Powder4

A short help file

- About the program
- About this version and author
- Features
- Available forms (or windows...)
- The menus
- Notice

About this program. Some history.

This program consists of a collection of tools for powder X-ray powder diffraction. This is a variant of a previously one, known as Powder3 , 2..etc, and down to Convert v3 which was capable of various data file conversions. These functions are still available in this program. The goal is to write a comprehensive package for the X-ray diffraction on powders, I have to say that this version is far from that goal. This small help file should give some info about this program, probably a heuristic method will be better for the user. However, at this moment the conversion function is still the main function of this program. A graphic interface for peak hunting and peak profile analysis was also added.

Since I start working on X-ray powder diffractometry I noticed that almost each program required a different form of the data file (quite normal, different authors, different programs). There was quite a pain to convert the data file from an instrument to an appropriate format for use with another software. So, not willing to do the conversion by hand, I wrote some small routines in some variants of Basic and/or FORTRAN (which has a very good Format function) to do the job. There were no fancy interfaces or help. I had to write then nicer routines for some of my colleagues. And I had then the (good ???) idea to write a more general program for such types of conversion. This was Convert 1.0. That version was written when doing my PhD at Université Paris Sud (1994; this wasn't what I supposed to do...!); it was written in a french version of Visual Basic 3.0. Another version and a bug hunting followed when I was working at University of Bucharest. The version 3 was written in '97 at University of Tokyo (Visual Basic v5, a japanese version; so some error messages may look funny on your computer).

About this version and author

For the exact version look in the About form (a window is called here Form), the help file may not be up to date. ***Due to a severe lack of time as well as a lack of motivation (I have no ideea who is using the program anymore because there is no feedback from the users) the development of this program will probably stop.***
This is the version 4 of Powder, a 32 bits version and can run in Windows 95 or NT 3.5 or, I hope, above. I didn't find any bugs in this version but I think that it may contain some (an even number...). If you find any, hopefully sooner, please let me know and I will try to correct them. I can not guarantee a support to this program since I have a job; that will depend entirely on my free time. However, before asking me something read the help file and follow the advises in the notice.

The author of this program is:

Dr. N. Dragoe
LPCES-ICMMO, Université Paris Sud
Bat. 410
91405 Orsay, France

nita.dragoe@lpces.u-psud.fr

back to top

Features

It is useful mainly for people doing X-ray diffraction experiments although the program can be used with different data types or instruments (I am using it for conversion of UV/VIS spectra ASCII data where the X values are in a decreasing order; so a negative step can revert this...).
This program can read and write some of the data files I know : DBWS, GSAS, Philips, Siemens, Scintag, ASCII files, MXP18 data, WPPF.... Some other files can also be read with this program.
A quite different function was added recently : a unit cell refinement calculations and HKL generation functions.

Forms/Pages

There are several different windows in the program, called here **Forms** or **Pages**.

About-which contains the version number and with the program starts. This form can be viewed at any time from the Help/About menu. To start the program you must click on the icon or in the text area in the form.

Main form- which is practically the old Convert 3 program.

Merge files - two functions for merging files, useful for variable counting time (hereafter VCT) data acquisitions

Graphic - GUI data management, background removal, peak hunting, etc

Rietveld/gDBWS - Rietveld file preparation and graphical interface

UnitCell-is a form added for utilities related to unit cell refinement

Main form - menus

The main form of the program has one text region where some info is printed from time to time and few commands available in a menu. This area is called “report pad” , refer to the information printed here during the execution of this program. Most of the output, except the saved files, will be listed here.

There are few menus in this form. Click on the appropriate keyword for more information.

File

Open

Save as

Merge XY

Merge XYZ

Batch mdi -> GSAS

Save Report pad

Shell to Notepad

Quit

Header

I don't have a header...

Ignore first ...

Graphic

Unitcell

Rietveld

Tools

Read HSTDMP from GSAS lst file

Read REFLIST from GSAS/rfl file

Compute d/angle

Remove strange characters

Truncate data

Swap X, Y

Show report

Clear report

Help

About

Contents - html file

Author WWW page

CCP14 web page

back to top

Main Form -> File

This menu contains the following sub menus:

Open

Save as

Merge XY

Merge XYZ

Batch mdi -> GSAS

Save Report pad

Shell to Notepad

Quit

Main Form > File -> Open

This command open a raw data file and read the data in memory. You should choose the file format you have otherwise strange effects may occur. The program accepts the following commands (as sub-menus) while in File-Open menu:

DBWS file

GSAS-CW ESD and STD

LHPM

Philips PC-UDF

Riet 7

Scintag data

Siemens, ascii

WPPF/Profit

Line

The first value as Y

The first 2 values as X, Y

The first 3 values as X, Y, Z

Y-free ascii

XY free ascii

XYZ free ascii

Mac Science (Win NT), binary

MXP18 (unix) binary

Philips RD/SD, binary

Custom

DBWS file

In DBWS format there is a first line with : starting 2theta, step, final 2theta and a title (in FORTRAN is 3F8, A56). This is the first line in the file, called in *Powder* as **header.** Note that, like in all cases in this program, the starting 2 theta is the first value and not start_2theta+step as is the case elsewhere !!
The rest of the file are in 8F7,1X format. Only the first 56 spaces are read since some old data files have a comment or card number in the columns 72 through 80 (this area was reserved for comments in the old days). The values read from the header will be later used to generate the X values, if necessary. Although it reads a title here, the program will prompt you to set or modify the title when trying to save the data. You can use this command only for data files having 8 values on a line, otherwise you may obtain strange results.
Refer to the Header commands if you want to find out how to ignore the header read from this file. This may be useful when wanting to change the value of the 2theta (although I can not see the reason for doing this).

GSAS - CW ESD and GSAS - CW STD

GSAS is the well known program written by A.C. Larson and Bob von Dreele from LANL.
You can read a GSAS-ESD constant wavelength. In *Powder* there are some limitations regarding Gsas files. Some of the records (except starting two theta, step and the number of points) are ignored in this program. For instance "Instrument parameter" which is optional in the Gsas file is completely ignored here, as well as BINTYP info (read the GSAS manual for more information). The data is read as 5 values and corresponding ESD's on a line until the EOF marker is found. The normal GSAS data may have few BANK records in a file, here the first BANK record is read.
Same general comments are available for STD file; ten values in each line are accepted here. For use of the GSAS files I strongly recommend to read the instructions in the GSAS User Manual. Note that in the new version of GSAS a file converter is available.

LHPM

This format is similar to that of DBWS but having 10 data on each line. Here only the first 64 characters on a line are considered.

Philips/Panalytical PC-UDF

With this function you can read a Philips (now Panalytical) UDF file. The UDF files which I know have a header (about 20 lines) and then the diffraction data as eight characters in a row, separated by commas. *Powder* is looking for the **RawScan** statement then reads all the data. From the UDF header most of the fields are ignored, this applies as well when writing the UDF file. If you don't want to use a header, the same file can be read by **Y, free ascii** command.

Riet7

This format has a header on four lines with a title and some other details (monochromator, date, etc). The important part is on a line with starting 2theta, step and ending 2theta each having 8 characters. Then the Y data are read as one value per line, the 2theta are computed from the header info.

Scintag data

This file type is similar to that of Philips, i.e. a big header which is mostly ignored by *Powder* and 2theta, intensity and esd. Although it is not convenient, the same file can be read ignoring the header, as XYZ free ascii.

Siemens ascii

The format of this file is quite similar to the Philips or Scintag ones, a header followed by data written as 8 values on each line. As before, from the header information only some fields are requested.

WPPF/Profit

WPPF and Profit are two different programs written by Hideo Toraya. These programs can read two distinct data files either as 8 or 10 values in a line. The program detect the data type you have and read it either as 8 or 10 values in a row, as appropriate. From the header of this file, some info as the wavelength or counting time are ignored.

Line

the first value, as Y
the first 2 values, as XY
the first 3 values, as XYZ

On this menu, three commands are available giving the opportunity for reading only the first value, the first 2 values, or the first 3 values from each line. This is useful only if there are more data in a row than x, y. These commands do not allow a header, you will be prompted for additional information if necessary. If the data file contains some titles, use the available commands to ignore these lines, see the Header menu.

Y; free ascii

You can read Y values no matter the format is, as long as the data are separated by comma or tab or spaces and no empty lines are in between values.

X, Y; free ascii

Read X and the corresponding Y in any format as long as the values are in pairs (i.e. in each line the final value must be for an Y value.)

X, Y, Z; ascii free

Read X, Y, Z from any ascii file. Z is usually considered the ESD value for each Y. However, in this program no ESD estimations/correction are made, if necessary Z will be filled with 1.0.

MAC Science NT, binary

This is a binary data file from the Mac Science diffractometers on NT systems. Read some details from a header as well as X and Y. Instructions relative to ASCII file do not apply here (for instance, ignore lines, etc), this is a binary file.

MXP-18 binary

This is a binary data file from the Mac Science MXP18 rotating anode diffractometers.
Read some details from a header as well as X and Y. Instructions relative to ASCII file do not apply here (for instance, ignore lines, etc), this is a binary file.

Philips/Panalytical RD/SD

A different format came from Philips and now is in use with the recent Panalytical diffractometers : the RD and SD ; these are binary files and **Powder** is able to read the RD and SD 3 to 7. Note that recently, an additional format is available from Philips/Panalytical the **.xrdml**. As of now there is no function associated with this format. I recommend the use of a UDF file format instead of the RD files.

Custom

This function, coupled with the **Ignore lines** command, allows the reading of files on columns (or even diagonally !!). A small window will ask for the number of point **n1** to be read following by “skipping” **n2** points. For instance, reading an ASCII file having three columns : **n1**=1 and **n2**=2 will allow the reading of the first column while **n1**=3 and **n2**=2 will read the third one. If **n2** is different than 2 then the data read is on “diagonal”. Give it a try but make sure to check the output (note that some UNIX characters may be considered “data so the output of this function may be affected by this; a solution is to apply the “**Remove strange characters”** function to this kind of files).

back to top

Main form -> File -> Save As

Write the *last set of data read* in the selected format. If necessary, the program will ask some details about the data, such as the 2 theta... Some notes applies for each format. The program accepts the following commands (as sub-menus):

- DBWS
- GSAS - CW ESD GSAS - CW STD
- LHPM
- Philips UDF, Siemens, Scintag
- RIET7
- WPPF/Profit
- Y, XY or XYZ ascii
- DPLOT file

DBWS file

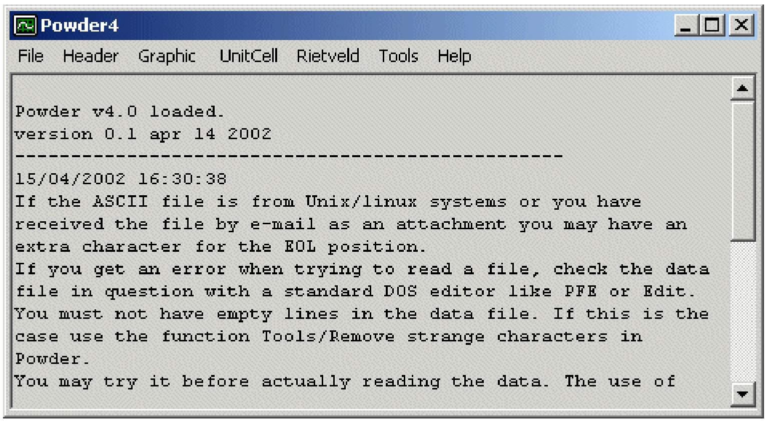
This command save a file in a DBWS file format.

GSAS - CW ESD , CW STD

The output file for GSAS is limited to some constants. For more advanced data type use the built-in convert program from the GSAS package (or edit the file manually). No "Instrument parameter" is written in the output file, the BANK number is always 1. Check the data file for 80 characters in length, GSAS requires this (*Powder* fill with spaces up to 80 chars but if you edit this files with other editors check if you still have 80 characters on each line).

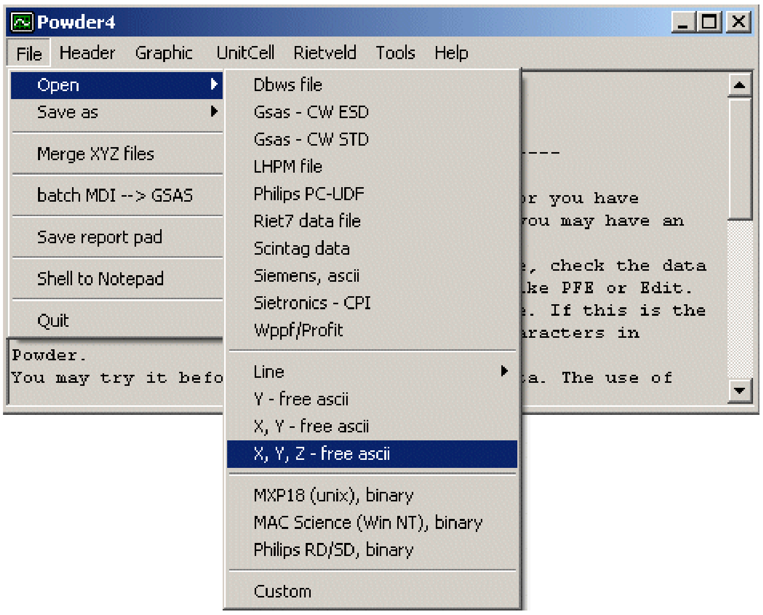
A small example of using GSAS conversion follows (as shown on www.ccp14.ac.uk) :

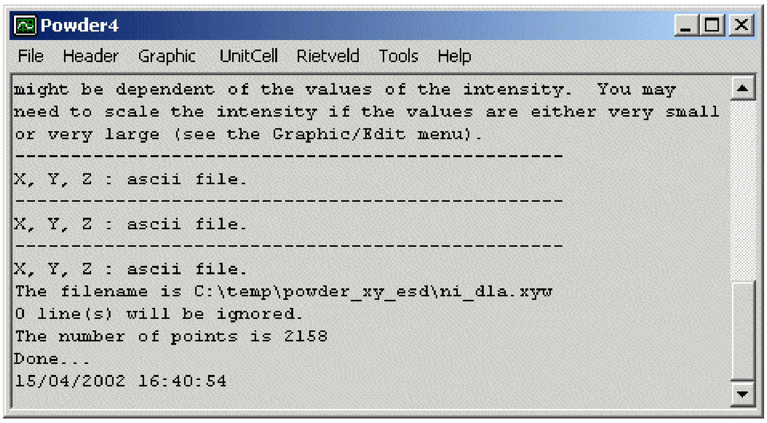
With the latest Powder4 for Windows by Nita Dragoe will allow you to convert an x,y,esd powder diffraction file in column format into GSAS ESD format.
First run Powder for Windows



```
Powder v4.0 loaded.
version 0.1 apr 14 2002
-------------------------------------------------
15/04/2002 16:30:38
If the ASCII file is from Unix/linux systems or you have
received the file by e-mail as an attachment you may have an
extra character for the EOL position.
If you get an error when trying to read a file, check the data
file in question with a standard DOS editor like PFE or Edit.
You must not have empty lines in the data file. If this is the
case use the function Tools/Remove strange characters in
Powder.
You may try it before actually reading the data. The use of
```

Click on **File, Open, X Y Z Free ASCII** and select the column format data file.

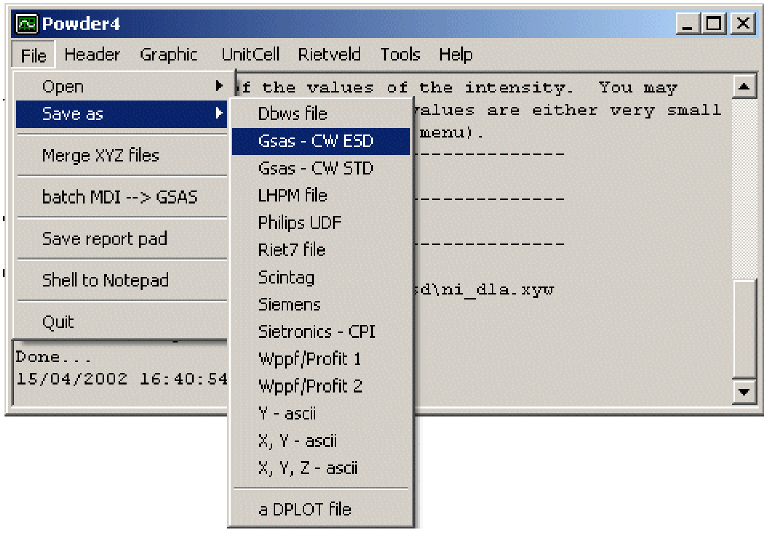




```
might be dependent of the values of the intensity.  You may
need to scale the intensity if the values are either very small
or very large (see the Graphic/Edit menu).
--------------------------------------------------
X, Y, Z : ascii file.
--------------------------------------------------
X, Y, Z : ascii file.
--------------------------------------------------
X, Y, Z : ascii file.
The filename is C:\temp\powder_xy_esd\ni_dla.xyw
0 line(s) will be ignored.
The number of points is 2158
Done...
15/04/2002 16:40:54
```

Click on **File, Save as, GSAS - CW ESD**. Provide the filename (preferably with a .gsa or .gs file extension.) **Done!**

It is advisable to look at the resulting file and compare the top and bottom with the original data to see the conversion did go OK.



LHPM

The LFPM format is similar to that of DBWS but having 10 data on each line.

Philips UDF, Siemens ascii and Scintag

In Philips, Siemens and Scintag files, most of the header is fixed, you can manually change the values to your preferences.

WPPF/Profit 1 or 2

Both format types in WPPF/Profit neglect the wavelength value and the counting time. The wavelength will be always of copper Kalpha 1. You can change later this value, see the Wppf or Profit user manual for this.

Riet7

This format has a header on four lines with a title and some other details (monochromator type, date, etc). The important part is on a line with starting 2theta, step and ending 2theta each having 8 characters. Then the Y data are saved as one value per line. As for the UDF format the info in the header are always the same and not relevant to this program. You may need to change these values by using a classical a text editor.

Y; X, Y and X, Y, Z, ASCII

Not much to explain here, the output is in ASCII with one record per line. The first line is a date stamp of the **Powder4**.

Dplot file

This command save the data file in a **Dplot** file. Dplot is a very good freeware program for scientific graphs. I quote from the Dplot help file: "DPLOT is a general purpose X-Y plotting program that allows you to view, manipulate, and produce hard copies of data in a variety of formats". The package can be found at http://www.dplot.com

back to top

Main Form -> Header

There are two submenus in the **Header** option :

I don't have a header

Selecting this option (it is ON if there is a check mark on the menu) allows the reading of data file which do not have a header definition. This has no other meaning that instructing the program not to “look" after the 2theta, and so on.

Ignore first : none...

With this option, ***n*** lines from data file can be ignored. The user should choose how many lines to be ignored when reading a data file. For instance, if you have an ascii data file and in the first 3 lines there are some comments, you can choose Ignore first then input the value 3. In this case the program will ignore the first 3 lines in your data file. You can ignore as many lines as you want (limited to 32000 though !).
Another possible use: if you want to read a DBWS file and modify the 2 theta values, select ***I don't have a header*** option and then ***Ignore 1st line***. The program will skip the 1st line and then start to read data directly, not searching for the X values. It will ask for it at a later occasion, if necessary.

back to top

Graphic

If the program has successfully read a file the data in memory is kept and this function will open the graphic routine. If necessary the X data will be asked as starting 2 theta and step 2 theta. Note that by opening the **Graphic page** the main page of the program will, temporarily, disappear. For more detailed explanations on how to use the graphic routines see the tutorial section.

Only the first 32000 pairs of data are shown in the graphic routine. If you have larger datafiles you can select, by removing some parts of the file with the command **Tools** -> Truncate data, which region to be seen in the graph.

Warning : depending upon the system you are using, the pegraphs.OCX may be required to be registered with regsvr32.exe *.ocx.

back to top

Unit cell

This is an independent routine in this program, no data is necessary to be read before starting these functions which are related mostly related to the indexation and refinement of the unit cell. Some practical details about these utilities are given in the tutorial, more can be found here.

back to top

Rietveld

This function is not well tested and it is not anymore under development. I had no time to make a help file for it, it is a GUI DBWS version with Rietveld graphs capabilities. Give it some try,...but unless you are familiar with DBWS I recommend you to check Winplotr/Fullprof or Expgui/Gsas programs.

back to top

Tools

The menu **Tools** contains the following commands:

Read HSTDMP from GSAS lst file

Read REFLIST from GSAS/rfl file

Compute d/angle

Remove strange characters

Truncate data

Swap XY

Show report

Clear report

back to top

Read HSTDMP from GSAS .lst file and Read REFLIST from GSAS .rfl file

These commands are related to GSAS package. The first one request a GSAS results file, *.lst. It will search in this file the occurrence of "Program HSTDMP" string. When it can find this, a file named _hstdmp.dat is created in the application directory (Warning - it will erase an old _hstdmp.dat file). This file contains the data output from HSTDMP program. If two or more statements are found the data set will be attached one after another without a separator. This command may depends on the GSAS version you have. You can use the _hstdmp.dat file for another application (your preferred graph editor..)

Read Reflist from GSAS/rfl file request a .rfl file written by GSAS. This file can be saved by the Tools/Reflist menu command with the option R/ascii file. With this function you can read h,k,l Fo and esd(Fo) from Gsas and save a Shelx .hkl file (for structure determination or use of these data with another software). The program *Powder* will ask for a batch number which is an optional field in Shelx (for those making batch refinements). The output file will be merged. The format in the output file is as required by SHELX 97, I don't know if works with older versions but I guess it does so..

Remove strange characters

This option allows to skip all characters from the extended code and from command code (smaller than 30). It is useful when the data come from some Unix software which have a different ascii character set, as my MXP18. It is a time consuming option (this is relative, 5 seconds with 5000 data on my laptop). To be honest, often I use this command without an obvious reason (some files, depending on the programs saving them, give errors when read by Powder. I guess that this is related to the EOL chars).

Truncate data

This option allows to (irreversibly !) delete some parts from the data file in memory (no changes are ever made to the input files). It may become useful if you want to analyse a smaller region of the data file or in the rare case of data files larger than 32k pairs of data. Note that the program (in theory) can read 2 Gb sets of data !!

Compute d/angle

Toggle a small utility for computing d or diffraction angle for a given radiation. When working with this routine, the report -a kind of activity log- is not visible anymore; however it is still there (if you want to read the report toggle OFF this function and ON the Show report command)

Swap X, Y

This function interchange the X and Y data in memory. Generally it is not useful for X-ray diffraction data but for other type of data file.

Show report

In the main form, a text area shows what is going on, what was the filename, how many lines it found in it, and so on. All output from the Unit Cell routine is directed here. May also be useful for some debugging purposes.
You can toggle the visibility of this report pad (it is still in memory). From time to time (when this pad has 32kb) the report will be appended to a file called _rpbackup.dat and will be erased from the screen. This file is in the working directory (Warning- NOT the application directory); take care and clean this file from time to time as it can get very large...

Clear report

A self explaining command, be careful : no UNDO is available.
You can also clear or copy/paste data to this report at any time. Use the Right_Click button of the mouse to see a PopUp Menu with common commands under Win95 (as Select, Copy,...).

back to top

Help

About

Shows the About window of the program. Nothing interesting there except the version number of the program (or the author ?..). When you have trouble and want to ask some questions (***after** reading this help file and **after** you have already let me know you use this program* !), report this version number.

Contents-html file

Open this Help file in the default browser. You can also edit this file and change whatever you like (I will be glad to put your comments in the future help files!)

Author WWW page

Open the default browser and go to my WWW page (there might be an update or another version of this program). The page location is read from the ASCII file _PwdInternalData.txt from the application directory. You can edit this file and change the URL of the author page. If this file isn't there, the program will try at the address internally stored.

back to top

Unit Cell /Powder Cell

This form will help you in refining a unit cell and to generate hkl and d's. It is also useful when reading some data (it can read a specified column from the file, etc..).

There are few menus which will are discussed later in this file. The unit cell, both for cell refinement and hkl computing, can be selected from the listbox named **Cell**, just click on it. In its right side there is a similar one where you can select the **Wavelength** or, if you use something rare -as a synchrotron- put the actual value in the associated text field.

The grid in the middle of the image is referred here as **Data grid**, where you -or the program- will put some data. You can change the data type in the column 4 just by clicking on the top of the grid (you can toggle between d/A, theta, 2 theta or E/keV for energy dispersive data). You can put a lot of data here but you have to be patient, in this case the program is rather slow, although I am not sure about the upper limit of the MSFlex grid, I guess you can put more than 15000 rows...). For refinement you have to insert data there, no empty lines allowed, the order is meaningless.

The grid in the right part of the form is the standard grid where you should insert the expected and observed values for your standard material.

Important note: use the same data type as in the data grid, therefore you can put here, d, theta or 2theta depending on what you have in the data grid..

The data inserted in the standard grid will be used for a calibration, if and only if, the associated check box is ON (named *Use standard*). The correction for 2theta is made with an interpolation polynomial for the difference in theta/2theta or d relative to theta. The program computes such a difference with a pseudo-Inverse routine i.e. an over-determined system (frankly speaking, a least squares!).

You should remember some mathematical basics, the polynomial interpolation may give strange results. However the corrections are printed on the report pad in the main form; check them out. In this version, the polynomial degree can be comprised between 0 and 8; although I recommend a value between 1 and 4. Two observations here:

-do not use a high degree polynomial (I suggest you not to go over 4 or 5) unless you have a lot of data somehow homogeneously distributed over the 2theta domain (about 20 to 30 data for a good conditioning) and not too far from the initial and final values.

-0 polynomial is not a zero error if the inserted data is d.

There are checkboxes associated with text fields, such as a, b, c...etc. In the refinement, the meaning is to refine or not the corresponding parameter; there is no meaning for them in the hkl generation routine. The lower left part shows a text field meaning refinements **Cycles**. In this example, when the method of refinement is Least squares (see later), 10 refinement cycles will be made.

Another important note: by weight In this program the value of w in the relation is considered:

w(Yo-Yc), so don't put the esd's on the column 5 but 1/esd.

The available menus are (they are quite self-explaining) :

File

Ignore : none

Similar to the Ignore function from the Main form. The program will ask how many lines from a data file should be ignored. Usually, the data files contains some headers and comments, by this command one can import data by ignoring few lines and thus avoiding editing and saving it under other name, etc.. Initially this menu will have the caption "Ignore:none" and after activated this menu will show how many lines will be ignored.

Import ASCII : 4 or 5 values on line

This function will import data from an ascii file and put these data in grid. Note: each record should contain four data or five data (the last is for a weight). The selection between the two option is made with submenus. These data can be either each record on one line either sequentially in an ascii file. If the four values are read, the weight column will be filled with 1. Special functions for editing the weight are available in the **Edit** menu.

If the data file are in a different format it still can be read with the command **Input data in column**, available in **Edit** menu.

Import calibration data

A two column ascii file will be read into standard grid. **Warning:** *If the data in the standard grid are not the same type as those in the data grid, you'll get a mess.*

Export data grid to graphic

Export data grid to ASCII file

The data from grid can be saved into an ascii file. This function is useful either for storing the datafile for further reference or to export a generated hkl, d pattern.

Export standard grid to ASCII file

Quit

Close the Cell refinement form and loose all the data you have in grid.

Data

Sort

Set data type to : 2 theta, theta, d, energy

Change data type to : 2 theta, theta, d, energy

Insert row

Delete row

Delete column

These functions refer to the data grid only. You can insert or delete rows in the grid, if you want to modify your data (delete column will delete data but not the column...). Delete row is in fact equivalent to a **Cut** command and is very useful because you can not have empty lines in the data grid (the program will "read" how many data you actually have by checking an empty line.)

Add to Column
a constant value
a column
1/column
to weight:1
Multiply Column
with a constant value
with column
with 1/column

These commands are similar, they will add a value (or multiply with one) to a column in the data grid. The program will ask which column you want to modify (H is considered the first column). You can add or multiply weight with the value which is in another column (but the same row). By this mechanism you can change the weighting scheme for the refinement.
Take care: if you put 0 in the weight column that data row will be neglected in the refinement.

Input data in column

This is a very powerful function which allows you to read only one column from a data file (or read some data and skip others in a regular manner). The program will ask in which column the data are to be inserted (again, the column 1 is H). The second question is which values will be skipped (read one values and then skip **n**), you'll be asked about this n; n=0 means to read all the data.

Clear data grid

You can clear one or both of the grid at any time.

Set the number of rows

When inserting data from files, to avoid errors the program automatically will increase the number of rows (often to much). By this command you can set the number of rows to a reasonable value. Sometimes this can save some time. If the grid has (because of unfortunate input files..) 1000 rows but you have only 100 data points it is better to set the data rows to 100 to save some memory (anyway, in my opinion -from time to time- 5 seconds more or less it is not a big deal !).

Paste the DICVOL results in data grid

Standard

Change standard data

Input standard data in column

Insert row

Delete row

Delete column

Clear standard grid

Cell

Assign hkl to peaks

Refinement of unit cell parameters

The unit cell parameters are calculated by least squares (all three methods have some least squares procedures inside) and I recommend you to use the first option (however, is the only one who gives the standards deviations for the cell parameters).

Both **least squares** and **overdetermined system, Newton** use the same algorithm of Newton, the difference is in the solution of the linear system (first one is a Crout type and the second one is a Gauss-Jordan). The second method is less convergent than the first one because of a dumping factor I imposed, but seems to be more stable. You should check if you arrived at a convergence, the values for the sq. deviations at each cycle are printed.

A fancy called **multidimensional search** is just a search for the minimum of the squared difference between Q and its expression reciprocal terms. This is done by a modified Interval halving method. I will explain this method because is very easy to put in code (although its value is limited) and because it is not a recommended method for refinement, especially for lower symmetries.

Starting from an initial guess (no matter how far from the minimum) one can find the minimum of a function $f=(y_1-y_2)^2$ where y_1 is an experimental value and y_2 is a model depending of some parameters $x_1, .., x_n$ which have to be estimated. If n is not too high one can make a search in an interval and compute the function f at each step, the minimum f will be an estimate of the solution x. The error will depend on the step size and on the fact that the actual minim is comprised in the search range.
In this function, if the minimum is not comprised in the search range, the width will be adjusted toward the minimum.
This procedure is implemented here (test it if you like but think how long it will take!). The **width** shows how far from the initial point it will search the minimum, the **steps/cycles** is how many steps will make on each variable on each interval and **refine** shows how many times to do this with a halving of the width for each cycle.
For each **refine** the step will be smaller with a 1/q factor where q is the number of **refine** in the textbox. The fields **Width** and **refine** are used only in the search algorithm and have no meaning in the least squares.

It is very slow because, for **n** parameters problem one need $\text{steps}^{\mathbf{n}}$ functions evaluations and this multiply by **refine** value.
It is a very time consuming tehnique but probably in ten years from now....
Actually, the reason of existence for this function here is related to educational purposes (manual indexations...). One can make an indexation according to Ito's procedure and get any strange triclinic cell. By a subsequent Delaunay transformation we can get a better cell.

Note: the results are printed in the Report pad and not in the Cell Form.

At this moment I don't know much about the accuracy of this program compared to other known programs (the minimizations use Double precision for variables but Visual Basic is not made for scientists!). I think that the eventual error is smaller than 0.001A which for me is good enough.
I am waiting some input from you here...

Automatic indexing (by DICVOL, ITO and TREOR)

Compute hkl, d, theta

Both commands perform the same calculation tasks but the output is directed either to the report pad or to data grid.
The HKL and d are computed for the cell you have and with the parameters found in the Cell form.The hkl limits and 2theta are to be input by the user as well as Bravais symbols.

HKLGEN

Decimate

This menu contains three functions which are inspired by the Armel LeBail's Overlap. Reading Fullprof FOU, Gsas RFL and/or Shelx HKL files, eliminating the reflections which are too close (the two theta limit will be requested) and save what's left in a Shelx .hkl file for further direct methods or whatever. The cell values are taken from the Cell form, the symmetry is not requested.

back to top

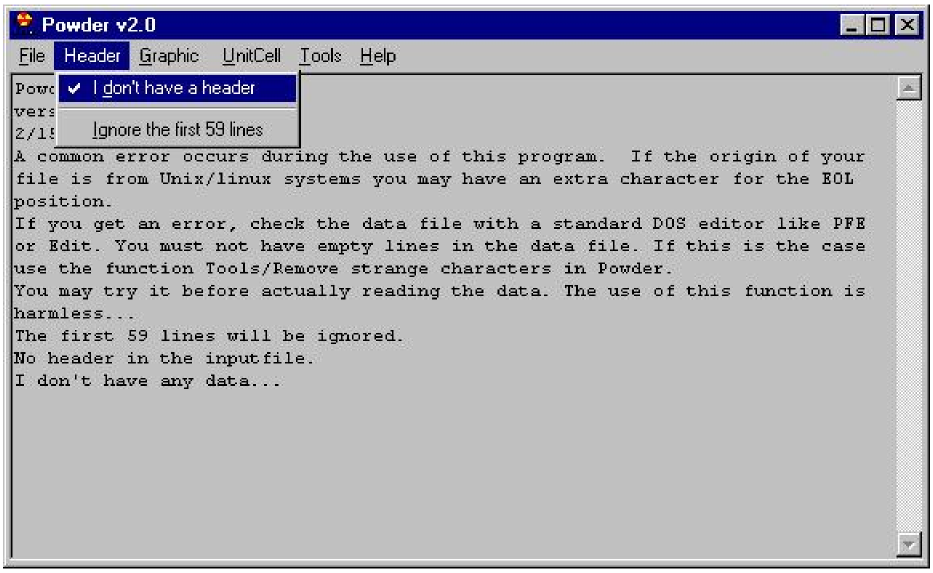
Tutorial

Basic run through Powder4

Let's take the data of NAC available at http://pcb4122.univ-lemans.fr/powdif/low_fwhm_and_rp.html#NAC , in another tutorial by Armel LeBail. The data are in a zip file and some explanations about the conditions used can be seen in the mentioned page. Grab the file and unzip it. Before attempting to convert it look at it with an ASCII editor like PFE. We can see some resemblance with the Siemens format but this is not a Siemens format though. We look how many header lines are there (59) and open this file with the program Powder 2.0 with the following options:

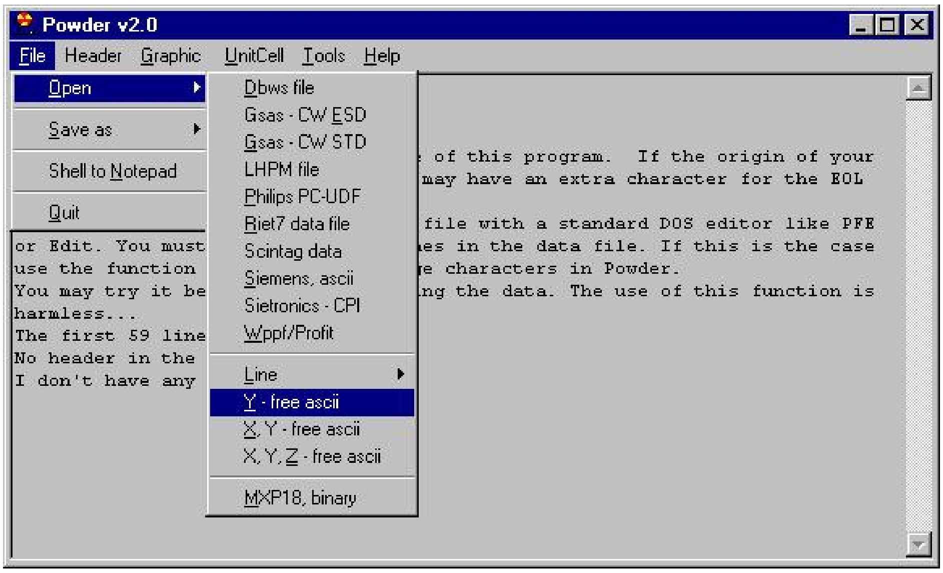
-I don't have a header

-Ignore the first 59 lines



```
Powc
vers
2/1!
A common error occurs during the use of this program.  If the origin of your
file is from Unix/linux systems you may have an extra character for the EOL
position.
If you get an error, check the data file with a standard DOS editor like PFE
or Edit. You must not have empty lines in the data file. If this is the case
use the function Tools/Remove strange characters in Powder.
You may try it before actually reading the data. The use of this function is
harmless...
The first 59 lines will be ignored.
No header in the inputfile.
I don't have any data...
```

Then open the file as Ascii, and choose the file in question.



```
of this program.  If the origin of your
may have an extra character for the EOL

file with a standard DOS editor like PFE
or Edit. You must    es in the data file. If this is the case
use the function     e characters in Powder.
You may try it be    ng the data. The use of this function is
harmless...
The first 59 line
No header in the
I don't have any
```

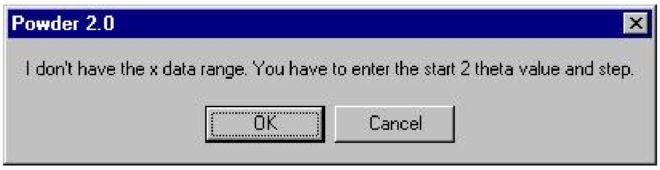
If this is successful you will see some messages (this is what is called Report Pad in this program), in this case what we can see is:

```
-----------------------------------------------
Y data file, ascii
The filename is C:\WINDOWS\Desktop\NAC-test-2.UXD
59 line(s) will be ignored.
The number of points is: 6501
Done...
2/15/99 5:26:39 PM
--
```

NOTE: I have noticed a frequent error in the use of this program. When the data file has been e-mailed without being zipped an extra EOL character appears (I guess it is related to different definition of EOL for DOS and Windows ?). If you look at this kind of file with DOS/Edit you can see alternating empty lines with data. These empty lines will "confuse" the program. Therefore you should remove them by the command Tools/Remove strange characters. The same procedure should be used for files coming from Unix and flavors of Unix.

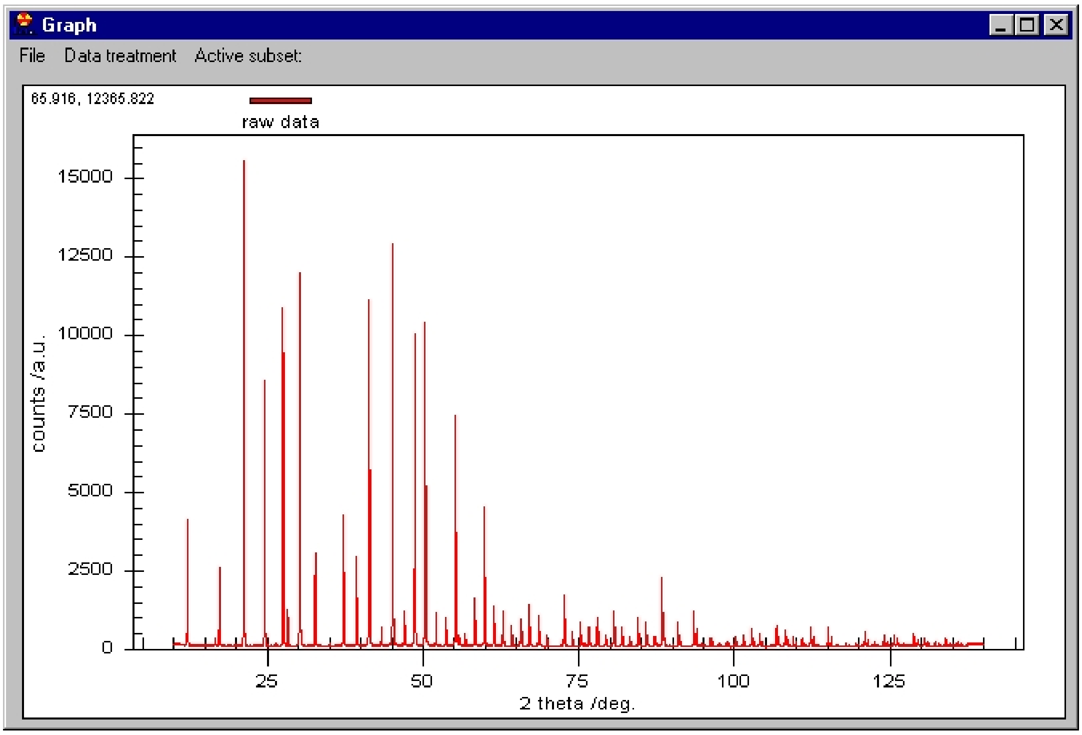
--

We can directly inspect the data once read by choosing Graphic in the menu. We get this message



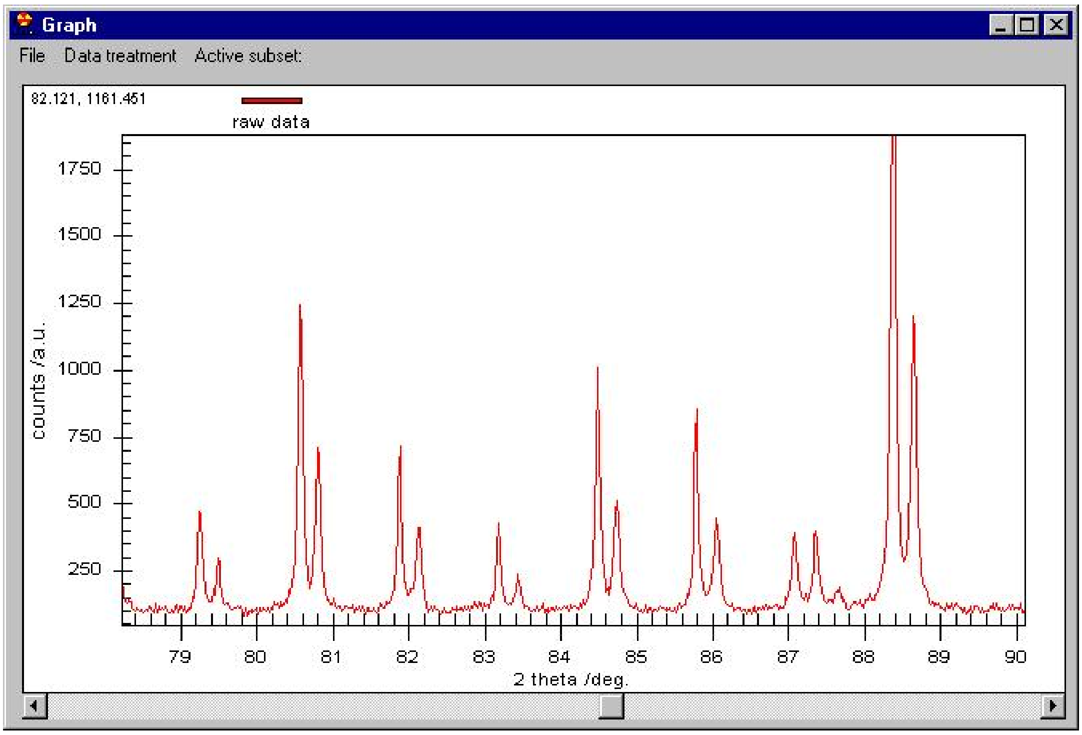
I don't have the x data range. You have to enter the start 2 theta value and step.

and we answer 10 for 2 theta and 0.02 for step (this message comes when the program does not know the starting value and step). Following, the Graphic window is shown:



The scale is automatically adjusted to fit all the data. In the top left corner we can see two values 65.916, and 12365...: these are the coordinates of the mouse on the screen. You can roughly inspect the data by this method.

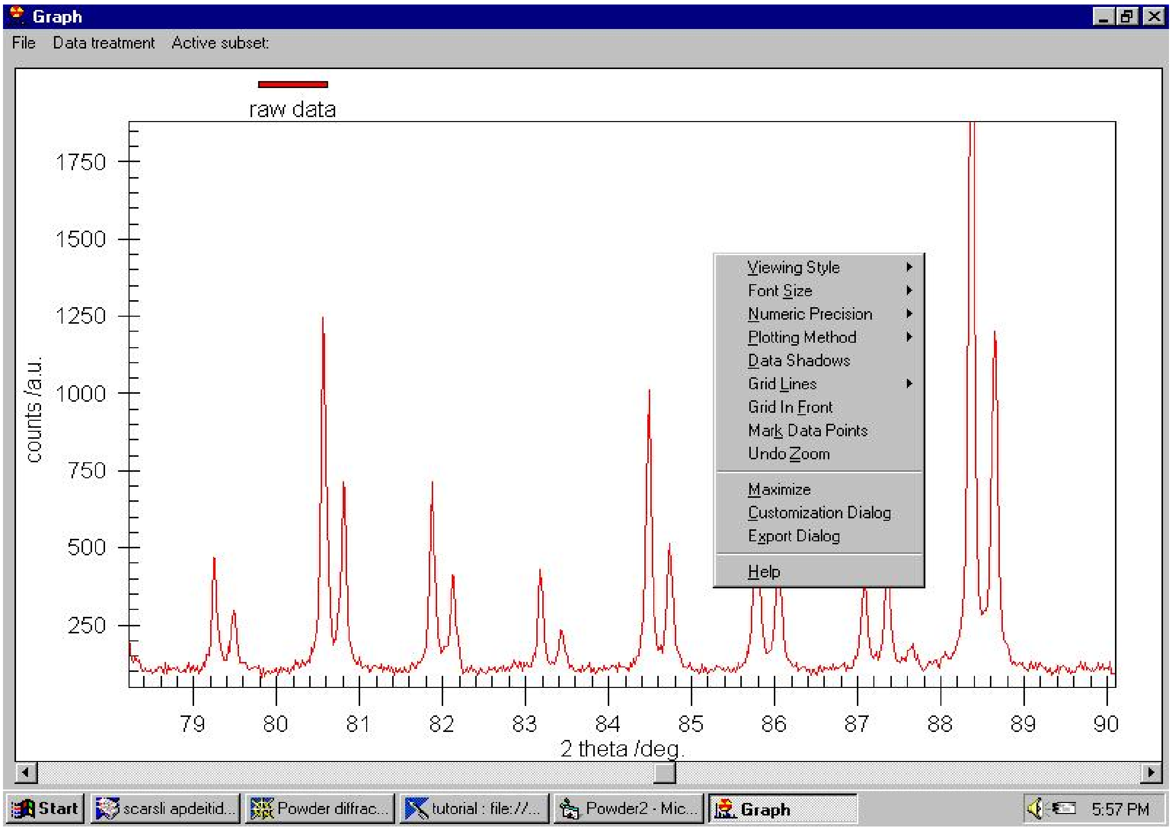
You can zoom as you like by dragging the mouse on the screen:



On the zoomed graph a horizontal bar appears. Clicking on it you can move to the right or left by keeping the same zoom as here. You can go back to the initial zoom by right-clicking on the graph area and choosing**Undo zoom**.

Several other menus are available here, most of them are easy to understand.

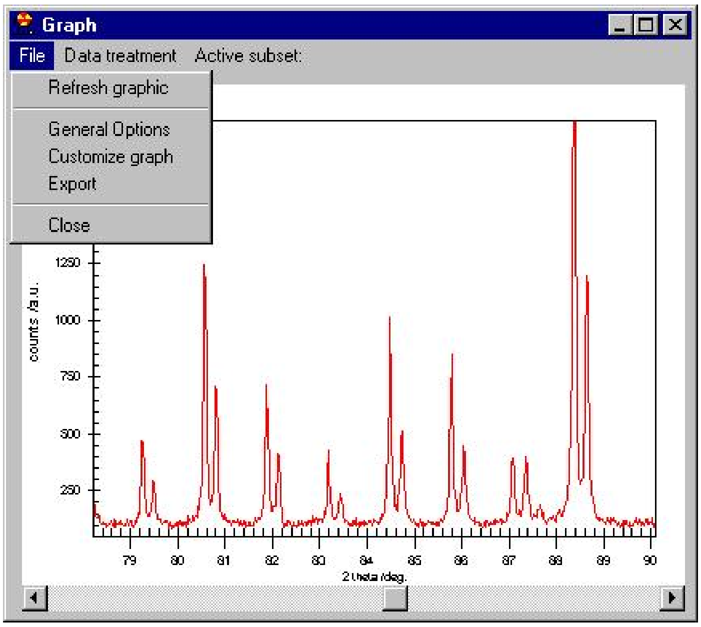
When you right-click on the graph an important menu allows changing some parameters:



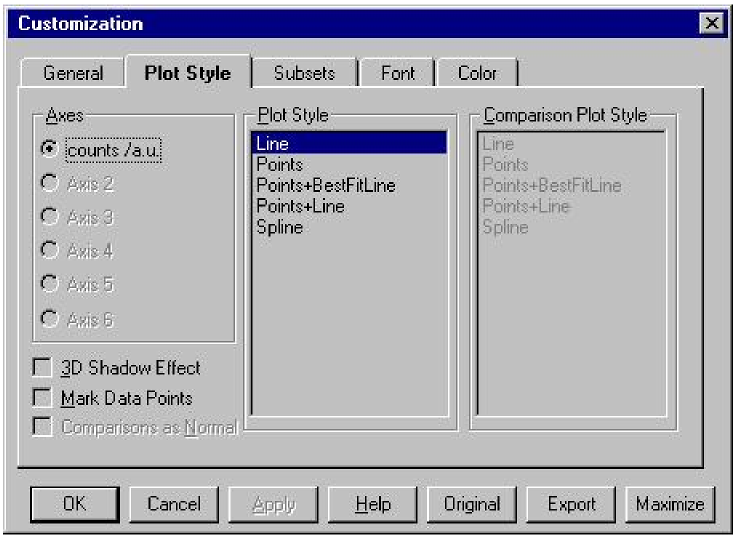
Note that we can resize the graph at any time. The menu (which can be invoked -for people hating the mouse- by **File/General Options** command as well) contains:

Viewing style (monochrome or color)
Font size
Numeric precision
Plotting method
Grid lines
Grid in front
Mark data points
Undo zoom
Maximize
Customization dialog
Export dialog
Help

Some of these menus are self explanatory; note that **Mark Data Points** is extremely slow.
There are two interesting options here: **Customization Dialog** and **Export Dialog,** both of them available also from the **File** menu.



By **Customization Dialog** (equivalent with **File/Customize Graph**) we open the following menu.



The most important command here is **Subsets** which is described later.

The second menu is **Data treatment** which allows:

-smooth by Savitzky Golay, Adjacent averaging or a Moving Window algorithm;

-remove background (automatic or manual),

-remove K alpha 2,

-Find peaks,

-Add peaks (not shown here but removing some of the peaks can be done easily, just click on the marker)

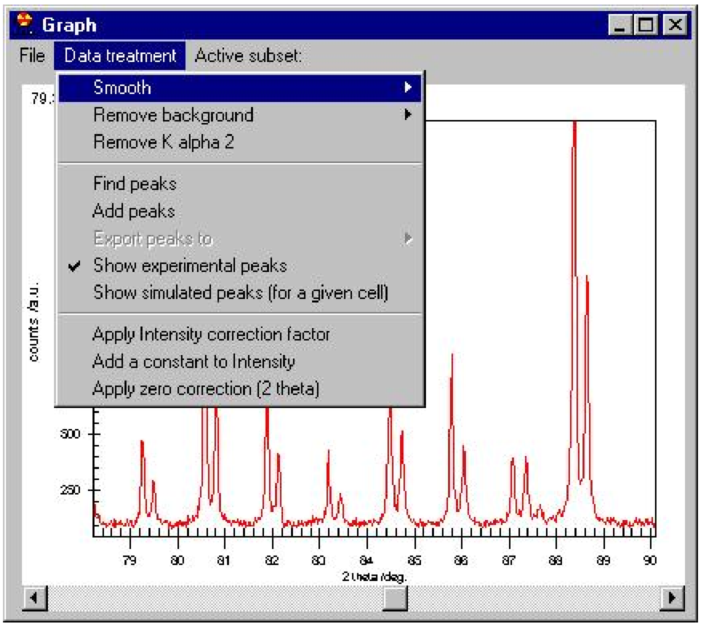
-Export peaks (when you have already determined them)

-Show experimental peaks

-Show simulated peaks

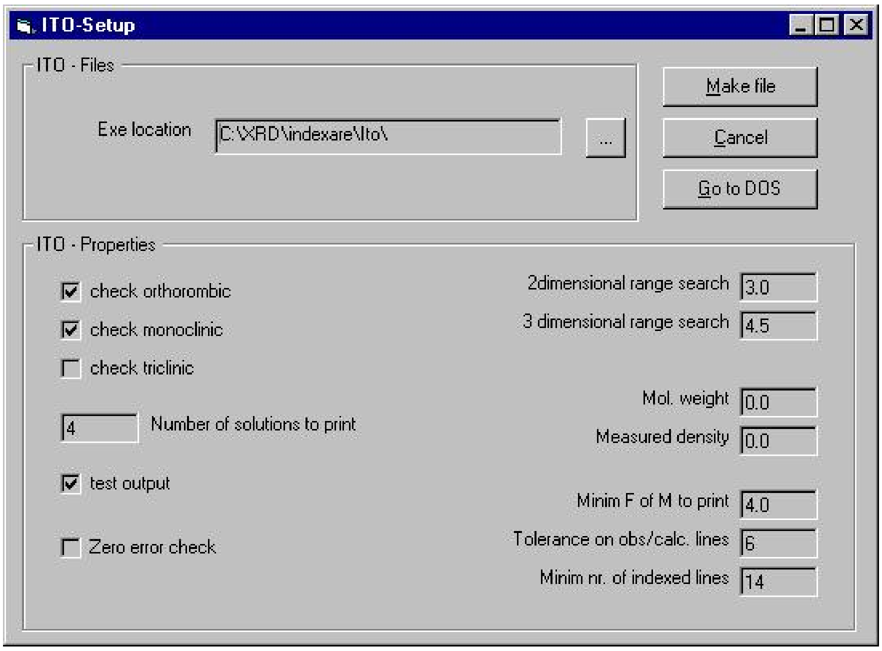
-Intensity correction

-zero correction



The values detected are sent to the grid as 2theta and the intensity as weight (the intensity is however ignored hereafter).

You can try to index these by Ito or Dicvol methods. Let's try ITO, select **Cell/ Automatic indexing/ Use Ito** and the following screen appears:



ITO - Files

Exe location: C:\XRD\indexare\Ito\

ITO - Properties

☑ check orthorombic

☑ check monoclinic

□ check triclinic

4 Number of solutions to print

☑ test output

□ Zero error check

2dimensional range search: 3.0

3 dimensional range search: 4.5

Mol. weight: 0.0

Measured density: 0.0

Minim F of M to print: 4.0

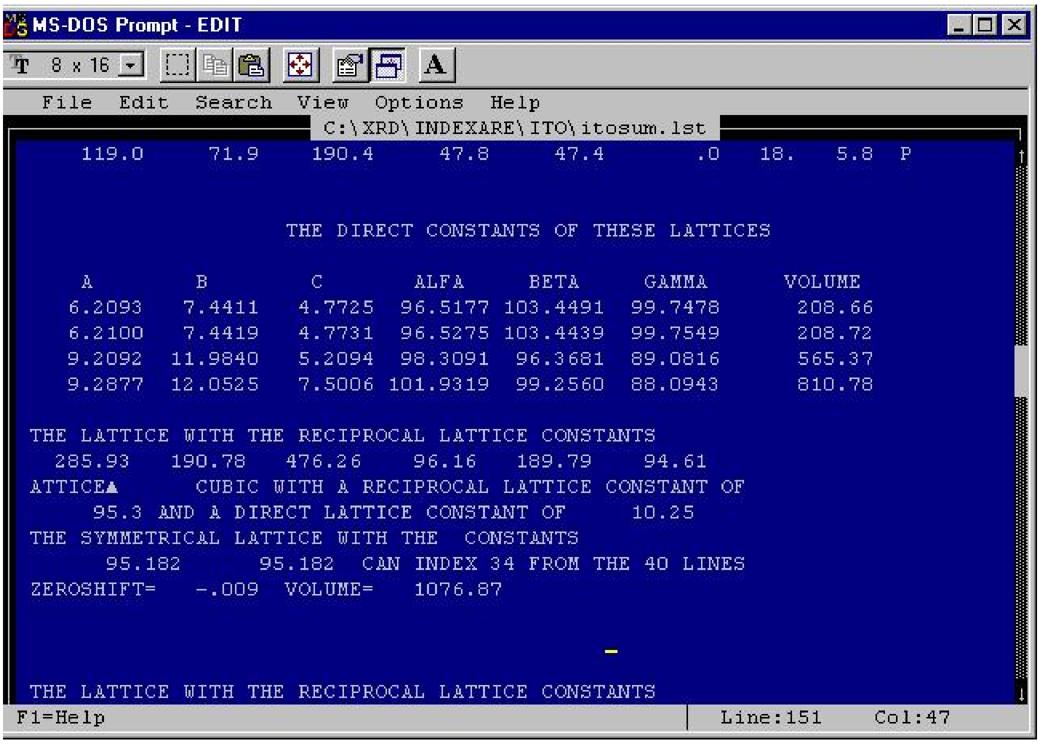
Tolerance on obs/calc. lines: 6

Minim nr. of indexed lines: 14

Only the basic options in ITO can be set up here (NOTE: ITO and DICVOL are independent programs which you should install before using the program Powder. These two well known programs are neither written nor recommended or supported in any way by me!). The data files saved by Powder 2.0 are**ito.inp** and **dicvol.inp** in the location chosen by Exe location textbox.

In the EXE location field you have to select the location of the program Ito or a directory where the input file ITO.INP should be created when pressing Make file button. You have to go to the DOS end start ITO by yourself.

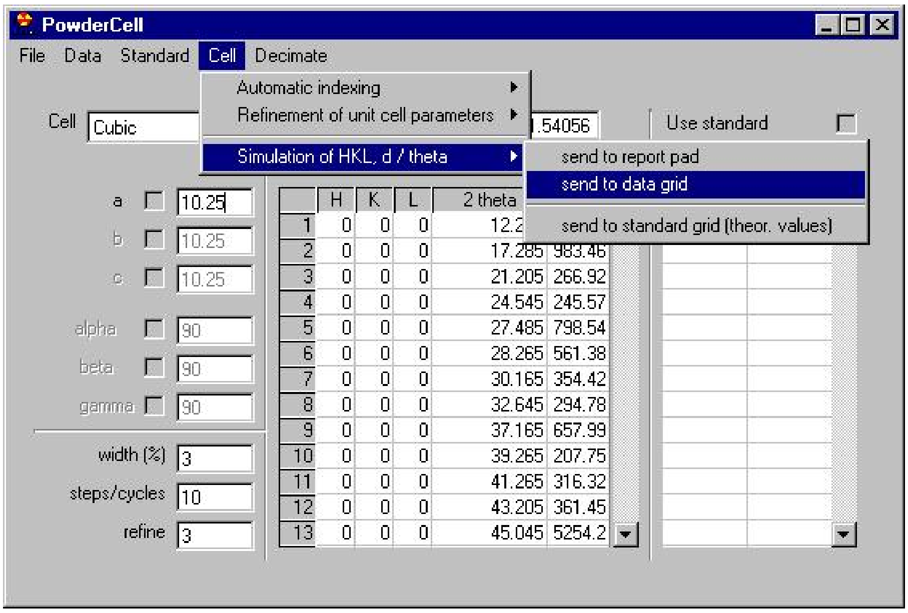
It gives several solutions (among the suggestions the true solution <much easier to decide when you know the true result, :)>,...)


MS-DOS Prompt - EDIT
8 x 16
File   Edit   Search   View   Options   Help
C:\XRD\INDEXARE\ITO\itosum.lst
F1=Help
Line:151   Col:47

```
     119.0      71.9     190.4      47.8      47.4        .0    18.    5.8   P


                      THE DIRECT CONSTANTS OF THESE LATTICES

     A         B         C        ALFA      BETA      GAMMA       VOLUME
    6.2093    7.4411    4.7725   96.5177  103.4491   99.7478       208.66
    6.2100    7.4419    4.7731   96.5275  103.4439   99.7549       208.72
    9.2092   11.9840    5.2094   98.3091   96.3681   89.0816       565.37
    9.2877   12.0525    7.5006  101.9319   99.2560   88.0943       810.78

 THE LATTICE WITH THE RECIPROCAL LATTICE CONSTANTS
   285.93    190.78    476.26     96.16    189.79     94.61
 ATTICE▲      CUBIC WITH A RECIPROCAL LATTICE CONSTANT OF
      95.3 AND A DIRECT LATTICE CONSTANT OF      10.25
 THE SYMMETRICAL LATTICE WITH THE  CONSTANTS
       95.182       95.182  CAN INDEX 34 FROM THE 40 LINES
 ZEROSHIFT=   -.009  VOLUME=   1076.87



 THE LATTICE WITH THE RECIPROCAL LATTICE CONSTANTS
```

If you want you can check that one of the solutions is the correct one.

Go to the UnitCell window and enter the value of the unit cell you want to check and the command simulate


PowderCell
File Data Standard Cell Decimate
Automatic indexing
Refinement of unit cell parameters
Simulation of HKL, d / theta
send to report pad
send to data grid
send to standard grid (theor. values)

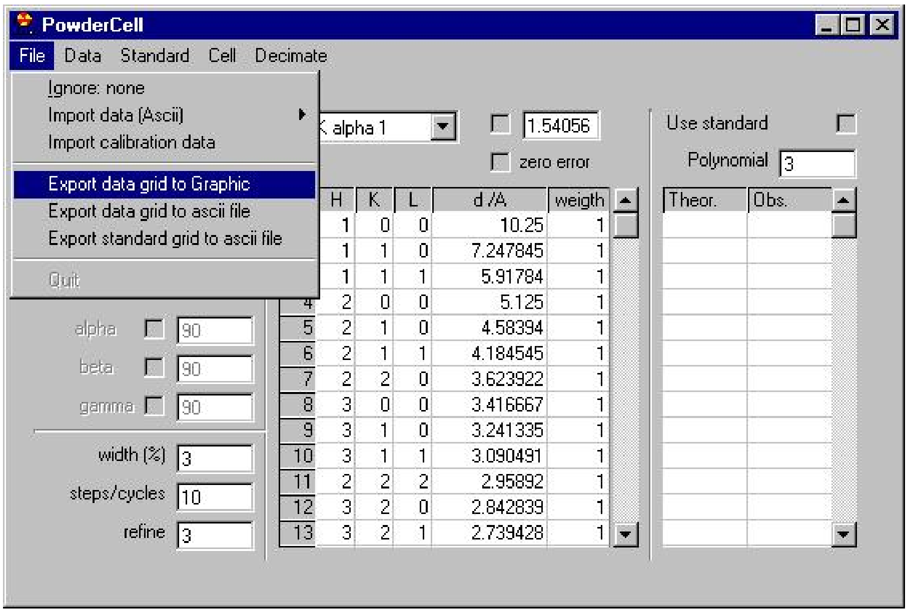
Cell Cubic

.54056 Use standard □

a □ 10.25
b □ 10.25
c □ 10.25
alpha □ 90
beta □ 90
gamma □ 90
width (%) 3
steps/cycles 10
refine 3

	H	K	L	2 theta	
1	0	0	0	12.2	
2	0	0	0	17.285	983.46
3	0	0	0	21.205	266.92
4	0	0	0	24.545	245.57
5	0	0	0	27.485	798.54
6	0	0	0	28.265	561.38
7	0	0	0	30.165	354.42
8	0	0	0	32.645	294.78
9	0	0	0	37.165	657.99
10	0	0	0	39.265	207.75
11	0	0	0	41.265	316.32
12	0	0	0	43.205	361.45
13	0	0	0	45.045	5254.2

The program will compute the theoretical values of 2 theta; select Send DataGrid to Graphic


PowderCell
File Data Standard Cell Decimate
Ignore: none
Import data (Ascii)
Import calibration data
Export data grid to Graphic
Export data grid to ascii file
Export standard grid to ascii file
Quit

K alpha 1 □ 1.54056 Use standard □

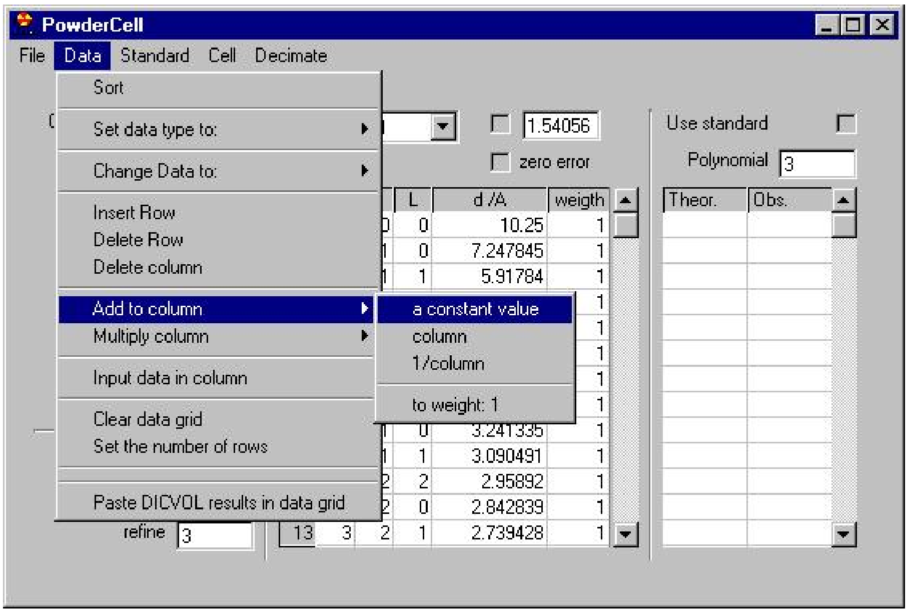
□ zero error Polynomial 3

alpha □ 90
beta □ 90
gamma □ 90
width (%) 3
steps/cycles 10
refine 3

	H	K	L	d /A	weigth
	1	0	0	10.25	1
	1	1	0	7.247845	1
	1	1	1	5.91784	1
4	2	0	0	5.125	1
5	2	1	0	4.58394	1
6	2	1	1	4.184545	1
7	2	2	0	3.623922	1
8	3	0	0	3.416667	1
9	3	1	0	3.241335	1
10	3	1	1	3.090491	1
11	2	2	2	2.95892	1
12	3	2	0	2.842839	1
13	3	2	1	2.739428	1

Theor.	Obs.

and these values will be marked with crosses on the graphic. Note: the intensity at which the crosses are put are determined by the value stored in the column Weight. You can easily add a constant to these values

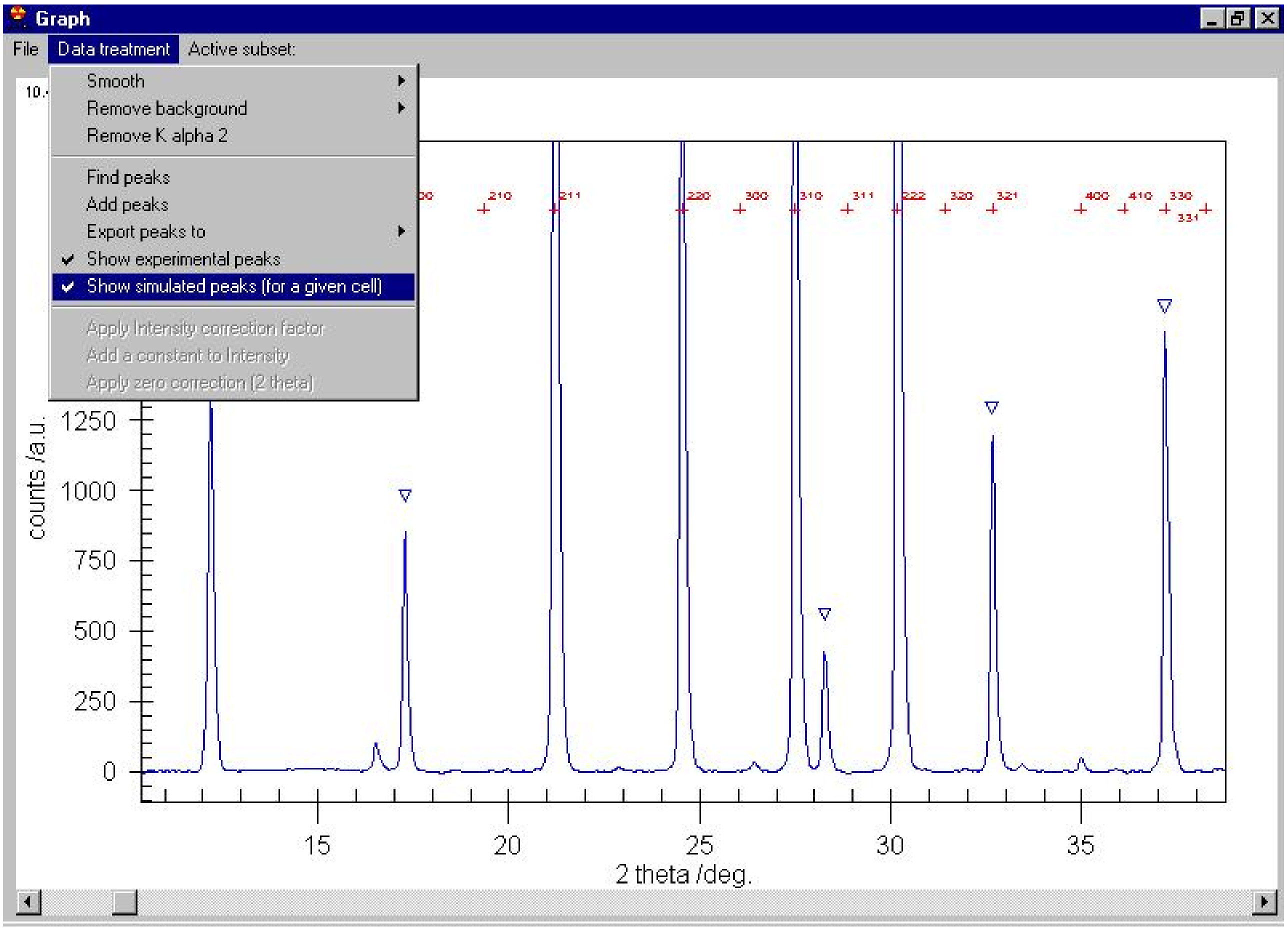


☐ 1.54056

	L	d /A	weigth
0	0	10.25	1
1	0	7.247845	1
1	1	5.91784	1
			1
			1
			1
			1
			1
1	0	3.241335	1
1	1	3.090491	1
2	2	2.95892	1
2	0	2.842839	1
13 3 2	1	2.739428	1

let' say 2000...

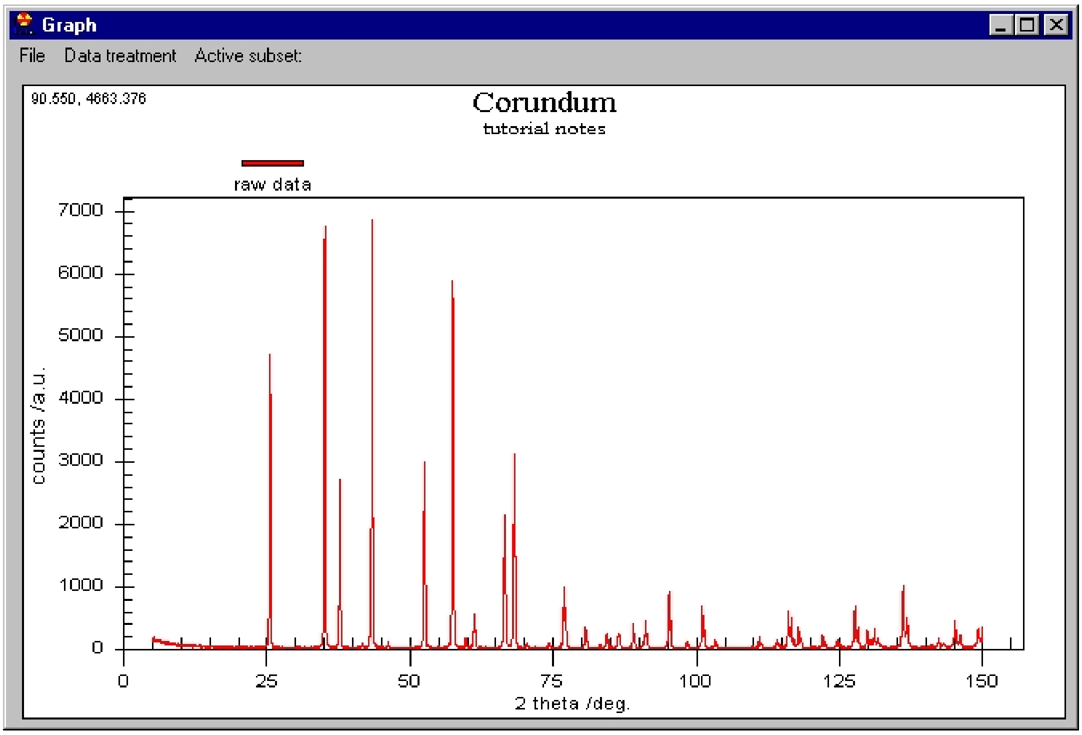
Make sure that the menu Show simulated values is checked. You get this kind of figure...



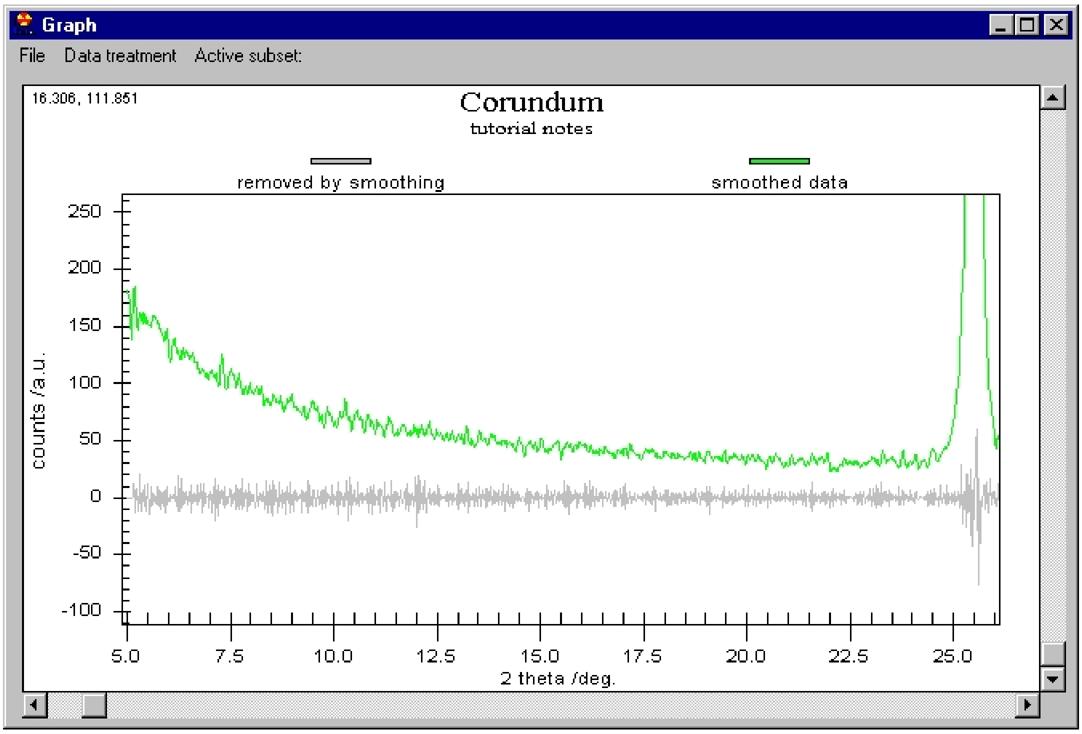
You can print or export this data as you wish.

When exiting the Graphic window, if you want to keep some data (for instance Smoothed data) make sure that the **Smoothed** data is checked in the **Active Subset** menu.

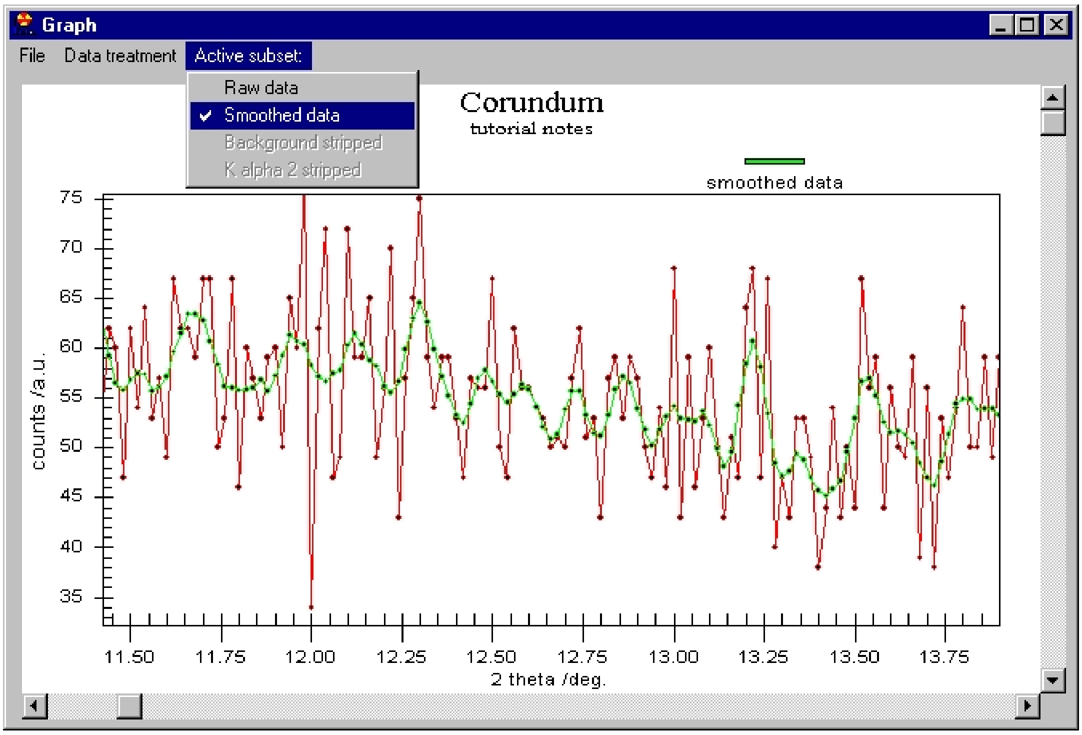
You can see 8 graphics at a time (but is difficult to understand them). Let's see corundum (a DBWS file provided by Lachlan Cranswick). We know that is a good DBWS data file so we can read it directly by **File/Open/DBWS file** from the main part of the program. Then we choose **graphic** and add a title and subtitle to the graphic. We get this.



We can apply a smooth to this and then check the result (here is shown the smoothed data and what was removed by smoothing).

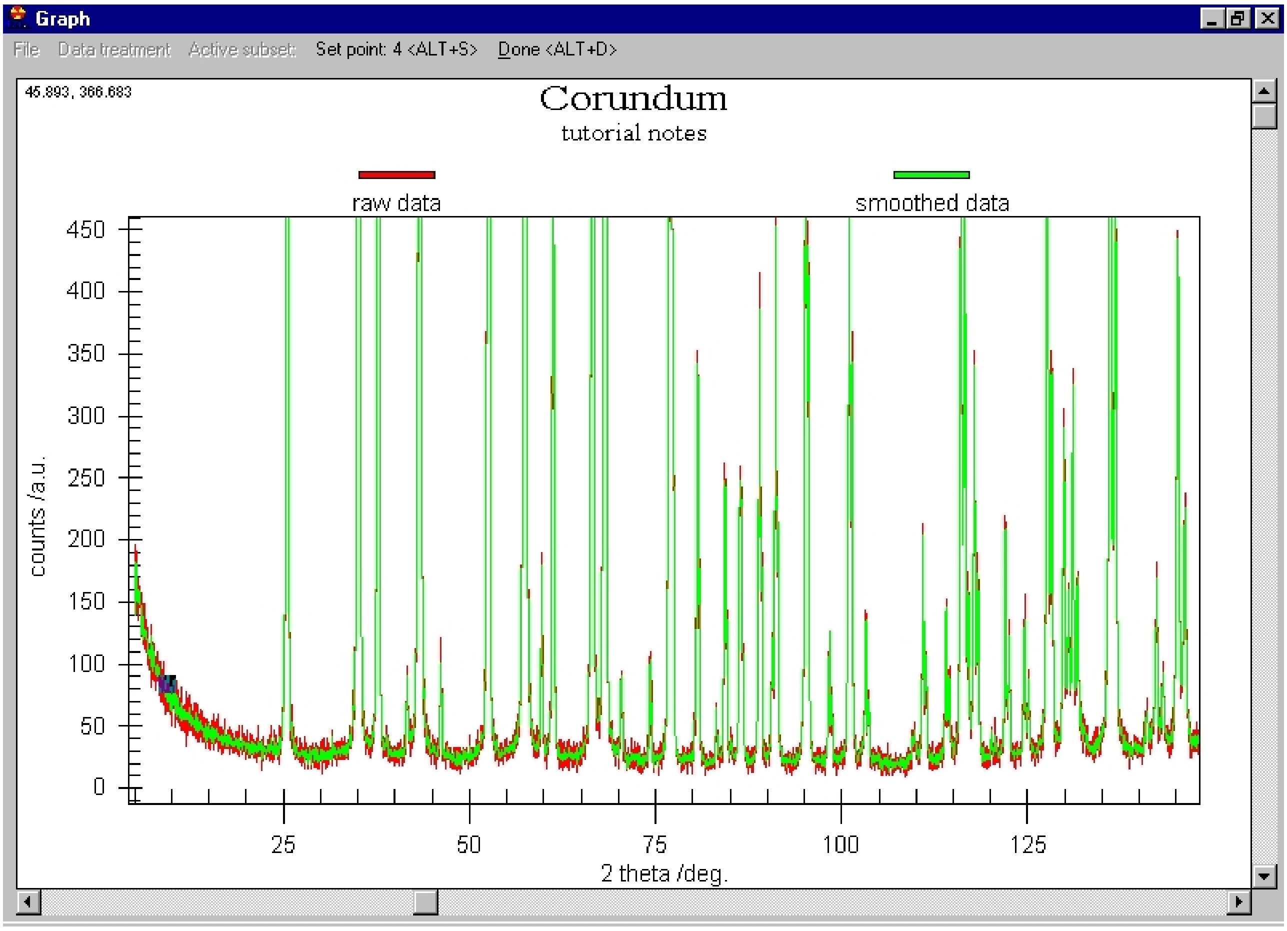


We should remove the Background now. If the Background is rather small and can be well defined by a low degree polynomial you can apply a **Manual Correction** (in Remove Background menu). If we want to remove the background of the Smoothed data we should choose **Active subsets/Smooth data.** In the following figure the Smoothed and Raw data are shown (note the point markers, when showing them be veeeeeery patient).

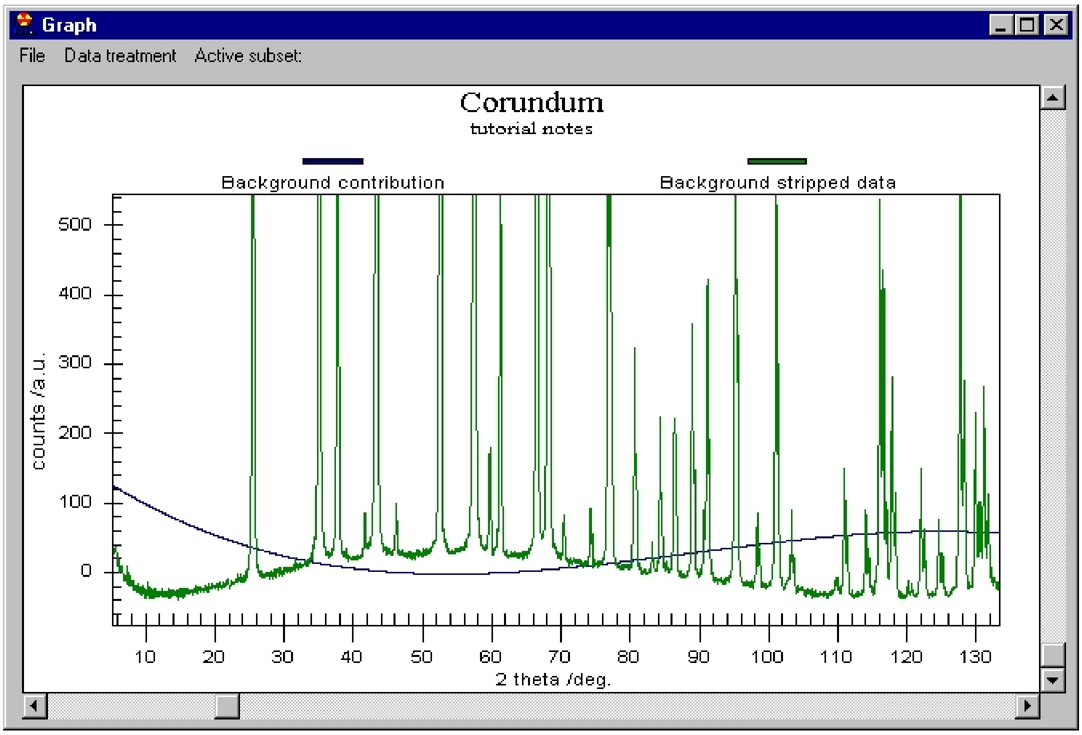


From now on, as long as the Smoothed Data menu remains checked, all the modifications will be performed on Smoothed data. If you smooth again the old smooth data will be replaced with the new values.

Manually removing the Background requires choosing some data points. First we should choose the polynomial degree, let's say 3 is enough (the program can accept a degree up to 8 but I do not recommend going to that value). Then we choose data points (the more the merrier) by moving a cursor through points). Choose a point by moving the cursor then Click on Set point or Alt+S to keep the value of the cursor for interpolation. When finished choose Done.

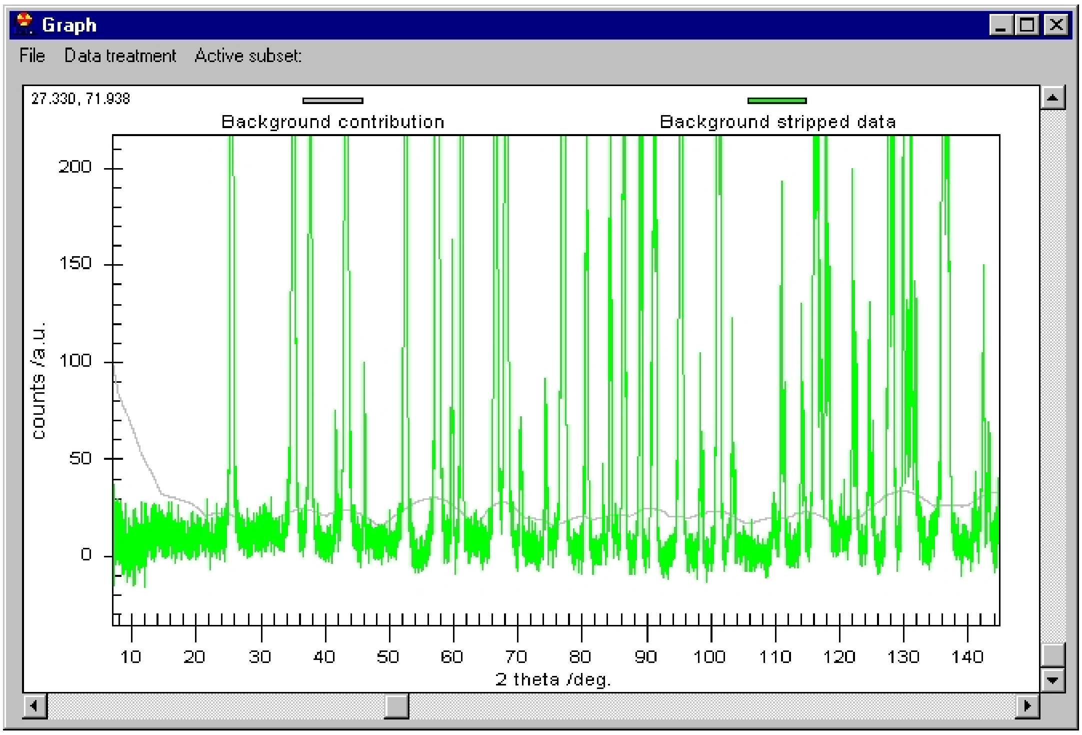


The program will compute the interpolation polynomial based on the values you choose. I selected for this tutorial 12 points and I got this ugly results:

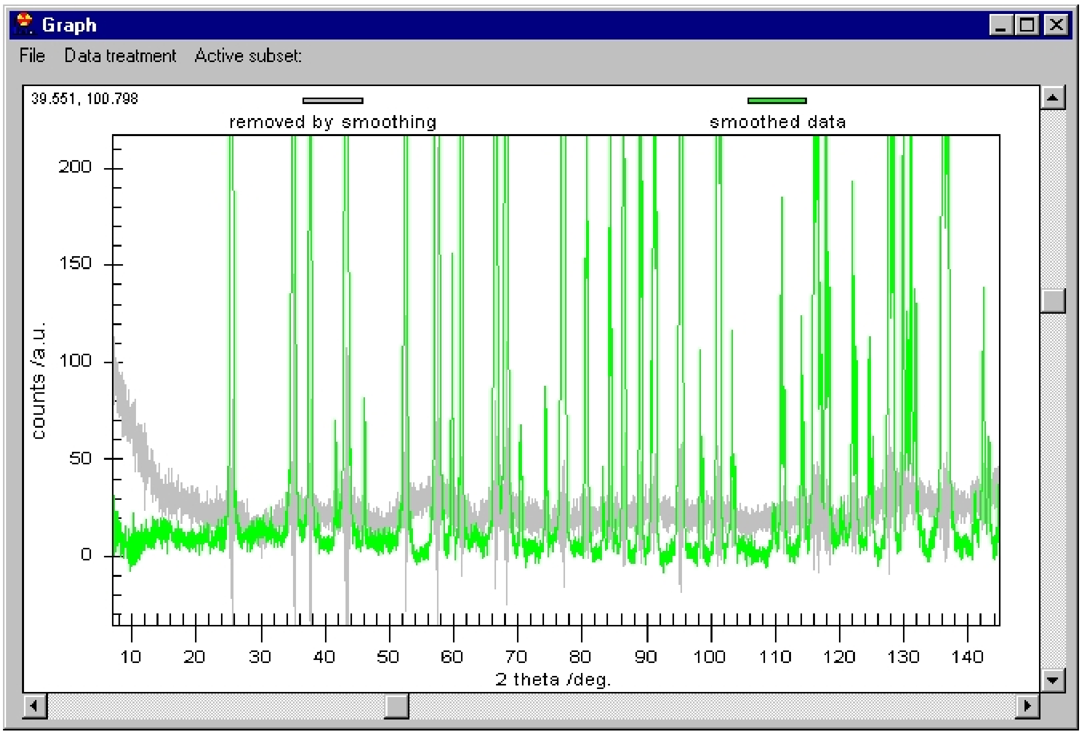


The Background contribution is shown so as you can see what it has computed (this is an example of how should not be the background; you should do better than this).

Now let's remove the background in a better way (from the raw data, we choose Active Subset/Raw data) and get this.

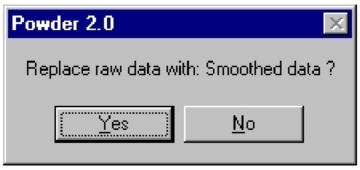


And now let's smooth the **Background removed data** (we should choose**Active Subset/Background stripped data**) we obtain this:



Note that the Smoothed data contains the Background removed and smoothed data.

If we want to quit the Graphic Window now we will get a message:



Powder 2.0

Replace raw data with: Smoothed data ?

That is, if the **Active Subset/Smoothed data** is checked you will be asked if the data which you read should be replaced by the Smoothed data. If you do not choose an **Active subset** no matter what modifications you do in the Graphic window the read data will not be replaced.

Enjoy and try it extensively to learn how it works.

Final note: Remember to register if you'll ever want to ask something or to receive updates and bug reports. If not, you are on your own.

--

Small print: Use the program at your own risk...

Merging and normalising data

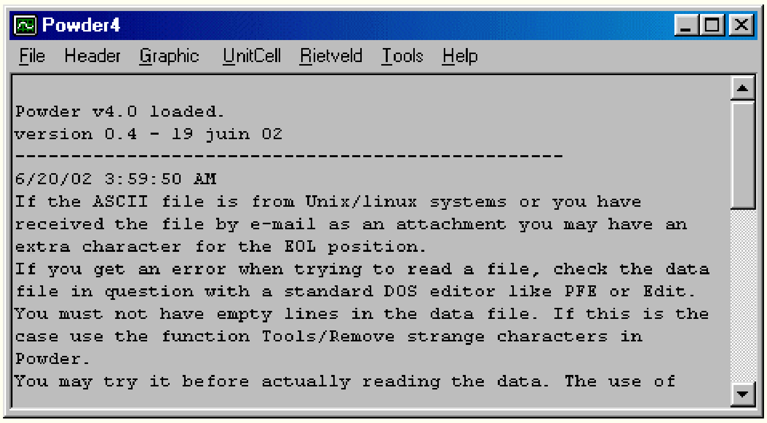
Some details about these functions (from www.ccp14.ac.uk).

With the latest Powder4 for Windows by Nita Dragoe will allow you to merge and normalise up to 10 files into a single file (calculating appropriate ESDs for each count). For this you need to give each pattern a "monitor" value (counting time is probably the best way for most scans). This is done via the Powder v4 interface as shown below.

Following are a few tips on how to use the latest Powder4 (i.e. version 0.3b) if you want to merge powder XRD files. WARNING: this is a dangerous operation, do not massage your data; make sure you know what you are doing.

First run Powder for Windows (make sure you have the latest versions)

A few instructions are listed on this panel ; read this from time to time, it contains some useful hints on what's going on with the program.

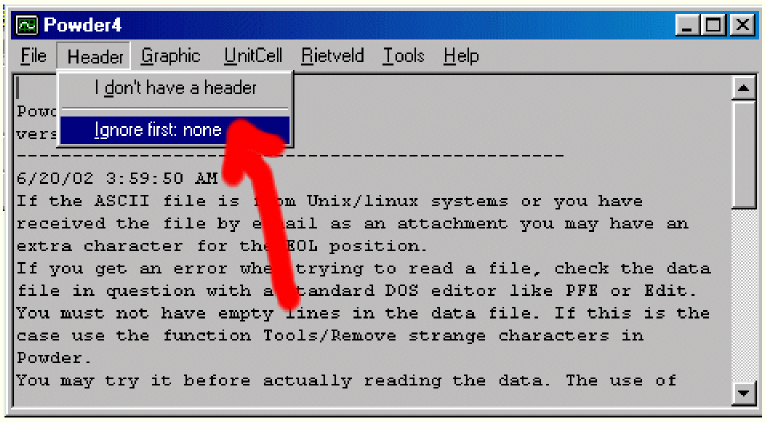


```
Powder v4.0 loaded.
version 0.4 - 19 juin 02
--------------------------------------------------
6/20/02 3:59:50 AM
If the ASCII file is from Unix/linux systems or you have
received the file by e-mail as an attachment you may have an
extra character for the EOL position.
If you get an error when trying to read a file, check the data
file in question with a standard DOS editor like PFE or Edit.
You must not have empty lines in the data file. If this is the
case use the function Tools/Remove strange characters in
Powder.
You may try it before actually reading the data. The use of
```

We are going to merge 10 files. Normally you have to conver the files into column XY, or XY_ESD format. In the above example data, the UDF files have been converted into XY format using Convert.

All these files have the first line as header, so we'll have to "tell" the program how to handle this. First, we click on Header/I don't have a header (this is optionally here but in many cases it's important to instruct the software that there is no header to look for). Then we click on Header/Ignore menu, just like in the following picture:

Now this is important : you have to instruct the program to ignore the first line otherwise you'll get a mess. Alternatively you can delete all the comments from the file but it's better to keep the original header in place (do not mix up the files!!!).

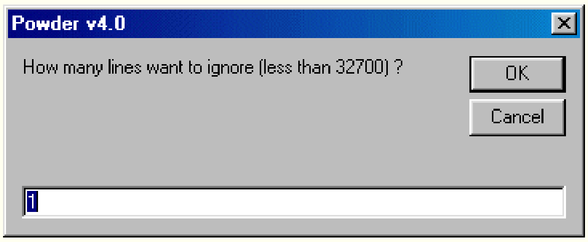


```
Powd
vers
---------------------------------------------------------------------------
6/20/02 3:59:50 AM
If the ASCII file is    om Unix/linux systems or you have
received the file by e  ail as an attachment you may have an
extra character for the EOL position.
If you get an error whe  trying to read a file, check the data
file in question with a  tandard DOS editor like PFE or Edit.
You must not have empty lines in the data file. If this is the
case use the function Tools/Remove strange characters in
Powder.
You may try it before actually reading the data. The use of
```

A small window will appear in which you insert (an integer) how many lines will be ignored.

Cancel or 0 have the same effect.

After that you can see listed in the menu Header/Ignore how many lines are going to be ignored. By *line*, this means everything up to an EOL character.



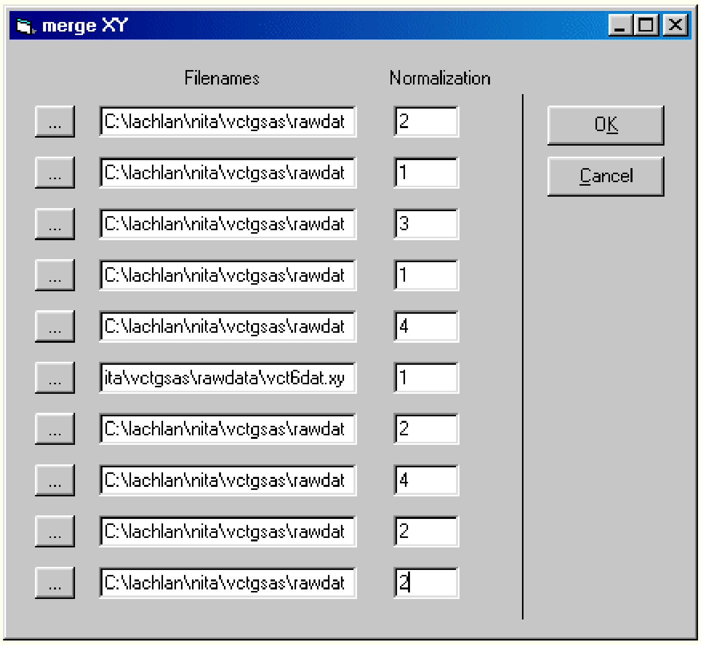
How many lines want to ignore (less than 32700) ?

1

All the Merging files have to be in the same format (the same number of comments, the same step, the same wavelength). It is not necessary to have overlapping ranges, you may as well just add to files say one having data from 20 to 90 the other from 90 to 120.

There are two possibilities for merging files: either XY ascii files or XYZ ascii files. For the first case the ESD will be computed internally; then everything is the same.

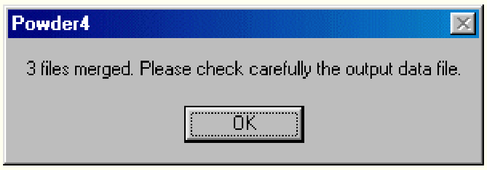
In the following we will merge the three files : we choose File/Merge XY files (**File, Merge XY Files**) and add the files via the dialog box. Give the appropriate "normalization / monitor" value to the right of the file name. In this case, they are all 1 (equally weighted or 1 second per step in the counting time)



Filenames	Normalization
C:\lachlan\nita\vctgsas\rawdat	2
C:\lachlan\nita\vctgsas\rawdat	1
C:\lachlan\nita\vctgsas\rawdat	3
C:\lachlan\nita\vctgsas\rawdat	1
C:\lachlan\nita\vctgsas\rawdat	4
ita\vctgsas\rawdata\vct6dat.xy	1
C:\lachlan\nita\vctgsas\rawdat	2
C:\lachlan\nita\vctgsas\rawdat	4
C:\lachlan\nita\vctgsas\rawdat	2
C:\lachlan\nita\vctgsas\rawdat	2

Now click **OK**

You will be prompted to output to a filename (enter this and the directory you wish the file to be place in); then you should get the following style of "OK" message:



Powder4

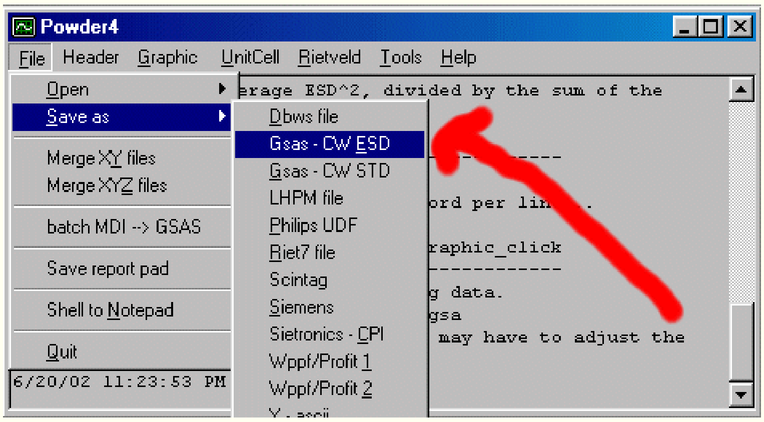
3 files merged. Please check carefully the output data file.

This creates a file in the following style of format (2-theta, counts, ESD, ready to be converted into other formats such as GSAS)

```
 -> merged 15/07/02 21:16:56
   17.00 ,          9.00 ,         1.50
   17.02 ,          6.50 ,      1.27475
   17.04 ,          6.00 ,      1.22474
   17.06 ,          4.50 ,      1.06066
   17.08 ,          5.50 ,       1.1726
   17.10 ,          5.00 ,      1.11803
   17.12 ,          9.50 ,       1.5411
   17.14 ,          4.00 ,         1.00
   17.16 ,          6.50 ,      1.27475
   17.18 ,          7.00 ,      1.32288
   17.20 ,          7.50 ,      1.36931
   17.22 ,          6.00 ,      1.22474
   17.24 ,          5.00 ,      1.11803
   17.26 ,          5.00 ,      1.11803
   17.28 ,          7.00 ,      1.32288
   17.30 ,          6.50 ,      1.27475
   17.32 ,          7.00 ,      1.32288
   17.34 ,          7.50 ,      1.36931
```

The Merged data is still in memory so you can still save this into other formats, such as XYZ, ASCII or GSAS

select **File, Save As, Gsas - CW ESD**

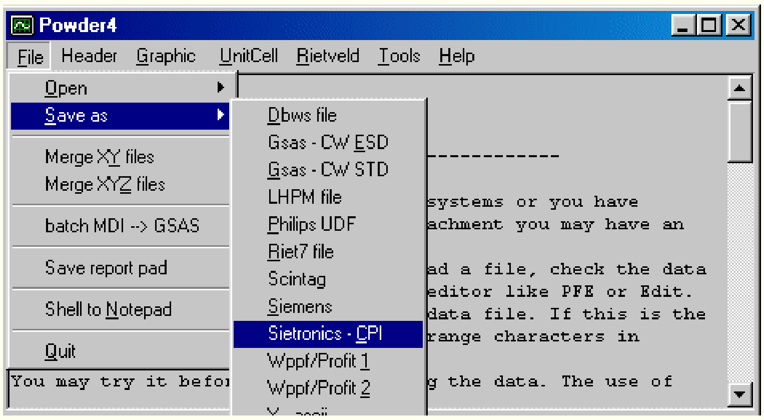


```
6/20/02 11:23:53 PM
erage ESD^2, divided by the sum of the
------------
ord per lin  .
raphic_click
------------
g data.
gsa
 may have to adjust the
```

Following is the top of the newly created GSAS file

```
Sample Title
BANK 1 6145 1229 CONST 1700 2 0 0 ESD
     9.0     1.5     6.5     1.3     6.0     1.2     4.5     1.1
     5.0     1.1     9.5     1.5     4.0     1.0     6.5     1.3
     7.5     1.4     6.0     1.2     5.0     1.1     5.0     1.1
     6.5     1.3     7.0     1.3     7.5     1.4    10.0     1.6
     7.0     1.3     3.5     0.9     8.0     1.4     7.5     1.4
     6.0     1.2    10.5     1.6     7.0     1.3     6.5     1.3
     5.0     1.1     6.5     1.3     7.0     1.3     6.5     1.3
    11.0     1.7     9.0     1.5     8.5     1.5     6.5     1.3
    10.5     1.6     8.5     1.5    10.5     1.6    11.0     1.7
     7.5     1.4     8.0     1.4    10.5     1.6     5.5     1.2
     7.5     1.4    11.0     1.7     8.5     1.5    13.0     1.8
    10.0     1.6    20.5     2.3    18.5     2.2    14.0     1.9
    17.0     2.1    23.0     2.4    24.5     2.5    22.5     2.4
    34.0     2.9    55.0     3.7    62.0     3.9    77.5     4.4
   127.5     5.6   128.5     5.7   107.0     5.2    67.5     4.1
    19.5     2.2    16.5     2.0    13.0     1.8    11.0     1.7
```

If you are in the mood to create other files (e.g., CPI, just select **File, Save As, Sietronics - CPI**. However, note that the ESDs will not be included in this format.



```
------------
systems or you have
achment you may have an

ad a file, check the data
editor like PFE or Edit.
data file. If this is the
range characters in

g the data. The use of
You may try it befo
```

Following is the top of the newly created CPI file

```
SIETRONICS XRD SCAN
17
139.9
0.02
Cu
1.54056
16-1-1998
1
SampleIdent
SCANDATA
9
6.5
6
4.5
5.5
5
9.5
4
6.5
7
7.5
6
5
5
7
6.5
7
7.5
10
5
7
3.5
8
```

Notice

There are some limitations in the use of this program and probably there are some bugs too. It may be slow but it can read as many data as you have, a special attention was paid to the "friendly" characteristic. Some comments may help you out of trouble.
And now, the usual stuff:

License Agreement

I you want to use this software, you are requested to send an e-mail to the author, stating your name, institution and the version you have. This is for bug reports and updates. Future correspondence regarding this program will be made only to the people on my "user list" where you can stay as long as you want (and to encourage you, I will not answer to any questions if you are not on that list).

back to top