ABSORB

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And with a lot help from Andrzej among many others including Charlie Burnham and Larry Finger!

Implementing absorption corrections with a physical basis

X-rays only

DACs

Go through the issues and illustrate how to do it in ABSORB



ABSORB 7

- ABSORB has been developed for 50 years!
 - Which makes it older than the operating systems!
 - Originally a program for crystals in air (Burnham, 1966)
 - Some development for DACs (Finger 1970s, 80s)
 - Further development for DACs and special cases (Angel, 2004)
 - Absorb controlled by text files
- ABSORB-7 and ABSORB-GUI
 - Angel and Gonzalez-Platas (2012)
 - Separation of Absorb 'engine' and input
 - Allows direct integration in to commercial software
 - GUI for stand-alone users.
- ABSORB-7 remains a research tool
 - Lots of options
 - And some weird stuff (thanks to Clivia!)

ABSORB workflow



Absorption in the DAC: geometry





Absorption in the DAC: defining beam paths



- The DAC is fixed to the goniometer head
- A natural Cartesian reference basis is therefore a basis fixed to the goniometer head (phi-axis system)
- Define beam paths on this axial system

Busing, Levy (1967) Acta Cryst 22:457



Defining beam paths in SHELX hkl files

Direction cosines of I-beam and D-beam

- relative to crystal reciprocal axes
- Works for any detector type
- Only snag is absolute orientation of crystal not specified
- Requires UB matrix for conversion to phi-axis

📃 exa	ample2	34.hkl - Notepa	d		x
<u>F</u> ile	<u>E</u> dit	F <u>o</u> rmat <u>V</u> iew	<u>H</u> elp		
-6	1	0 5.7973	8 0.31172	1-0.23997-0.53208 0.97037-0.84621 0.02801-0.02828	-
-0 -6	ย 0	0 1069.7	8 22.2590	1-0.38709-0.38470 0.92172-0.92207 0.02437-0.02457 1-0.38537-0.38646 0.92012-0.92208 0.06957 0.02019	
-6	0	2 486.68	1 9.87660	1-0.38113-0.39070 0.91738-0.91820 0.11445 0.06531	
-6	0	3 0.0000	0 1.04327	1-0.37941-0.39243 0.91128-0.91321 0.15981 0.10988	
-6	-1	4 910.07	5 0.05248	1-0.52157-0.25023 0.82945-0.95490 0.19968 0.16005	
-6	-1	3 1.8482	6 0.12085	1-0.52550-0.24621 0.83652-0.96241 0.15495 0.11481	
-6	-1 -1	2 20.242	3 0.58635 2 0 38137	1-0.52868-0.24304 0.84164-0.96751 0.10998 0.06983 1-0 53164-0 24006 0 84446-0 97044 0 06500 0 02484	
-6	-1	0 6.0759	5 0.31568		
-6	-2	0 746.42	3 14.6594		
-6 -6	-2 -2	1 21.511	0 0.56781 1 7.67619	1-0.66655 0.70465 0.74271-0.99467 0.06024 0.02964 1-0.66421-0.10732 0.74007-0.99144 0.10526 0.07458	
	2	2 071.70	1 1.01017		Ψ.
					▶



Phi-axis definitions

- The definition of the phi-axis system, and thus U and UB, is different in different software!
 - Axis directions when diffractometer circles at zero





ABSORB works in Busing-Levy



Diffractometer parameters

AbsorbGUI Program
File Run Help
Diffractometer Experiment Crystal Model DAC
Definitions
Diffractometer Agilent Instrument (XCalibur, SuperNOVA) Vavelength Mo Lamb1: 0.70926
Axis rotation direction relative to Busing-Levy
2Theta: Same/Positive 🐨 Omega: Same/Positive 🐨 Chi: Same/Positive 🐨 Phi: Same/Positive 🐨
Coordinate System
X (Diffrac) = -Y (Busing-Levy) Y (Diffrac) = +X (Busing-Levy) Z (Diffrac) = +Z (Busing-Levy)
ile: C:\Users\rangel\Documents\RJA_Software\Reduce_Refine\Absorb\Absorb7\test\Spring2012\testcrystaldac.exp Date: 25/06/2012

And the UB matrix can now be given

AbsorbGUI Program le Run Help	constat.		in the	240			-	_ _ X
) ,								
Diffractometer Experim	ent Crystal Model	DAC						
Title	atau audas Coustal das	ariband in target of la	ld of feens					
Absorb Example: Ne	ctangular Ciystal des	cribed in terms of n	IKI OF FACES					
Cell Parameters								
a	Ь	с	alpha	beta	gamma			
Orientation Matrix (U	B)							
[✓] Containing wavel	ength (i.e: CrysAlis)		0.072410	-0.103570	-0.000750			
			-0.003440	-0.003600	0.089900			
			, 	1				
Reflection Controls								
2Theta limits:	0.00 - 180.00	S	cale Factor:	1.00				
-Reflections in Rfine	e Format							
Unobserved criteri	um: 0.00		Reflections mar	ked as rejected	will be retained			
Negative Intensities	Кеер	r Ir	ntensities with neg	ative sigmas:	Кеер 🔻			
e: C:\Users\ra	ngel\Documents\F	UA Software\Red	luce Refine\Abs	orb\Absorb7\	test\Examples 2012	\crystalair2.exp	Date:	25/06/2012
	inger (bocaments (i				test (examples_corr	(crystalanziexp	Pater	25,00,2012

DAC Absorption

Diamond Anvil Cell Definition	nystar moder of the		
V Active DAC absorption of	correction DAC Type: Gen	eral 🔻	DAC Phi Orientation: 0.00
Anvils			Gasket
	Incident-beam side		📝 Active
	Thickness (mm): 1.4	Absorp. coeff. (mm^-1): 0.00	Thickness (microns): 60.00
	Diffracted-beam side		Radius hole (microns): 95.000
Identical Anvils	Thickness (mm): 1.4	Absorp. coeff. (mm^-1): 0.00	Absorp coeff (mm^-1); 200,000
			Illuminated fraction: 0.20
Backing Plates			niuminated fraction: 0.20
C Active	Incident-beam side		Mar Free
	Thickness (mm): 0.0	Absorp. coeff. (mm 1): U.UU	
Lidentical Dec. Distan	Diffracted-beam side		Active
Identical blac. Flates	Thickness (mm): 0.0	Absorp. coeff. (mm^-1): 0.00	Linear abs. coeff. 0.00 (mm^-1):
Opening Angle			
Identical Opening	Incident-beam side: 45.00	Diffracted-beam side: 45.00	
	43.00	40.00	PSI Curve Information

DAC with beryllium seats

File Run Help		
Diffractometer Experiment	Crystal Model DAC	
	ns porrection DAC Type: General 💌	DAC Phi Orientation: 0.00
		Gasket
00 00	Thickness (mm): 1.4 Absorp. coeff. (mm^-1): 0.00	Active Thickness (microns): 60.00
- Carelin	Diffracted-beam side	Radius hole (microns): 95.000
A los	Thickness (mm): 1.4 Absorp. coeff. (mm^-1): 0.00	Absorp. coeff. (mm^-1): 200.000
		Illuminated fraction: 0.20
	Incident-beam side	
	Thickness (mm): 4.0 Absorp. coeff. (mm^-1): 0.00	Medium
	Diffracted-beam side	Active
	Thickness (mm): 0.0 Absorp. coeff. (mm^-1): 0.00	Linear abs. coeff. 0.00 (mm^-1):
	Incident-beam side: 40,00 Diffracted-beam side: 45.00	PSI Curve Information



The DAC on the goniometer

		4	
Diffractometer Experiment	Crystal Model DAC		
Mamona Anvii Celi Derinia		val 💌	DAC Phi Orientation: 20.00
Active DAC absorption			
Anvils	Incident-beam side		Gasket V Active
	Thickness (mm): 1.4	Absorp. coeff. (mm^-1): 0.00	Thickness (microns): 60.00
🖂 latan Karal Ang Ja	Diffracted-beam side		Radius hole (microns): 95.000
V Identical Anvils	Thickness (mm): 1.4	Absorp. coeff. (mm^-1): 0.00	Absorp. coeff. (mm^-1): 200.000
Backing Plates			Illuminated fraction: 0.20
✓ Active	Incident-beam side		
in the state	Thickness (mm): 4.0	Absorp. coeff. (mm^-1): 0.00	Medium
	Diffracted-beam side		Active
Identical Bac. Plates	Thickness (mm): 0.0	Absorp. coeff. (mm^-1): 0.00	Linear abs. coeff. 0.00 (mm^-1):
Opening Angle			
Identical Opening	Incident-beam side: 40.00	Diffracted-beam side: 40.00	PSI Curve Information

Small beam case

File Run Help Image: Second	Image: Spherical Spherical Using DAC Description ed Gasket Spherical Using DAC Description Image: Spherical Spherical Using DAC Description Image:	
File: C:\Users\rangel\Documents\RJA_Software	Reduce_Refine\Absorb\Absorb7\test\Spring2012\Boehler_Almax.exp	Date: 25/06/2012



Describing the crystal

- □ The crystal position in the DAC can be described by:
 - The Miller indices and distances of faces
 - Coordinates of the corners
- In addition, the position of the crystal relative to the gasket centre must be defined for gasket shadowing:







- **Capture an image**
- Beware of optical inversion and rotation!
- □ View from which side?
 - Write it down!!!
 - Name the file!!!
- This is the real view from detector side





← 250um →

The gasket gives you a length scale





- Make a simple representation of the crystal
 - Using Gaussian grid
 - Must be convex





← 250*um* →

- Measure the x,z coordinates of corners
- Scale from gasket to convert to um:

	X	z
А	-82	0
В	-38	-63
С	+58	-14
D	+36	+77
Е	-48	+67





Now load the crystal information

), Dj 🖻 🗐 🖶 🔍) 🕜 🖪	
Diffractometer Experiment Crystal Model DAC		
Crystal absorption		
Active		
Empirical Formula		icient
K2 Zr Si3 09	Z: 6	n-1): 0.0000
Lrystal Description		
Model: Faces (Corners)	Better n	ot for HP data
DAC Small Beam	Filled Gasket Soberical Using DAC Description the U.C.	nanges with Pl
Faces (HKL)		langes man .
Origi Faces (XYZ) Filled Gasket		
Spherical	XYZ Corner	^
	1	
Grid points for Gaussian Integration	2	
	4	
	5	
Verbose information		

Now load the crystal information

📅 AbsorbGUI Program	
File Run Help	
Diffractometer Experiment Crystal Model DAC	
Crystal absorption Image: Crystal absorption <	
Empirical Formula Absorption coefficient	
Image: Image	0.0000
Crystal Description Model: Faces (Corners)	
Faces (Corners) Faces (HKL) Faces (XYZ) Filled Gasket Spherical Using DAC Description	
Origin (Offset)	
XYZ Corner	A
1 -82 0 0	
Grid points for Gaussian Integration 2 -38 0 -63	_
x y z 3 580-14	_
8 ▼ × 8 ▼ × 8 ▼ 4 30077	-
Verbose information	
File: Not File defined	Date: 28/06/2012



Now load the crystal information

AbsorbGUI Program	Į	X
File Run Help		
Diffractometer Experiment Crystal Model DAC		
Crystal absorption		
I Active ■ I Active		
Empirical Formula Absorption coefficient		
	0.0000	
Crystal Description		
Model: Faces (Corners)		
Faces (Forners) Eaces (HK11) Eaces (XYZ1) Filled Gasket Spherical Using DAC Description		
Origin (Offset) Beam size much smaller than crystal (i.e. Synchroton conditions)		
Crystal on DAC		
✓ Active Crystal sitting: Anvil on incident beam side	•	
Thiskness (missens): 50.00		
Thickness (hictoris).		
File: Not File defined	Date:	28/06/2012



Ready to run the program

Diffractometer Experiment Crystal Model DAC				
Crystal absorpti Active Empirical Forr © K2Zr Crystal Descr Model: Faces (Corners) Faces (Corners) Faces (HKL) Faces (XYZ) Fill Origin (Offset)	Run the program	Absorption coefficient Mu (mm -1): Dian I (i.e. Synchroton conditions) nvil on incident beam side s): 50.00	0.0000	
File: Not File defined			Date:	28/06/2012

Test models with psi scans or equivalents



Other DAC options

	PD data	DAC_psiscan.exp - Notepad
	- Cotting engles in file	<u>File Edit Fo</u> rmat <u>V</u> iew <u>H</u> elp
	Setting angles in file	# Experimental File using AbsorbGVI Program
	Parities	# TITLE Absorb Example: Psiscan data of a crystal in DAC
	No UB	# Diffractometer Information WAVEL 1 0.70926 PARITY 1 1 1 1 BLAXES 1 2 3
	Crystal	# Experimental Information CELL 5.50220 5.65080 7.86560 90.049 89.975 90.007 ABSORB LESSTHAN 2.000
	Co-ords of corners	# Crystal Absorption Model ABSORB MU 12.200 ABSORB MODEL XYZCORNER 4 3 4 0
_		ABSORB CORNER -44.000 0.000 61.000 ABSORB CORNER 73.000 0.000 32.000 ABSORB CORNER -69.000 0.000 -32.000 ABSORB CORNER -69.000 0.000 -60.000
	DAC description	ABSORB CORNER -44.000 60.000 61.000 ABSORB CORNER 73.000 60.000 32.000
	Transmission curve	ABSORB CORNER -69.000 60.000 -32.000 ABSORB CORNER 48.000 60.000 -60.000
	Gasket	# DAC Absorption Model DAC TYPE 5 DAC OPEN 40.00 40.00 DAC GASKET 114.00 140.00 30.00 0.20 DAC ABSPSI CURVE 0.61



Absorption in DAC – key points

- For corrections on a physical basis, the DAC, crystal and data must be described on a common reference system
 - Orientation matrix
 - Axial systems
- **Keep things simple:**
 - DAC face-on to beam at circles zero
 - Use hkl files with d-cosines
- Do inter-frame detector scaling afterwards
- Diamond dips and other outliers:
 - Identify in averaging or in refinement (Fo vs Fc)



For previous users

Key changes to experiment (exp) files from Absorb 6:

- Absorption coefficients now in mm-1
- DAC component sizes in mm
- New handling of negative intensities
- New options to describe crystals in DACs
- Distinction between DAC and phi-axis coordinate systems

