# Chapter 4.1

## NUMERICAL

Louis J. Farrugia Dept. of Chemistry University of Glasgow G4 9DS, Scotland, U.K. email: <u>louis@chem.gla.ac.uk</u> These methods provide mathematically exact absorption corrections, providing of course that the input data are correct. The first two methods require that the crystal faces have been indexed, and the distances of the crystal faces from the centre of rotation have been measured. This can be very difficult to do accurately.

The reflection file that is actually used for input (though indicated as *name.HKL* in the following descriptions) is the file selected through the "Set HKL File" menu item If more than one SHELX style reflection file is found, the following window is displayed :

Select HKL File					
More than one SHELX reflection file has been found. Please select a file.					
The selected reflection file will be used for all refinements and other jobs requiring reflection data, e.g. AssignSpaceGroup. If the standard reflection file is the only one found, this dialog box will not be displayed.					
RUC012.HKL	Standard reflection file	dcos			
	Analytical absorption correction	dcos			
CAD4.HKL	SHELX file of unknown origin/processing	dcos			
CAD4HKL.HKL	SHELX file of unknown origin/processing				
CAMEL_JOCKEY.HKL	_JOCKEY.HKL Camel-Jockey empirical correction				
CYLINDRICAL.HKL	Cylindrical crystal correction	dcos			
DELABS.HKL	DELABS refined correction				
DIFABS.HKL	DIFABS refined correction				
GAUSSIAN.HKL	Gaussian quadrature absorption correction	dcos			
MULTISCAN.HKL	Spherical harmonic absorption correction				
NEEDLE.HKL	Needle crystal correction (CAD4)	dcos			
PLATON.HKL	SHELX file of unknown origin/processing	dcos			
PLATON_XOR.HKL	SHELX file of unknown origin/processing	dcos			
PSIABS.HKL	Psi-scan empirical correction	dcos			
SHELXA.HKL	SHELXA spherical harmonic correction				
SPHERICAL.HKL	Spherical crystal correction	dcos			
XABS2.HKL	XABS2 Fourier refined correction				
	<u>K</u>	<u>C</u> ancel			

This window indicates whether the reflection file contains direction cosines. For many absoprtion corrections, these are essential. The Serial data processing programs XCAD4/PROFIT will normally include direction cosines in their output reflection files, but this will not normally be the case for Area detector processed data.

The auxilliary program XtalView is provided as a useful check that the proposed set of indices and distances accurately describe the crystal. XtalView reads the file FACES.DEF (section 4.1.6) and allows the user to edit it interactively. This file is read by the programs ANALYTICAL and GAUSSIAN.

## 4.1.1 - ANALYTICAL Absorption Correction

🔀 Absorption - Analytical	
Analytical absorption correction, implemented in de Meulenaar & Tompa, Acta Cryst (1965) A19, Alcock, Cryst Computing (1970) (Ed F. R. Ahmed	1014. and
Linear absorption coef mu (mm-1) 2.657	Options Check for validity of direction cosines Print all corrected reflections in list file Retain direction cosines in output file
	<u>Q</u> K <u>C</u> ancel

## Synopsis

The procedure of de Meulenaar, Tompa and Alcock as implemented in PLATON is used. The required data are the indices of the faces (which must make up a closed polyhedron) and the distance of the centre of rotation of the polyhedron to each face (in mm). The required files are name.INS and name.HKL. The latter file MUST contain direction cosines corresponding to the cell in the name.INS file. The direction cosines are removed in the output file.

## Files read

name.INS (for unit cell information only).

name.HKL - uncorrected reflection file.

FACES.DEF - face indices and distances. This file may also be created interactively by the program.

## Files written

ANALYTICAL.HKL - absorption corrected reflection file ABSORB.LST - PLATON list file giving a list of the applied corrections ABSORB.CIF - CIF containing relevant information

## 4.1.2 - GAUSSIAN Absorption Correction

🗙 Absorption - Gaussian	
Absorption correction by Gaussian quadrature, Coppens, Leiserowitz & Rabinovic, Acta Cryst	
To use the automatic grid selection, leave NX=	NY=NZ=0.
Linear absorption coef mu (mm-1) 2.657 Number of grid points NX: 0 Number of grid points NY: 0	Options Check for validity of direction cosines Print all corrected reflections in list file Retain direction cosines in output file
Number of grid points NZ : 0	
	<u>Q</u> K <u>C</u> ancel

## Synopsis

The method of Gaussian integration as implemented in PLATON is used. The required data are the indices of the faces (which must make up a closed polyhedron) and the distance from the centre of rotation of the polyhedron to each face (in mm). The required files are name.INS and name.HKL. The latter file MUST contain direction cosines corresponding to the cell in the name.INS file. The direction cosines are removed in the output file.

## Files read

name.INS (for unit cell information only) name.HKL - uncorrected reflection file FACES.DEF - face indices and distances. This file may also be created interactively by the program.

## Files written

GAUSSIAN.HKL - absorption corrected reflection file ABSORB.LST - PLATON list file giving a list of the applied corrections ABSORB.CIF - CIF containing relevant information

## 4.1.3 - SPHERICAL Absorption Correction

🗙 Spherical Crystal Absorption 🛛 👔	X
Spherical absorption approximation, to provide a theta dependence of the absorption surface. If muR exceeds 10.0, the program is halted as the errors become too great. The corrections are interpolated from data in the International Tables.	
Mean radius of crystal(mm)	
<u>O</u> K <u>C</u> ancel	

## Synopsis

This provides a correction for spherical crystals, effectively the theta dependence of the absorption surface. If muR exceeds 10.0 the program is aborted, as the errors will be too large. The only required input is the mean spherical radius of the crystal in mm. The correction is calculated by interpolation of data from International Tables. If direction cosines are present in the input reflection file, these will be echoed to the output file.

Files read

name.INS (for unit cell information only) name.HKL - uncorrected reflection file

Files written SPHERICAL.HKL - absorption corrected reflection file ABSORB.CIF - CIF containing relevant information

## 4.1.4 - CYLINDRICAL Absorption Correction

🔀 Cylindrical Crystal Absorption	
	o provide a theta dependence of the absorption surface. Ited as the errors become too great. The corrections ational Tables.
Mean radius of crystal(mm) 🛛 🛓	<ul> <li>Display relative correction factor</li> <li>Display absolute transmission factor</li> <li>Retain direction cosines in output file</li> </ul>
	<u>O</u> K <u>C</u> ancel

## Synopsis

This provides a correction suitable for cylindrical or needle crystals, where the crystal is mounted along the cylinder axis. If muR exceeds 10.0 the program is aborted, as the errors will be too large. The only required input is the mean cylindrical radius of the crystal in mm. The correction is calculated by interpolation of data from International Tables. If direction cosines are present in the input reflection file, these will be echoed to the output file.

## Files read

name.INS (for unit cell information only) name.HKL - uncorrected reflection file

## Files written

CYLINDRICAL.HKL - absorption corrected reflection file ABSORB.CIF - CIF containing relevant information

## 4.1.5 - NEEDLE Absorption Correction

🔀 NEEDLE Absorption Correction - Control Panel					
Absorption correction for needle-shaped crystals, specific for CAD4 data (bisecting mode). An optional correction can be made for capillary absorption (give non-zero wall thickness).					
The following assumptions apply :					
<ol> <li>The crystal is a needle with an approximately spherical cross-section, which is smaller than the x-ray beam.</li> </ol>					
<ol> <li>The needle dimension is much longer than the x-ray beam width.</li> <li>The needle and spindle (goniometer phi) axes coincide.</li> </ol>					
0.71073 Wavelength (Å)	2.6567 🛉 Crys	tal absorption coef	ř. (mm-1)		
0.15 🔹 X-ray beam radius (mm)	0.1 🗘 Capi	llary absorption co	eff. (mm-1)		
0.1 Crystal radius (mm)	16 🔹 No. g	No. grid points for crystal radius			
0 🔶 Capillary wall thickness (mm)	16 🔹 No. g	No. grid points for crystal length			
		<u>0</u> K	<u>C</u> ancel		

## Synopsis

This provides a CAD4 specific correction suitable for cylindrical or needle crystals, where the crystal is mounted along the spindle axis. It will be useful for crystals where the needle axis is very much longer than the X-ray beam width. A GUI is presented for further information and user input. If direction cosines are present in the input reflection file, these will be echoed to the output file.

## Files read

name.INS (for unit cell information only) name.HKL - uncorrected reflection file

## Files written

NEEDLE.HKL - absorption corrected reflection file ABSORB.CIF - CIF containing relevant information

## 4.1.6 The FACES.DEF File

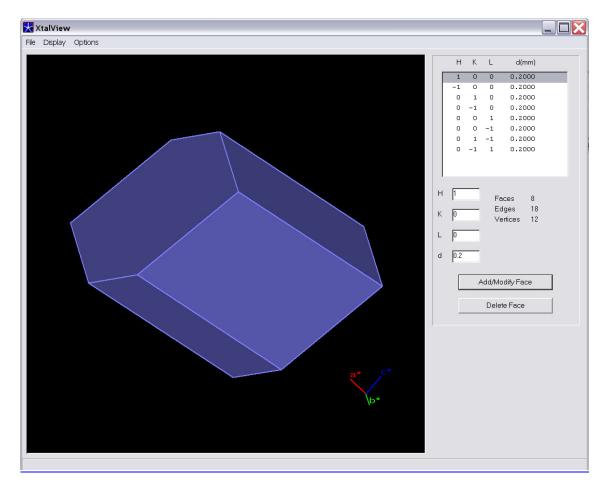
This file contains the face index and distance information about the crystal. It is a SHELX style free format text file with four-character data identifiers. It may either be created manually with a text-editor, created by the ANALYTICAL or GAUSSIAN absorption correction programs, or by the visualisation program XtalView. The format is as follows

TITL	te	st c	ryst	al shape	for Na	WO4.2H2O		
CELL	8.	4797	10	.5930 13	.8527	90.0	90.0	90.0
FACE	0	0	1	0.0387				
FACE	0	0	-1	0.0387				
FACE	1	0	0	0.1225				
FACE	-1	0	0	0.1225				
FACE	0	1	0	0.1375				
FACE	0	-1	0	0.1375				
FACE	0	1	2	0.0825				
FACE	0	-1	-2	0.0825				
FACE	0	1	-2	0.0825				
FACE	0	-1	2	0.0825				
FACE	1	0	1	0.1050				
FACE	-1	0	-1	0.1050				
FACE	-1	0	1	0.1050				
FACE	1	0	-1	0.1050				
ABSC	18.	669						
GRID	5							

The TITL card is optional if you want to put a title in the picture

The GRID entry allows the user to modify the default setting of the %age accuracy required (5%). It is only used for the GAUSSIAN correction in the Area Detector programs

## 4.1.7 The XTALVIEW program



This program allows the user to visualise the crystal faces in the file FACES.DEF and also to edit the dimensions of add new faces. The viewpoint may be changed interactively by mouse dragging and the reciprocal space axial directions are also rotateds