Processing of Bruker & Nonius CCD data
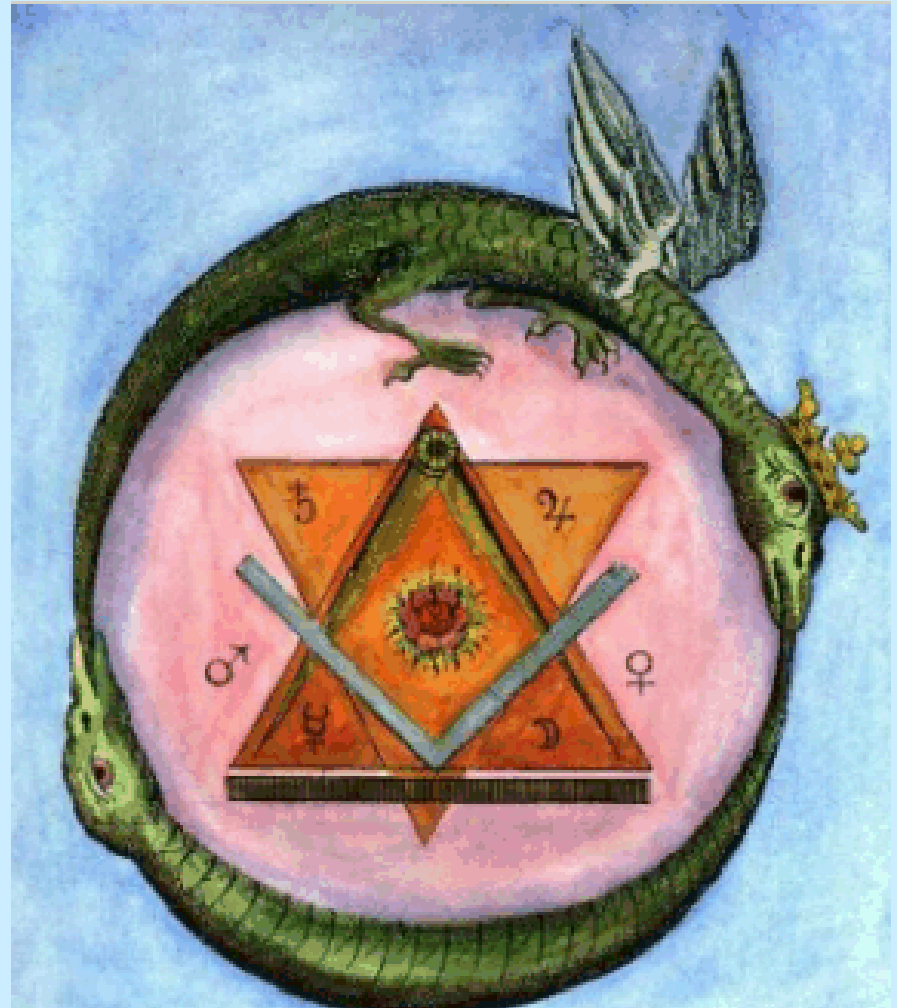
Louis J. Farrugia,
Department of Chemistry
University of Glasgow
GLASGOW G12 8QQ
Scotland

Processing of Bruker & Nonius CCD data

There are two main stages of CCD data processing :

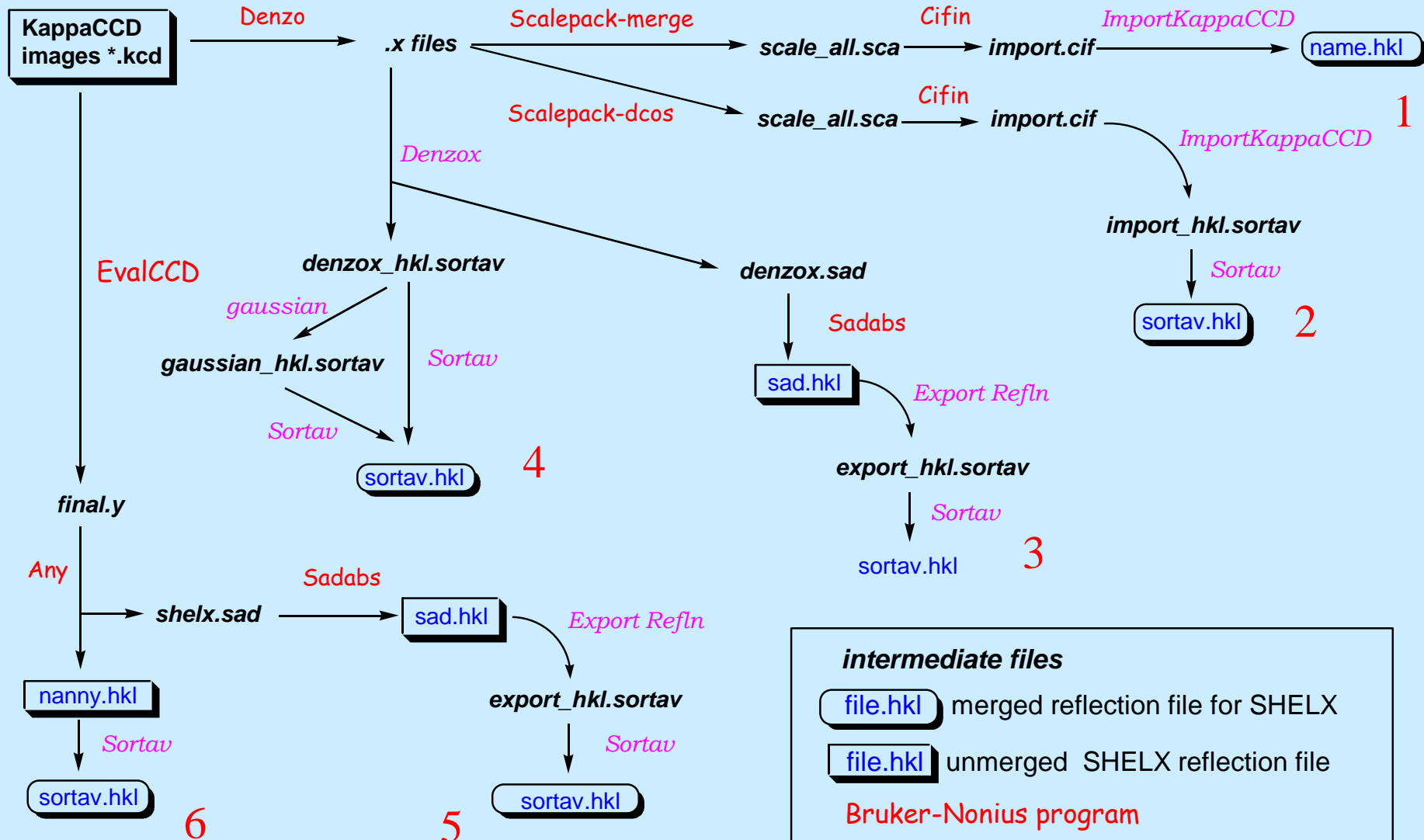• Frame integration - extraction of intensities from images
• Post processing - error modelling, merging, abs corrections, *etc*

First procedure is specialised, involves many corrections to the raw CCD data - no facilities in WinGX for this (**Denzo**, **EvalCCD**, **SAINT**).

Second procedure is handled in *WinGX* by a variety of routes

Frame integration relies on "black box" commercial software.

Single Crystal Software Workshop   ECM21-2003

Flowchart for data reduction of Bruker Nonius KappaCCD images

KappaCCD images *.kcd —**Denzo**→ **.x files** —**Scalepack-merge**→ **scale_all.sca** —**Cifin**→ **import.cif** —*ImportKappaCCD*→ name.hkl

1

.x files —**Scalepack-dcos**→ **scale_all.sca** —**Cifin**→ **import.cif** —*ImportKappaCCD*→ **import_hkl.sortav** —*Sortav*→ sortav.hkl **2**

.x files —*Denzox*→ **denzox_hkl.sortav**

.x files —*Denzox*→ **denzox.sad**

denzox_hkl.sortav —*gaussian*→ **gaussian_hkl.sortav**

denzox_hkl.sortav —*Sortav*→ sortav.hkl **4**

gaussian_hkl.sortav —*Sortav*→ sortav.hkl

denzox.sad —**Sadabs**→ sad.hkl —*Export Refln*→ **export_hkl.sortav** —*Sortav*→ sortav.hkl **3**

KappaCCD images *.kcd —**EvalCCD**→ **final.y**

final.y —**Any**→ **shelx.sad** —**Sadabs**→ sad.hkl —*Export Refln*→ **export_hkl.sortav** —*Sortav*→ sortav.hkl **5**

final.y → nanny.hkl —*Sortav*→ sortav.hkl **6**

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intermediate files

file.hkl merged reflection file for SHELX

file.hkl unmerged SHELX reflection file

Bruker-Nonius program
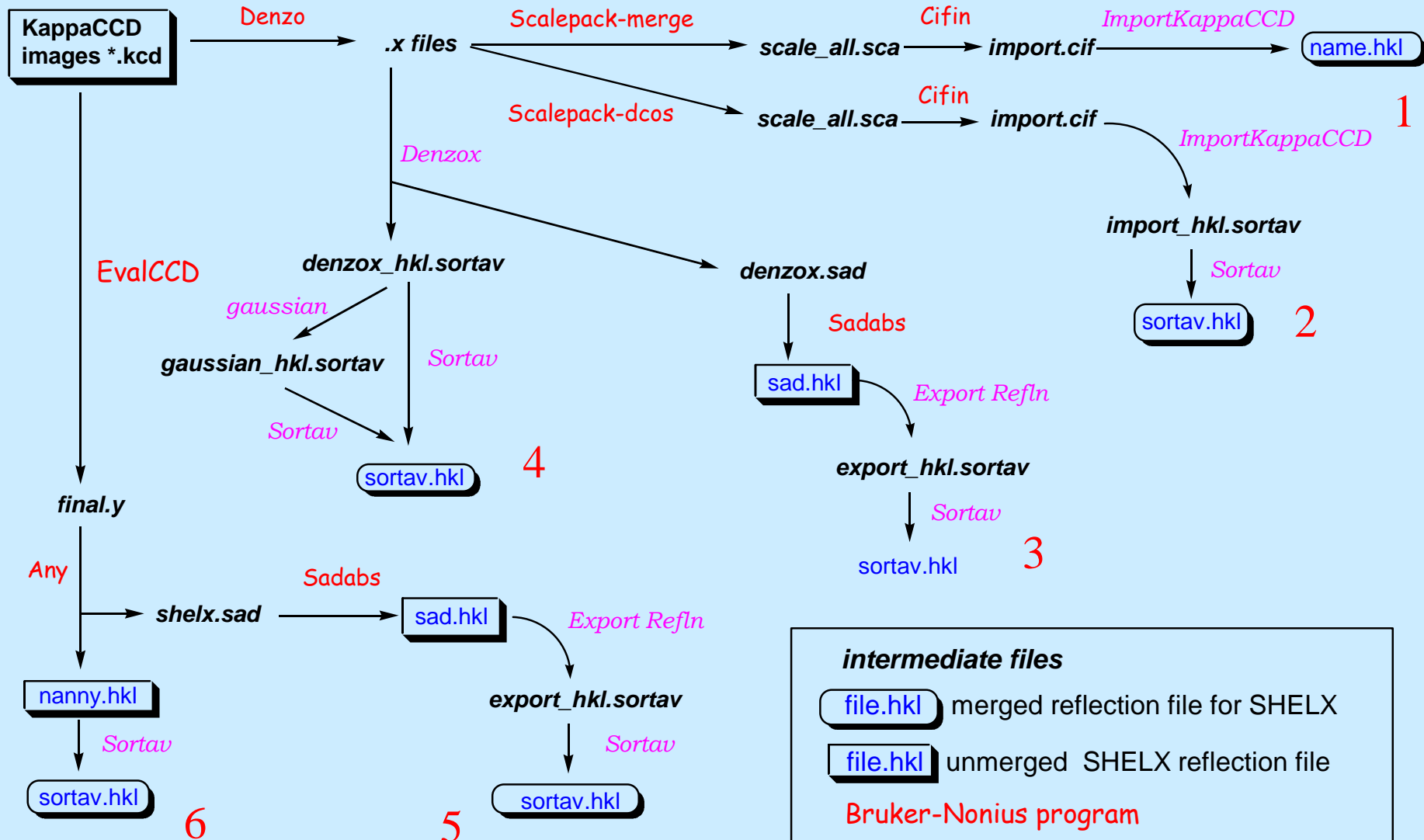
WinGX program

Denzox program

Denzox was written by Bob Blessing, but extensively modified in Glasgow

- Reads .x files produced by Denzo and concatenates data
- does little massaging of reflection intensities
- transfers *fulls* transparently - no change in I or $\sigma(I)$
- simple statistical summation of *partials* to provide I and $\sigma(I)$
- some rejection criteria - χ^2 of fit, partials at edge of scan set, $\sin\theta/\lambda$
- calculates direction cosines
- re-indexing for equivalent orientation matrices (NOT re-orientation !)
- various possibilities for batch number - frame, scan-set, experiment
- output file *denzox_hkl.sortav* for Sortav (or Gaussian abs correction)
- output file *denzox.sad* for Sadabs

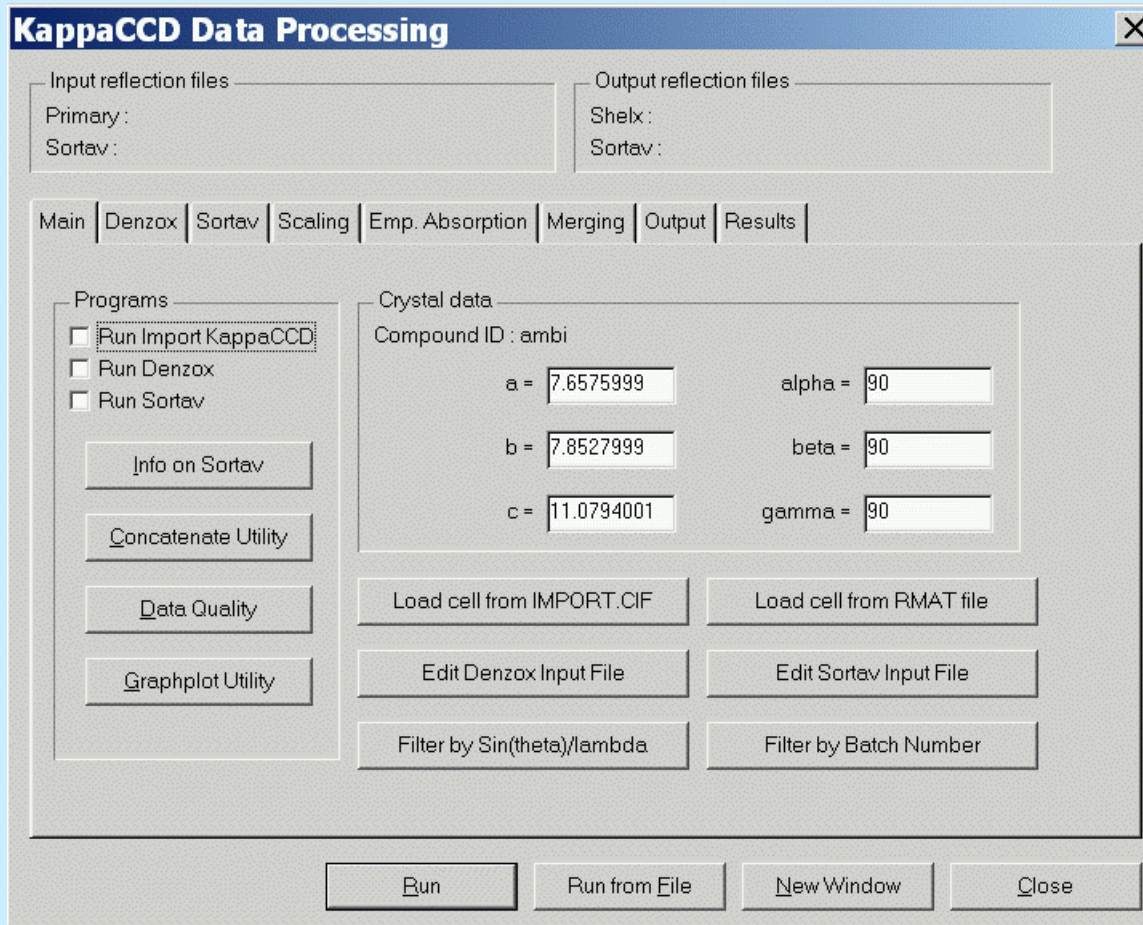**Flowchart for data reduction of Bruker Nonius KappaCCD images**

KappaCCD images *.kcd → *Denzo* → **.x files**

.x files → *Scalepack-merge* → **scale_all.sca** → *Cifin* → **import.cif** → *ImportKappaCCD* → name.hkl

1

.x files → *Scalepack-dcos* → **scale_all.sca** → *Cifin* → **import.cif** → *ImportKappaCCD* → **import_hkl.sortav** → *Sortav* → sortav.hkl

2

.x files → *Denzox* → **denzox_hkl.sortav**

denzox_hkl.sortav → *gaussian* → **gaussian_hkl.sortav**

gaussian_hkl.sortav → *Sortav* → sortav.hkl

denzox_hkl.sortav → *Sortav* → sortav.hkl

4

.x files → *Denzox* → **denzox.sad** → *Sadabs* → sad.hkl

sad.hkl → *Export Refln* → **export_hkl.sortav** → *Sortav* → sortav.hkl

3

KappaCCD images *.kcd → *EvalCCD* → **final.y**

final.y → *Any* → **shelx.sad** → *Sadabs* → sad.hkl

sad.hkl → *Export Refln* → **export_hkl.sortav** → *Sortav* → sortav.hkl

5

final.y → nanny.hkl → *Sortav* → sortav.hkl

6

intermediate files

file.hkl merged reflection file for SHELX

file.hkl unmerged SHELX reflection file

Bruker-Nonius program

WinGX program

Processing of KappaCCD data

Summary of Data Reduction methods for AMBI (ammonium bitartrate)

	Denzo Scalepack-m	Denzo Scalepack	Denzo Denzox/Sadabs	Denzo Denzox/Sortav	EvalCCD Sadabs	EvalCCD Sortav
	route1	**route2**	**route3**	**route4**	**route5**	**route6**
Total unique	1930	1923	1931	1935	1952	1953
Observed	1641	1684	1684	1700	1523	1525
R(sigma)	0.0366	0.0357	0.0316	0.0332	0.0427	0.0436
w$R2$	0.810	0.0818	0.0795	0.0792	0.0813	0.0815
$R1$ (obs)	0.0326	0.0336	0.0331	0.0334	0.0385	0.0385
$R1$ (all data)	0.0428	0.0429	0.0426	0.0425	0.0618	0.0619
$\Delta\rho$	0.18 -0.22	0.20 -0.22	0.23 -0.20	0.23 -0.18	0.20 -0.22	0.19 -0.21

Processing of KappaCCD data

KappaCCD Data Processing

Input reflection files
Primary :
Sortav :

Output reflection files
Shelx :
Sortav :

Main | Denzox | Sortav | Scaling | Emp. Absorption | Merging | Output | Results

Programs
☐ Run Import KappaCCD
☐ Run Denzox
☐ Run Sortav

Info on Sortav

Concatenate Utility

Data Quality

Graphplot Utility

Crystal data
Compound ID : ambi

a = 7.6575999   alpha = 90

b = 7.8527999   beta = 90

c = 11.0794001   gamma = 90

Load cell from IMPORT.CIF   Load cell from RMAT file

Edit Denzox Input File   Edit Sortav Input File

Filter by Sin(theta)/lambda   Filter by Batch Number

Run | Run from File | New Window | Close

Load cell from various sources

Filter data by $\sin(\theta)\lambda$ or batch no.

Quick view of data quality by Weiss or Diederich - Karplus programs

Concatenation of input files

Plots (Sortav)

Most data reduction programs handled through KappaCCD GUI

Processing of KappaCCD data

Data Quality ✕

This utility provides a quick indication of data quality using several different statistical descriptors, i.e. the standard Rmerge (Rsym), the redundancy-independent Rmeas (Rrim), the pooled coefficient of variation (PCV) or the precision indicating R-value (Rpim).   For a definition of these descriptors, see :

K. Diederichs and P.A. Karplus, Nature Struct. Biology (1997) 4, 269,   and
M. S, Weiss, J. Appl. Cryst (2001), 34, 130.

15963 reflections read from BATCH1_HKL.SORTAV   Diederichs/Karplus data quality analysis finished
3079 unique merged reflections for Laue class   2/m
15962 observations in merging calculations

Reflection data | R-values | Rmrg I-values

Bin	Res (A)	Rmeas (%)	(Rsym) (%)	PCV (%)	<I-over-sigma> measured data	<I-over-sigma> merged data	fract (%)	multi
1	1.27	4.9 (	4.4)	7.8	40.0 (40.0)	105.5 (****)	13.8	6.8
2	0.90	4.3 (	4.0)	7.3	32.2 (32.2)	94.6 (94.6)	17.5	8.5
3	0.76	4.8 (	4.5)	8.6	24.8 (24.8)	68.3 (68.3)	15.6	7.6
4	0.68	6.0 (	5.5)	10.5	20.0 (20.0)	52.6 (52.6)	14.1	6.9
5	0.63	6.6 (	6.0)	10.4	15.1 (15.1)	37.9 (38.0)	12.6	6.3
6	0.59	7.9 (	7.1)	12.3	12.9 (12.9)	29.1 (29.9)	9.9	5.1
7	0.56	7.6 (	6.2)	10.2	12.7 (12.6)	20.6 (21.6)	5.1	2.6
8	0.53	9.5 (	7.7)	13.3	9.9 (9.9)	15.5 (16.6)	4.2	2.5
9	0.51	12.9 (	10.2)	18.3	7.8 (7.7)	12.0 (12.7)	4.4	2.4
10	0.49	15.4 (	12.3)	23.1	7.2 (7.1)	10.1 (11.3)	2.9	2.1
Totals		5.0 (	4.5)	16.4	22.4 (22.6)	45.8 (49.0)	100.0	5.2

Load cell from various sources

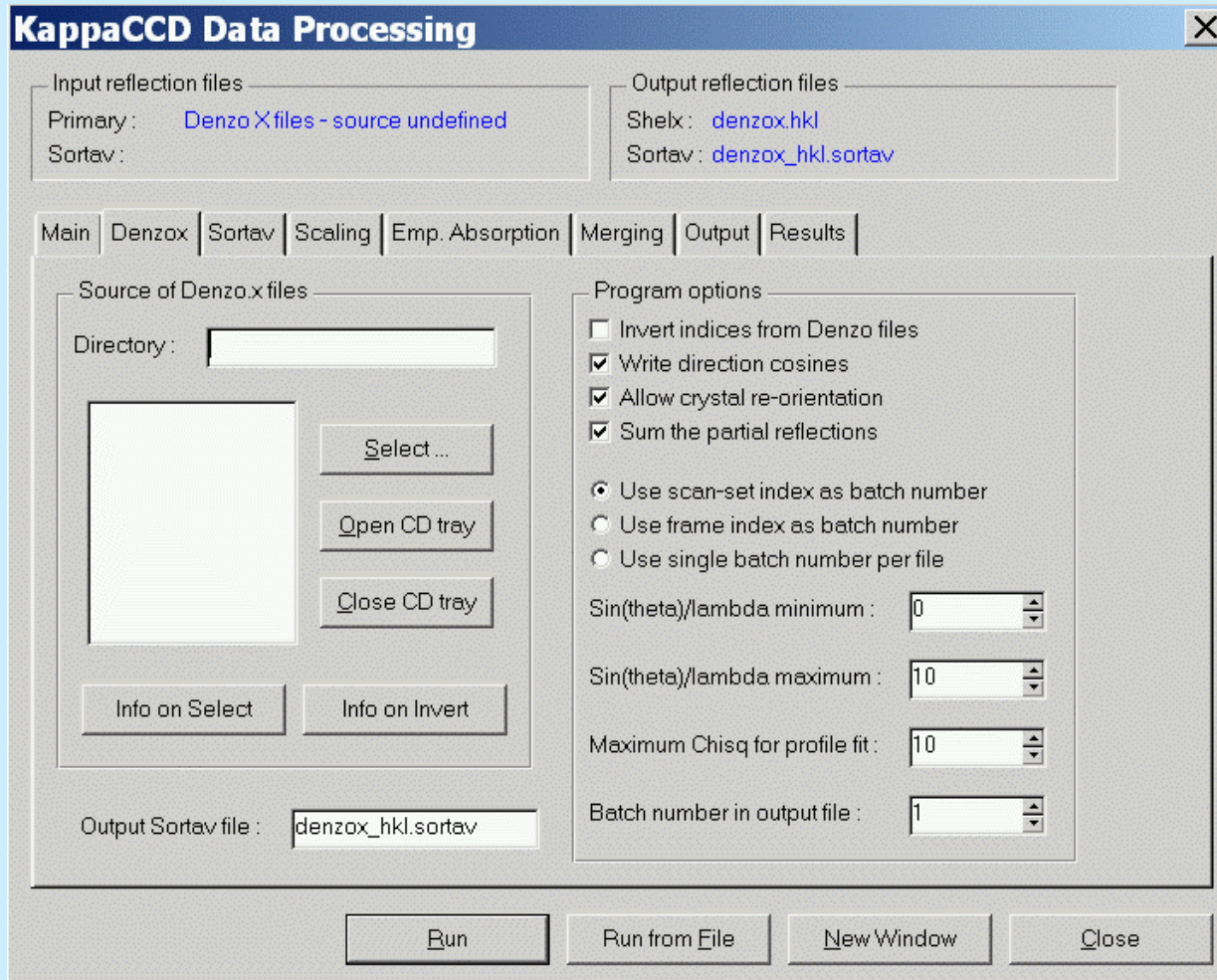Filter data by $\sin(\theta)\lambda$ or batch no.

Quick view of data quality by Weiss or Diederich - Karplus programs

Concatenation of input files

Plots (Sortav)

Most data reduction programs handled through KappaCCD GUI
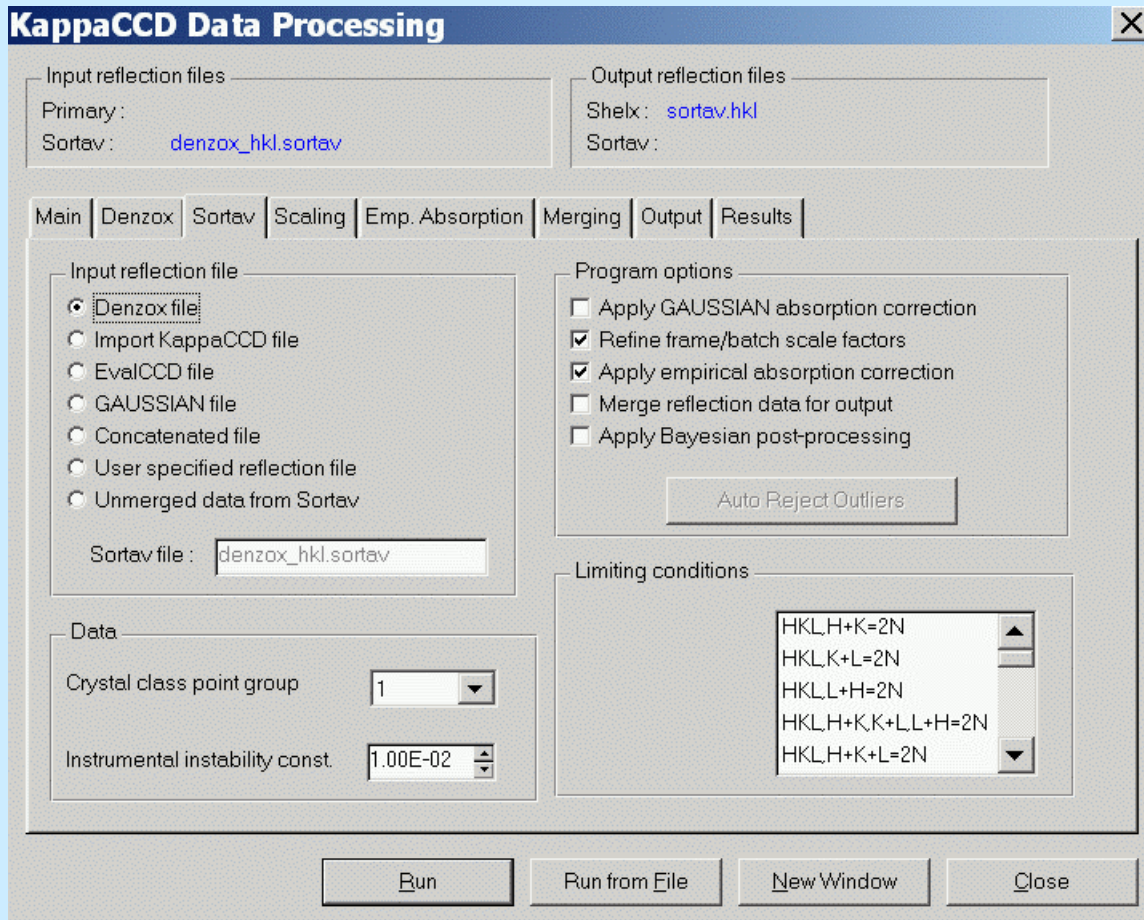
Denzox GUI

Denzox by R. Blessing

Reads .x files and calculates direction cosines + batch no.'s

Writes out files *denzox_hkl.sortav* *denzox.sad*

Reads .x files from disk files or CD-ROM

Allows for Denzo matrix variation

Sortav GUI

KappaCCD Data Processing

Input reflection files
Primary :
Sortav :   denzox_hkl.sortav

Output reflection files
Shelx :   sortav.hkl
Sortav :

Tabs: Main | Denzox | Sortav | Scaling | Emp. Absorption | Merging | Output | Results

Input reflection file
- ⦿ Denzox file
- ○ Import KappaCCD file
- ○ EvalCCD file
- ○ GAUSSIAN file
- ○ Concatenated file
- ○ User specified reflection file
- ○ Unmerged data from Sortav

Sortav file :   denzox_hkl.sortav

Data
Crystal class point group   1
Instrumental instability const.   1.00E-02

Program options
- ☐ Apply GAUSSIAN absorption correction
- ☑ Refine frame/batch scale factors
- ☑ Apply empirical absorption correction
- ☐ Merge reflection data for output
- ☐ Apply Bayesian post-processing

Auto Reject Outliers

Limiting conditions
HKL,H+K=2N
HKL,K+L=2N
HKL,L+H=2N
HKL,H+K,K+L,L+H=2N
HKL,H+K+L=2N

Run   Run from File   New Window   Close

Sortav by R. Blessing
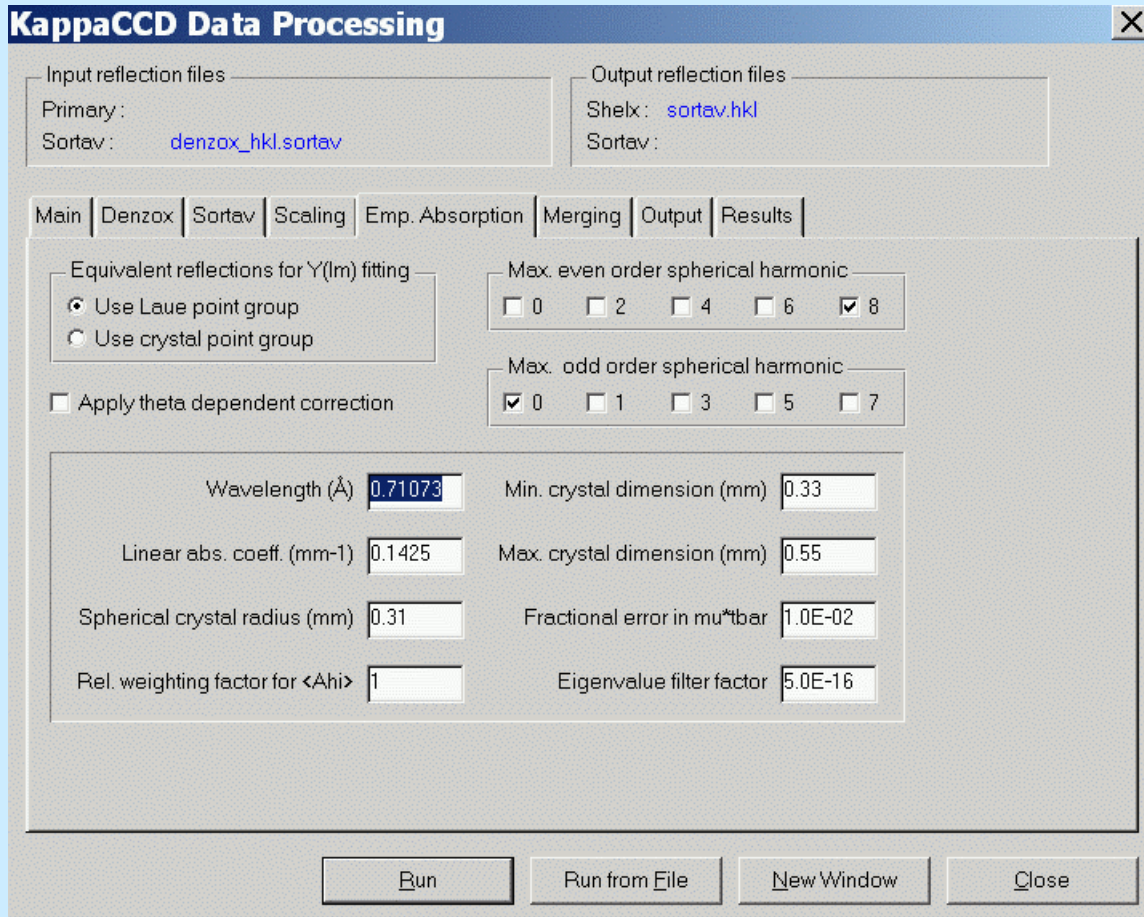
Pre-correction with GAUSSIAN abs. cor.

Apply frame/batch scaling

Apply empirical absorption correction

Data merging & reject outliers

Check that parameters are correct !
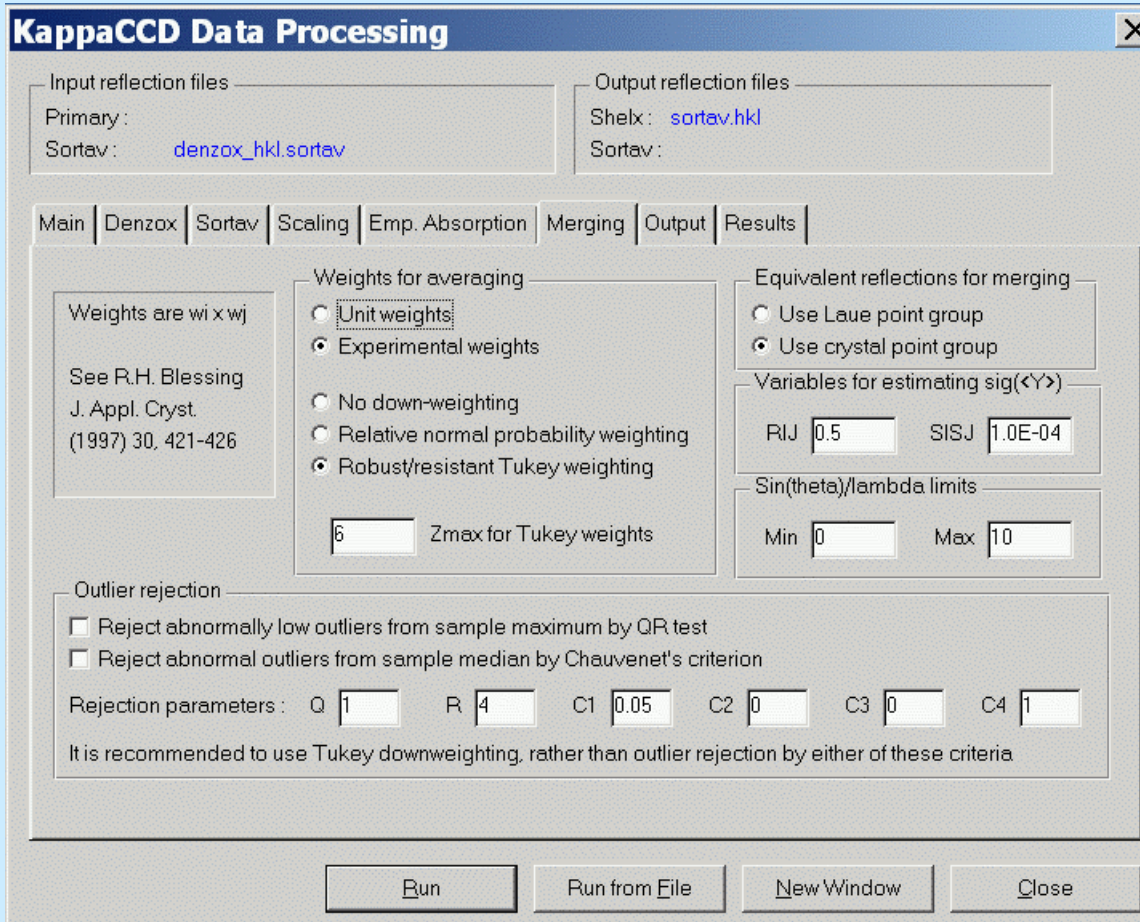
Sortav GUI - Empirical absorption

KappaCCD Data Processing

Input reflection files
Primary :
Sortav :   denzox_hkl.sortav

Output reflection files
Shelx :   sortav.hkl
Sortav :

Main | Denzox | Sortav | Scaling | Emp. Absorption | Merging | Output | Results |

Equivalent reflections for Y(lm) fitting
- ⦿ Use Laue point group
- ○ Use crystal point group

Max. even order spherical harmonic
☐ 0   ☐ 2   ☐ 4   ☐ 6   ☑ 8

☐ Apply theta dependent correction

Max. odd order spherical harmonic
☑ 0   ☐ 1   ☐ 3   ☐ 5   ☐ 7

Wavelength (Å)	0.71073	Min. crystal dimension (mm)	0.33
Linear abs. coeff. (mm-1)	0.1425	Max. crystal dimension (mm)	0.55
Spherical crystal radius (mm)	0.31	Fractional error in mu*tbar	1.0E-02
Rel. weighting factor for <Ahi>	1	Eigenvalue filter factor	5.0E-16

[Run]   [Run from File]   [New Window]   [Close]

Requires sufficient data redundancy

Uses spherical harmonics to model absorption anisotropy

Odd harmonics used to model non-centrosymmetric systematic errors

θ-dependency also possible

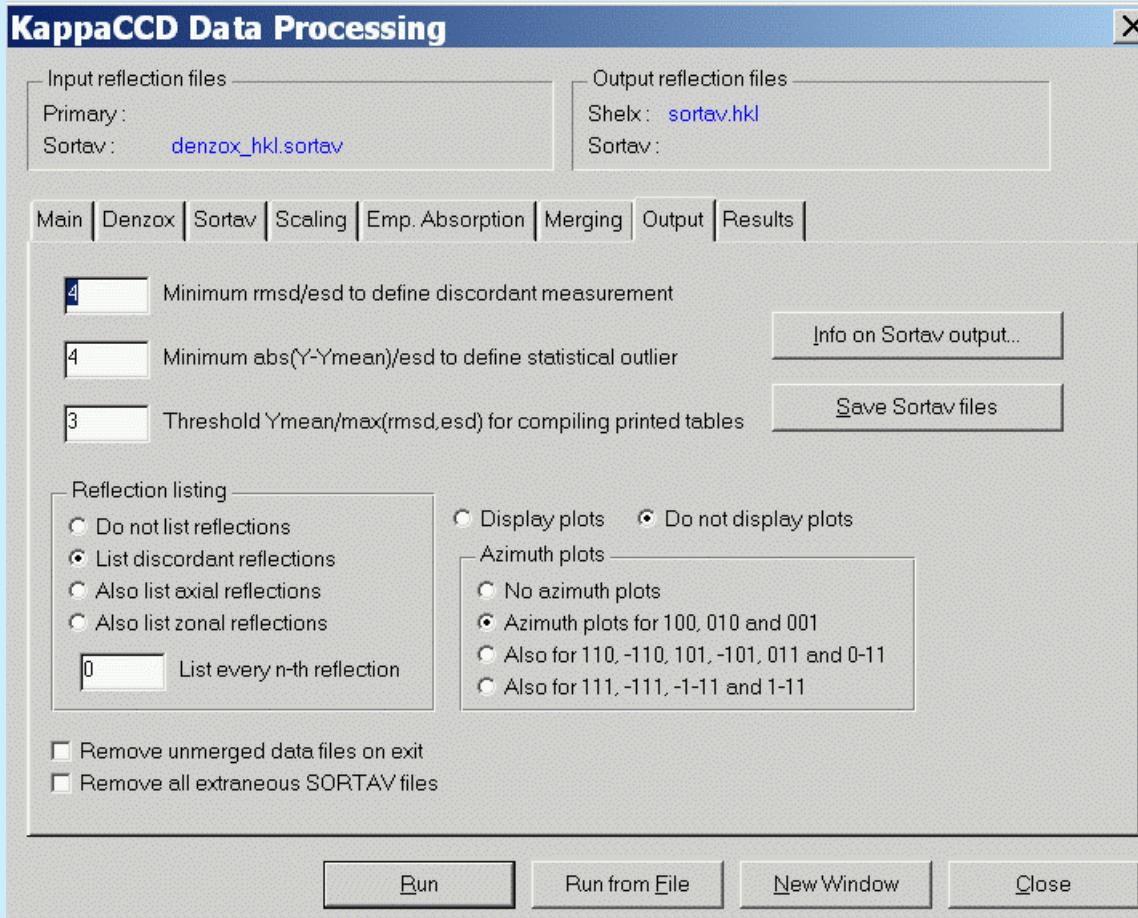R. H. Blessing, *Acta Crystallogr., Sect A* 1995, 51, 33-38.

Sortav GUI - Merging

Several schemes for outlier rejection

Normally use Tukey downweighting and rejection of outliers
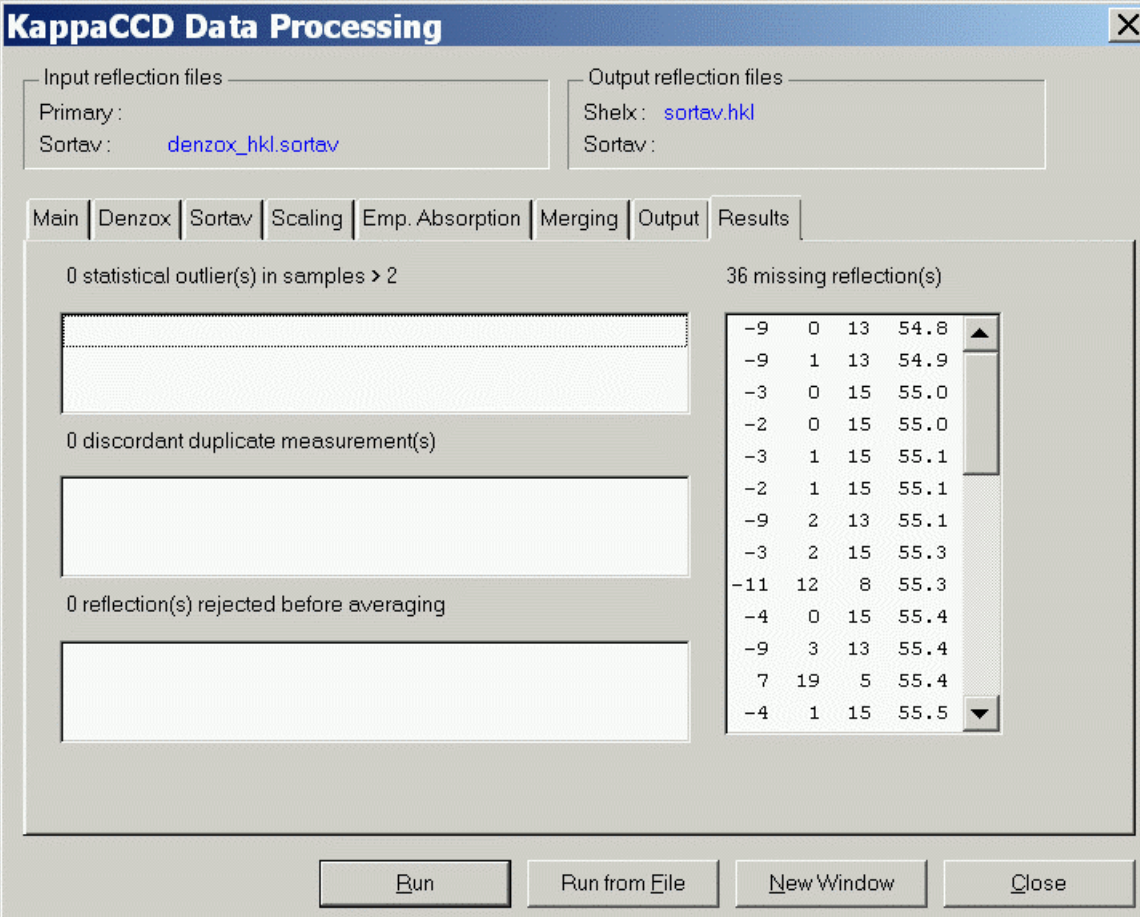
"Autoreject Outliers" until no further ones found

R. H. Blessing, *J Apply Cryst* 1997, 30, 421-426

Sortav GUI - Output

KappaCCD Data Processing

Input reflection files
Primary :
Sortav :   denzox_hkl.sortav

Output reflection files
Shelx :   sortav.hkl
Sortav :

Main | Denzox | Sortav | Scaling | Emp. Absorption | Merging | Output | Results

4   Minimum rmsd/esd to define discordant measurement

4   Minimum abs(Y-Ymean)/esd to define statistical outlier

3   Threshold Ymean/max(rmsd,esd) for compiling printed tables

Info on Sortav output...

Save Sortav files

Reflection listing
- ○ Do not list reflections
- ● List discordant reflections
- ○ Also list axial reflections
- ○ Also list zonal reflections
- 0   List every n-th reflection

○ Display plots   ● Do not display plots
Azimuth plots
- ○ No azimuth plots
- ● Azimuth plots for 100, 010 and 001
- ○ Also for 110, -110, 101, -101, 011 and 0-11
- ○ Also for 111, -111, -1-11 and 1-11

☐ Remove unmerged data files on exit
☐ Remove all extraneous SORTAV files

Run | Run from File | New Window | Close

Save intermediate results here

Controls number of plots and verbosity of output.

Remove unwanted files

R. H. Blessing, *J Apply Cryst* 1997, 30, 421-426

Sortav GUI - Results

Shows outliers and rejected reflections

Shown missing reflections

R. H. Blessing, *J Apply Cryst* 1997, 30, 421-426

Processing of SMART/SAINT data

SMART-SAINT RAW File(s) Summary

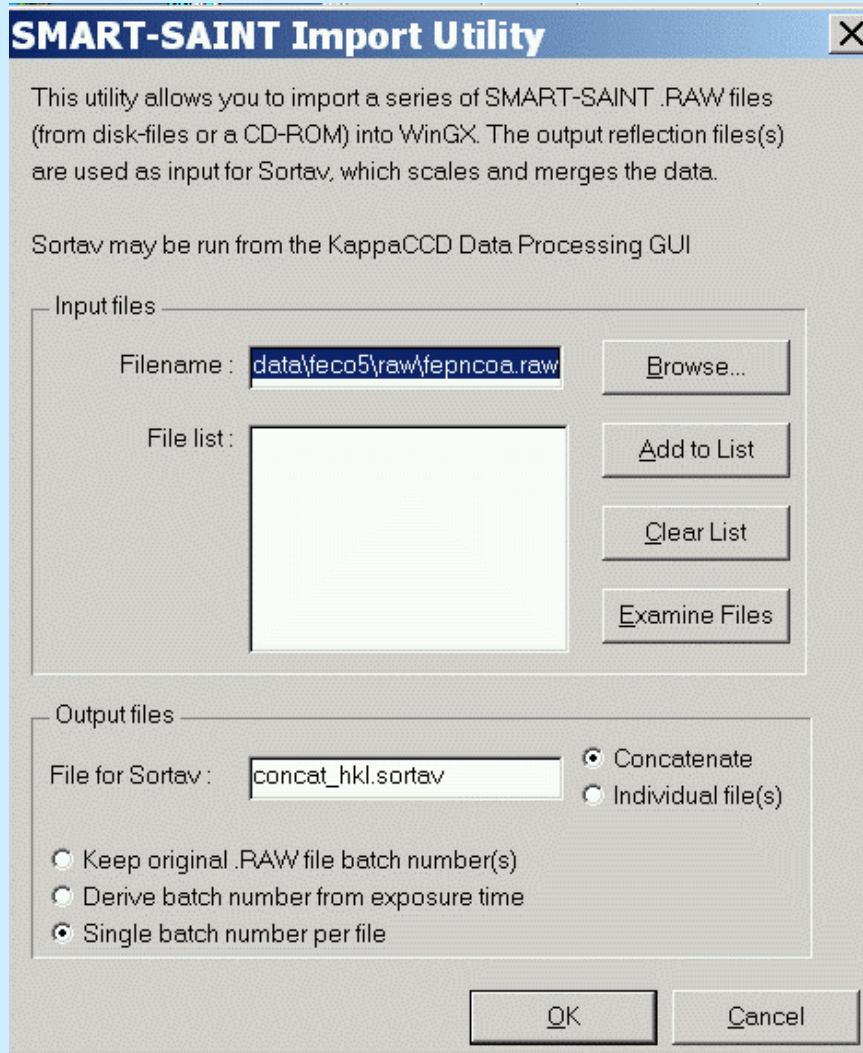|	Intensity ranges		Exposure time (min)		Frame numbers		No. reflns
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	
FEPNCOA	-146.72099	58332.6992	4	596	4	597	2452
FEPNCOB	-74.964996	58147.3007	602	1200	3	600	2474
FEPNCOC	-68.375999	53412.6015	1203	1799	4	600	2504
FEPNCOD	-100.32299	41693.3984	1802	2054	3	255	1034
FEPNCOE	-62.098999	105834	2056	2354	3	600	1969
FEPNCOF	-31.742000	93237	2356	2654	3	600	1981
FEPNCOG	-32.820999	100514	2656	2953	4	598	1975
FEPNCOH	-92.516998	103624	2956	3253	4	598	1969
FEPNCOI	-42.411998	172722	1	99	3	596	989
FEPNCOJ	-32.826999	148280	101	200	4	600	999
FEPNCOK	-58.915000	109070	200	299	3	600	1020
FEPNCOL	-29.718000	140085	1	100	4	598	961
FEPNCOM	-28.516000	162600	100	200	3	600	988
FEPNCON	-55.997001	190336	200	299	5	598	963
FEPNCOO	-439.25399	551677	299	310	3	596	1031
FEPNCOP	-290.51699	533255	310	319	4	600	1043
FEPNCOQ	-107.28900	444682	319	329	3	600	1047
NFEPNCOA	-97.099998	44182.3007	4	596	4	597	2452
NFEPNCOB	-64.400001	44854	602	1200	3	600	2474
NFEPNCOC	-44.900001	49512.5	1203	1799	4	600	2504

OK   Cancel

Reads in all .RAW files found in directory

Provides ranges of Intensities, exposure time, frame numbers.

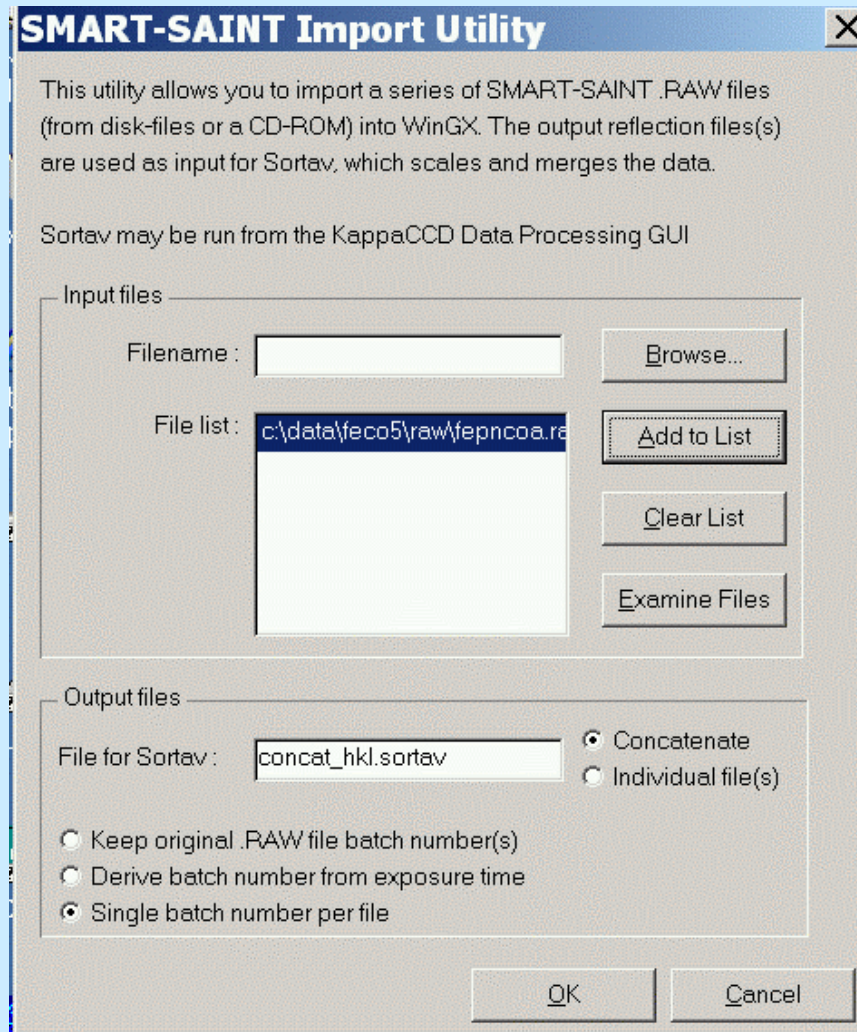Necessary to provide batch numbers for scaling.

In this example, files should be processed in *four* batches

Processing of SMART/SAINT data

Choose which sets of RAW files to concatenate

Processing of SMART/SAINT data

SMART-SAINT Import Utility

This utility allows you to import a series of SMART-SAINT .RAW files (from disk-files or a CD-ROM) into WinGX. The output reflection files(s) are used as input for Sortav, which scales and merges the data.

Sortav may be run from the KappaCCD Data Processing GUI

Input files

Filename : [   ]   Browse...

File list : c:\data\feco5\raw\fepncoa.ra   Add to List

Clear List

Examine Files

Output files

File for Sortav : concat_hkl.sortav   ⦿ Concatenate   ○ Individual file(s)

○ Keep original .RAW file batch number(s)
○ Derive batch number from exposure time
⦿ Single batch number per file

OK   Cancel

Choose which sets of RAW files to concatenate

Decide on way to derive batch numbers for scaling

Write file *concat_hkl.sortav* which is exact translation of the data in RAW file.

Suitable for merging *etc* with Sortav