

TOPAZ

GSAS/EXPGUI refinement example

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Single Crystal Neutron Diffraction Experiment

The tutorial is for locating hydrogen atoms on the water molecules in scolecite using neutron data measured on TOPAZ.

Scolecite is a member of *nat* zeolites with a porous aluminosilicate framework analogous to natrolite but a different placement of the calcium cation and water molecules in the zeolite channels.

A block-shaped crystal with the dimension of $0.62 \times 1.19 \times 1.63$ mm-cubed was cut from a prismatic scolecite crystal originated from India, and mounted with cyanoacrylate glue on the tip of a polyamide capillary to minimize the scattering from the mount. The initial orientation matrix was obtained in ISAWEV at 100 K. Using this orientation matrix, an optimized data collection strategy was calculated in CrystalPlan [1]. Data were collected using 39 crystal orientations for an estimated 99.7% coverage of symmetry-equivalent reflections to d_{\min} of 0.5 Å. An estimated 90% of the measured reflections would be measured more than once. Each orientation was measured for approximately 1.5 h.

Scolecite	$\text{CaAl}_2\text{Si}_3\text{O}_{10} \cdot 3\text{H}_2\text{O}$
Temperature	100(2) K
Radiation source	TOF neutron
wavelengths	0.4 – 3.2 Å
Crystal system	Monoclinic
Space group	C c
a, Å	6.5120(1)
b, Å	18.9545(4)
c, Å	9.7582(2)
β, °	108.921(1)
V, Å³	1139.39(4)
Z	4

[1] Zikovskiy J., Peterson P. F., Wang X. P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *J. Appl. Cryst.* **44**, 418-423 (2011)

Scolecite Data

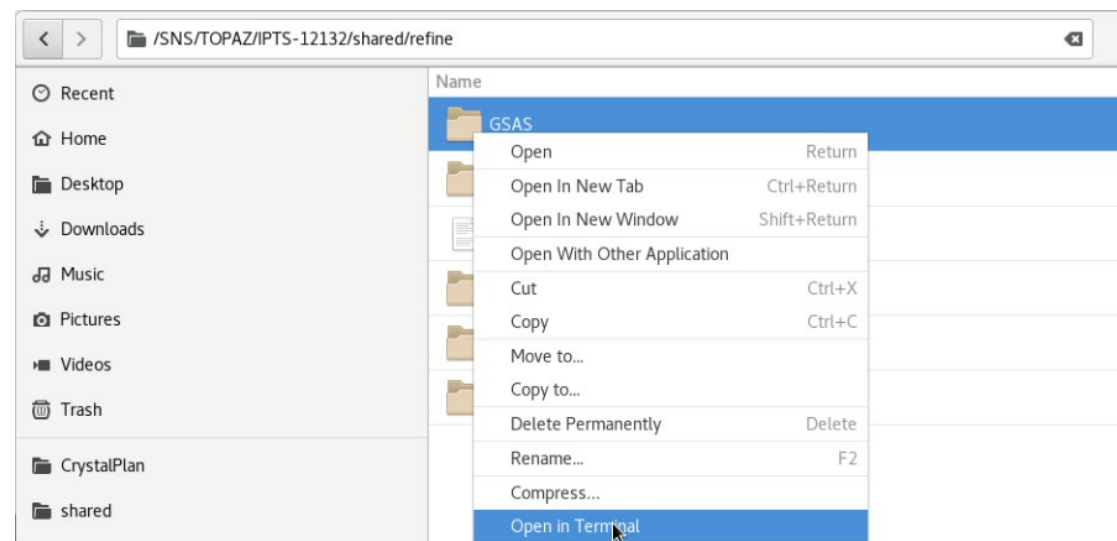
- Single crystal diffraction data are saved in /SNS/TOPAZ/IPTS-12132/data
 - Event NeXus
TOPAZ_#####_event.nxs
- **100 K Data Set**
 - Run numbers 7450:7488
- **295 K Data Set**
 - Run numbers 10074:10079,10081:10084

Start EXPGUI

- Copy the hkl data *scolecite100K_Monoclinic_C.hkl* to GSAS folder from `/SNS/TOPAZ/IPTS-12132/shared/SC100K/` to the working directory `/SNS/TOPAZ/IPTS-12132/shared/refine/GSAS/`

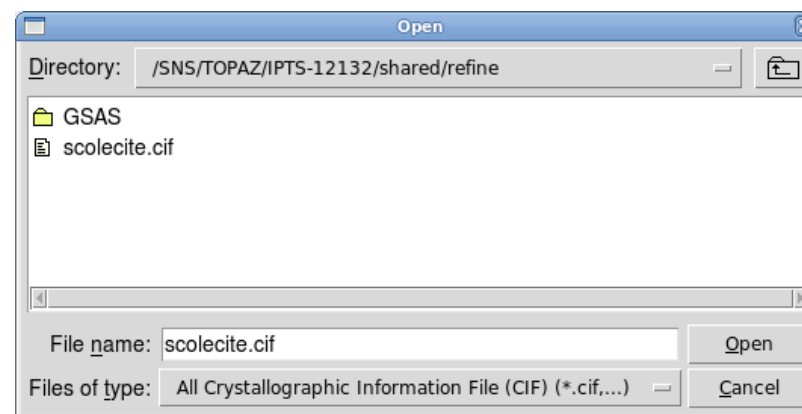
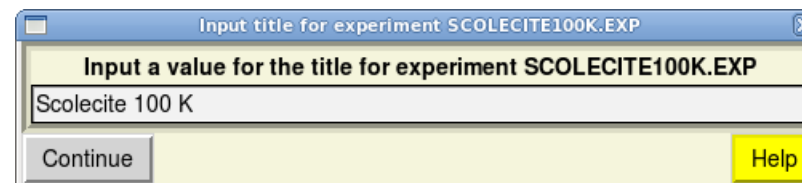
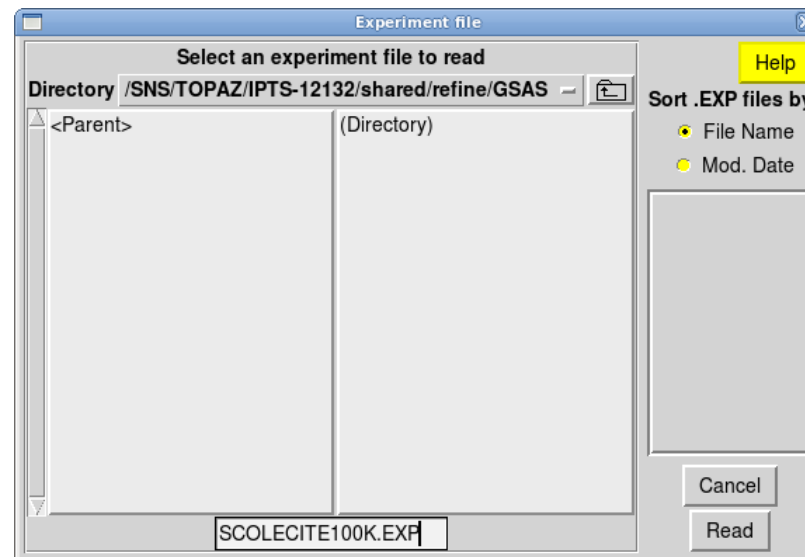
Open a terminal window in `/SNS/TOPAZ/IPTS-12132/shared/refine/GSAS` [as shown below]

- Type `ls *.hkl` to verify the hkl data is available
- Execute **expgui** in terminal window to start the GSAS program
- Start EXPGUI by typing `expgui`

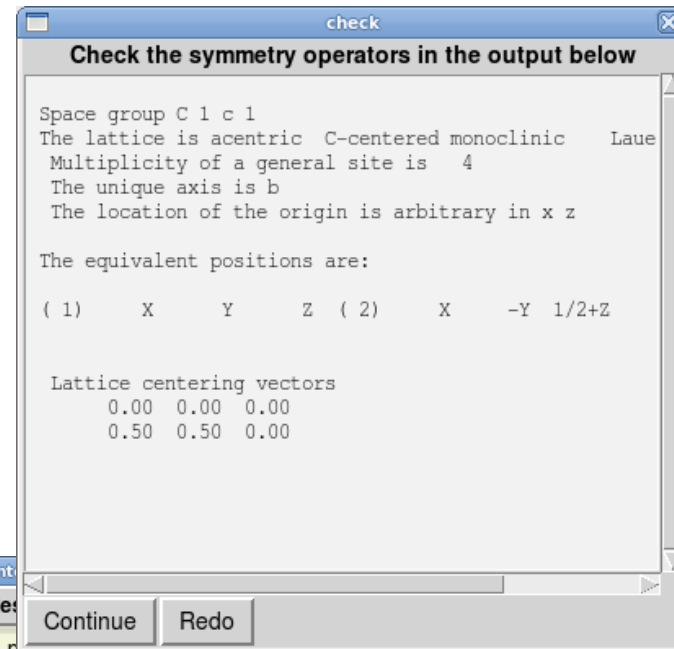
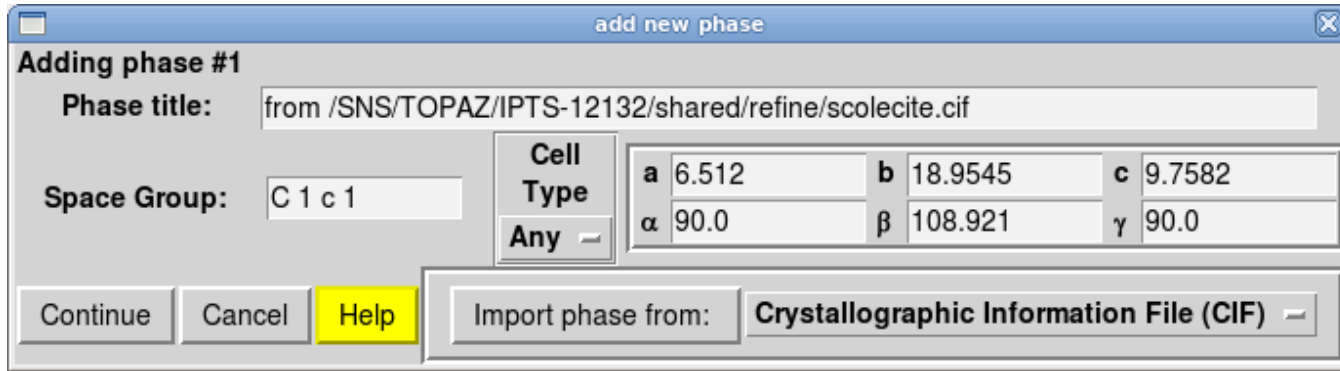


Create GSAS EXP

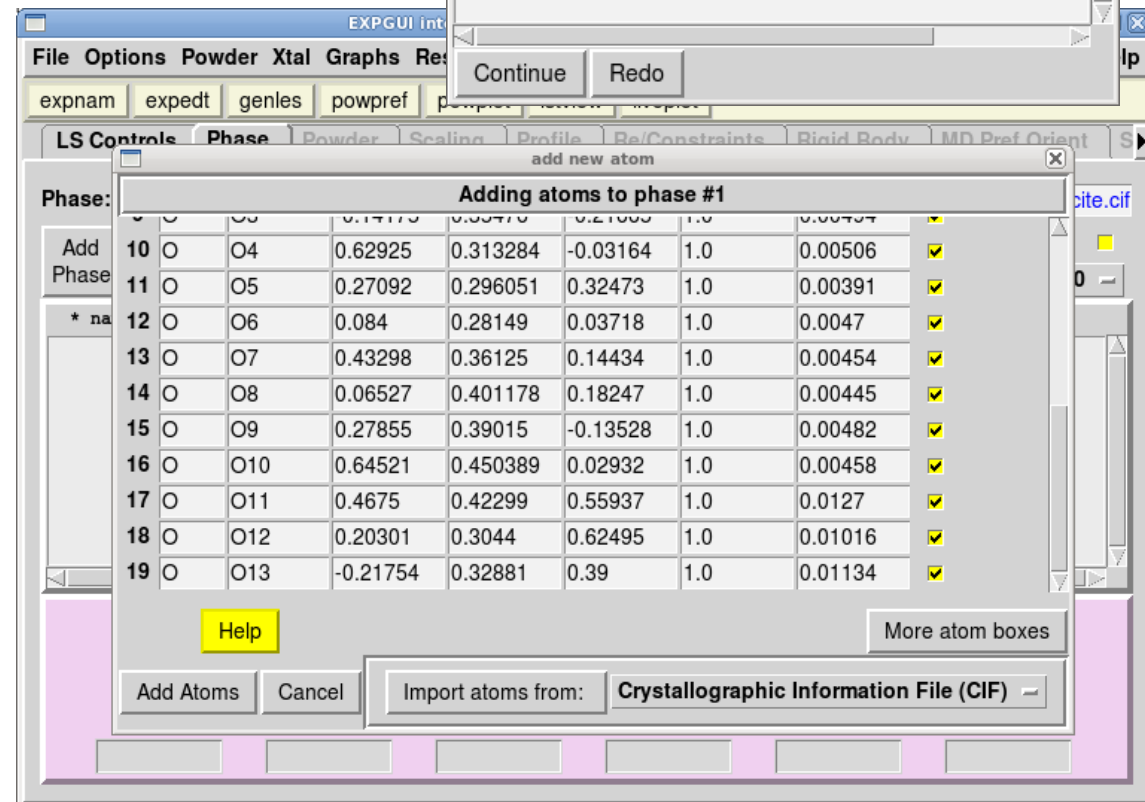
- Type in SCOLECITE100K.EXP
- Click Read
- Click Create
- Input Soclecite 100 K
- Click Continue
- Click Add Phase
- Select scolecite.cif from
/SNS/TOPAZ/IPTS-12132/shared/refine



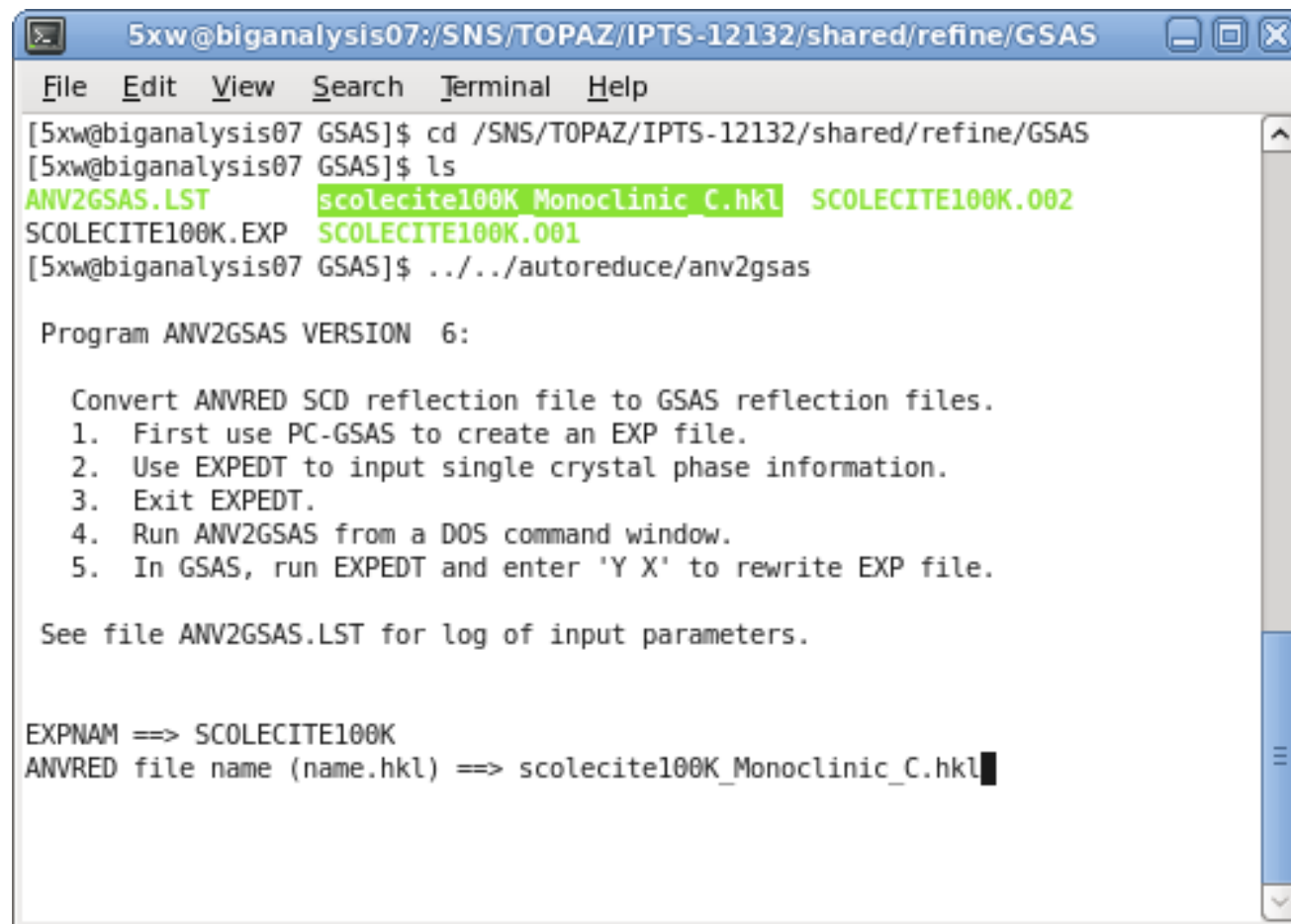
Import scolecite cif



- Save SCOLECITE100K.EXP
- Click File
Save



Convert TOPAZ Data to GSAS Format



```
5xw@biganalysis07:/SNS/TOPAZ/IPTS-12132/shared/refine/GSAS
File Edit View Search Terminal Help
[5xw@biganalysis07 GSAS]$ cd /SNS/TOPAZ/IPTS-12132/shared/refine/GSAS
[5xw@biganalysis07 GSAS]$ ls
ANV2GSAS.LST          scolecite100K Monoclinic C.hkl  SCOLECITE100K.002
SCOLECITE100K.EXP    SCOLECITE100K.001
[5xw@biganalysis07 GSAS]$ ../../autoreduce/anv2gsas

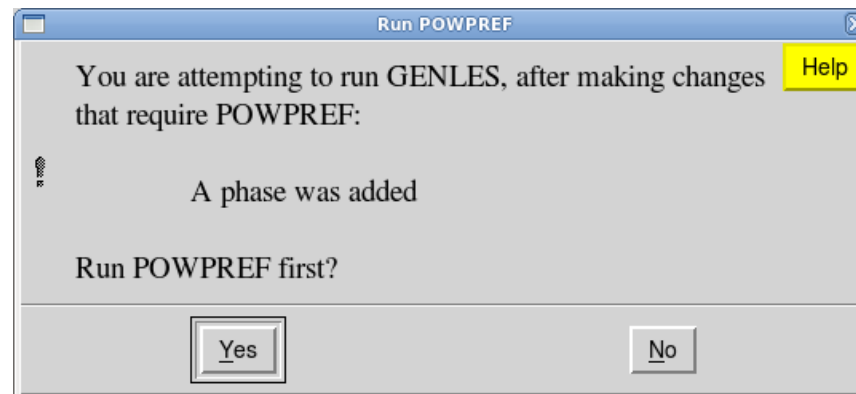
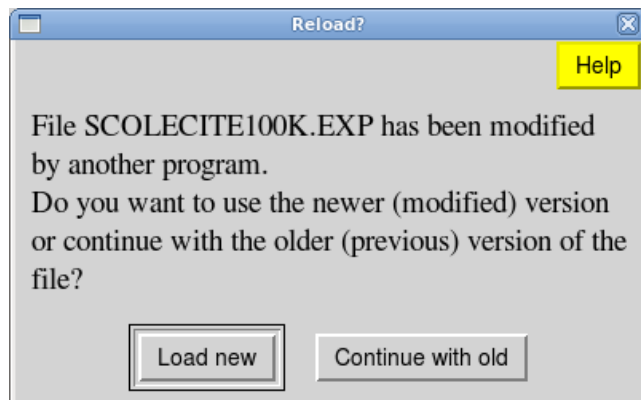
Program ANV2GSAS VERSION 6:

Convert ANVRED SCD reflection file to GSAS reflection files.
1. First use PC-GSAS to create an EXP file.
2. Use EXPEDT to input single crystal phase information.
3. Exit EXPEDT.
4. Run ANV2GSAS from a DOS command window.
5. In GSAS, run EXPEDT and enter 'Y X' to rewrite EXP file.

See file ANV2GSAS.LST for log of input parameters.

EXPNAM ==> SCOLECITE100K
ANVRED file name (name.hkl) ==> scolecite100K_Monoclinic_C.hkl
```

Run Genles



```
genes -- SCOLECITE100K
Histogram 32 Type SNT Nobs = 578 R(F) = 0.1430 CHI**2= 3.9105E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 33 Type SNT Nobs = 624 R(F) = 0.1340 CHI**2= 2.3571E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 34 Type SNT Nobs = 581 R(F) = 0.1380 CHI**2= 3.1818E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 35 Type SNT Nobs = 602 R(F) = 0.1282 CHI**2= 2.4296E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 36 Type SNT Nobs = 601 R(F) = 0.1376 CHI**2= 3.8937E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 37 Type SNT Nobs = 578 R(F) = 0.1273 CHI**2= 2.3064E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 38 Type SNT Nobs = 607 R(F) = 0.1332 CHI**2= 3.3471E+04
                    Nfree= 0 Rfree= 0.0000
Histogram 39 Type SNT Nobs = 592 R(F) = 0.1287 CHI**2= 2.7690E+04
                    Nfree= 0 Rfree= 0.0000

Single crystal Rw(Fo) = 0.205 for 23062 observations

CPU times for matrix build 1.05 sec; matrix inversion 0.00 sec
Final variable sum((shift/esd)**2) for cycle 2: 0.00 Time: 1.05 sec
Convergence was achieved and
STOP GENLES terminated successfully statement executed
Press Enter to continue
```


EXPEDT Difference Map Setup

```
expedt -- SCOLECITE100K
-----
Program EXPEDT Version Fedora
A menu driven routine to edit .EXP files
Distributed on Fri Dec 9 19:56:47 2011
-----

Allen C. Larson and Robert B. Von Dreele
Manuel Lujan, Jr. Neutron Scattering Center, MS-H805
Los Alamos National Laboratory, Los Alamos, NM 87545

Copyright, 2000, The Regents of the University of California.

The last history record is :
HSTRY 7 GENLES Fedora Jun 24 17:47:38 2014 Sdsq= 0.113E+07 S/E= 0.279E-02

Is this the file you wish to use? (<?>,D,K,Q,R,Y) >Y F
Experiment title:
Scolecite 100 K
The last history record is :
HSTRY 7 GENLES Fedora Jun 24 17:47:38 2014 Sdsq= 0.113E+07 S/E= 0.279E-02

Enter desired map (<?>,DELF,FCLC,FOBS,NFDF,PTSN,DPTS,EXIT) >DELF
```

```
The last history record is :
HSTRY 9 EXPEDT Fedora Jun 24 17:52:02 2014 File cleanup only
Is this the file you wish to use? (<?>,D,K,Q,R,Y) >Y F
Experiment title:
Scolecite 100 K
The last history record is :
HSTRY 9 EXPEDT Fedora Jun 24 17:52:02 2014 File cleanup only
Enter desired map (<?>,DELF,FCLC,FOBS,NFDF,PTSN,DPTS,EXIT) >DELF
Enter section desired (X,Y,Z - choose Z for DSN6 maps) >Z
Do you wish to specify individual map steps for each axis (Y/<N>)? >Y
The a-axis is 6.512000 A
Current del-x is 0.2000 A
Enter Fourier calculation spacing in Angstroms along the a-axis >0.1
The new del-x is 0.1018 A
The cell will be divided into 64 points along x
The b-axis is 18.954500 A
Current del-y is 0.2000 A
Enter Fourier calculation spacing in Angstroms along the b-axis >0.1
The new del-y is 0.0987 A
The cell will be divided into 192 points along y
The c-axis is 9.758200 A
Current del-z is 0.2000 A
Enter Fourier calculation spacing in Angstroms along the c-axis >0.1
The new del-z is 0.0976 A
The cell will be divided into 100 points along z
Old x limits are 0.0000 to 0.0000
Enter minimum and maximum values of x in fractions of the cell edge >0 1
New x limits are 0.0000 to 1.0000
Old y limits are 0.0000 to 0.0000
Enter minimum and maximum values of y in fractions of the cell edge >0 1
New y limits are 0.0000 to 1.0000
Old z limits are 0.0000 to 0.0000
Enter minimum and maximum values of z in fractions of the cell edge >0 1
New z limits are 0.0000 to 1.0000
At least one asymmetric part of the unit cell is included in the Fourier.
Enter new list of histogram numbers in the order you wish them to be read.
The last occurrence of a reflection will be used.
Include histogram (0 to terminate list) >1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 0
Enter FOURIER map option (<?>,A,C,D,E,F,H,I,L,P,R,S,T,W,X) >
```

Search for Hydrogen

- Run fourier
- Run forsrh

Min rho = -1.00000 No. of peaks = 10 Peaks saved? N

Phase I.D. from /SNS/TOPAZ/IPTS-12132/shared/refine/scolecite.cif
 Data I.D. Scolecite 100 K
 Map type is DELF

The following peaks were found

	Rho	X	Y	Z
1	-2.283	0.0326	0.0317	0.0522
2	-2.176	0.6695	0.2444	0.0977
3	-2.009	0.6502	0.3418	0.3137
4	-1.986	0.7738	0.1890	0.2279
5	-1.910	0.7386	0.3007	0.4579
6	-1.845	0.0805	0.1045	0.1249

Total CPU time for FORSRH was 0.09 seconds

Archiving SCOLECITE100K.EXP as SCOLECITE100K.OOA

EXPGUI interface to GSAS: SCOLECITE100K.EXP

File Options Powder Xtal Graphs Results Calc Macro Import/Export

expnam expdet genes

LS Controls Phase P

Phase: 1 Replace

Add Phase

forplot
polplot
powplot
ortep
rawplot
fourier
forsrh
gsas2map
liveplot
vrstplot
widplt
absplt
seqplot
mustrplot

title: from /SNS/TOPAZ/IPTS-12132/shared/refine/scolecite.cif

b 18.954500 c 9.758200 Edit Refine Cell
 beta 108.9210 gamma 90.0000 Cell damping 0

	name	type	ref/damp	Occupancy	Uiso
1	Ca1	CA	0 U0 0	1.0000	0.00301
2	Al1	AL	0 U0 0	1.0000	0.00036
3	Al2	AL	0 U0 0	1.0000	0.00062
4	Si1	SI	0 U0 0	1.0000	0.00076
5	Si2	SI	0 U0 0	1.0000	0.00059
6	Si3	SI	0 U0 0	1.0000	0.00096
7	O1	O	0 U0 0	1.0000	0.00317
8	O2	O	0 U0 0	1.0000	0.00245
9	O3	O	0 U0 0	1.0000	0.00258
10	O4	O	0 U0 0	1.0000	0.00383

0.392820 0.446630 4 1.0000 0.00301
 0.467060 0.064070 4 1.0000 0.00036
 0.212070 0.399570 4 1.0000 0.00062
 0.379700 -0.000050 4 1.0000 0.00076
 0.332770 0.167980 4 1.0000 0.00059
 0.417930 -0.202020 4 1.0000 0.00096
 0.478944 -0.092680 4 1.0000 0.00317
 0.449884 -0.360630 4 1.0000 0.00245
 0.354760 -0.216650 4 1.0000 0.00258
 0.313284 -0.031640 4 1.0000 0.00383

Ca1 0.108280 0.392820 0.446630 1.000000
 0.004090

Add New Atoms
 Modify Atoms

Add Hydrogen from DELF Map Output

EXPGUI interface to GSAS: T:/IPTS-12132/shared/refine/GSAS/SCOLECITE100K.EXP (modified)

File Options Powder Xtal Graphs Results Calc Macro Import/Export Help

expnam expedt genes powpref powplot lstview liveplot

LS Controls Phase Powder Scaling Profile Re/Constraints Rigid Body MD Pref Orient SH Pref Orient

Phase: 1 Replace title: Scolecite Neutron Structure at 100 K

add new atom

Adding atoms to phase #1

#	Atom type	Name	x	y	z	Occ	Uiso	Use Flag
1	H	H1	0.0326	0.0317	0.0522	1.0	0.025	<input checked="" type="checkbox"/>
2	H	H2	0.6695	0.2444	0.2444	1.0	0.025	<input checked="" type="checkbox"/>
3	H	H3	0.6502	0.3418	0.3137	1.0	0.025	<input checked="" type="checkbox"/>
4	H	H4	0.7738	0.1890	0.2279	1.0	0.025	<input checked="" type="checkbox"/>
5	H	H5	0.7386	0.3007	0.4579	1.0	0.025	<input checked="" type="checkbox"/>
6	H	H6	0.0805	0.1045	0.1249	1.0	0.025	<input checked="" type="checkbox"/>

Refine Cell

Cell damping 0

83
87
06
75
04
62
59
46
76
78

Add New Atoms
Modify Atoms

Help More atom boxes

Add Atoms Cancel Import atoms from: PowderCell .CEL file

Refine Histogram Scale Factors

$R_w(F_o) = 0.117$

EXPGUI interface to GSAS: SCOLECITE100K.EXP

File Options Powder Xtal Graphs Results Calc Macro Import/Export Help

expsnam expdet genes powpref powplot lstview liveplot

LS Controls Phase Powder Scaling Profile Re/Constraints Rigid Body MD Pref Orient SH Pref Orient

Phase: 1 Replace title: Scolecite Neutron Structure at 100 K

Add Phase a 6.512000 b 18.954500 c 9.758200 Edit Refine Cell
Cell Cell damping 0

* name	type	ref/damp	fractional coordinates			Mult	Occupancy	Uiso
16 O10	O	0 0 0	0.645210	0.450389	0.029320	4	1.0000	0.00259
17 O11	O	0 0 0	0.467500	0.422990	0.559370	4	1.0000	0.00946
18 O12	O	0 0 0	0.203010	0.304400	0.624950	4	1.0000	0.00876
19 O13	O	0 0 0	-0.217540	0.328810	0.390000	4	1.0000	0.00978
20 H1	H	0 0 0	0.032600	0.031700	0.052200	4	1.0000	0.02500
21 H2	H	0 0 0	0.669500	0.244400	0.244400	4	1.0000	0.02500
22 H3	H	0 0 0	0.650200	0.341800	0.313700	4	1.0000	0.02500
23 H4	H	0 0 0	0.773800	0.189000	0.227900	4	1.0000	0.02500
24 H5	H	0 0 0	0.738600	0.300700	0.457900	4	1.0000	0.02500
25 H6	H	0 0 0	0.080500	0.104500	0.124900	4	1.0000	0.02500

Add New Atoms
 X U F 0 0 0 Modify Atoms

Refine Extinction

```
Editing single crystal extinction parameters:

Phase no. 1
Title: from /SNS/TOPAZ/IPTS-12132/shared/refine/scolecite.cif
Extinction for crystal element no. 1:
Current extinction type:
Secondary Type-I      Value = 1.0000E-10 Refine(N)  Damp flag = 0
Lorentzian model used
Single crystal extinction editing option? (<?>,C,D,L,T,V,X) >T
Select new extinction type:
Enter extinction type flag, (<?>,0-4) >1
Do you want to use the Lorentzian model (Y/<N>)? >Y
Current extinction type:
Secondary Type-I      Value = 1.0000E-10 Refine(N)  Damp flag = 0
Lorentzian model used
Single crystal extinction editing option? (<?>,C,D,L,T,V,X) >V
The Type-I refinement flag was changed to Y
Current extinction type:
Secondary Type-I      Value = 1.0000E-10 Refine(Y)  Damp flag = 0
Lorentzian model used
Single crystal extinction editing option? (<?>,C,D,L,T,V,X) >X
Enter overall parameter to be edited (<?>,E,H,S,T,X) >X
Select editing option for Least Squares calculation
(<?>,A,B,F,H,L,O,R,S,T,X) >X
```

Search for Missing Hydrogen

- Set Occupancy to 0.0 for H2
- Run fourier
- Run forsrh

$R_w(F_o) = 0.047$ for 23062 observations

Min rho = -1.00000 No. of peaks = 10 Peaks saved? N

Phase I.D. Scolecite Neutron Structure at 100 K

Data I.D. Scolecite 100 K

Map type is DELF

The following peaks were found

	Rho	X	Y	Z
1	-1.860	0.6725	0.2439	0.1139

Total CPU time for FORSRH was 0.09 seconds

Archiving SCOLECITE100K.EXP as SCOLECITE100K.O18

EXPGUI interface to GSAS: SCOLECITE100K.EXP

File Options Powder Xtal Graphs Results Calc Macro Import/Export Help

expname expedt genes powpref powplot lstview liveplot

LS Controls Phase Powder Scaling Profile Re/Constraints Rigid Body MD Pref Orient SH Pref Orient

Phase: 1 Replace title: Scolecite Neutron Structure at 100 K

Add Phase Edit Cell Refine Cell Cell damping 0

* name	type	ref/damp	fractional coordinates			Mult	Occupancy	Uiso/Uij		
16 O10	O	X0 U0 0	0.646675	0.450412	0.045228	4	1.0000	0.00379	0.00303	0.00752
17 O11	O	X0 U0 0	0.468890	0.422966	0.575123	4	1.0000	0.00740	0.00846	0.02066
18 O12	O	X0 U0 0	0.204595	0.304336	0.640831	4	1.0000	0.01930	0.00534	0.00637
19 O13	O	X0 U0 0	-0.216005	0.328808	0.405921	4	1.0000	0.00634	0.01620	0.01058
20 H1	H	X0 U0 0	0.034432	0.031353	0.068086	4	1.0000	0.02065	0.01575	0.03370
21 H2	H	0 0 0	0.672500	0.243900	0.113900	4	1.0000	0.02500		
22 H3	H	X0 U0 0	0.652008	0.341130	0.329107	4	1.0000	0.01525	0.03128	0.02427
23 H4	H	X0 U0 0	0.776030	0.191189	0.242537	4	1.0000	0.03831	0.03515	0.01222
24 H5	H	X0 U0 0	0.741605	0.299799	0.473425	4	1.0000	0.02678	0.03714	0.02460
25 H6	H	X0 U0 0	0.081177	0.105890	0.140258	4	1.0000	0.02038	0.02219	0.03900

Editing atom #21 -- H2 Add New Atoms

Refinement Flags: X U F Damping: X 0 U 0 F 0 Modify Atom

Label H2 Coordinates 0.672500 0.243900 0.113900 Occupancy 1.000000

Uiso 0.025000

Extinction Coefficient and Final R Factors

Single crystal extinction coefficients for element 1 of phase 1 are:

Parm. : Type-l

Value : 1.286E-04

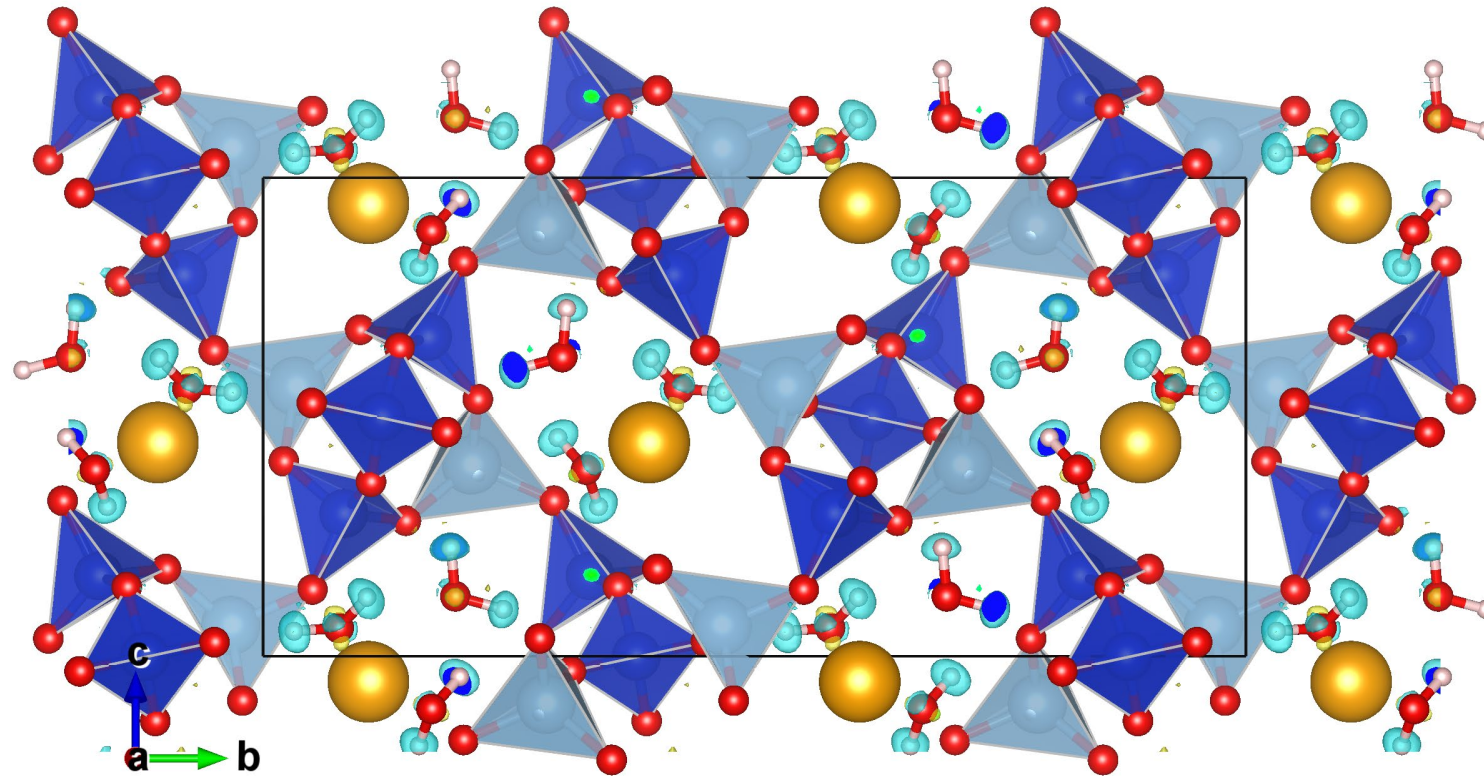
E.s.d. : 1.290E-06

Shft/esd : 0.01

Sum single crystal extinction (shift/error)**2 : 0.00

	All data	I>0.0	I>3*sigl
NREFL=	22823	22823	22303
Rw(F**2)	0.076	0.076	0.076
R(F**2)	0.074	0.074	0.074
Rw(F)	-----	0.038	0.038
R(F)	-----	0.043	0.042

DELF Map Generated from GSAS



Refine ADPs for All atoms

$R_w(F_o) = 0.046$ for 23062 observations

$R_w(F_o) = 0.038$ for 22823 observations
With 5σ cut-off

76 EXPGUI interface to GSAS: SCOLECITE100K.EXP

File Options Powder Xtal Graphs Results Calc Macro Import/Export Help

expname expdet genles powpref powplot lstview liveplot

LS Controls Phase Powder Scaling Profile Re/Constraints Rigid Body MD Pref Orient SH Pref Orient

Phase: 1 Replace title: Scolecite Neutron Structure at 100 K

Add Phase a 6.512000 b 18.954500 c 9.758200 Edit Refine Cell
 alpha 90.0000 beta 108.9210 gamma 90.0000 Cell damping 0

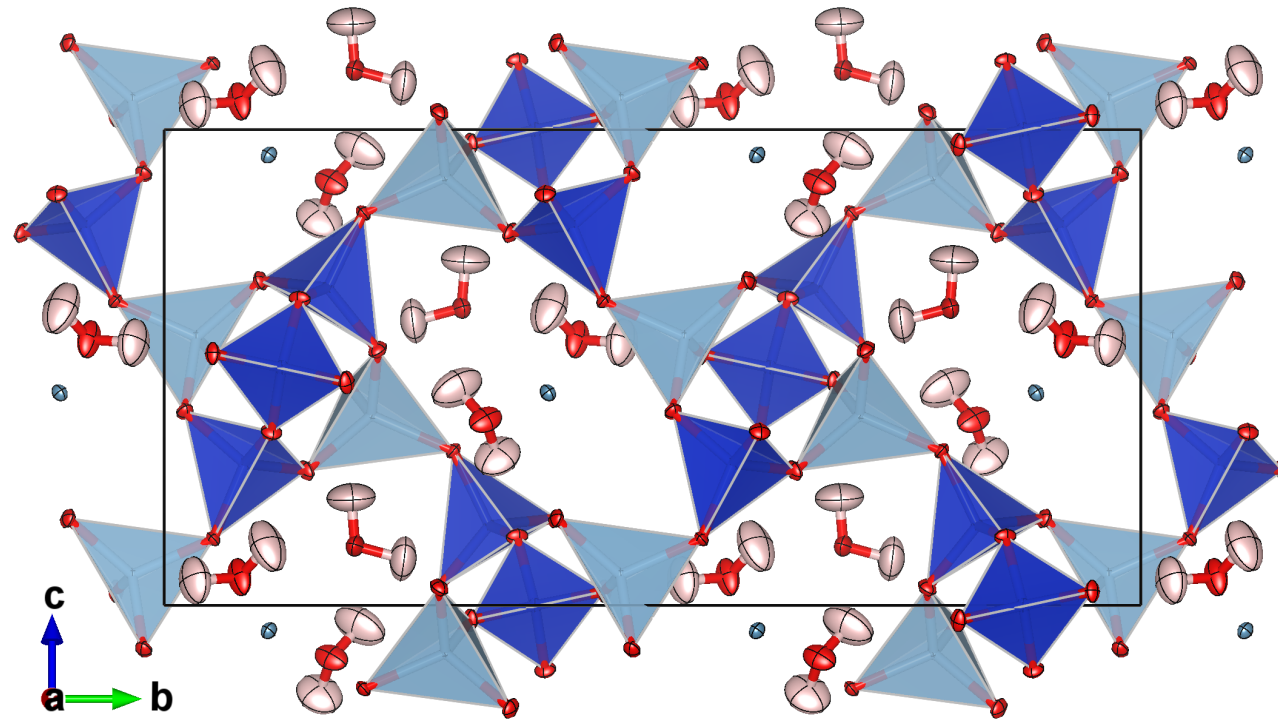
* name	type	ref/damp	fractional coordinates			Mult Occupancy	Uiso/Uij			
1 Ca1	CA	XO U0 0	0.109894	0.392835	0.462617	4	1.0000	0.00542	0.00369	0.00425
2 Al1	AL	XO U0 0	0.922100	0.467089	0.080047	4	1.0000	0.00335	0.00176	0.00280
3 Al2	AL	XO U0 0	0.335478	0.212071	0.415555	4	1.0000	0.00291	0.00194	0.00303
4 Si1	SI	XO U0 0	0.501206	0.379720	0.015858	4	1.0000	0.00213	0.00242	0.00298
5 Si2	SI	XO U0 0	0.209810	0.332737	0.183945	4	1.0000	0.00241	0.00217	0.00229
6 Si3	SI	XO U0 0	0.029908	0.417906	-0.186136	4	1.0000	0.00302	0.00196	0.00224
7 O1	O	XO U0 0	-0.002754	0.478962	-0.076640	4	1.0000	0.00734	0.00402	0.00399
8 O2	O	XO U0 0	-0.003215	0.449884	-0.344659	4	1.0000	0.00603	0.00293	0.00311
9 O3	O	XO U0 0	-0.140211	0.354738	-0.200714	4	1.0000	0.00549	0.00406	0.00580
10 O4	O	XO U0 0	0.630787	0.313303	-0.015699	4	1.0000	0.00603	0.00374	0.00765

X U F 0 0 0
 Add New Atoms
 Modify Atoms
 H2 0.672500 0.243900 0.113900 1.000000
 0.025000

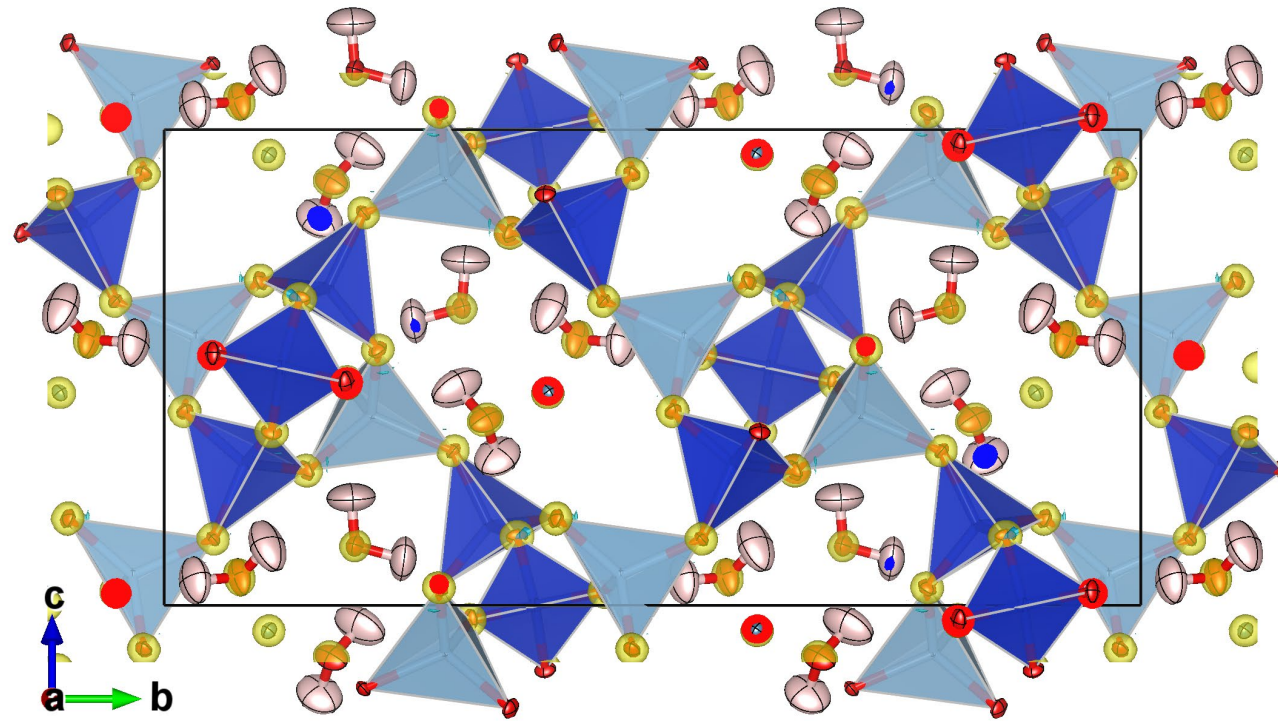
Single crystal $R_w(F_o) = 0.038$ for 22823 observations

	All data	I>0.0	I>3*sigI
NREFL=	22823	22823	22303
$R_w(F^{**2})$	0.076	0.076	0.076
$R(F^{**2})$	0.074	0.074	0.074
$R_w(F)$	-----	0.038	0.038
$R(F)$	-----	0.043	0.042

Neutron Structure from GSAS Refinement



Overlay of Fobs to the Neutron Structure



SHELX2016

```
TITL Scolecite from Neutron TOF Laue
REM Space group Cc
CELL 0.70000 6.5120 18.9545 9.7582 90.000 108.921 90.000
ZERR 4 0.0001 0.0004 0.0002 0.000 0.001 0.000
LATT -7
SYMM +X,-Y,1/2+Z
SFAC Al 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 =
      0.00000 0.00000 3.44900 0.000 0.000 0.072 1.430 26.982
SFAC Ca 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 =
      0.00000 0.00000 4.70000 0.000 0.000 0.170 1.970 40.078
SFAC H 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 =
      0.00000 0.00000 -3.73900 0.000 0.000 30.194 0.320 1.008
SFAC O 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 =
      0.00000 0.00000 5.80300 0.000 0.000 0.001 0.660 15.999
SFAC Si 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 =
      0.00000 0.00000 4.14910 0.000 0.000 0.052 1.170 28.086

UNIT 8 4 24 52 12
TEMP -173
ACTA 50.0

SIZE 1.63 1.19 0.62
L.S. 5
BOND $H
equiv $1 x,-y+1,z+1/2
equiv $2 x+1,y,z+1
equiv $3 x-1/2,-y+1/2,z+1/2
equiv $4 x,y,z+1
equiv $5 x-1,y,z
HTAB 011 010_$1
HTAB 011 03_$2
HTAB 012 04_$3
HTAB 012 09_$4
HTAB 013 06_$3
HTAB 013 07_$5
HTAB 013 05_$5
CONF
FMAP -2
PLAN -10
CONN 4 $Al $Si
LIST 3

WGHT 0.020900
FVAR 0.31665
CA1 2 0.108325 0.392807 0.446642 11.00000 0.00549 0.00402 =
      0.00445 -0.00039 0.00195 0.00026
```

Or add NEUT before SFAC in the .ins file for neutron structure refinement

NEUT
SFAC Al Ca H O Si

- Run ref2hk program from the GSAS folder
\$../../reduction/ref2hkl
Select option 8 as the output format
- Copy SCOLECITE100K.hkl to a working folder
\$ cd /SNS/TOPAZ/IPTS-12132/shared/refine/SHELX
- Refine the scolecite structure
- \$../shelx2013/shelxl scolecite100k

SHELX2016

wR2 = 0.0573 for 8171 data
and 2 / 226 parameters
GooF = S = 1.069;
Restrained GooF = 1.068 for 2 restraints
R1 = 0.0306
for 8151 Fo > 4sig(Fo) and 0.0307
for all 8171 data
wR2 = 0.0573,
GooF = S = 1.069,
Restrained GooF = 1.068 for all data

```
5xw@biganalysis04:/SNS/TOPAZ/IPTS-12132/shared/refine/SHELX
File Edit View Search Terminal Help
[5xw@biganalysis04 GSAS]$ cd /SNS/TOPAZ/IPTS-12132/shared/refine/SHELX
[5xw@biganalysis04 SHELX]$ ls
scolcite100k.cif  SCOLECITE100K.hk4  scolcite100k.ins  scolcite100k.res  shelxl.lst  wingx.log
scolcite100k.fcf  scolcite100k.hkl  scolcite100k.lst  scolcite.lst  wgxScreen.log
[5xw@biganalysis04 SHELX]$ ../shelx2013/shelxl scolcite100k

*****
+ SHELXL - CRYSTAL STRUCTURE REFINEMENT - MULTI-CPU VERSION +
+ Copyright(C) George M. Sheldrick 1993-2014 Version 2014/3 +
+ scolcite100k started at 17:18:31 on 25-Jun-2014 +
*****

Command line parameters: scolcite100k -a50000 -b3000 -c624 -t19

-a sets the approximate maximum number of atoms including hydrogens.
-b sets the maximum number of full-matrix parameters (leave unchanged for
  CGLS). For example -b9000 allows refinement of 1000 anisotropic atoms or
  3000 with BLOC 1. For a 32-bit version, -b times the square root of the
  number of threads should not exceed about 65500. -c sets the reflection
  buffer size. This depends on the CPU cache size but will rarely need
  changing. -t sets the number of threads, otherwise the multi-CPU version
  sets this equal to the number of available CPUs. For optimal performance
  on systems with hyperthreading, usually the hyperthreading should be
  switched off or -t used to halve the number of threads; e.g. -t4 rather
  than -t8 for an Intel i7 processor.

Running 19 threads on 32 processors

Read instructions and data

** Warning: DISP instructions may be required for this wavelength **
Data: 8171 unique, 0 suppressed R(int) = 0.0598 R(sigma) = 0.0583
Systematic absence violations: 0 Bad equivalents: 2
wR2 = 0.0573 before cycle 1 for 8171 data and 226 / 226 parameters
GooF = S = 1.069; Restrained GooF = 1.068 for 2 restraints
Mean shift/esd = 0.007 Maximum = 0.026 for U23 02 at 17:18:32
Max. shift = 0.000 A for Al2 Max. dU = 0.000 for 04
wR2 = 0.0573 before cycle 2 for 8171 data and 226 / 226 parameters
GooF = S = 1.068; Restrained GooF = 1.068 for 2 restraints
Mean shift/esd = 0.002 Maximum = 0.010 for U23 02 at 17:18:32
Max. shift = 0.000 A for Al2 Max. dU = 0.000 for 04
wR2 = 0.0573 before cycle 3 for 8171 data and 226 / 226 parameters
GooF = S = 1.068; Restrained GooF = 1.068 for 2 restraints
Mean shift/esd = 0.000 Maximum = -0.001 for 05F at 17:18:32
Max. shift = 0.000 A for H1 Max. dU = 0.000 for H3
wR2 = 0.0573 before cycle 4 for 8171 data and 226 / 226 parameters
GooF = S = 1.068; Restrained GooF = 1.068 for 2 restraints
Mean shift/esd = 0.000 Maximum = -0.001 for y 08 at 17:18:32
Max. shift = 0.000 A for 08 Max. dU = 0.000 for H4
wR2 = 0.0573 before cycle 5 for 8171 data and 226 / 226 parameters
GooF = S = 1.068; Restrained GooF = 1.068 for 2 restraints
Mean shift/esd = 0.000 Maximum = 0.001 for y 05 at 17:18:32
Max. shift = 0.000 A for All Max. dU = 0.000 for H4
wR2 = 0.0573 before cycle 6 for 8171 data and 2 / 226 parameters
GooF = S = 1.069; Restrained GooF = 1.068 for 2 restraints
R1 = 0.0306 for 8151 Fo > 4sig(Fo) and 0.0307 for all 8171 data
wR2 = 0.0573, GooF = S = 1.069, Restrained GooF = 1.068 for all data
0 atoms may be split and 0 atoms NPD
R1 = 0.0256 for 4407 unique reflections after merging for Fourier
Highest peak 1.09 at 0.5811 0.0596 0.1236 [ 0.65 A from 02 ]
Deepest hole -0.76 at 0.7053 0.4237 0.0474 [ 0.63 A from 010 ]

*****
+ scolcite100k finished at 17:18:33 Total elapsed time: 1.88 secs +
*****
[5xw@biganalysis04 SHELX]$
```

WinGx Nuclear Density Map

The image shows a composite screenshot of the WinGx v2013.3 software interface. The main window displays the Fourier Map Status dialog, the Fourier Map Control Panel, the Select Atoms dialog, and the MapView Control Panel. The main window also shows the structure factors and phases read from the FCF file, the number of grid points, and the function values in the map range.

Fourier Map Status Dialog:

- WinGx binary outputs. This may take several minutes. Min. and max. contours are 0.3 and 0.8 electron A³.
- Hit FOURIER MAP
- Close

Fourier Map Control Panel:

- Structure factor file for Fourier map (LIST 3 FCF): scolecite100k - list
- Options: Slant-plane Fourier, Include F(000), Write MCE File
- Projection: X axis, Y axis, Z axis
- Atoms to define slant plane: [Browse...]
- Fourier coefficients: Fo - Fc, Fo, Fc, Fo*2 (Patt), Fc*2 (Patt)
- Summation limits: Resolution in Å, summation limits in Angstroms. X min: 3, Y min: 3, Z min: 0; X max: 3, Y max: 3, Z max: 0; resolution: 0.1, 0.1, 0.1
- Rejection criteria: Fo/Sig(Fo): 0, Sin(theta)/lambda minimum: 0, Phase Fc/Fo: 0, Sin(theta)/lambda maximum: 10

Select Atoms Dialog:

- 07
- 08
- 09
- 010
- 011
- 012
- H1
- H2
- H3
- OK
- Cancel

MapView Control Panel:

- General controls: Plot linewidth: 1, Number of contour levels: 15, Max. function value: 140.0408, Min. function value: 45.8931, Overlay contours: , Draw zero contour level:
- Colour Table: GRAY-SCALE, HEAT, RAINBOW, BGRW, SERPENT
- Rendering and Iso-surfaces: Use single colour surface, Use colour table surface, Render surface shiny, Positive level iso-surface, Negative level iso-surface
- Annotations: Draw atoms, Draw bonds, Label atoms, Draw cps/bpaths, Draw title string
- 3D-Relief Plots: Tilt angle (deg): 30, Skew angle (deg): 45, Cut-off: 154, Vertical accentuation: 0.25, Magnification: 1, Movie delay time: 0.1

Main Window Text:

Structure factors and phases read from FCF file

4452 reflections have been expanded to 8800 reflections

Number of grid points in Fourier map	nx	ny	nz
	61	61	1

Calculating Fourier summations ...

Function values in the map range from -45.893 to 140.041

2D Mapfile written to file : slant001.mpf : export.fou

SLANT-PLANE & 3D FOURIER PROGRAM - version of May 2007

Reflection data will be read from file : scolecite100k - list3.fcf

Unit cell parameters	a	b	c	alpha	beta	gamma
	6.512	18.955	9.758	98.0	108.9	98.0

Structure factors and phases read from FCF file - type - 3

4452 reflections have been expanded to 8800 reflections

Number of grid points in Fourier map	nx	ny	nz
	61	61	1

Calculating Fourier summations ...

Function values in the map range from -45.893 to 140.041

2D Mapfile written to file : slant001.mpf : export.fou

TOPAZ Data Format SHELX HKLF2 Laue + TOF

```
hklFile.write( 3*'%4d' % (h, k, l)
```

```
+ 2*'%8.2f' % (fsq, sigfsq)
```

```
+ '%4d' % hstnum
```

```
+ 2*'%8.5f' % (wl, tbar)
```

```
+ 6*'%9.5f' % (dir_cos_1[0], dir_cos_2[0], dir_cos_1[1], dir_cos_2[1], dir_cos_1[2], dir_cos_2[2])
```

```
+ '%6d' % (curhst)
```

```
+ '%7d' % (seqnum)
```

```
+ '%7.4f%4d%9.5f%8.4f%7.2f%7.2f\n'
```

```
+ % (transmission, dn, twoth, dsp, col, row) )
```

h, k, l	peak indices
fsq	peak intensity
sigfsq	sigma
hstnum:	a number from 1 to the number of crystal settings
wl:	neutron wavelength
tbar:	the absorption weighted path length
dir_cos_1:	the three direction cosines of a*, b* and c* with the reverse incident beam
dir_cos_2:	the three direction cosines of a*, b* and c* with the scattered beam
curhst:	the actual run number
seqnum:	a number from 1 to the number of peaks
transmission:	the transmission after absorption
dn:	the detector number
twoth:	2-theta angle
dsp:	d-spacing
col:	detector column pixel number
row:	detector row number