/ prefix".

The figure below shows the "Automatic mode parameters" window at this point.



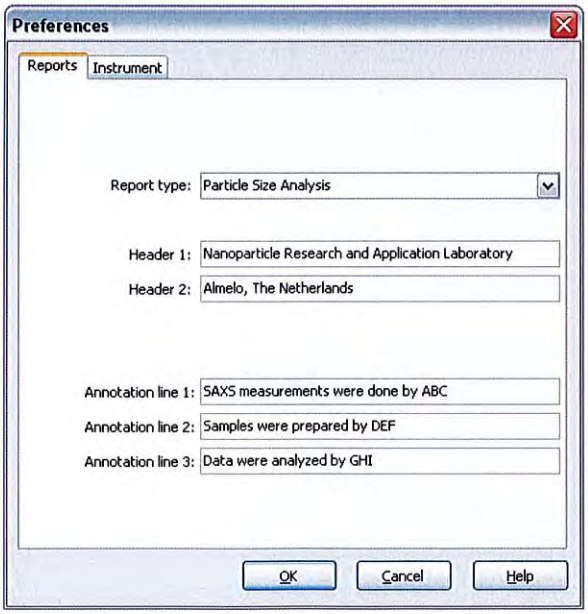
2.2.3 Setting your Preferences

Before running the automatic analysis, you should set your preferences.

- Click [Preferences...] . The "Preferences" window with two tabs opens.
- In the "Reports" tab you can set your preferences for the analysis report that is to be created:

  In the "Report type" text box open the drop-down list (▾) and choose between the options "Particle Size Analysis" and "Particle Size Analysis (compact)". The first one is a two-page report, whereas the second one is a more compact, 1-page report.

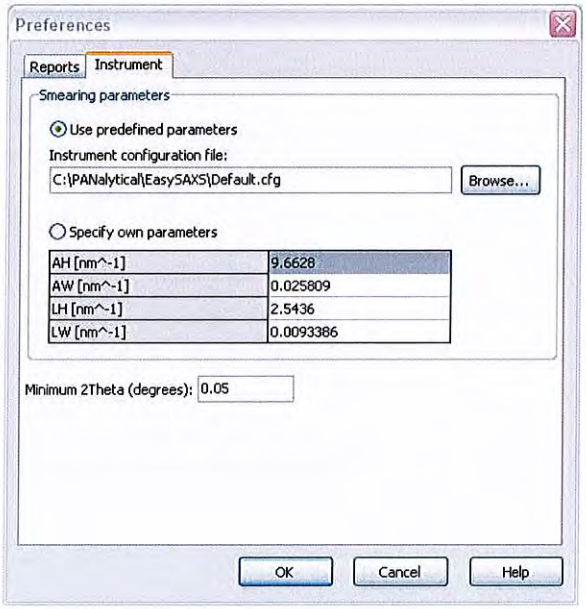
  The report file can be customized by specifying up to two lines to be displayed in the header, and up to three lines for annotations at the end of the report (not in case of the compact report).



- Next select the "Instrument" tab.

  The "Instrument configuration file" text box must contain the configuration file (*.cfg) that corresponds to the experimental setup with which the files to be analyzed were measured. You can do this by selecting "Use predefined parameters" and then clicking on [Browse...]. These files are delivered with EasySAXS and can be found in the directory *C:\PANalytical\EasySAXS*. The choice of the configuration file should be made according to Appendix E. For all the example measurement files that were delivered with EasySAXS select "Default.cfg".

  In the field "Minimum 2Theta (degrees)", you should enter the 2Theta position of the first data point measured with the beam attenuator removed. In all the example measurement files this value is 0.05 degrees, which is also the default setting in this field. The position can also be identified by plotting the measurement file in Data Viewer and searching for the position close to zero 2Theta where the intensity increases in a stepwise manner. If you are not sure about this value, you may also set it to 0.00. EasySAXS will then automatically determine the actual value.

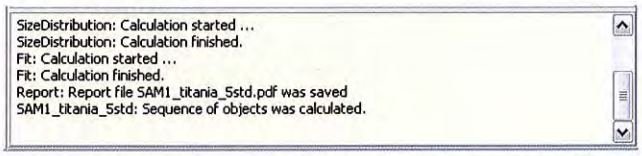


- Click [OK].

You have now provided all of the settings required for the analysis and we are ready to start the particle size distribution analysis of the selected titania nanopowder sample.

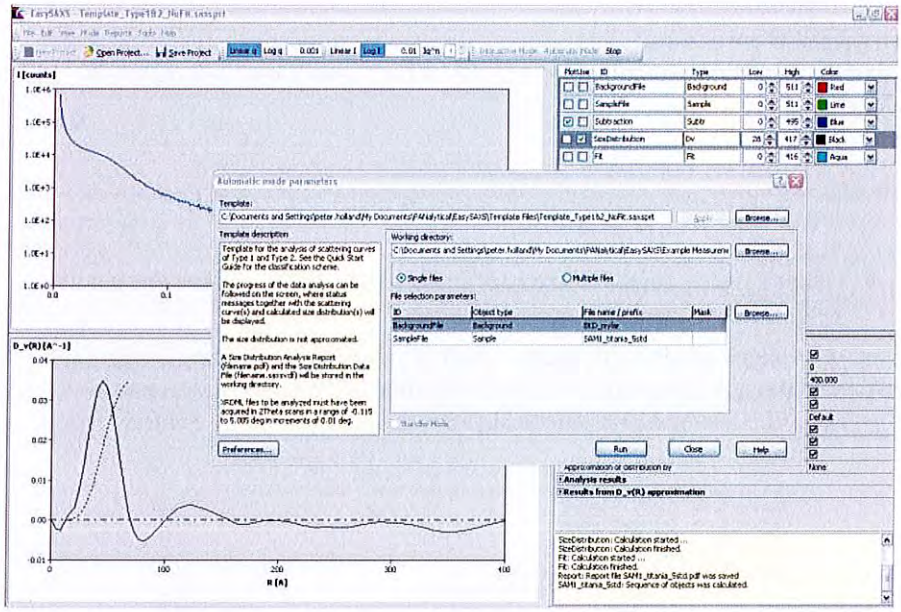
2.2.4 Executing Automatic Data Analysis on Single Data Files

- Click [Run] in the "Automatic mode parameters" window, this window will disappear and data analysis will start.
- The sequence of steps, such as loading data files, performing background subtraction, calculation of size distribution and creation of an analysis report is performed automatically. See the messages in the Status Message Pane.



Once the sequence of automatic processing is finished, the "Automatic mode parameters" window will reappear on the screen. The Main Graphics pane displays the background-corrected SAXS data and the Additional graphics pane the particle size distribution plot.

The system is ready to be set up for another analysis, if wanted.



- Click [Close] to close the "Automatic mode parameters" window.

2.2.5 Closing EasySAXS

- Close EasySAXS by clicking on *File – Exit* in the Menu Bar.

2.2.6 Inspecting the Analysis Results

When the automatic data processing has finished, the following files are created in the working directory (in this example "*C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Example Measurement Files*" as we specified on page 2 - 5):

- An **analysis report file** "*samplefilename*.pdf" containing all relevant details and results from data analysis.
- The **size distribution data** in ASCII format ("*samplefilename*.saxsvdf").
- A log file "*templatefilename*.txt" in which the sequence of analysis steps has been recorded.

2.2.6.1 Analysis Report File

In *Preferences - Report* we opted for the full 2-page report. It can be found in the working directory and is identified as "*samplefilename*.pdf".

Among other items, this analysis report lists the analyzed data files, the analysis template used, and the absorption factor of the sample. The background-corrected scattering curve and the (volume-weighted) size distribution curve are shown in two separate graphs. Parameters derived from the size distribution, such as average particle radius, standard deviation of the distribution and surface-to-volume ratio of the particles are also shown.

The absorption factor of the sample determined to 2.2 indicates that the sample thickness was chosen appropriately. If this value had exceeded 5, the measurement should preferably be repeated with a thinner sample to obtain best possible results.

For the titania sample that we have just analyzed the median particle radius (R50) could be determined to 44.0 Å and the relative standard deviation of the size distribution to approximately 24%. The surface-to-volume ratio (S/V) was calculated from the distribution curve to 0.0736 $Å^{-1}$. Knowing that the measured titania (anatase) sample is made of particles having a mass density of 3.9 g/cm^3, following the instructions given in the Help File, we can calculate the specific surface area as follows: S/m = 10000 / 3.9 x 0.0736 = 189 m^2/g. These results are in good agreement with those obtained from the same sample by electron microscopy and by gas adsorption (BET).

For detailed explanations about all the entries in the analysis report please see the on-line Help.

Nanoparticle Research and Application Laboratory
Almelo, The Netherlands

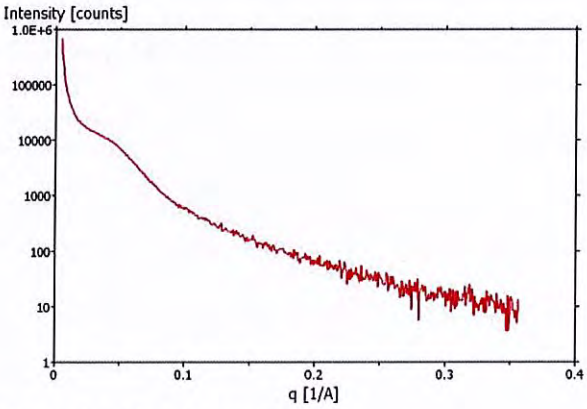
PANalytical

Report on Particle Size Analysis

Sample and Analysis Details

Sample file:	**SAM1_titania_5std.xrdml**
Background file:	BKD_mylar.xrdml
Analysis software:	EasySAXS V. 2.0.0.373
Analysis template:	Template_Type1&2_NoFit.saxsprt
Working directory:	C:\Documents and Settings\peter.holland\My Documents\PANalytical\EasySAXS\Example Measurement Files

Background-Corrected Scattering Curve

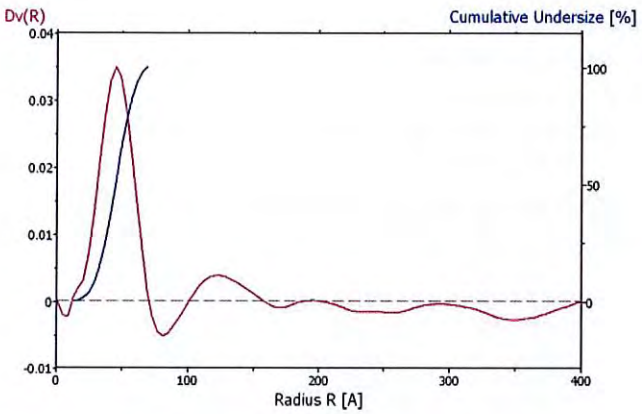


Absorption factor of sample: 2.206

Nanoparticle Research and Application Laboratory
Almelo, The Netherlands

PANalytical

Size Distribution by Volume



Results based on Size Distribution by Volume

Most frequent radius:	44.0 A
Average radius:	43.8 A
R20:	34.3 A
R50:	44.0 A
R80:	53.5 A

Relative standard deviation: 24.13 %

Surface-to-volume ratio S/V: 0.0736 1/A

Annotations

SAXS measurements were done by ABC

Samples were prepared by DEF

Data were analyzed by GHI

09.11.2010 13:01:58

Page 2 of 2

2.2.6.2 Size Distribution File

The size distribution file ("*samplefilename*.saxsvdf") is an ASCII file containing the particle volume-weighted size distribution data in the following format;

Column 1: Radius in units of Å or nm as indicated in the first line,
Column 2: probability by volume Dv(R),
Column 3: error estimation.

When using the analysis templates provided the radius is always given in units of Angstroms. Any plotting program may be used for displaying this data file in a graph. The data may also be loaded in the Data Viewer.

SAM1_titania_5std - Notepad

File Edit Format View Help

```
units=angstroms
  0.000000000E+0  0.000000000E+0  0.000000000E+0
  4.000000000E+0 -2.120994766E-3  1.686054284E-4
  8.000000000E+0 -2.282778405E-3  1.866949379E-4
  1.200000000E+1  2.765704174E-4  1.764867976E-4
  1.600000000E+1  1.886440162E-3  1.879929725E-4
  2.000000000E+1  3.173631423E-3  2.038350883E-4
  2.400000000E+1  7.189682945E-3  1.907878554E-4
  2.800000000E+1  1.349926834E-2  1.755389767E-4
  3.200000000E+1  2.068135685E-2  1.948949545E-4
  3.600000000E+1  2.772263473E-2  2.383182533E-4
  4.000000000E+1  3.288683381E-2  2.311661221E-4
  4.400000000E+1  3.496987286E-2  2.131867672E-4
  4.800000000E+1  3.361336793E-2  2.326339167E-4
  5.200000000E+1  2.896862940E-2  2.546875418E-4
  5.600000000E+1  2.219283483E-2  2.632617900E-4
  6.000000000E+1  1.469726129E-2  2.780472859E-4
  6.400000000E+1  7.531523436E-3  2.830622486E-4
  6.800000000E+1  1.696753588E-3  2.752937422E-4
  7.200000000E+1 -2.260362251E-3  2.619076686E-4
```

2.2.6.3 Log File

The sequence of analysis steps have been recorded in the log file "*templatefilename*.txt".

Template_Type1&2_NoFit - Notepad

File Edit Format View Help

```
09.11.2010 16:11:44 Template_Type1&2_NoFit.saxsprt: Automatic processing started...
09.11.2010 16:11:44 BackgroundFile: File BKD_mylar.xrdml was correctly loaded.
09.11.2010 16:11:44 SampleFile: File SAM1_titania_5std.xrdml was correctly loaded.
09.11.2010 16:11:44 Subtraction: Calculation started ...
09.11.2010 16:11:44 Subtraction: Calculation finished.
09.11.2010 16:11:44 SizeDistribution: Calculation started ...
09.11.2010 16:11:45 SizeDistribution: Calculation finished.
09.11.2010 16:11:45 Fit: Calculation started ...
09.11.2010 16:11:45 Fit: Calculation finished.
09.11.2010 16:11:45 SAM1_titania_5std: Sequence of objects was calculated.
```

2.3 AUTOMATIC ANALYSIS OF MULTIPLE DATA FILES

Having understood how to set up the analysis of single data files, it is now a straightforward procedure to analyze multiple data files in a batch.

In this example, we will analyze all the example measurement files that have the prefix "SAM2" in their file name. As is suggested by their file names, the scattering curves all have the characteristics of a "Type 2 scattering curve".

Accordingly, to analyze this type of data files we have to choose an analysis template (*.saxsprt) that was made for "Type 2 scattering curves".

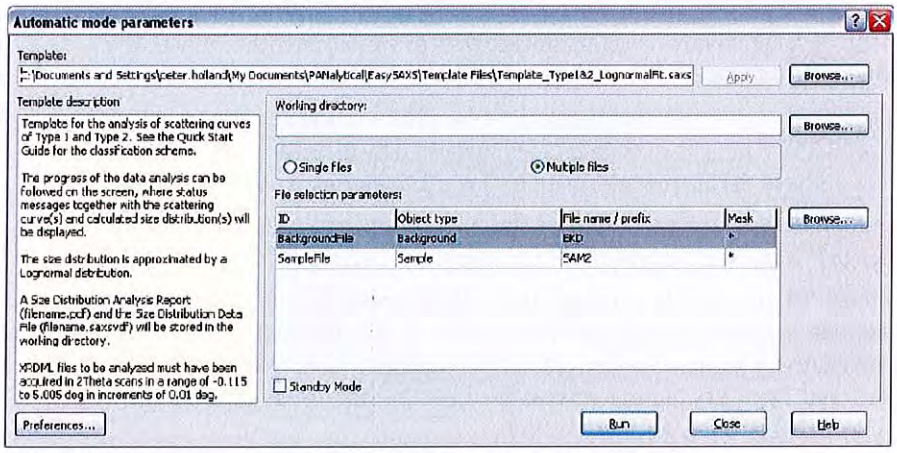
From the list of available template files delivered with EasySAXS we select "Template_Type1&2_LognormalFit.saxsprt". As can be readily understood from the template file name, and as explained in Appendix B, this template was made for Type 1 and Type 2 scattering curves; this time the size distribution curves will be approximated by a log-normal function.

2.3.1 Setting Up and Executing an Automatic Data Analysis

- To load this template double-click on the template file "Template_Type1&2_LognormalFit.saxsprt" in the Windows Explorer.

  EasySAXS is launched with this template.
- When clicking on the Start button in the Main toolbar The "Automatic mode parameters" window will open.

The "Automatic mode parameters" window already contains some default entries for the selected template and will initially look as shown below.



We now have to complete and modify the entries as follows:

- The working directory, being the directory in which the to be analyzed data files and the corresponding background file are located, can be specified by clicking on the [Browse...] button next to this entry field.
- As this time we want to analyze several sample files in a batch, the "Multiple files" option button must be selected (the default).
- Next, under "File selection parameters", we have to select the multiple example measurement files to be analyzed. To make this selection, EasySAXS uses the concept of a "File name prefix" combined with a "Mask".

  The "Mask" entry is only available in the "Multiple files" mode and is interpreted as a wild card character to specify multiple files whose names all start with the specified "File name prefix". For example, the asterisk "*" acts as a wild card character for any number of characters, and the question mark "?" for single characters.

  In this example, we want to analyze all example measurement files that have the prefix "SAM2" in their file name. The corresponding background file is "BKD_mylar_1.xrdml". The background was measured only once at the very beginning, followed by the measurements of the actual samples.

NOTE: For the analysis of multiple samples, the background MUST have been measured BEFORE the samples. If there have been repeated background measurements, as a rule, the background last measured before a given sample will be taken for the background-subtraction procedure with that sample file.

With the (default) entry "BKD" as the "File name prefix" for the background, in combination with the asterisk "*" symbol as the (default) setting for the "Mask", all background files in the working directory which have a file name starting with "BKD" will be used. In our example there is only one such background file available.

In the same way, with the (default) entry "SAM2" as the "File name prefix" for the Sample File, in combination with the asterisk "*" symbol as the (default) setting for the "Mask", all sample files in the working directory which have a file name starting with "SAM2" will be evaluated.

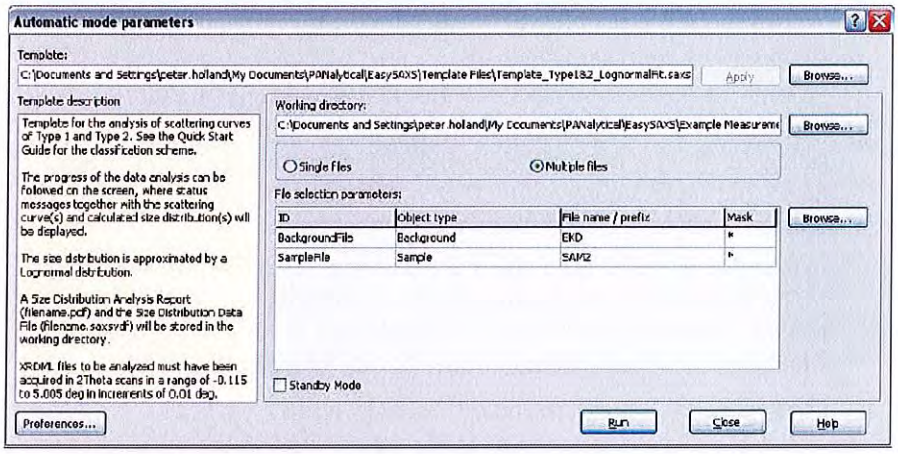
- Click [Preferences...]. The "Preferences" window opens.

  As before, select "Reports" tab and set your preferences for the analysis report that is to be created:

  This time select "Particle Size Analysis (compact)", the compact, 1-page report.

- Click [OK] to accept these settings

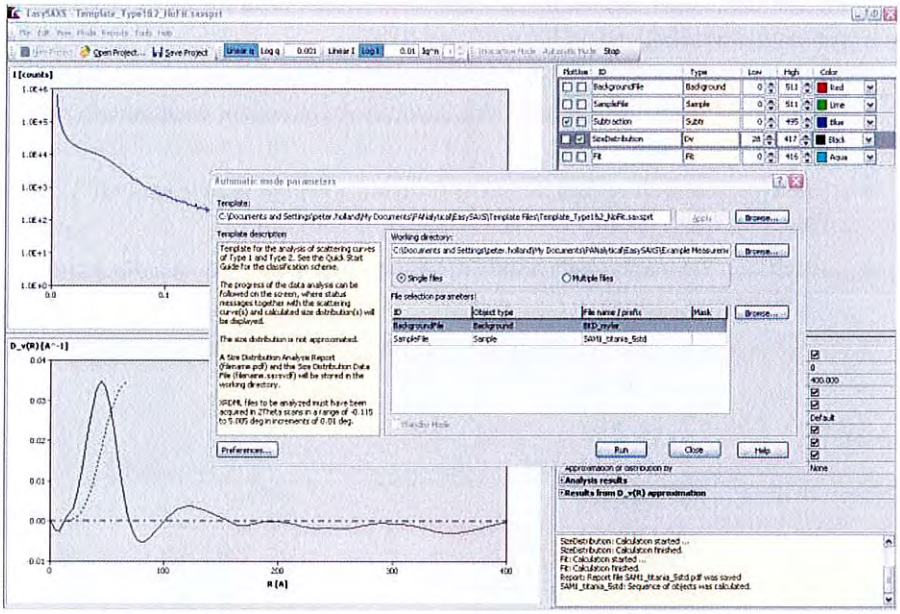
Now that we have applied the settings as shown below, we are ready to start the automatic particle size distribution analysis of multiple data files.



- Click [Run] in the "Automatic mode parameters" window. This window will then disappear and data analysis will start.
- The Status Message Pane informs you about the ongoing sequence of data processing steps, such as loading data files, performing background subtraction, calculation of size distribution and creation of an analysis report. You can also follow the results of the calculations in the two graphics panes.

  EasySAXS reads the time stamp of the measurements from the xrdml files to be analyzed and processes them in the same sequence as they were measured. This is why, as already mentioned earlier, it is essential that the background measurement was done before the sample measurements.

Once all data files have been processed, the "Automatic mode parameters" window will reappear on the screen.



- Now close the "Automatic mode parameters" window by clicking on the [Close] button.

NOTE: To stop multiple data analysis during the course of analysis, click on the [Stop] button in the Main Toolbar. This will, possibly with some delay, cause the "Automatic mode parameters" window to reappear on the screen. You can then either perform another analysis or exit EasySAXS.

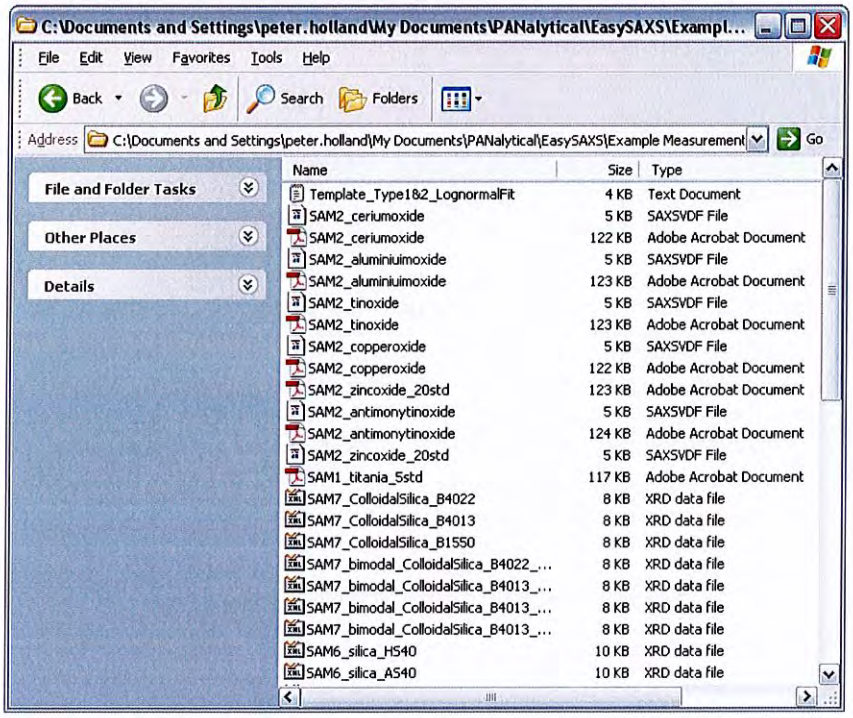
2.3.2 Closing EasySAXS

- Close EasySAXS by clicking on *File – Exit* in the Menu Bar.

2.3.3 Inspecting the Analysis Results

When the automatic data processing has finished, the following files are created in the working directory for each of the analyzed files:

- An analysis report file "*samplefilename*.pdf" containing all relevant details and results from data analysis. One such file is created for each sample file analyzed.
- The size distribution data in ASCII format ("*samplefilename*.saxsvdf"). One such file is created for each sample file analyzed.
- A log file "*templatefilename*.txt" in which the sequence of analysis steps for all samples has been recorded.



2.3.3.1 Analysis Report File

In *Preferences - Reports* we opted for the "compact" 1-page report. The reports on all of the analyzed samples can be found in the working directory and are identified as "samplefilename.pdf".

As an example, we shall look at the report "SAM2_zincoxide_20std.pdf" from a zinc oxide nanopowder.

In the graph, the volume-weighted size distribution is displayed in red, the approximated log-normal function in green, and the undersize cumulative in blue. The average particle radius was determined to approximately 200 Å. As is the case with most type 2 scattering curves, the deduced size distribution is relatively broad with a long tail towards larger particles.

Nanoparticle Research and Application Laboratory
Almelo, The Netherlands

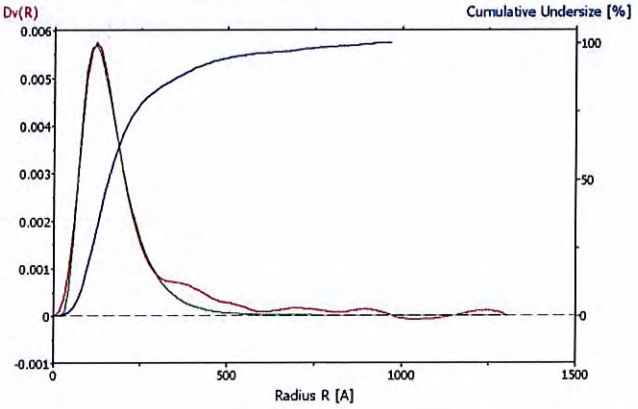
PANalytical

Report on Particle Size Analysis

Sample and Analysis Details

Sample:	SAM2_zincoxide_20std.xrdml
Background:	BKD_mylar.xrdml
Analysis software:	EasySAXS V. 2.0.0.373
Analysis template:	Template_Type1&2_LognormalFit.saxsprt
Working directory:	C:\Documents and Settings\peter.holland\My Documents\PANalytical\EasySAXS\Example Measurement Files

Size Distribution by Volume



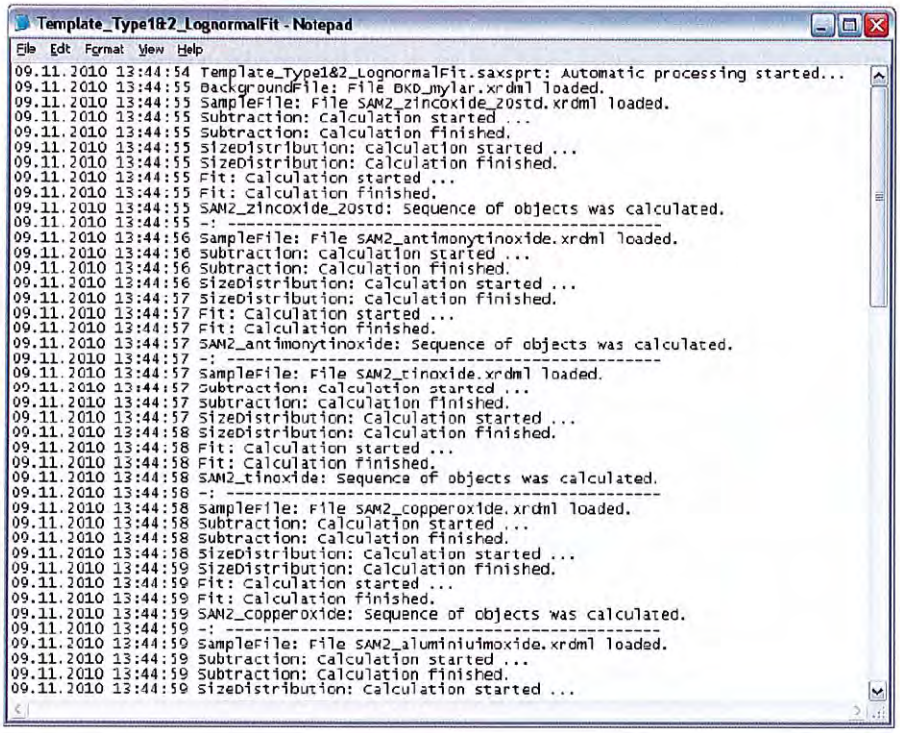
Results based on Size Distribution by Volume

		Log-normal approximation
Most frequent radius:	117.0 A	
Average radius:	201.2 A	
R20:	97.7 A	95.2 A
R50:	152.9 A	143.4 A
R80:	263.7 A	216.0 A
Relative standard deviation:	77.68 %	48.68 %
Surface-to-volume ratio S/V:	0.0226 1/A	0.0235 1/A
Absorption factor of sample:	1.621	

09.11.2010 14:02:47

2.3.3.2 Log File

The sequence of analysis steps for all of the samples has been recorded in the log file "*templatefilename*.txt".



```
Template_Type1&2_LognormalFit - Notepad
File Edit Format View Help
09.11.2010 13:44:54 Template_Type1&2_LognormalFit.saxsprt: Automatic processing started...
09.11.2010 13:44:55 BackgroundFile: File BKD_mylar.xrdml loaded.
09.11.2010 13:44:55 SampleFile: File SAM2_zincoxide_20std.xrdml loaded.
09.11.2010 13:44:55 Subtraction: Calculation started ...
09.11.2010 13:44:55 Subtraction: Calculation finished.
09.11.2010 13:44:55 SizeDistribution: Calculation started ...
09.11.2010 13:44:55 SizeDistribution: Calculation finished.
09.11.2010 13:44:55 Fit: Calculation started ...
09.11.2010 13:44:55 Fit: Calculation finished.
09.11.2010 13:44:55 SAM2_zincoxide_20std: Sequence of objects was calculated.
09.11.2010 13:44:55 -: ----------------------------------------------
09.11.2010 13:44:56 SampleFile: File SAM2_antimonytinoxide.xrdml loaded.
09.11.2010 13:44:56 Subtraction: Calculation started ...
09.11.2010 13:44:56 Subtraction: Calculation finished.
09.11.2010 13:44:56 SizeDistribution: Calculation started ...
09.11.2010 13:44:57 SizeDistribution: Calculation finished.
09.11.2010 13:44:57 Fit: Calculation started ...
09.11.2010 13:44:57 Fit: Calculation finished.
09.11.2010 13:44:57 SAM2_antimonytinoxide: Sequence of objects was calculated.
09.11.2010 13:44:57 -: ----------------------------------------------
09.11.2010 13:44:57 SampleFile: File SAM2_tinoxide.xrdml loaded.
09.11.2010 13:44:57 Subtraction: Calculation started ...
09.11.2010 13:44:57 Subtraction: Calculation finished.
09.11.2010 13:44:57 SizeDistribution: Calculation started ...
09.11.2010 13:44:58 SizeDistribution: Calculation finished.
09.11.2010 13:44:58 Fit: Calculation started ...
09.11.2010 13:44:58 Fit: Calculation finished.
09.11.2010 13:44:58 SAM2_tinoxide: Sequence of objects was calculated.
09.11.2010 13:44:58 -: ----------------------------------------------
09.11.2010 13:44:58 SampleFile: File SAM2_copperoxide.xrdml loaded.
09.11.2010 13:44:58 Subtraction: Calculation started ...
09.11.2010 13:44:58 Subtraction: Calculation finished.
09.11.2010 13:44:58 SizeDistribution: Calculation started ...
09.11.2010 13:44:59 SizeDistribution: Calculation finished.
09.11.2010 13:44:59 Fit: Calculation started ...
09.11.2010 13:44:59 Fit: Calculation finished.
09.11.2010 13:44:59 SAM2_copperoxide: Sequence of objects was calculated.
09.11.2010 13:44:59 -: ----------------------------------------------
09.11.2010 13:44:59 SampleFile: File SAM2_aluminiumoxide.xrdml loaded.
09.11.2010 13:44:59 Subtraction: Calculation started ...
09.11.2010 13:44:59 Subtraction: Calculation finished.
09.11.2010 13:44:59 SizeDistribution: Calculation started ...
```

2.3.3.3 Size Distribution File

The size distribution Dv(R) for each of the samples analyzed, is stored in ASCII format ("*samplefilename*.saxsvdf").

2.4 AUTOMATIC STANDBY ANALYSIS OF MULTIPLE FILES

With EasySAXS you can also perform on-line data analysis during a batch measurement involving multiple samples. As soon as a measurement file is completed and saved in the working directory, it will be loaded and processed by EasySAXS.

In this case we will do a routine measurement and analysis of a series of nano-titania samples.

2.4.1 Setting Up Data Collector and Initiating SAXS Measurements

In Data Collector, we prepare a sample changer batch program that will first measure the background, and then the whole series of available nano-titania samples. We make sure to use an "absolute scan program" that performs 2Theta scans in the range from –0.115 to 5.005 deg with a step size of 0.01 deg, using a point detector. This is very important to ensure compatibility of the measurement files with the analysis templates provided with EasySAXS.

For the background measurement say we decide to choose a file name starting with "BKD", such as "BKD_mylar_1" or "BKD_EmptyHolder_1.xrdml" etc. For the sample measurements say we decide to choose file names starting with "Titania", such as "Titania_Batch123" or "Titania_anatase_3" and so on. In the sample changer batch program we further decide to have the measurements files to be stored in the directory "*C:\X'Pert Data\Peter Holland\EasySAXS\Titania*".

Once all the settings have been done, we start the series of SAXS measurements.

For more details about how to set up and perform a measurement with Data Collector on PANalytical XRD systems refer to the "SAXS/WAXS Application Guide" that is delivered together with the SAXS hardware.

2.4.2 Selecting and Loading a Template that Runs in Standby Mode and Applies a Gaussian Fitting on the Size Distribution

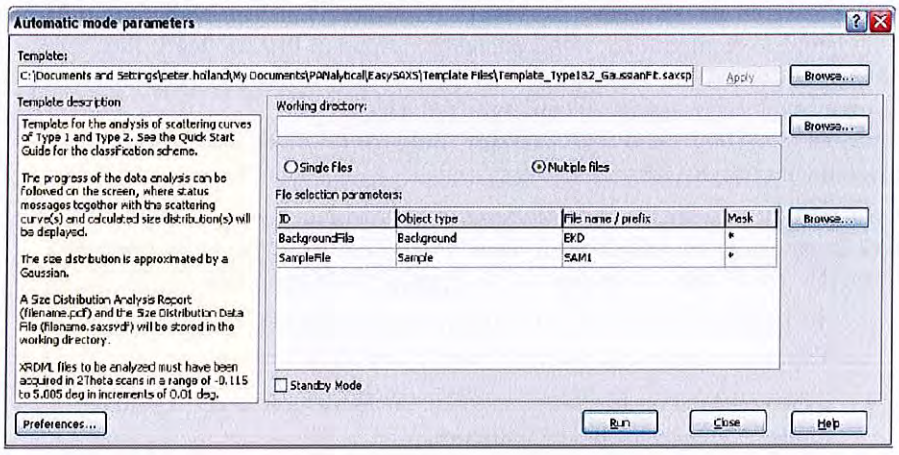
Now we will set up EasySAXS to analyze these data automatically as soon as a sample measurement is finished. Suppose that from previous experiments on similar titania samples we already know that an analysis template that was made for Type1 data is most suitable for data analysis and that the size distribution can be best approximated by a Gaussian. Accordingly, we select "Template_Type1&2_GaussianFit.saxsprt" from the list of available templates delivered with EasySAXS.

As you can see from the template file name, and as explained in Appendix A to this Quick Start Guide, this template was made for Type 1 and Type 2 of scattering curves; the size distribution curves will be approximated by a Gaussian function.

- To load this template double-click on the template file in the Windows Explorer, which will launch EasySAXS with this template.
- When clicking on the Start button in the Main toolbar The "Automatic mode parameters" window will open.

2.4.3 Setting Up and Executing the Automatic Data Analysis

The "Automatic mode parameters" window already containing some default entries for the selected template will initially look as shown below.



We now have to complete and modify the entries as follows:

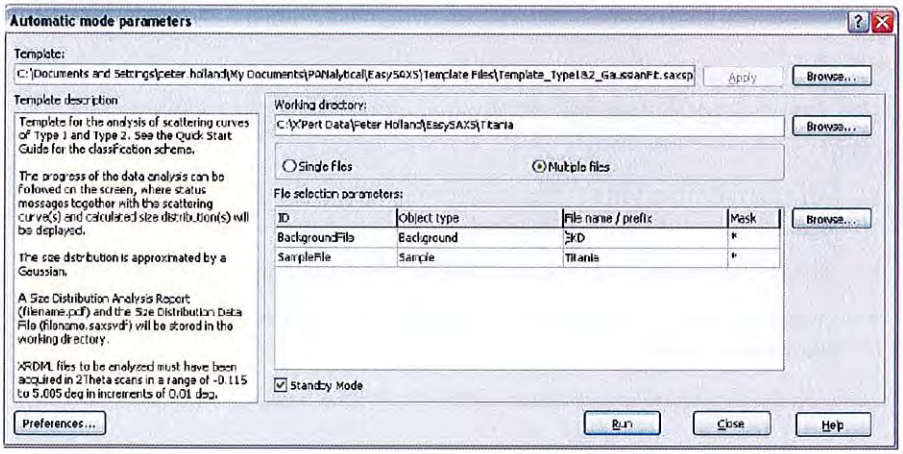
- Enter the working directory. This is the directory in which the to be analyzed data files and their corresponding background file will be located. You can select the required directory by clicking on the Browse... button next to this text box. We have to make sure here to choose the directory in which the measurement files collected by Data Collector will be stored. In this example "*C:\X'Pert Data\Peter Holland\EasySAXS\Titania*".
- As this time we want to analyze several sample files in a batch, the "Multiple experiments" radio button must be selected (the default).
- Next, under "File selection parameters", we have to select the measurement files to be analyzed. When setting up the sample changer batch program in Data Collector we decided to use the file name prefix "BKD" for a background measurement, and the prefix "Titania" for a measurement of a titania sample. We thus enter these prefixes in the corresponding columns and we use the asterisk "*" symbol so that all files with these prefixes in their file name will be analyzed.

- Check the "Standby mode" check box so that EasySAXS is set up to wait for incoming xrdml measurement files and, provided that their file names are according to the selection rule specified in the "Automatic mode parameters" setup window, to start data analysis as soon as a background and a sample measurement was completed and stored in the working directory. If previously measured files with these names are found in the working directory, they will be processed immediately. Thus, it does not matter whether you first start the SAXS measurement with Data Collector and afterwards start EasySAXS in standby mode or vice-versa. In both cases, all relevant data files will be analyzed.
- Click [Preferences...]. The "Preferences" window opens.

  As before, select "Reports" tab and set your preferences for the analysis report that is to be created:

  Choose the required report type.

Now that we have applied the settings as shown below, we are ready to start the automatic analysis of multiple data files in standby mode.



- Click [Run] in the "Automatic mode parameters" window. EasySAXS is now waiting for incoming files to be processed. This is shown in the Status Message Pane:

```
Template_Type1&2_GaussianFit.saxsprt: Automatic processing started...
BackgroundFile: File BKD_mylar.xrdml loaded.
Info: Automatic processing finished, now waiting for the next file...
```

  The Status Message Pane informs you about the ongoing sequence of data processing steps, such as loading data files, performing background subtraction, calculation of size distribution and creation of an analysis report.

  You can also follow the results of the calculations in the two graphics panes.

When all samples have been measured and analyzed we can stop EasySAXS running in standby mode by clicking on the [Stop] button in the Main Toolbar. When you do so, the "Automatic mode parameters" window will reappear on your screen. You may now do another analysis or exit this window by clicking [Close].

2.4.4 Inspecting the Analysis Results

For each of the analyzed data files, the following files are created in the working directory:

- An analysis report file "samplefilename.pdf" containing all relevant details and results from data analysis.
- The size distribution data in ASCII format ("samplefilename.saxsvdf").
- A log file "templatefilename.txt" in which the sequence of analysis steps has been recorded.

PANalytical

Chapter 3

Using the Interactive Mode

3.1 ABOUT THE INTERACTIVE MODE ..... 3 - 3

3.2 INTRODUCTION TO THIS CHAPTER..... 3 - 4

3.3 SIZE DISTRIBUTION ANALYSIS IN INTERACTIVE MODE ON A NANOPOWDER..... 3 - 5

3.3.1 Starting EasySAXS ..... 3 - 5
3.3.2 Setting the Project Properties ..... 3 - 6
3.3.3 Setting your Preferences..... 3 - 6
3.3.4 Opening Background and Measurement Files ..... 3 - 7
3.3.5 Subtracting the Background from the Sample Data ..... 3 - 10
3.3.6 Removing the Background and Sample Data from the Graph..... 3 - 12
3.3.7 Determining the Size Distribution Dv(R)..... 3 - 13
3.3.8 Creating a Size Distribution Analysis Report ..... 3 - 18
3.3.9 Saving the Project..... 3 - 20
3.3.10 Exporting Data..... 3 - 20
3.3.11 Creating an analysis template..... 3 - 20
3.3.12 Exiting EasySAXS ..... 3 - 21

3.4 FINAL REMARKS ON THE INTERACTIVE MODE..... 3 - 22

3.1 ABOUT THE INTERACTIVE MODE

The Interactive mode gives advanced users and experts easy access to a wide variety of advanced data treatment and analysis options via the respective toolbars.

Primary data handling options include background-correction, desmearing, data smoothing and scaling of intensities. Data analysis options range from Guinier and pair distance distribution function analysis, to Porod analysis and size distribution determination. Furthermore, it is possible to simulate and fit SAXS data for a variety of particle models, namely spheres, ellipsoids, cylinders, platelets and lamellar structures. Effects of slit smearing can directly be taken into account in the analysis, simulation and fitting procedures.

All parameter settings for these operations can be controlled in the Object Inspection Pane. Each treatment or analysis operation results in the creation of a new data file (in the following referred to as "object"). Each object is assigned a specific ID and Type descriptor. In the Object List Pane all objects are listed and conveniently accessible to be selected for plotting or further analysis.

Two zoomable graphics panes allow to view and compare scattering curves, and to display analysis results. Objects may also be exported as ASCII files.

All the details of the various evaluations made can be saved in a project file (*.saxsprj). The interactive mode can also be used to create analysis templates (*.saxsprt) that can then be applied for automated particle size distribution analysis in the Automatic mode.

The interactive mode supports data files in the PANalytical XRDML format, as well as ASCII files of background-corrected data.

NOTE: The user interface of the interactive mode is explained in section 1.5.1.

3.2 INTRODUCTION TO THIS CHAPTER

In this Chapter we will first explain step-by-step how to determine the particle size distribution from experimental SAXS data in the Interactive mode. We will save the sequence of analysis in a project file (*.saxsprj) and also create a template file (*.saxsprt) that can be then used for automatic analysis.

To do these tasks we will use the example measurement files (*.xrdml) that were supplied with EasySAXS (for details see Appendix D). After installation and first start of the program, they are located at:

C:\Users
<currentuser>\Documents\PANalytical\EasySAXS\Example Measurement Files

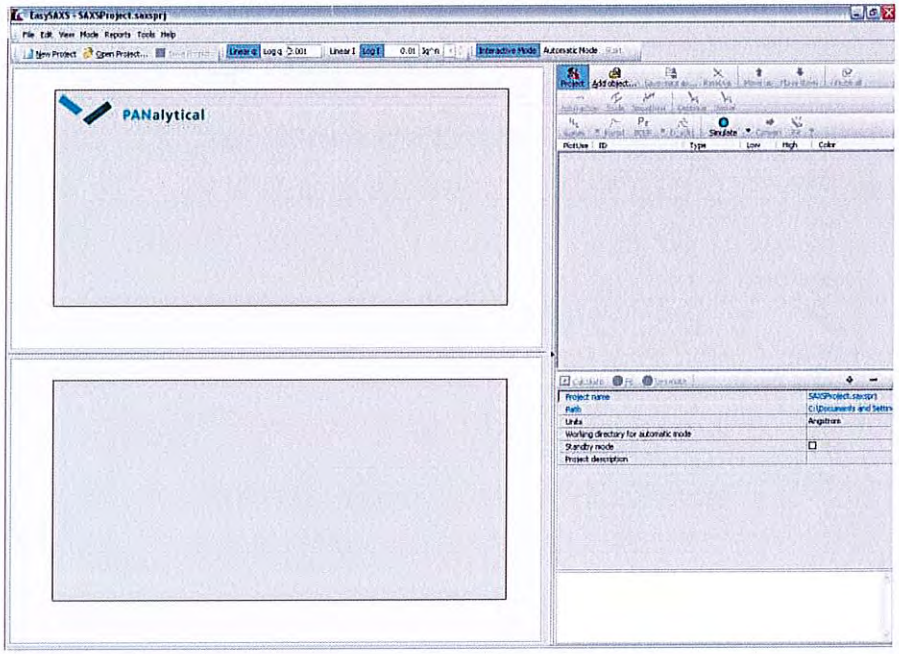
For detailed information about the analysis options that are available in the Interactive mode, refer to the EasySAXS on-line Help.

3.3 SIZE DISTRIBUTION ANALYSIS IN INTERACTIVE MODE ON A NANOPOWDER

In this section we will perform a size distribution analysis on the measurement file "SAM1_titania_10std.xrdml" from a nano-titania powder sample. The corresponding background file to be used is "BKD_mylar.xrdml"

3.3.1 Starting EasySAXS

- Either double-click on the EasySAXS icon EasySAXS, or select **Start** – *All Programs - PANalytical EasySAXS - EasySAXS*. By default, EasySAXS always starts up in the Interactive mode.



3.3.2 Setting the Project Properties

You will see that the

button is activated and the (default) properties of the project are displayed in the Object Inspection pane.

Project name	SAXSProject.saxsprj
Path	C:\Documents and Settin
Units	Nanometer
Working directory for automatic mode	
Standby mode	☐
Project description	

- At this point of time, in the field "Units" choose from a drop-down list whether you would like to use Angstroms or Nanometers as units. In this example, we decided to work with Nanometers. This means that, for example the scattering vector q will have units of nm^{-1}, and the Radius R units of nm.

  In all other fields we use the default entries.

3.3.3 Setting your Preferences

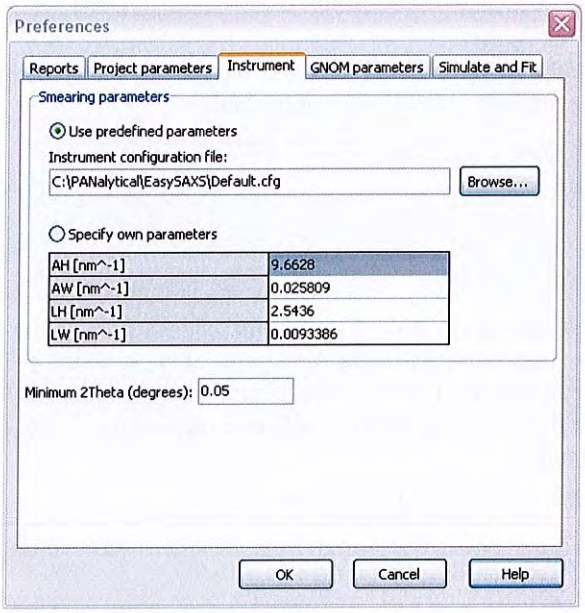
- Select *Tools - Preferences...* in the Menu Bar and a window with five tabs will open.

  In the "Reports" tab you can customize the "Header" and "Annotation" lines in the 2-page analysis report.

  In the "Instrument" tab it is important to select the "Instrument configuration file" that corresponds to the experimental SAXS setup on a PANalytical XRD system that was used for the measurement of the data files to be analyzed. Refer to Appendix E which relates the experimental setups with the configuration file (*.cfg) to be chosen here. For the example measurement files always use "Default.cfg".

  It is usually appropriate to enter the value of the first data point that was measured with the beam attenuator removed (or above the beam stop) in the "Minimum 2Theta" text box. You may also choose to enter 0.00; if you do that EasySAXS will determine this position automatically. For all the example measurement files use a value of 0.05 deg 2 Theta.

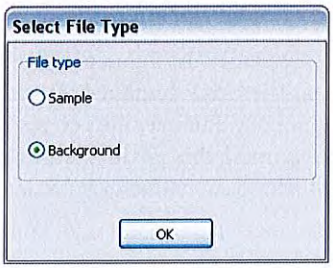
  Details about the other tabs can be found in the on-line Help. There is no need to make changes for this example.



3.3.4 Opening Background and Measurement Files

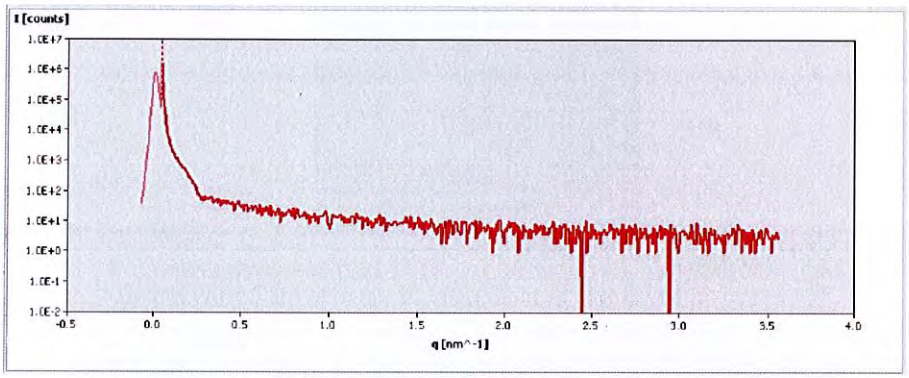
- Click on Add object... and then browse to the required file, in this example "BKD_mylar.xrdml" and click Open.

  You must then click on the appropriate option button to define what kind of file it is; in this case it is a "Background" file.



- Click [OK] and several things occur:

  A graph of the background measurement is shown in the Main Graphics pane.



Information about the plot of this object is shown in the Object List pane.

PlotUse	ID	Type	Low	High	Color
☑ ☑	_BKD_mylar	Background	16	511	Red

Information about the background file is shown in the Object Inspection pane.

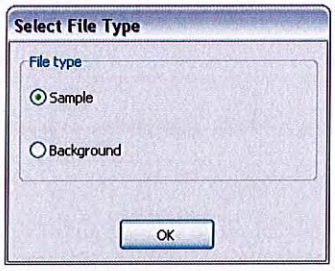
Calculate Fit Terminate

Object properties	
Object ID	_BKD_mylar
Object type	Background
Use object for next operation	☑
Plot object	☑
Lower limit of curve	16
Higher limit of curve	511
Multiplier	1
Original file name	C:\Documents and Settin
File Name Prefix	BKD_MYLAR
File Name Mask	
Wavelength [nm]	0.1541874

Now you need to open the actual sample measurement file.

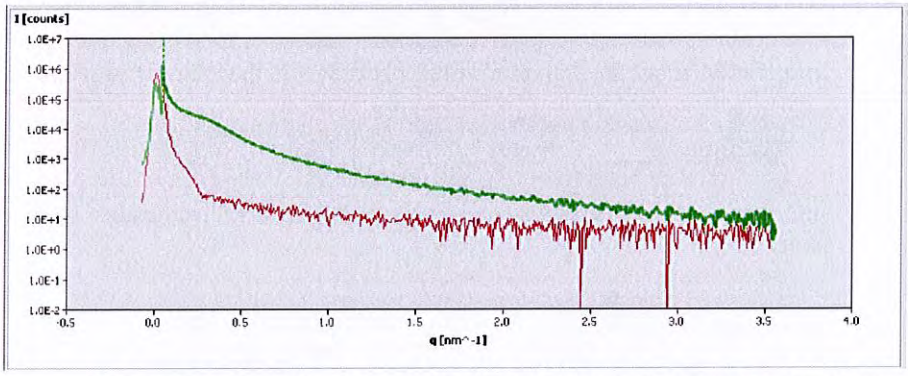
- Click on [Add object...] and then browse to the required file, in this example "SAM1_titania_10std.xrdml" and click [Open].

You must then click on the appropriate option button to define what kind of file it is; in this case it is a "Sample" file.



- Click [OK] and several things occur:

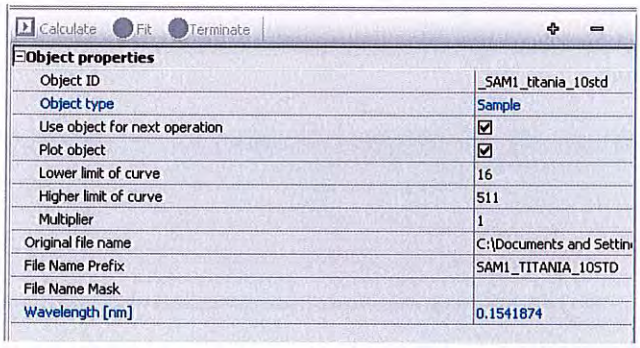
  The curve of the sample measurement is added to the Main Graphics pane.



Information about the plot of both of the objects is shown in the object list pane.

PlotUse	ID	Type	Low	High	Color
☑ ☑	_BKD_mylar	Background	16	511	Red
☑ ☑	_SAM1_titania_10std	Sample	16	511	Lime

Information about the sample file is shown in the Object Inspection pane.

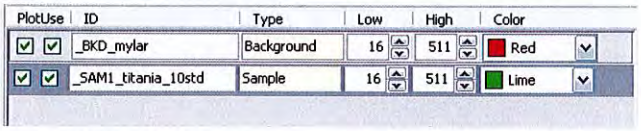


3.3.5 Subtracting the Background from the Sample Data

The next task is to correct the sample measurement for the background. To do so, we subtract the background intensities from that of the sample. In this subtraction procedure it is important to take into account the absorption effect of the sample. To be able to do so, we included the direct beam profile in the measurements.

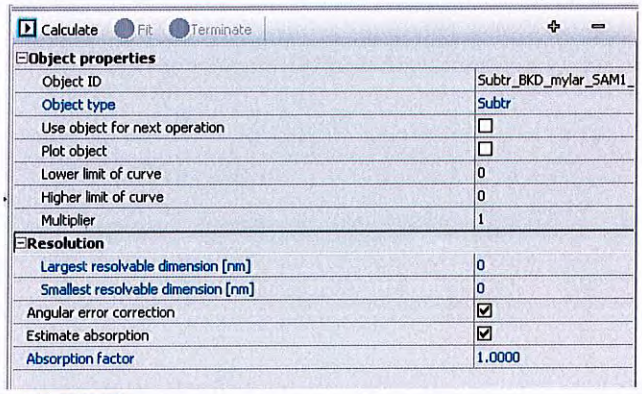
- Make sure that both of the plots in the object list pane (sample and background) have their "Use" check boxes checked and then click on Subtraction in the object treatment toolbar.

  A new object of type "Subtr" appears in the object list pane.



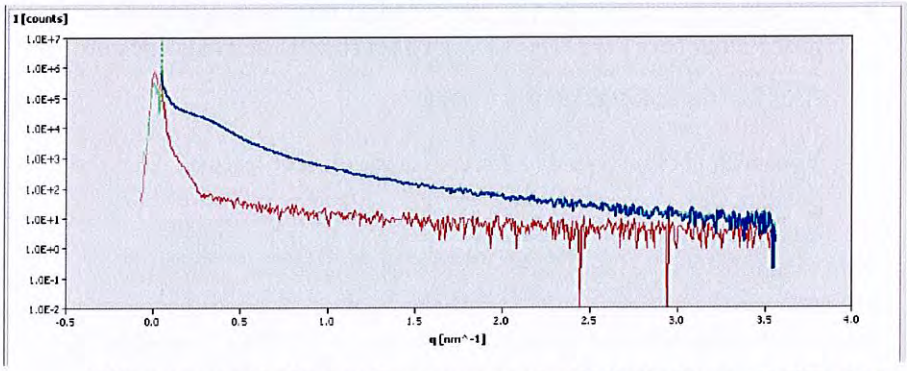
The parameters/options for the subtraction procedure yet to be performed are listed in the Object Inspection pane. Before we perform the subtraction we must ensure that the parameters are correctly set.

- Check the "Estimate absorption" check box (for an automatic absorption correction based on the profile of the direct beam) and the "Angular error correction" check box. No other specific settings are required in this example.



- Click Calculate.

  You will see that a third curve, in this example in blue, has been added to the Main Graphics pane.



This new curve is the sample measurement with the background subtracted from it. In this example, you see that for such strongly scattering powder sample throughout the whole measured angular range, the intensity of the background is nearly negligible compared to the intensity of the sample; the green (Sample) curve is mostly overlaid by the blue (Subtr) curve.

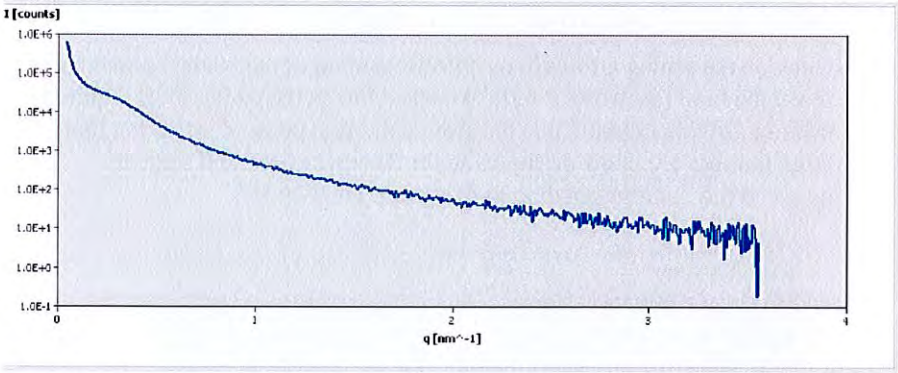
3.3.6 Removing the Background and Sample Data from the Graph

The background corrected data is all that will be needed for the size distribution analysis. We can therefore remove the other two curves:

- In the object list pane, clear the background and sample files "Use" and "Plot" check boxes.

PlotUse	ID	Type	Low	High	Color
☐ ☐	_BKD_mylar	Background	16	511	Red
☐ ☐	_SAM1_titania_10std	Sample	16	511	Lime
☑ ☑	Subtr_BKD_mylar_SAM1_t	Subtr	0	495	Blue

You will see that the background and sample curves are no longer displayed in the Main Graphics pane.



From the inspection of the background-corrected curve we can clearly identify it as being of "Type 1" according to the classification scheme detailed in Appendix A.

Characteristics of a scattering curve of Type 1:

> "A single, relatively pronounced, broad hump in the decaying scattering curve with a point of inflection clearly visible."

3.3.7 Determining the Size Distribution Dv(R)

3.3.7.1 Setting the limits

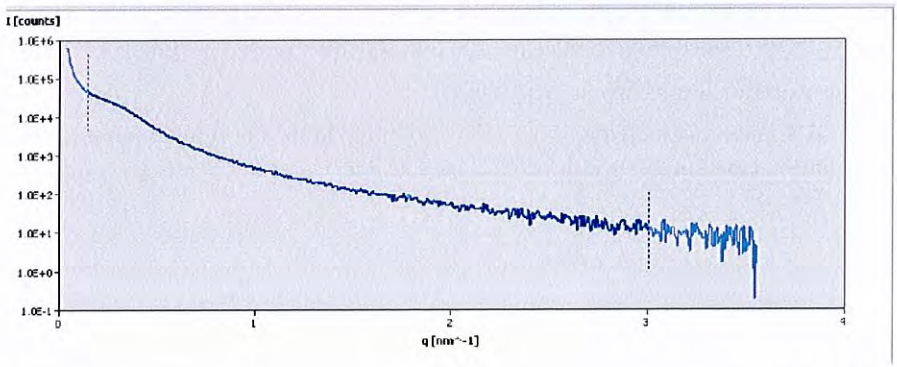
Now set the lower and upper limits of the background corrected curve, to define the interval of the scattering curve that is to be used for the subsequent size distribution analysis.

To determine the size distribution for this Type 1 of scattering curve in the Interactive mode, we follow the instructions given in the Appendix as follows:

Recommended settings for the evaluation of the size distribution in the Interactive mode:

- Set the "Lower Limit" q_{min} around the (first) point of inflection in the background-corrected scattering curve, the "Higher Limit" q_{max} around 3 nm^{-1} (0.3 $Å^{-1}$). The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 40 nm (400 Å) or smaller.
- Click on the setting arrows in the "Low" column of the "Subtr" object and move the limit (shown as a vertical dashed line in the graph) to its required position. In this example it is the around the first point of inflection (data point number 15). Now do the same for the upper limit and set it at $q_{max} = 3\ nm^{-1}$, corresponding to data point number 417.

PlotUse	ID	Type	Low	High	Color
☐ ☐	_BKD_mylar	Background	16	511	Red
☐ ☐	_SAM1_titania_10std	Sample	16	511	Lime
☑ ☑	Subtr_BKD_mylar_SAM1_t	Subtr	15	417	Blue



3.3.7.2 Setting up and Performing the Analysis of the Size Distribution Dv(R)

- Click on the D_v(R) button in the object treatment toolbar.

  A new object of type "Dv" (short for "volume-weighted size distribution") appears in the object list pane:

PlotUse	ID	Type	Low	High	Color
☐ ☐	_BKD_mylar	Background	16	511	Red
☐ ☐	_SAM1_titania_10std	Sample	16	511	Lime
☑ ☐	Subtr_BKD_mylar_SAM1_t	Subtr	15	417	Blue
☐ ☐	Dv_Subtr_BKD_mylar_SAM	Dv	0	402	Black

Details about this object are shown in the scrollable Object Inspection pane. Non-editable fields are marked in blue color.

Calculate Fit Terminate

Object properties	
Object ID	Dv_Subtr_BKD_mylar_
Object type	Dv
Use object for next operation	☐
Plot object	☐
Lower limit of curve	0
Higher limit of curve	402
Multiplier	1
Desmearing	☑
R_min [nm] for evaluating D_v(R)	0
R_max [nm] for evaluating D_v(R)	50.000
Zero condition at R = R_min	☑
Zero condition at R = R_max	☑
Calculation mode	Default
Estimate q_min	☐
Estimate q_max and R_max	☐
Normalization of D_v(R)	☑
Approximation of distribution by	None
Analysis results	
Average radius [nm]	0
Most frequent radius [nm]	0
R20 [nm]	0
R50 [nm]	0
R80 [nm]	0
Relative standard deviation [%]	0
Surface-to-volume ratio S/V [nm^-1]	0
Results from D_v(R) approximation	
R20 [nm]	0
R50 [nm]	0

You must now adjust the settings in the Object Inspection pane as appropriate for this type of scattering curve.

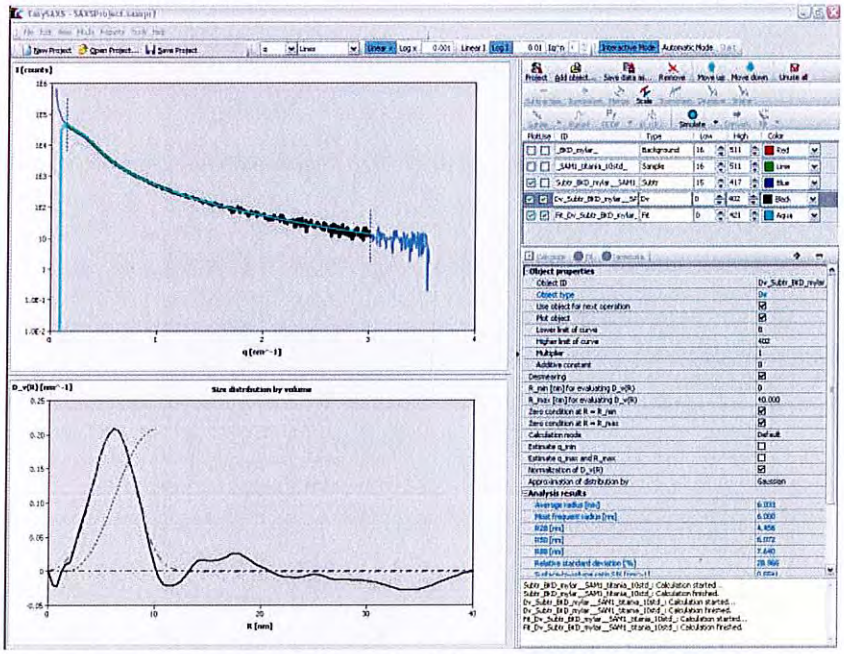
- As the experimental data were acquired with a line focus, they are affected by some instrumental smearing. This must be taken into account by checking the "Desmearing" check box (which is the default setting). The instrumental parameters are taken from the instrumental configuration file (*.cfg) selected previously in "Preferences - Instrument".
- It is important to make a first guess about the maximal size (radius) R_max of the particles in the sample. As a start, you may follow the instructions given in Appendix A of this Quick Start Guide. Accordingly, in this example, we set "R_max for evaluating Dv(R)" to 40 nm.
- In this example, we would like to approximate the size distribution Dv(R) by a Gaussian function. Thus, in the field "Approximation of distribution by", we select "Gaussian" from the drop-down menu.

The default entries in all other fields are suitable and do not need to be changed in this example.

Calculate Fit Terminate

Object properties	
Object ID	Dv_Subtr_BKD_mylar_
Object type	Dv
Use object for next operation	☐
Plot object	☐
Lower limit of curve	0
Higher limit of curve	402
Multiplier	1
Desmearing	☑
R_min [nm] for evaluating D_v(R)	0
R_max [nm] for evaluating D_v(R)	40.000
Zero condition at R = R_min	☑
Zero condition at R = R_max	☑
Calculation mode	Default
Estimate q_min	☐
Estimate q_max and R_max	☐
Normalization of D_v(R)	☑
Approximation of distribution by	Gaussian
Analysis results	
Average radius [nm]	0
Most frequent radius [nm]	0
R20 [nm]	0
R50 [nm]	0
R80 [nm]	0
Relative standard deviation [%]	0
Surface-to-volume ratio S/V [nm^-1]	0
Results from D_v(R) approximation	
R20 [nm]	0
R50 [nm]	0

- Click Calculate to start the calculation of the size distribution.



When the calculation is completed the (volume-weighted) size distribution curve (solid line) is shown in the additional graphics pane together with the undersize cumulative distribution curve (short-dashed black line) and the approximated Gaussian (long-short dashed, blue line).

In this example, a major maximum in the size distribution curve, centered around approximately R = 6.0 nm, can be observed. The major volume fraction of the particles has a radius in the range between 1 and 12 nm and the distribution is well approximated by a Gaussian. The minor oscillations in the distribution curve close to zero and towards larger particle radii may be regarded as insignificant: these are unavoidable artifacts from the Fourier transformation procedure.

NOTE: A new object of type "Fit" is created in the object list pane. The plot of this object is displayed in the Main Graphics pane in aqua color. It is calculated by a back-transformation of the Dv(R) function. Within the limit settings defined in section 3.3.7.1, it should be a good fit to the experimental data (dark blue curve), as it is the case in this example.

In the Object Inspection pane of the type Dv object you can inspect the analysis results.

Analysis results	
Average radius [nm]	6.033
Most frequent radius [nm]	6.000
R20 [nm]	4.456
R50 [nm]	6.072
R80 [nm]	7.640
Relative standard deviation [%]	28.966
Surface-to-volume ratio S/V [nm^-1]	0.5541
Results from D_v(R) approximation	
R20 [nm]	4.470
R50 [nm]	6.138
R80 [nm]	7.805
Relative standard deviation [%]	32.371
Surface-to-volume ratio S/V [nm^-1]	0.5716

The sequence of calculations previously done is displayed in the status message pane.

```
Subtr_BKD_mylar_SAM1_titania_10std: Calculation started ...
Subtr_BKD_mylar_SAM1_titania_10std: Calculation finished.
Dv_Subtr_BKD_mylar_SAM1_titania_10std: Calculation started ...
Dv_Subtr_BKD_mylar_SAM1_titania_10std: Calculation finished.
Fit_Dv_Subtr_BKD_mylar_SAM1_titania_10std: Calculation started ...
Fit_Dv_Subtr_BKD_mylar_SAM1_titania_10std: Calculation finished.
```

3.3.8 Creating a Size Distribution Analysis Report

Next we want to create a report on the size distribution analysis.

- Select *Reports* in the Menu Bar and choose between "Particle Size Analysis" and "Particle Size Analysis (compact)".

NOTE: On computer systems using double byte fonts (for example: Japanese and Chinese), only compact reports can be displayed. In such a case, the only option is to select "Particle Size Analysis (compact)" here.

In this example, we choose the compact 1-page report which will be displayed in a PDF viewer. By default the report will automatically be saved in *C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\User data\My Reports*.
You may change the default directory in *Preferences – Reports*.

Nanoparticle Research and Application Laboratory
Almelo, The Netherlands

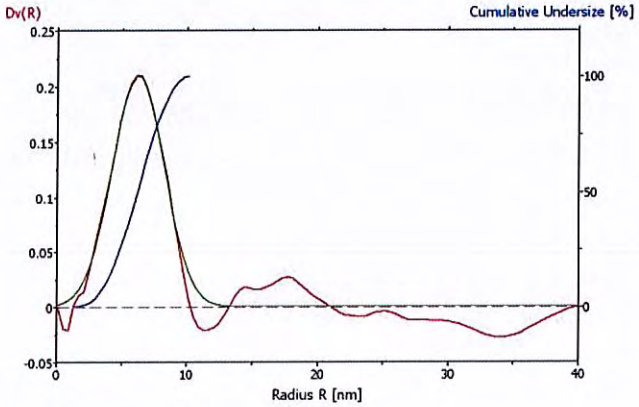
PANalytical

Report on Particle Size Analysis

Sample and Analysis Details

Sample:	SAM1_titania_10std.xrdml
Background:	BKD_mylar.xrdml
Analysis software:	EasySAXS V. 2.0.0.375
Analysis template:	SAXSProject.saxsprj
Working directory:	

Size Distribution by Volume



Results based on Size Distribution by Volume

		Gaussian approximation
Most frequent radius:	6.00 nm	
Average radius:	6.03 nm	
R20:	4.46 nm	4.47 nm
R50:	6.07 nm	6.14 nm
R80:	7.64 nm	7.81 nm
Relative standard deviation:	28.97 %	32.37 %
Surface-to-volume ratio S/V:	0.5541 1/nm	0.5716 1/nm
Absorption factor of sample:	2.220	

10.11.2010 15:01:29

3.3.9 Saving the Project

We have now completed a size distribution analysis in the Interactive mode. We can save the whole sequence of analysis steps together with all objects and parameter settings in a project file (*.saxsprj).

- Select *File - Save Project As...* and then select the folder where you want to store the project file, give it its new name and click [Save].

3.3.10 Exporting Data

It is possible to export any of the objects listed in the object list pane as an ASCII file. For example, to export the background-corrected data, in the object list pane put the focus on the object with the type "Subtr", then click on [Save data as...] in the Project toolbar. Specify a file name (*.dat) and the directory and then click [Save]. In the same way, you can, for example, export the size distribution data as a *.saxsvdf file.

These exported files can then be viewed or further treated with other plotting or analysis programs.

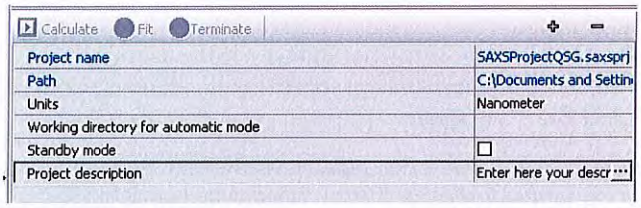
3.3.11 Creating an analysis template

If you have a series of samples of the same type as the one just analyzed, you may apply the same evaluation procedure with the same settings in an automated way using the Automatic mode of EasySAXS. In order to be able to automate this evaluation you have to save the sequence of analysis steps in a template file (*.saxsprt).

Before doing this, first you may want to define some default settings that will be displayed in the "Automatic mode parameters" window when starting up the template file.

- Click on the [Project] button in the project toolbar. In the Object Inspection pane in the field "Project description" you can enter some information for the user of the template. This information will be displayed in the "Template description" text box of the "Automatic mode parameters" window.

  If you check the "Standby mode" box, it will also automatically be checked in the "Automatic mode parameters" window.



Furthermore, in the Object Inspection panes corresponding to the background and sample file, respectively, you can enter a default "File name prefix" and "File name mask" for the Automatic mode.

You may now save the template by:

- Selecting *File - Save Project As...*

  Select the type for a template (*.saxsprt), enter a template file name and click [Save].

The new project template is then saved. It can be loaded from the Automatic mode of EasySAXS to perform an automatic analysis as explained in Chapter 2.

NOTE: A template file once saved can only be (partially) viewed, it cannot be modified. On the other hand, a project file can fully be viewed and modified. Therefore, it is good practice when creating an analysis template, to first save the project file upon which the template is to be based, and then to save the project as a template file. To modify a template later on, just load the corresponding project file, apply and save the changes there, and then save it as the modified template file.

For this reason, for all template files delivered with EasySAXS, the corresponding project files are also included (see Appendix C). This allows you to inspect all the specific settings that are used in the template files. You may also modify the settings in the analysis, and then save this as a new template that is further optimized for your specific samples.

After installation and first start of the program, the project files are located at *C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Project Files* (see also Appendix C of this Quick Start Guide).

3.3.12 Exiting EasySAXS

You may exit EasySAXS by selecting *File - Exit* in the Menu Bar.

3.4 FINAL REMARKS ON THE INTERACTIVE MODE

In this chapter of the Quick Start Guide we have described the principles and concepts of using EasySAXS in the interactive mode. For this we have taken size distribution analysis as one example.

Furthermore, using the same type of procedures, several additional analysis options are available in EasySAXS, such as Guinier and Porod analysis, simulation and fitting, and pair distance distribution function determination. These are not explicitly explained in this Guide. For detailed step-by-step instructions as well as worked examples for each procedure, refer to the EasySAXS on-line Help.

PANalytical

Appendix A

Classification of Scattering Curves into Different Types

A.1 INTRODUCTION ..... A - 3

A.2 TYPE 1 SCATTERING CURVES ..... A - 5

A.3 TYPE 2 SCATTERING CURVES ..... A - 9

A.4 TYPE 3 SCATTERING CURVES ..... A - 13

A.5 TYPE 4 SCATTERING CURVES ..... A - 16

A.6 TYPE 5 SCATTERING CURVES ..... A - 20

A.7 TYPE 6 SCATTERING CURVES ..... A - 25

A.8 TYPE 7 SCATTERING CURVES ..... A - 28

Appendix A. Classification of Scattering Curves into Different Types

A.1 INTRODUCTION

Samples differing significantly with respect to the range of particle or pore size, size polydispersity, agglomeration behavior and so on, show characteristic differences in their scattering curves.

In this Appendix we define seven different types of scattering curves based on the characteristics of the as-measured and/or background-corrected data. Size distribution profiles that are typical for a certain type are also shown. In some cases, electron micrographs that are representative of the type being described are displayed.

Furthermore, the recommended analysis templates for each type are listed. Also recommendations are given for suitable parameter settings when performing data analysis in the Interactive Mode.

Example files provided by PANalytical that are representative of a given type are also listed.

A.1.1 Concave and Convex

In this Appendix we sometimes use the terms concave and convex, by this we mean:

Concave the scattering curve has a slight "dip" as shown in Figure A.1.

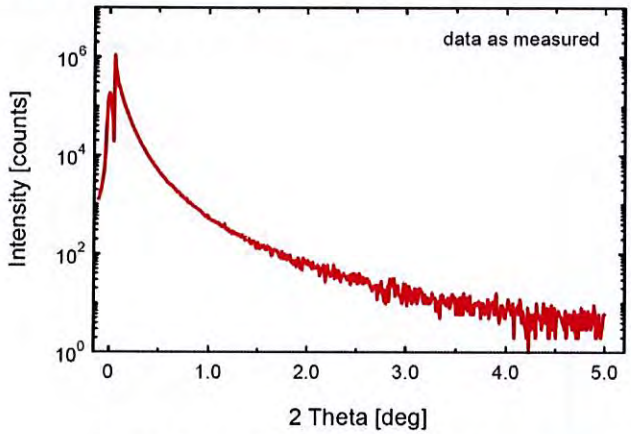


Figure A.1 : Concave Scattering Curve

Convex the scattering curve has a slight "rise" as shown in Figure A.2.

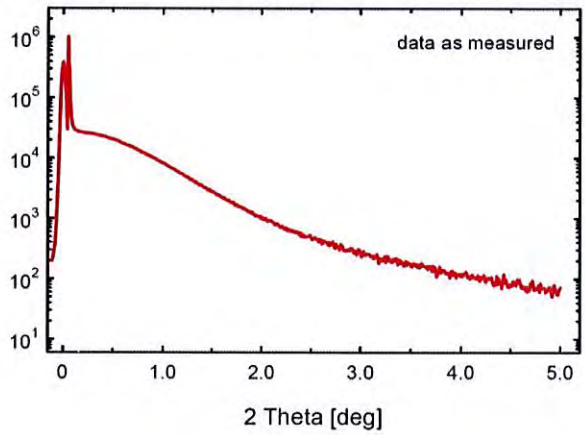


Figure A.2 : Convex Scattering Curve

A.2 TYPE 1 SCATTERING CURVES

A.2.1 Characteristics of the Scattering Curve

This type has a single, relatively pronounced, broad hump in the decaying scattering curve with a clearly visible point of inflection as shown in Figure A.3 and Figure A.4.

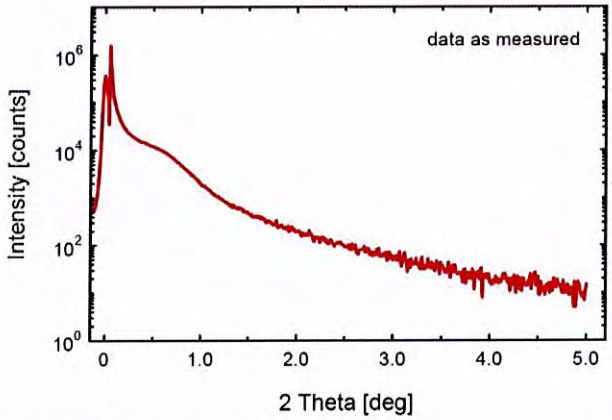


Figure A.3 : Type 1 Scattering Curve - as measured

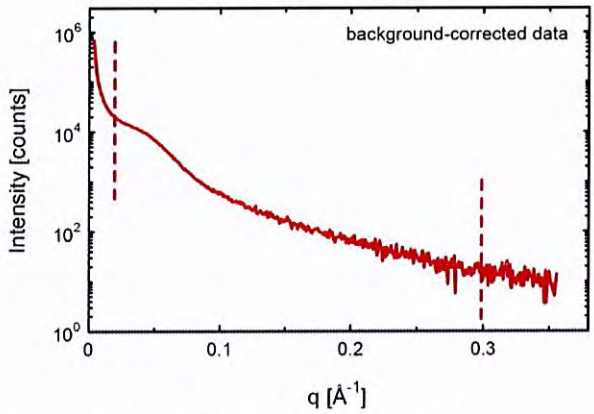


Figure A.4 : Type 1 Scattering Curve - background corrected

A.2.2 Sample Type

Spherical particles/pores with a well-defined size distribution that often can be best approximated by a Gaussian. The average radius is probably below 20 nm (200 Å).

The tendency of primary particles to agglomerate is only moderate.

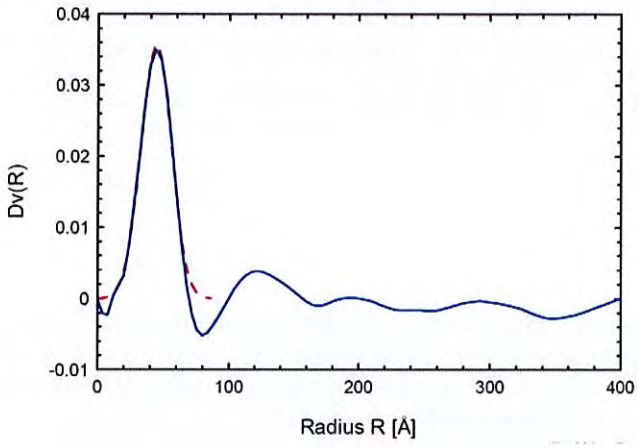


Figure A.5 : A typical size distribution from the analysis of type 1 scattering curves, together with a Gaussian approximation (dashed line)

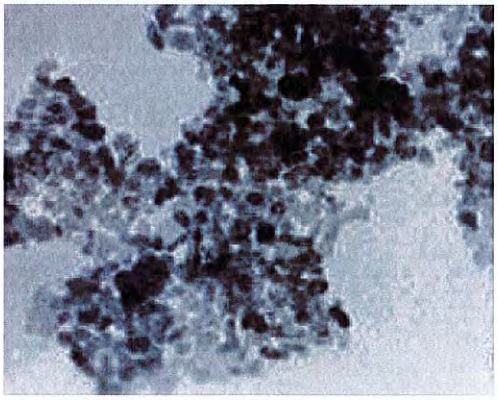


Figure A.6 : Example of an electron micrograph that is typically obtained from samples with type 1 scattering curves

A.2.3 Recommended Analysis Templates

Template Name (*saxsprt)	Approximation of Size Distribution Dv(R)
Template_Type1&2_NoFit	None
Template_Type1&2_GaussianFit	Gaussian (often most suitable for Type 1)
Template_Type1&2_LognormalFit	Log-normal (often most suitable for Type 2)

A.2.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} around the (first) point of inflection in the (background-corrected) scattering curve, the "Higher Limit" q_{max} around 3 nm^{-1} (0.3 $Å^{-1}$); see the dashed lines in Figure A.4. The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 40 nm (400 Å) or smaller.

A.2.5 Remarks

Normally this type of scattering curve leads to reliable analysis results with EasySAXS. Minor oscillations towards higher particle radius that may be observed in the distribution curve Dv(R) in most cases have no meaning and may thus be neglected (exception: multimodal distribution).

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.2.6 Available Example Data Files

Sample files:

- SAM1_titania_0501.xrdml
- SAM1_titania_0502.xrdml
- SAM1_titania_PAN.xrdml
- SAM1_porous_alumina_pm103.xrdml
- SAM1_porous_alumina_pm104.xrdml
- SAM1_titania_bimodal_PLVU0075_10std_1to1.xrdml
- SAM1_titania_bimodal_PLVU0075_10std_1to2.xrdml
- SAM1_titania_bimodal_PLVU0075_10std_2to1.xrdml

Background file:

- BKD_mylar.xrdml (for all sample files).

A.3 TYPE 2 SCATTERING CURVES

A.3.1 Characteristics of the Scattering Curve

This type has a steadily decaying scattering curve of concave shape without a visible hump or point of inflection as shown in Figure A.7 and Figure A.8.

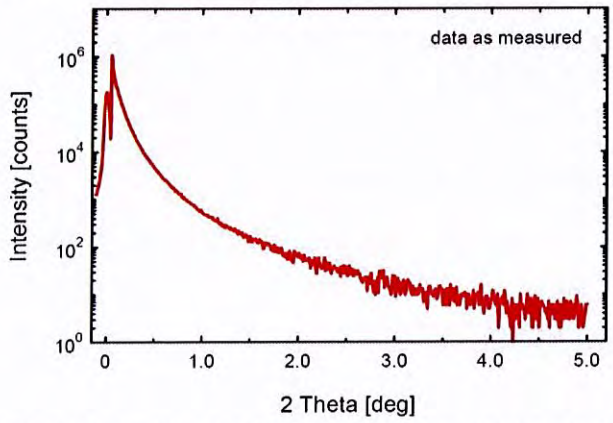


Figure A.7 : Type 2 Scattering Curve - as measured

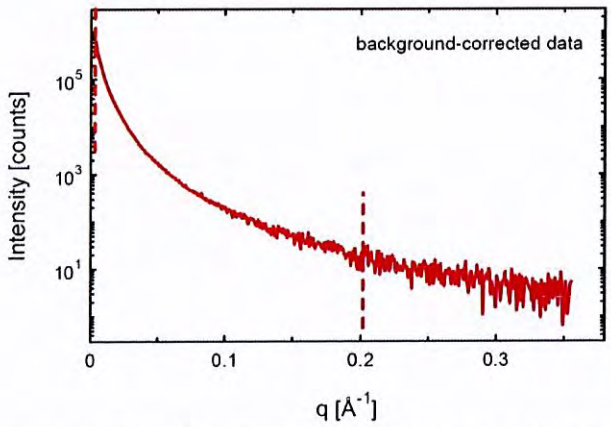


Figure A.8 : Type 2 Scattering Curve - background corrected

A.3.2 Sample Type

Spherical particles/pores with a broad size distribution that often can be best approximated by a log-normal function.

There could be a tendency of primary particles to agglomerate.

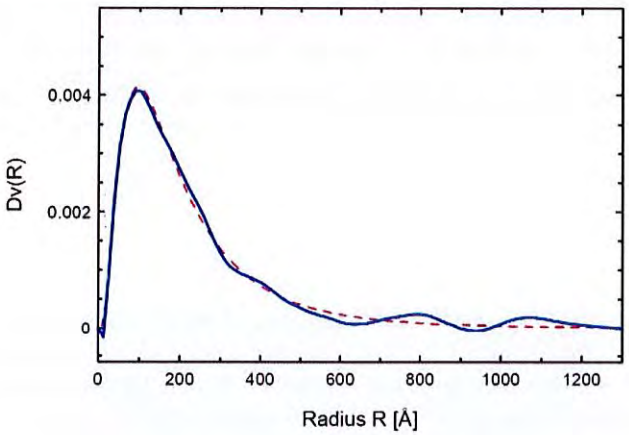


Figure A.9 : A typical size distribution from the analysis of type 2 scattering curves, together with a log-normal approximation (dashed line)

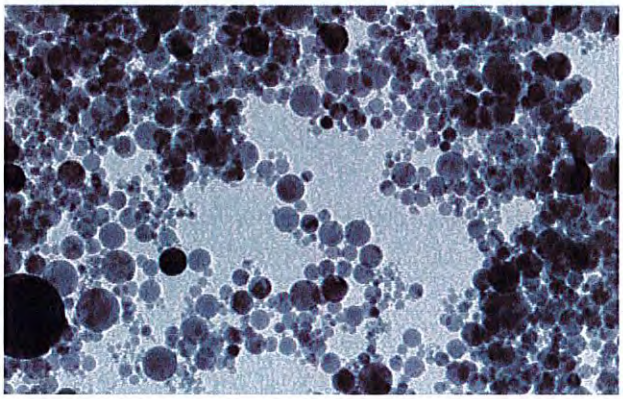


Figure A.10 : Example of an electron micrograph that is typically obtained from samples with type 2 scattering curves

A.3.3 Recommended Analysis Templates

Template Name (*saxsprt)	Approximation of Size Distribution Dv(R)
Template_Type1&2_NoFit	None
Template_Type1&2_GaussianFit	Gaussian (often most suitable for type 1)
Template_Type1&2_LognormalFit	Log-normal (often most suitable for type 2)

A.3.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} at the first data point of the (background-corrected) scattering curve, the "Higher Limit" q_{max} around 2 nm^{-1} (0.2 $Å^{-1}$); see the dashed lines in Figure A.8. The maximum particle radius "R_max for evaluating Dv(R)" is to be set to a relatively large value, such as 130 nm (1300 Å).

A.3.5 Remarks

This type of scattering curve leads to reasonable analysis results with EasySAXS provided that the particles are spherical and that the R80 value does not exceed the upper resolution limit of the measurement (approximately 50 nm for the particle radius). If it does exceed the upper resolution limit of the measurement the solution is likely to be unreliable. Minor oscillations towards higher particle radii that may be observed in the distribution curve in most cases have no meaning and therefore, can be neglected (exception: multimodal distribution).

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.3.6 Available Example Data Files

Sample files:

- SAM2_aluminiuimoxide.xrdml
- SAM2_antimonytinoxide.xrdml
- SAM2_ceriumoxide.xrdml
- SAM2_copperoxide.xrdml
- SAM2_tinoxide.xrdml
- SAM2_zincoxide_20std.xrdml

Background file:

- BKD_mylar.xrdml (for all sample files).

A.4 TYPE 3 SCATTERING CURVES

A.4.1 Characteristics of the Scattering Curve

This type has a relatively slowly but steadily decaying scattering curve having a convex shape at smallest angles (Guinier's law), and not showing oscillations. An upturn of the scattering curve in just a small region at smallest angles may be observed as shown in Figure A.11 and Figure A.12.

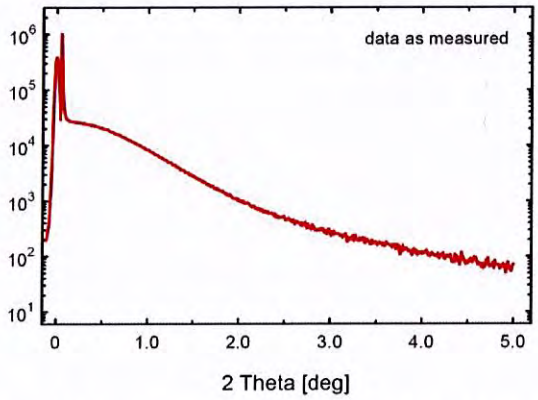


Figure A.11 : Type 3 Scattering Curve - as measured

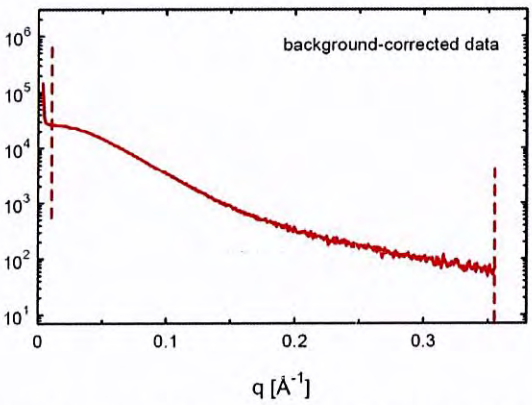


Figure A.12 : Type 3 Scattering Curve - background corrected

A.4.2 Sample Type

Spherical particles/pores with a well-defined size distribution that often can be approximated by either a log-normal or Gaussian distribution. The average radius is very small, well below 10 nm (100 Å).

The tendency of primary particles to agglomerate is quite moderate.

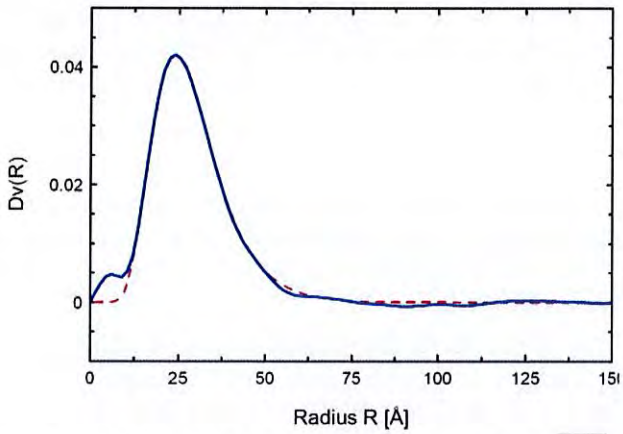


Figure A.13 : A typical size distribution from the analysis of type 3 scattering curves, together with a log-normal approximation (dashed line)

A.4.3 Recommended Analysis Templates

Template Name (*saxsprt)	Approximation of Size Distribution Dv(R)
Template_Type3_NoFit	None
Template_Type3_GaussianFit	Gaussian
Template_Type3_LognormalFit	Log-normal

A.4.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} at a relatively small value in the scattering curve so as to just exclude the region where the sharp upturn of the background-corrected scattering curve is observed, the "Higher Limit" q_{max} around 3.5 nm^{-1} (0.35 $Å^{-1}$) or higher); see the dashed lines in Figure A.12. The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 20 nm (200 Å) or smaller.

A.4.5 Remarks

Normally this type of scattering curve leads to reliable analysis results with EasySAXS. Minor oscillations towards higher particle radius that may observed in the distribution curve in most cases have no meaning and may thus be neglected (exception: multimodal distribution).

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.4.6 Available Example Data Files

Sample files:

- SAM3_porous_silica_dav635.xrdml
- SAM3_porous_silica_gd12.xrdml

Background file:

- BKD_mylar.xrdml (for all sample files).

A.5 TYPE 4 SCATTERING CURVES

A.5.1 Characteristics of the Scattering Curve

This type has a very weak, broad hump in the decaying scattering curve with a point of inflection hardly visible as shown in Figure A.14 and Figure A.15.

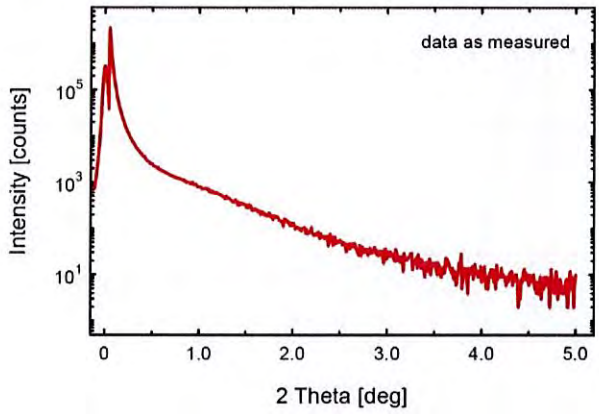


Figure A.14 : Type 4 Scattering Curve - as measured

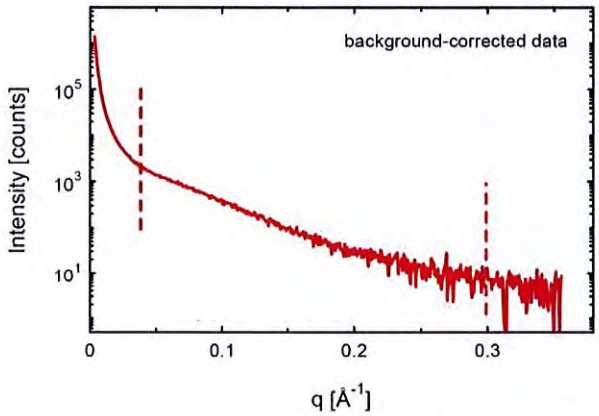


Figure A.15 : Type 4 Scattering Curve - background corrected

A.5.2 Sample Type

Spherical particles/pores with probably a well-defined size distribution. The average radius is probably below 20 nm (200 Å).

The tendency of primary particles to agglomerate is relatively strong.

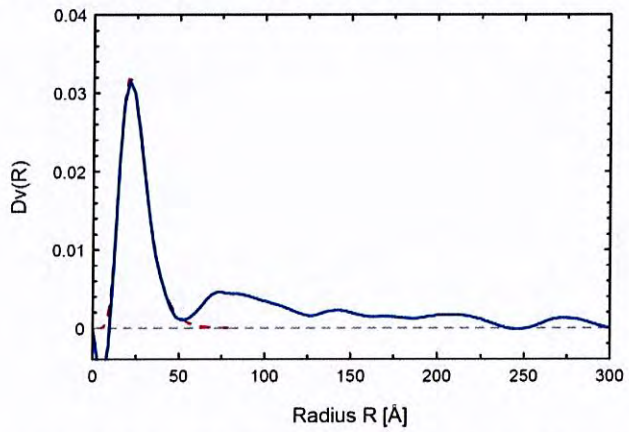


Figure A.16 : A typical size distribution from the analysis of type 4 scattering curves, together with a log-normal approximation (dashed line)

A.5.3 Recommended Analysis Templates

Template Name (*saxsprt)	Approximation of Size Distribution Dv(R)
Template_Type4_NoFit	None
Template_Type4_GaussianFit	Gaussian
Template_Type4_LognormalFit	Log-normal

A.5.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} around the (first) point of inflection in the background-corrected scattering curve, the "Higher Limit" q_{max} around 3 nm^{-1} (0.3 $Å^{-1}$); see the dashed lines in Figure A.15. The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 40 nm (400 Å) or smaller.

A.5.5 Remarks

Often this type of scattering curve allows, at least, to estimate the most frequent radius with EasySAXS. However, due to the strong tendency to form agglomerates, the determined size distribution may not return to zero within an extended region towards larger radii (see Figure A.16). One should be careful not to misinterpret this finding with the existence of a larger fraction of primary particles of that larger size. The major peak position and its width, however, in many cases properly reflect the actual distribution reasonably well, whereas the long tail toward larger radii is insignificant.

In such a case, it is recommended to approximate the main peak of the distribution function with a log-normal or Gaussian function, while neglecting the long tail towards larger radii (see red dashed line in Figure A.16). All values derived from this approximated function are likely to give much more reliable results than those derived directly from the evaluation of the original Dv(R).

Values derived from the original Dv(R) profile are likely to contain strong overestimations in the R20, R50 and R80 values, a strong overestimation of the relative standard deviation and an underestimation of the surface-to-volume ratio. On the other hand, the value given for the "Most frequent radius" will be estimated correctly.

Analyzing the data in the Interactive mode of EasySAXS with optimized evaluation parameters could lead to further improvements in the results.

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.5.6 Available Example Data Files

Sample files:

- SAM4_titania_PLV0071.xrdml
- SAM4_titania_PLVU0075.xrdml

Background file:

- BKD_mylar.xrdml (for all sample files).

A.6 TYPE 5 SCATTERING CURVES

A.6.1 Characteristics of the Scattering Curve

At smallest angles the scattering curve exhibits a convex curvature (Guinier's law). A small upturn or decrease of the scattering intensity towards smallest angles is sometimes also observed, even in the background-corrected data. This may be either due to minor agglomeration effects or due to a small error in the background-subtraction. Furthermore, two or more distinct oscillations are observed in the decaying curve. As this type of scattering curve is mostly observed from nanoparticles at rather low concentrations, the signal-to-background ratio at higher angles is often small. Examples of type 5 scattering curve are shown in Figure A.17 and Figure A.18.

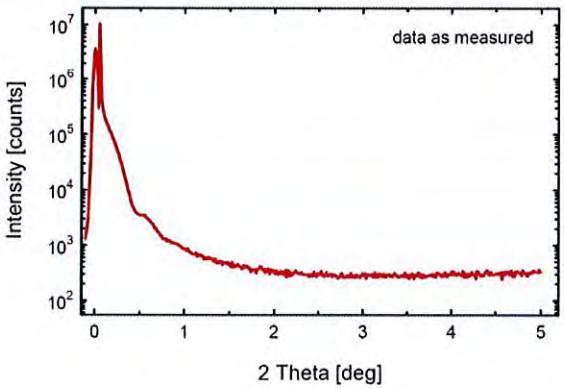


Figure A.17 : Type 5 Scattering Curve - as measured

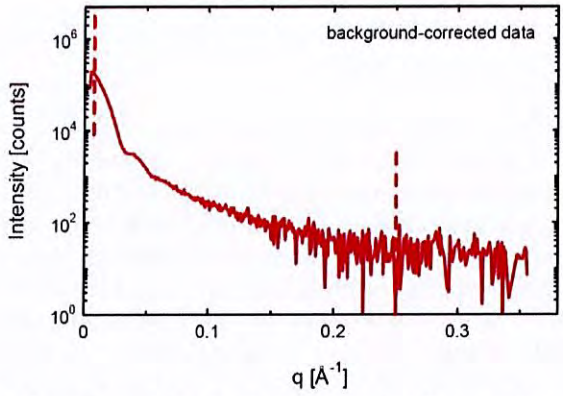


Figure A.18 : Type 5 Scattering Curve - background corrected

A.6.2 Sample Type

Samples containing distinct, isolated spherical particles with a very well-defined, narrow size distribution and a relative standard deviation of below 12%. The particles exhibit no or only minor interaction effects. Nanocomposites in which the incorporated nanoparticles are finely distributed in the matrix material, and have no or only little tendency to form agglomerates, may produce such type of scattering curve. This type of scattering curve is also often observed with dilute dispersions of (non-interacting) nanoparticles in a liquid.

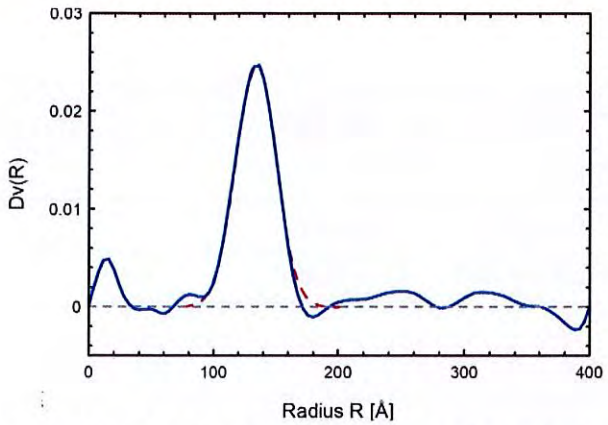


Figure A.19 : A typical size distribution from the analysis of type 5 scattering curves, together with a Gaussian approximation (dashed line)

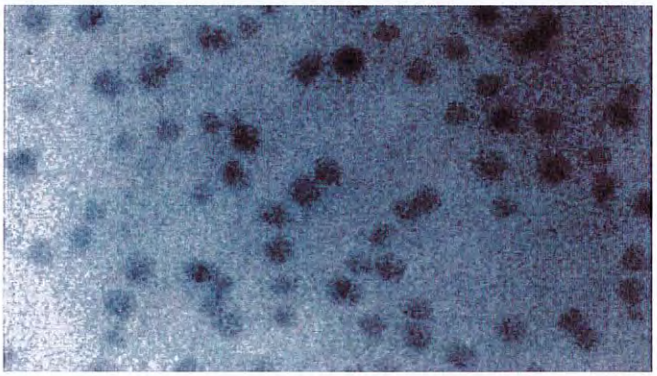


Figure A.20 : Example of an electron micrograph that is typically obtained from samples with type 5 scattering curves

A.6.3 Recommended Analysis Templates

Template Name (*saxprt)	Approximation of Size Distribution Dv(R)
Template_Type5_NoFit	None
Template_Type5_GaussianFit	Gaussian
Template_Type5_LognormalFit	Log-normal

A.6.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} at a relatively small value so as to just exclude the region where the sharp upturn or decrease of the (background-corrected) scattering curve is observed (if applicable), the "Higher Limit" q_{max} at 2.5 nm^{-1} (0.25 $Å^{-1}$) or higher; see the dashed lines in Figure A.18. The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 40 nm (400 Å) or smaller.

A.6.5 Remarks

This type of scattering curve usually leads to reliable analysis results with EasySAXS.

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.6.6 Available Example Data Files

Sample file:

- SAM5_PMMA_silica_nanocomposite_monodispersed.xrdml

Background file:

- BACK_PMMA_matrix.xrdml

NOTE: The example file is a measurement of a nanocomposites material, consisting of a PMMA matrix with incorporated, monodispersed silica nanoparticles. In such a case, the background measurement has to be performed on a reference sample of the PMMA matrix material without incorporated nanoparticles. The thickness of the reference sample must be the same as that of the nanocomposite sample.

A.7 TYPE 6 SCATTERING CURVES

A.7.1 Characteristics of the Scattering Curve

This type has two or more pronounced oscillations in the decaying scattering curve. At smallest angles, in the region around the first oscillation, a more or less pronounced peak may be superimposed. This is due to the influence of the structure factor S(q). A strong upturn of the scattering intensity towards smallest angles is often observed. An example is shown in Figure A.21 and Figure A.22.

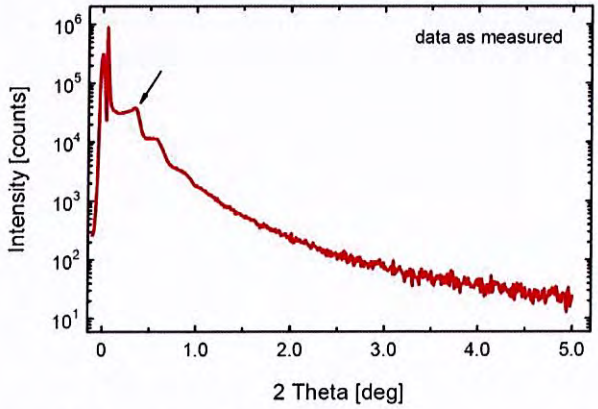


Figure A.21 : Type 6 Scattering Curve - as measured

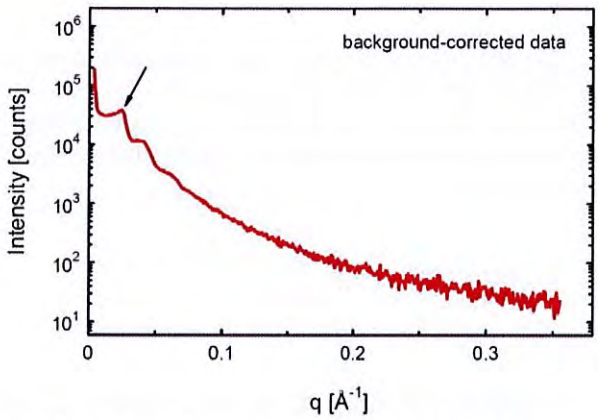


Figure A.22 : Type 6 Scattering Curve - background corrected

A.7.2 Sample Type

Particles with a very well-defined, narrow size distribution and a relative standard deviation of below 16%. Typical powder samples or concentrated dispersions of nanoparticles having such a narrow size distribution normally tend to have strong interparticle correlations (regular spatial ordering) that strongly influence the scattering profile, in particular at smallest angles.

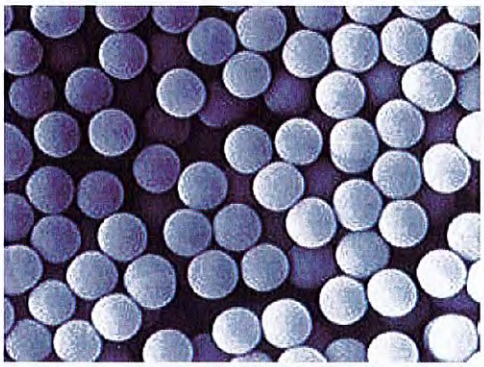


Figure A.23 : Example of an electron micrograph that is typically obtained from samples with type 6 scattering curves

A.7.3 Recommended Analysis Templates

None of the templates allows this type of data to be analyzed. However, in most cases at least a qualitative data interpretation is possible if you consider the following rules: the higher the observed oscillation frequency, the larger the particle size. The more oscillations observed and the more pronounced these are, the narrower the size distribution.

A.7.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Dv(R) analysis is not suitable, but fitting with the model "interacting spheres" could be tried.

A.7.5 Remarks

In most cases at least a qualitative data interpretation is possible if you consider the following rules: the higher the observed oscillation frequency, the larger the particle size. The more oscillations observed and the more pronounced these are, the narrower the size distribution.

A.7.6 Available Example Data Files

Sample files:
- SAM6_polystyrene.xrdml
- SAM6_silica_AS40.xrdml
- SAM6_silica_HS40.xrdml

Background file:
- BKD_mylar.xrdml (for all sample files).

Sample file:
- SAM6_PMMA_silica_nanocomposite_agglomorated.xrdml

Background file:
- BACK_PMMA_matrix.xrdml (for all sample files).

A.8 TYPE 7 SCATTERING CURVES

A.8.1 Characteristics of the Scattering Curve

Type 7 scattering curves have similar characteristics to types 3 and 5. But they refer specifically to weakly scattering samples (in particular dilute nanoparticle dispersions), for which relevant SAXS data can be measured only in a smaller angular range, up to approx. 3 deg 2Theta.

At smallest angles the scattering curve exhibits a convex curvature (Guinier's law). A small upturn or decrease of the scattering intensity towards smallest angles is sometimes also observed, even in the background-corrected data. This may be either due to minor agglomeration effects or due to a small error in the background-subtraction. Furthermore, possibly one or more distinct oscillations are observed in the decaying curve. The signal-to-background ratio at higher angles tends to be relatively poor. Examples of type 7 scattering curve are shown in Figure A.24 and Figure A.25.

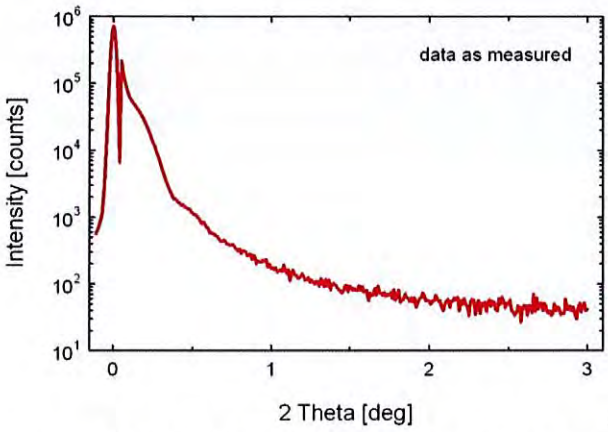


Figure A.24 : Type7 Scattering Curve - as measured

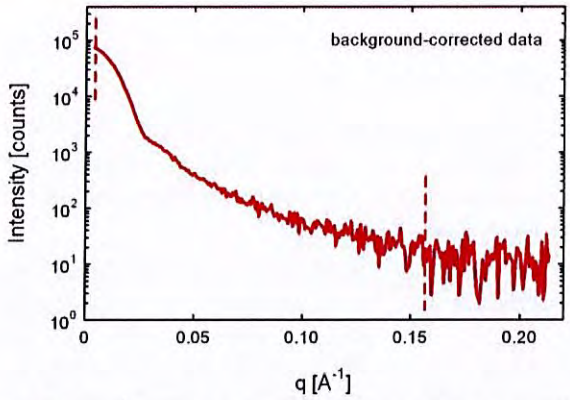


Figure A.25 : Type 7 Scattering Curve - background corrected

A.8.2 Sample Type

Samples containing distinct, isolated particles with a well-defined, narrow size distribution and a relative standard deviation of below 25%. The particles have a well-defined, spherical shape. They exhibit no or only minor interparticle interaction effects. This type of scattering curve is often observed with dilute dispersions of (non-interacting) nanoparticles in a liquid.

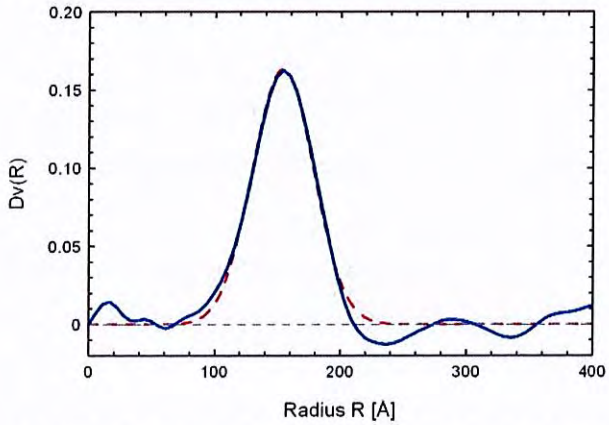


Figure A.26 : A typical size distribution from the analysis of type 7 scattering curves, together with a Gaussian approximation (dashed line)

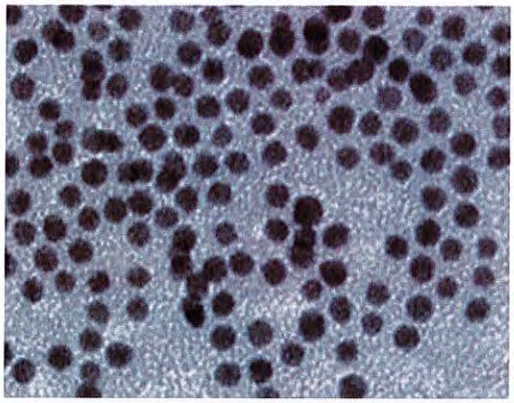


Figure A.27 : Example of an electron micrograph that is typically obtained from samples with type 7 scattering curves

A.8.3 Recommended Analysis Templates

Template Name (*saxprt)	Approximation of Size Distribution Dv(R)
Template_Type7_NoFit	None
Template_Type7_GaussianFit	Gaussian
Template_Type7_LognormalFit	Log-normal

A.8.4 Recommended Settings for the Evaluation of the Size Distribution in the Interactive Mode

Set the "Lower Limit" q_{min} at a relatively small value so as to just exclude the region where the sharp upturn or decrease of the (background-corrected) scattering curve is observed (if applicable), the "Higher Limit" q_{max} at around 2.0 nm^{-1} (0.20 $Å^{-1}$); see the dashed lines in Figure A.25. The maximum particle radius "R_max for evaluating Dv(R)" is to be set at 40 nm (400 Å) or smaller.

A.8.5 Remarks

This type of scattering curve usually leads to reliable analysis results with EasySAXS.

The templates were created under the assumptions that all particles have the same electron density, a spherical shape and an essentially homogeneous structure. If these assumptions are not met, the obtained results are NOT reliable.

A.8.6 Available Example Data Files

Sample files:
SAM7_ColloidalSilica_B4013
SAM7_ColloidalSilica_B4022
SAM7_ColloidalSilica_B1550
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to1
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to1
SAM7_bimodal_ColloidalSilica_B4022_B1550_2to1
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to2

Background file:
BLANK_DispersionMedium_water

NOTE: The example files are measurements of nanoparticle dispersions in water. In such a case, the background measurement was performed using the same capillary, but filled with pure water.

PANalytical

Appendix B

Template Files

NOTE: When starting EasySAXS for the first time, the available templates (*.saxsprt) will be copied automatically to the following folder.

C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Template Files

Samples differing significantly with respect to the range of particle or pore size, size polydispersity, agglomeration behavior etc., show characteristic differences in their scattering curves and accordingly may be classified in different types as detailed in Appendix A.

For each type there exist optimized parameter settings for the SAXS data analysis. These different settings that are to be applied in a preprogrammed sequence of analysis steps within EasySAXS are stored in the various analysis templates (*.saxsprt) supplied by PANalytical.

It is essential to select the most appropriate template for the analysis of a given measured scattering curve (*.xrdml). To do so, one has to compare the features observed in the scattering curve with those described in the Appendix A of this Quick Start Guide for the various types. This comparison should preferably be done based on the background-corrected curve as displayed in the analysis report, even though in many cases the curve as measured may also be used.

For each type of scattering curve, one may choose a dedicated template for running the analysis. One may choose between fitting the size distribution with either no function, with a monomodal Gaussian or with a monomodal log-normal distribution. Multimodal distributions cannot be fitted successfully.

The templates assume that the particles or pores are spherical and homogeneous. Strongly anisometric or inhomogeneous particles cannot be analyzed with these templates.

For each analyzed sample, a Size Distribution Analysis Report (filename.pdf) and the Size Distribution Data File (filename.saxsvdf) will be stored in the working directory.

NOTE: For a SAXS measurement to be analyzed with any of the templates listed in the table below, it is essential that it was performed with the PANalytical Data Collector software in 2Theta scans with –0.115 deg as start angle and 5.005 deg (3.005 deg for Type 7) as end angle, and in increments of 0.010 deg. The templates are not compatible with data that were acquired with different settings. As a consequence, they also cannot be used for the analysis of experimental data that were acquired with the ScatterX^{78} SAXS/WAXS attachment.

To run the template of your choice, click on the template file in the Windows Explorer. This will open EasySAXS with this template loaded. Then follow the instructions given in chapter 2.

Table B-1 : Analysis Templates Delivered with EasySAXS

Template Name (*.saxsprt)	Applicability	Approximation of Size Distribution Dv(R) by:
Template_Type1&2_NoFit	Types 1 and 2	None
Template_Type1&2_GaussianFit	Types 1 and 2	Gaussian (often most suitable for Type 1)
Template_Type1&2_LognormalFit	Types 1 and 2	Log-normal (often most suitable for Type 2)
Template_Type3_NoFit	Type 3	None
Template_Type3_GaussianFit	Type 3	Gaussian
Template_Type3_LognormalFit	Type 3	Log-normal
Template_Type4_NoFit	Type 4	None
Template_Type4_GaussianFit	Type 4	Gaussian
Template_Type4_LognormalFit	Type 4	Log-normal

Table B-1 : Analysis Templates Delivered with EasySAXS (Continued)

Template Name (*.saxsprt)	Applicability	Approximation of Size Distribution Dv(R) by:
Template_Type5_NoFit	Type 5	None
Template_Type5_GaussianFit	Type 5	Gaussian
Template_Type5_LognormalFit	Type 5	Log-normal
No template available	Type 6	
Template_Type7_NoFit	Type 7	None
Template_Type7_GaussianFit	Type 7	Gaussian
Template_Type7_LognormalFit	Type 7	Log-normal

PANalytical

Appendix C

Project Files

NOTE: When starting EasySAXS for the first time, the available project files (*.saxsprj) will be copied automatically to the following folder.

C:\Users\<currentuser>\Documents\PANalytical\EasySAXS\Project Files

For each of the template files (*.saxsprt) delivered with EasySAXS, also the corresponding project files (*.saxsprj) are provided. Corresponding template and project files have the same file name, and only differ in their file name extensions.

By opening a project file in the Interactive mode of EasySAXS, you may inspect all the specific settings that are used in the corresponding template file. In a project file you may also modify the settings used in the analysis so as to further optimize them for your specific samples of interest, then recalculate the objects, and finally save this as a new template file.

Detailed information about how to create and modify a project file and about how to create a template file can be found in Chapter 3.

PANalytical

Appendix D

Example Measurement Files

D.1 FILE LOCATIONS

NOTE: When starting EasySAXS for the first time, the available example measurement files (*.xrdml) will be copied to the following folder:

C:\Users
<currentuser>\Documents\PANalytical\EasySAXS\Example Measurement Files

Several example measurement files (*.xrdml) are delivered with the EasySAXS analysis software. They may be used for doing exercises in data analysis with EasySAXS in either the Automatic or Interactive Mode of the software.

These files contain SAXS curves of different types. The classification scheme in different types is detailed in the Appendix A.

Measurements from nanopowders, nanoparticle dispersions, porous materials and nanocomposites are included. To demonstrate the good capabilities of EasySAXS in analyzing multimodal particle size distributions, a few example files of this type are also given.

D.2 MEASUREMENT DETAILS

The example files were measured on a PANalytical XRD system with an experimental setup in air, using a focusing mirror and the PIXcel detector in 0D (receiving slit) mode. When analyzing these data with EasySAXS, make sure to select the "Default.cfg" configuration file in "*Preferences – Instrument*".

All measurements were done in 2Theta scans with the following settings:

Start angle	–0.115°
End angle	5.005° (3.005° for liquid nanoparticle dispersions)
Step size	0.010°
Measurement time	20 min. for powder samples, 60 min. for dilute dispersions

These data files are thus compatible with the analysis templates that are delivered with EasySAXS.

The powder data were collected fully automatically using an automatic sample changer in combination with the reflection-transmission spinner and an automatic beam attenuator. The activation range of the beam attenuator was set from –10° to 0.04° 2Theta. The first measured data point with the beam attenuator removed was at 0.05° 2Theta.

D.3 FILE NAMING CONVENTIONS

File name prefixes were used so as to differentiate a Background measurement from a Sample measurement in the following way:

File names denoting Background measurements have the prefixes

"BKD"	for all powder samples
"BACK"	for the nanocomposite sample
"BLANK"	for liquid dispersions

File names denoting Sample measurements have the prefix

"SAM", followed by a number.

Sample measurements with scattering curves classified as "Type 1" were assigned the file name prefixes "SAM1", "Type 2" data the prefix "SAM2", etc.

A list of the available example measurement files is given in the tables below.

Table D-1 : Background Files

File Name (*.xrdml)	Chemical Nature
BKD_mylar	Background for all files with scattering curves of types 1–4 (empty sample holder with mylar foils)
BACK_PMMA_matrix	Background for the nanocomposites (PMMA matrix without nanoparticles)
BLANK_DispersionMedium_water	Background for all liquid dispersions with scattering curves of Type 7 (capillary filled with the dispersion medium)

Table D-2 : Sample Files

File Name (*.xrdml)	Chemical Nature
SAM1_titania_5std	titania nanopowder
SAM1_titania_10std	titania nanopowder
SAM1_titania_0501	titania nanopowder
SAM1_titania_0502	titania nanopowder
SAM1_titania_PAN	titania nanopowder
SAM1_titania_bimodal_PLVU0075_10std_1to1	titania nanopowder, bimodal size distribution (1 : 1)
SAM1_titania_bimodal_PLVU0075_10std_1to2	titania nanopowder, bimodal size distribution (1 : 2)
SAM1_titania_bimodal_PLVU0075_10std_2to1	titania nanopowder, bimodal size distribution (2 : 1)
SAM1_porous_alumina_pm103	porous alumina nanopowder
SAM1_porous_alumina_pm104	porous alumina nanopowder
SAM2_aluminiuimoxide	aluminium oxide nanopowder
SAM2_antimonytinoxide	antimony tin oxide nanopowder
SAM2_ceriumoxide	cerium oxide nanopowder
SAM2_copperoxide	copper oxide nanopowder
SAM2_tinoxide	tin oxide nanopowder
SAM2_zincoxide_20std	zinc oxide nanopowder
SAM3_porous_silica_dav635	silica gel
SAM3_porous_silica_gd12	silica gel

Table D-2 : Sample Files (Continued)

File Name (*.xrdml)	Chemical Nature
SAM4_titania_PLV0071	titania nanopowder
SAM4_titania_PLVU0075	titania nanopowder
SAM5_PMMA_silica_nanocomposite_monodispersed	PMMA-silica nanocomposite; monodispersed nanoparticles
SAM6_polystyrene	powder of polystyrene nano-spheres
SAM6_silica_AS40	powder of silica nano-spheres
SAM6_silica_HS40	powder of silica nano-spheres
SAM6_PMMA_silica_nanocomposite_agglomerated	PMMA-silica nanocomposite; agglomerated nanoparticles

Table D-2 : Sample Files (Continued)

File Name (*.xrdml)	Chemical Nature
SAM7_ColloidalSilica_B4013	colloidal silica @ 3wt%
SAM7_ColloidalSilica_B4022	colloidal silica @ 3wt%
SAM7_ColloidalSilica_B1550	colloidal silica @ 3wt%
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to1	colloidal silica @ 3wt%; bimodal size distribution (1 : 1)
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to1	colloidal silica @ 3wt%; bimodal size distribution (1 : 1)
SAM7_bimodal_ColloidalSilica_B4022_B1550_2to1	colloidal silica @ 3wt%; bimodal size distribution (2 : 1)
SAM7_bimodal_ColloidalSilica_B4022_B1550_1to2	colloidal silica @ 3wt%; bimodal size distribution (1 : 2)

PANalytical

Appendix E

Configuration Files

E.1 INTRODUCTION

Configuration files (*.cfg) contain information that is related to the experimental setup that was chosen for performing SAXS measurements on a PANalytical XRD system. This information must be taken into account when analyzing SAXS data using EasySAXS.

The configuration files delivered with EasySAXS can be found in the folder: *C:\PANalytical\EasySAXS.*

Before performing data analysis with EasySAXS, you must make sure that you have selected the correct "Instrument configuration file" in "Preferences - Instrument".

E.2 CONFIGURATION FILES

Select the appropriate configuration file using the information given in the table below.

Configuration File Name (*.cfg)	Applicability
Default	Any of the experimental SAXS setups described in the "SAXS/WAXS Application Guide" that DO NOT include any of the following items: ◆ 2-bounce hybrid monochromator ◆ incident beam slit system ◆ secondary 0.04 rad Soller slits. Particularly, use this configuration file for ScatterX[78].
Configuration2	Any of the experimental setups described in the "SAXS/WAXS Application Guide" that DO include one or more of the following items: ◆ 2-bounce hybrid monochromator ◆ incident beam slit system ◆ secondary 0.04 rad Soller slits.

NOTE: To analyze any of the Example Measurement Files (see Appendix D), select "Default.cfg" as the configuration file.

4022 339 16083
Printed in The Netherlands

March 2014
Edition 3