

Chapter 3.1.2

DENZOX(KappaCCD)

Processing of Denzo .x files from Bruker-Nonius KappaCCD

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3.1.2.1 User's Instructions for the Program DENZOX

The programs **DENZOX** & **SORTAV** offer an alternative to *Scalepack* for processing the integration files on a Bruker-Nonius KappaCCD written by *Denzo(SMN)*. The *Denzo* .x files comprise a distillation of each image or frame and contain all the information necessary to create input reflection files for **Shelx** or **SORTAV**. The intensities in the .x files have been corrected for Lorentz-polarization effects and will only need a further absorption correction to be suitable for **Shelx** refinement. The program **DENZOX** makes no modifications to the intensities or sigma's of the fully recorded reflections, and merely sums the intensities of the partials and applies a simple statistical formula to obtain the sigma. The summed partial reflections are assigned an intensity-weighted average frame number.

DENZOX was originally written by R.H. Blessing (1997) but it has been heavily modified by L.J. Farrugia and K.W. Muir to automate the procedure and write out direction cosines. Versions for WinGX and LINUX are available. This manual describes the LINUX version -the WinGX version is run from a GUI, but the program and input files are identical.

Note that this version of **DENZOX** is highly KappaCCD specific and will only work with the *Denzo* .x files written by the latest versions of *Denzo (SMN)*.

The program is run in the following manner :

- Create the instruction file *denzox.input* using the utility program **MAKEDENZOX** (or the **DENZOX** GUI in WinGX).
- Optionally edit *denzox.input* to modify the action of **DENZOX**.
- Run the program **DENZOX** from the directory containing the *Denzo* .x files and *denzox.input*.

3.1.2.2 Running MAKEDENZOX

The utility program **MAKEDENZOX** creates the instruction file for **DENZOX**. It attempts to find an accurate unit cell, firstly from an RMAT file previously prepared using the *Nonius-Collect* programs *xmatrix* or *Phi-chi* scans. If no RMAT filename is entered when requested, the program then looks for *import.cif*, created by a previous run of *Scalepack/ Cifin*. The current directory, then the sub-directories maxus, maxus2, unscaled and scaled are consecutively searched for this file. If neither an RMAT file or *import.cif* is found, a default version of *denzox.ins* is created with zero entries for the unit cell. It is possible to manually edit this file and enter an accurate unit cell. If no unit cell is given in *denzox.ins*, **DENZOX** will use the cell from the first .x file. This is not at all desirable and it is *strongly* recommended to obtain the accurate unit cell from a source such as *import.cif*. The program **MAKEDENZOX** also searches the current directory for the scan-set

file-patterns of the .x files e.g. s01f####, s02f#### etc. Each scan-set is given an individual entry in *denzox.ins*. Any particular scan-set may be deselected for processing by commenting out the appropriate line.

3.1.2.3 Running DENZOX

This program takes as input the instruction file *denzox.ins* and a collection of *Denzo* .x files in the same directory. It is executed by typing **denzox** at the command line with no parameters.

A number of rejection criteria are applied to the full and summed partial reflections, and the program errs on the side of caution. All rejected reflections are noted in the log file, which also states the reason for rejection. Any measurements with negative sigma's indicate problems with integration. This may either be due to reflections lying too close to the edge of the CCD image, or to a reflection having a *bad pixel* in the integration sphere. Such reflections are always rejected. Some reflections have their intensities and sigma's written as *integers* rather than floating point numbers in the .x file. The *Denzo* manual states that these correspond to pixel overflows, but this does not seem to be the case always. Such "overflow" reflections are presently *not* rejected, but appear in the log file. If an individual reflection in a summed partial occurs in the first or last frame of a scan-set, the summed partial will always be rejected (since we don't know *a priori* if it extends further). When direction cosines are requested some additional criteria are applied. If the incremental oscillation angle for an individual reflection is *indeterminable*, then this reflection is always rejected. Under user control, a reflection may also be rejected if this angle is *unreliable* (see **REJECT** directive below). Finally, some other rejection criteria are also under user control, *i.e.* rejection on the basis of $\sin(\theta)/\lambda$ or χ^2 of profile fit. See below for details.

The program also checks whether all orientation matrices in the .x files are the same (within allowable re-orientation errors). If a change of orientation matrix is found, a warning is printed at the end. This situation can easily arise if the integration with *Denzox* is carried out manually for different scan-sets. While the chosen cell metrics may appear to be the same, the *Denzox* program can adopt alternative settings, so that the indices from different scan sets do not correspond. An automatic re-indexing is carried out to ensure that all indices in the resultant reflection files relate to the same (the first) orientation. The user should check *very carefully* that this re-indexing procedure is sensible. The matrices used for re-indexing (if any) are printed at the bottom of the listing file.

3.1.2.4 Program Instructions

The instruction file is read in free format. Each record starts with a tag defining the data which follows. Default values, where applicable, are given in square parentheses. The meaning of the input data is as follows :

DCELL *a b c alpha beta gamma*

The direct cell with lengths in Å and angles in degrees. Ideally it should be the most accurate cell available (see above). If all entries are given as zero, the unit cell will be taken from the first *Denzo* .x file.

RCELL $a^* b^* c^* \cos(\alpha^*) \cos(\beta^*) \cos(\gamma^*)$

This is the reciprocal cell corresponding to the above direct cell. If all entries are given as zero, the program will calculate the reciprocal cell from a non-zero DCELL entry, or failing that from the first *Denzo* .x file.

BMAT1 $B(1,1) B(1,2) B(1,3)$

The first 3 elements of the reciprocal space orthogonalization matrix B (as defined by Busing and Levy (1967) *Acta Cryst* **22**, 457-464) for the cell given in the DCELL entry above. If all entries are given as zero, the program will calculate the elements of the B matrix. Entries BMAT2 and BMAT3 are similarly defined.

UMAT1 $U(1,1) U(1,2) U(1,3)$

The first 3 elements for the orientation matrix U , such that the matrix UxB follows the same definition as the standard CAD4 orientation matrix. This matrix is the same as the U-matrix printed at the top of each .x file, except it is rotated by 180° about the y-axis (in the Nonius laboratory axial definition), *i.e.* the signs for all elements in rows 1 and 3 are interchanged. Entries UMAT2 and UMAT3 are similarly defined.

ALPHAK α

This is the Kappa-block support angle, which is ~ 50°. It is an instrumental constant determined in the factory and will not need changing. It is different for each KappaCCD goniometer. In the Windows version, this constant is read from the file KAPPASUPPORT.DAT, which should be placed in the WinGX files directory (default c:\wingx\files). In the absence of such a file, a default value of 50.0° is assumed.

INVERT (yes/no) [no]

Prior to the 2000 release of the *Nonius-Collect* software, the indices of reflections in the *Denzo* .x files used to be inverted. Select “yes” if you wish to invert the reflection indices to correct this bug. The effect of this bug is to invert the chirality of the model for non-centrosymmetric structures, *i.e.* it is serious. In the current release of the *Nonius-Collect* software this bug has been corrected and “no” should be selected. The program *makedenzox* will attempt to guess whether this inversion is required, but the method is not fool-proof. If you are uncertain as to whether inversion is needed on your system, then data should be collected on a crystal of known absolute configuration and both options tested.

DIRCOS (yes/no) [yes]

Select “yes” or “no” to write/not write direction cosines to the output reflection files. Older versions of the *Nonius-Collect* software produce *Denzo* .x files which

lack the goniostat parameters. As a result, it is not possible to calculate direction cosines with these .x files.

ORIENT (yes/no) [yes]

By default, the orientation matrix U is taken from the header of each individual .x file and used to recompute the UB matrix for the direction cosines. In this way, any changes in the crystal orientation which occurred during the data collection are taken into account, though the unit cell metrics remain fixed throughout. This is the preferred method. If “no” is selected the initial orientation is kept fixed throughout the processing. NOTE : if more than one orientation matrix is present, then selecting “no” will cause serious problems. The initial orientation is taken either from the UMAT entries, or from the first .x file if these entries are zero.

SCALEF (yes/no) [no]

By default, any scale-factor file *ScaleFactors.sortav* (created by the program *Sortav*) found in the working directory will be ignored. If you wish to use the individual frame scale factors from this file, select “yes”. Note that the scale-factors should be computed using only the *fully-recorded* reflections from the .x files, *i.e.* from a previous run of *DENZOX* with SUMPAR (see below) set to “no”.

SUMPAR (yes/no) [yes]

By default, the partial reflections will be summed and written to the output file. If you wish to calculate individual frame scale-factors with *Sortav*, then this should be done using the output file of *DENZOX* which contains only the *fully-recorded* reflections, *i.e.* select “no” for this option.

REJECT (yes/no) [yes]

In order to calculate accurate direction cosines, the exact oscillation angle ψ (about either goniometer ω or ϕ) needs to be determined for each reflection. This is done by computing an incremental angle $\delta(\psi)$ to be added to ψ_{mid} , the oscillation angle at the middle of the scan, so that Bragg condition is satisfied. The mean (absolute) value of this angle (DANG) is printed at the end of the listing file. It should normally be no more than half the scan-width.

There are two problem cases:

- For certain reflections this angle cannot be computed, presumably because the tip of the scattering vector crosses the sphere of reflection obliquely (*e.g.* for high χ values). All solutions for $\delta(\psi)$ are found to be imaginary, and the resulting reflection (almost always a partial) is rejected. These are denoted as having *indeterminable* oscillation angles.
- In other cases, the computed correction angle $\delta(\psi)$ is both greater than the estimated mosaicity *and* greater than 3 times the scan width, and must be regarded as dubious. These reflections are denoted as having *unreliable* oscillation angles.

If “yes” is selected (the default), then all reflections suffering from the second problem will be rejected from the output reflection files (though they are included in the summary statistics). In an average data set, generally fewer than 100 reflections are excluded in this fashion. If direction cosines are not calculated, these tests are not undertaken and the REJECT directive has no effect.

REINDX (yes/no) [yes]

If a change of orientation matrix is found, the program will by default re-index the reflections written to the output files, so that all indices refer to a single orientation matrix. To prevent this action, set this directive to “no” - this is generally very inadvisable.

BATCHN n [-1]

If a value greater than zero is given for n , this value will be written to the output files as the batch-number. If a value of zero is given, then the action is to assign batch numbers corresponding to the frame numbers for fully recorded reflections, or the intensity-weighted average frame number for partially recorded reflections. If a value less than zero is given, then the action is to assign batch numbers corresponding to the scan-set number, *i.e.* all reflections from .x files *s01f#####.x* will be given batch number 1. This is probably the most useful option and is the default action.

MAXINC (maxinc) [0.7]

If the cosine of the angle between the incident beam and the detector is greater than *maxinc* the reflection will be rejected. This is to eliminate reflections with very oblique incidence angles, since detectors may be less sensitive in this region. The default value generally results in only a small percentage of rejections - to reduce the number of rejections, reduce the value of *maxinc*.

STLMAX $\sin(\theta)/\lambda_{max}$ [10.0]

Reflections with $\sin(\theta)/\lambda$ greater than this value will be excluded from the output reflection files. The default value of 10.0 ensures that no reflection is rejected under this criterion for all reasonable values of λ .

STLMIN $\sin(\theta)/\lambda_{min}$ [0.0]

Reflections with $\sin(\theta)/\lambda$ less than this value will be excluded from the output reflection files. The default value of 0.0 ensures that no reflection is rejected under this criterion.

CHISQR χ^2 value [10.0]

Reflections with a χ^2 of profile fit greater than the given number will be excluded from the output reflection files. The default value of 10.0 generally results in very few rejections under this criterion. For partial reflections the *maximum* χ^2 value from the set is used as the criterion.

SCANSET pattern

Only .x files corresponding to this pattern will be read and used. Commenting out individual **SCANSET** lines (with a “#” as the first character) provides an easy way of controlling the scan-sets processed with **DENZOX**. By default all scan-set patterns found in the directory will be used.

3.1.2.5 Output files

Five output files are written :

- **DENZOX.LST** listing file - same as screen output
- **DENZOX.SAD** reflection file suitable for input to **SADABS**
- **DENZOX_HKL.SORTAV** reflection file suitable for input to **SORTAV**
- **DENZOX.LOG** log file showing all rejected and re-indexed reflections
- **DENZOX.MATRIX** the CAD4 style orientation matrix

The log file (which can become very large) contains information on all the rejected and re-indexed reflections. The number of rejections will of course depend partly on the values given in the above directives. If a significant number of reflections are rejected because of problems with the oscillation angles, then check your output very carefully. A sample of the records written to this file is :

```

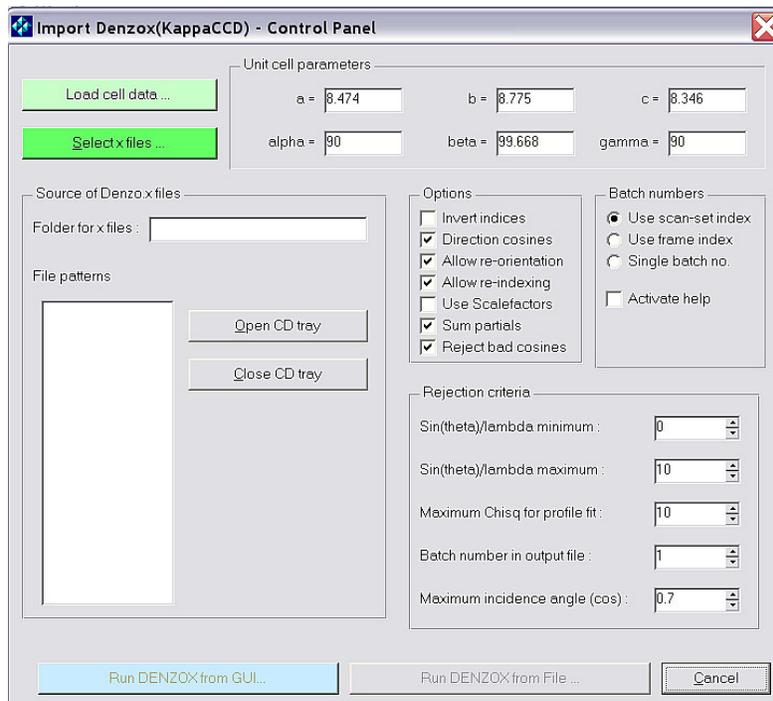
NEGSIGMA      4  -4  11  0   536.8   486.6   1.37 -51.1  0.775  449.6  615.9  2.477
OVERFLOW     11  -1   7  0 1990812 2033134   4.43 59024 0.804  226.4  615.4  2.519
CHISQREJ    of 10.5700   2   2  -2 28429.50   3
SCANEDGE    op                13   1   1 18796.80   1   2
OSCILANG    op 10.2480  -4   0   7   -11.70  151
BADORIEN    pp   8.1570  -3  20   4   102.53  604
SINTHLIM    of   0.0270  14   2   0 35976.70   2
RE-INDEX    op Original=   23   1  -12   New=  -23   1   12

```

All records start with an identifier which gives the reason for rejection. The **NEGSIGMA** and **OVERFLOW** records merely echo the relevant line from the *Denzo* .x file, but all other records have a similar format. All start with a two character reflection-status string, the first character "o" or "p" indicating an omega or phi scan, the second letter "f" or "p" indicating a full or partial reflections. A following floating point number (if present) is the value of the variable causing the rejection, e.g. for the **OSCILANG** and **BADORIEN** flags this number is the value of the computed incremental oscillation angle $\delta(\psi)$. Next come the reflection indices followed by the intensity. The next integer is the frame number (or intensity weighted frame number for partials), except for the **SCANEDGE** record which shows the first and last frame numbers for the individual partials comprising the summed partial. One of these will be at the start or end of a scan set. The **RE-INDEX** record shows the original and new reflection indices. These should be related by an operation of the Laue point group - see re-indexing matrices given at the end of the listing file.

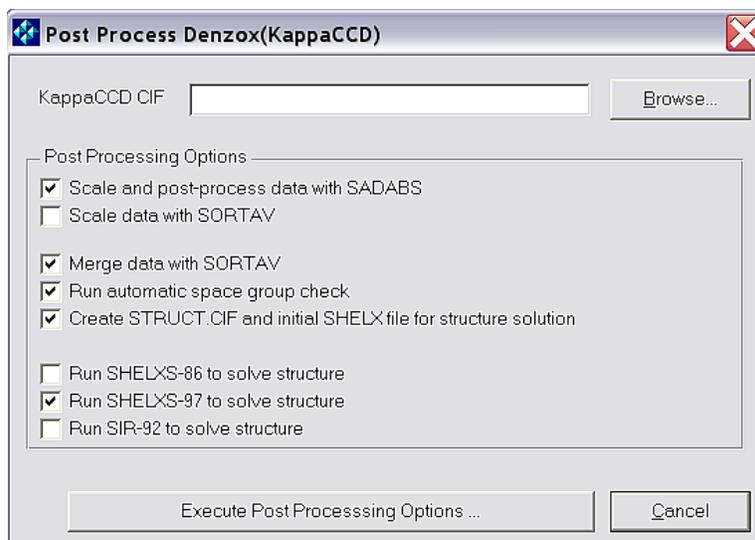
3.1.2.6 Running Denzox from WinGX

The program is run from its own GUI, obtained from the menu item *Data* → *Area Detectors* → *Import DENZOX(KappaCCD)* .



The creation of the input file *denzox.input* is automatic. The user merely needs to select the directory containing the *Denzo* .x files. If this directory contains the file *import.cif*, this will be automatically scanned for an accurate cell obtained by post refinement with *Scalepack*. Otherwise the user will either have to type in the accurate cell, or navigate to a suitable file (the filter on the navigate button indicates the available file formats). The *Denzo* .x files may reside on any disk drive (local or networked) or even on a CDROM. Click the "Select .x files" button and navigate to the directory of choice and then select *any* file in the directory. The required options are set via the GUI, but note that not all program options are accessible (defaults are mostly suitable). When both the .x file directory has been selected, and an accurate cell loaded, then the "Run DENZOX from GUI" button will become green (*i.e.* active). Click this button to run the program. All files are written to the current work directory, regardless of the source of the diffractometer files.

Text output is directed to the *WinGX* TextServer Window, and shows the progress of the calculation. The output reflection files *denzox_hkl.sortav* and *denzox.sad* are suitable as input for further processing (absorption corrections, scaling, rejection of outliers, merging) with the programs *SORTAV* or the Bruker-Nonius program *SADABS* respectively.



The Post-process Window then opens which allows the user to select a number of options for further processing of data and writing initial starting files for structure solution. The defaults should be appropriate in most cases. If a KappCCD file *import.cif* is present and is selected, this file is used to extract the information for the data reduction summary CIF, called *dreduc.cif*. It is not essential, but useful for the final stages of a structure report.