

5. Generate output file for structure determination

Usually, we would determine first the space group by generating reciprocal-space sections. However, this standard procedure is well described in many other tutorials and is skipped here. From now on, we assume that the point group is $\bar{3}m1$ and the space group is $P3_121$ (or $P3_221$).

Open the "Finalize integration" menu

Check the checkbox "kinematical"

Select the radio button "integrate profile"

Check the checkbox "frame scaling"

Select "Laue class for Scaling": $\bar{3}m1$

"Interframe correlation range": 4

"Interframe correlation weight": 0.3

Check the checkbox "dynamical"

Set the "virtual frames" parameters to number of frames = 2 and step between frames = 1.

Finalize integration

☒ kinematical

intensity estimation:

☐ fit profile
☒ integrate profile
☐ maximum intensity
☐ Bragg position

integration parameters: 1 min. number of reflections
1 reflection width multiplier

☒ frame scaling

Laue class for scaling: $\bar{3}m1$

Interframe correlation range: 4

Interframe correlation weight: 0.3

☒ dynamical

virtual frames: number of frames: 2
step between frames: 1

Click on "Finalize integration"

Laue class	Rint(obs)	Rint(all)	Nobs/Nall	redundancy
$\bar{3}$	17.59	20.11	292/ 468	3.338
$\bar{3}1m$	42.21	44.14	195/ 272	5.743
$\bar{3}m1$	18.70	20.99	204/ 294	5.313
6/m	42.81	44.51	187/ 256	6.102
6/mmm	43.43	44.86	138/ 177	8.825

From these stats we expect that the point group symmetry is $\bar{3}m1$.

Two output files (apart from the log files) are generated:

Qtz.cif_pets is the list of reflections for structure solution and kinematical refinement.

Qtz_dyn.cif_pets is the list of reflections for dynamical refinement.

PART 2 – Structure solution and kinematical refinement

1. Create new structure

Important! The data-processing procedure is almost never perfectly reproducible. Small differences in the indexing and cell refinement procedure may result in small differences of integrated intensities. If you want to be sure that you can reproduce the following part of the tutorial, do not use the file Qtz.cif_pets that you have just created, but use the file from the

folder "reference_cif_pets" provided with the example files. Using your own cif_pets file is also possible, but your results may differ from the results described in this tutorial.

Create a new subfolder "Jana" and copy Qtz.cif_pets into this folder

Start Jana2020

Main menu bar: "Structure" → "New"

Locate the folder Jana

Enter "quartz" as filename; "open"

This starts a new structure determination with jobname quartz in the new subfolder "Jana".

2. Import Wizard

The data import is automatically started.

[On the screen: Specify type of the file to be imported]

Select "known diffractometer formats"; NEXT

Select the format: "Pets electron diffractometer"

"Browse" for the file Qtz.cif_pets; "Open"

Data reduction file from:

Input file name: Qtz.cif_pets Browse

<input type="radio"/> Nonius-CCD	<input type="radio"/> Koala at ANSTO
<input type="radio"/> Bruker-CCD	<input type="radio"/> SCD-LANL
<input type="radio"/> Bruker-CCD (raw)	<input type="radio"/> Hasylab F1
<input type="radio"/> Oxford Diffraction-CCD	<input type="radio"/> Hasylab HUBER
<input type="radio"/> Rigaku-CCD	<input type="radio"/> Hasylab XDS
<input type="radio"/> IPDS Stoe	<input type="radio"/> 6T2 LBB
<input type="radio"/> D9-ILL, D23 or Trics-Zebra	<input checked="" type="radio"/> Pets electron diffractometer
<input type="radio"/> HeDi	<input type="radio"/> SENJU TOF
<input type="radio"/> ILL-Vivaldi	<input type="radio"/> Polarized neutrons
<input type="radio"/> ISIS SXD	<input type="radio"/> SHELX on I - abs.correction needed
<input type="radio"/> TOPAZ	

☐ Make the reflection file for dynamical refinement

NEXT

[On the screen: Complete/correct experimental parameters]

The unit cell parameters, radiation type and wavelength are correctly set. The sample was measured at room temperature.

NEXT

[On the screen: Define the reference cell]

We do not want to change anything here.

NEXT

1562 input reflections were properly handled.

OK

[On the screen: Define parameters for absorption and scaling procedure]

NEXT

The import wizard is complete. As a next step you can import another or modify the previously imported ones.

FINISH; OK;

3. Symmetry wizard

The symmetry wizard starts automatically after the import wizard. The symmetry wizard can be started separately by expanding "Reflection file" in the Command tree and selecting "Make space group test".

NEXT

We may adapt the tolerances for the determination of the crystal system and space group recognition. In this case, this is not needed.

Deselect "Search for higher symmetrical supercell"

NEXT;

Select "Ordered by R(int)"

Select Laue point group				
Crystal system	Point group	Rint(obs/all)	#averaged(obs/all)	Redundancy
Triclinic	-1	11.74/14.08	460/805	1.94
Trigonal	-3	17.46/20.11	288/468	3.338
Trigonal	-3m1	18.61/20.98	203/294	5.313
Monoclinic-setting "c"	2/m	35.93/37.98	334/510	3.063
Orthorhombic-setting "-a-b,a-b"	mmm	37.79/39.72	289/417	3.746
Orthorhombic-setting "b,-2a+b"	mmm	38.03/39.94	233/332	4.705
Orthorhombic-setting "a,a+2b"	mmm	41.30/43.22	236/352	4.438
Trigonal	-31m	42.18/44.16	193/272	5.743
Hexagonal	6/m	42.74/44.52	187/256	6.102
Hexagonal	6/mmm	43.34/44.88	137/177	8.825

☐ Ordered by Laue symmetry

☒ Ordered by R(int)

Details

Assuming that we have never heard of quartz before and we do not know the structure, we expect trigonal or hexagonal symmetry from the unit cell parameters. From the Rint overview during the data reduction with PETS2.0 we expect the point group -3m1, which also has a reasonable Rint in this overview.

Choose the point group "-3m1"; NEXT

[On the screen: Select cell centering]

Select "P"

We assume a primitive unit cell without R centering

NEXT

[On the screen: Select space group]

Characteristics for systematically absent reflections			
Space group	#obs/#all	ave(I/sig(I))	Figure of merit
P-3m1	0/0	-----	1.00000
P3m1	0/0	-----	1.00000
P321	0/0	-----	1.00000
P3221	4/12	7.395/3.192	2.76820
P3121	4/12	7.395/3.192	2.76820

4 observed reflections violate the space group P3₂21 and P3₁21. This may be attributed to dynamical diffraction effects.

Choose the space group "P3121"

Note that P3₂21 and P3₁21 form a pair of chiral space groups. Successful structure solutions in the two space groups are related by mirror symmetry and exhibit different absolute structure.

Within the kinematical approximation, the two corresponding enantiomorphs cannot be distinguished because both result in identical R factors. We will determine the correct absolute structure later when we perform the dynamical refinement.

NEXT

[On the screen: Final step of space group test]

FINISH

[On the screen: Processing refinement reflection file for Block1...]

In the next step the reflection file is generated from the hkl input file taking the determined symmetry into account.

NEXT;

782/1562 reflections read from input file

OK; OK;

Select "Perform averaging"

(At the bottom) "Sigma(I(ave)) from": Equivalents

PETS provides uncertainties based on detector and counting statistics. Due to the systematic errors introduced by the kinematical approximation, it is often advantageous to determine the standard uncertainties from reflection intensity statistics

NEXT

Summary after averaging

Rint(obs/all) = 10.88/20.19 for 186/466 reflections ...

OK; FINISH

OK

4. Structure solution

After finishing the symmetry wizard, the structure solution program starts automatically. If you need to start it manually, expand "Structure solution" in the Command tree of Jana2020 and double click "Run Superflip".

Enter the chemical formula: Si O₂

Formula units: 3

Repeat superflip: Number of runs: 5

Iteration scheme: CF

Starting model: Random phases

For peak search use: EDMA – fixed composition

"Run superflip"

The structure is solved by superflip.

OK

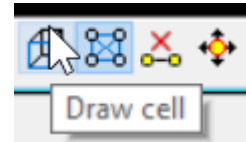
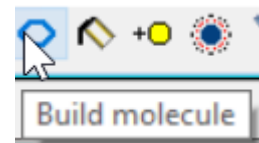
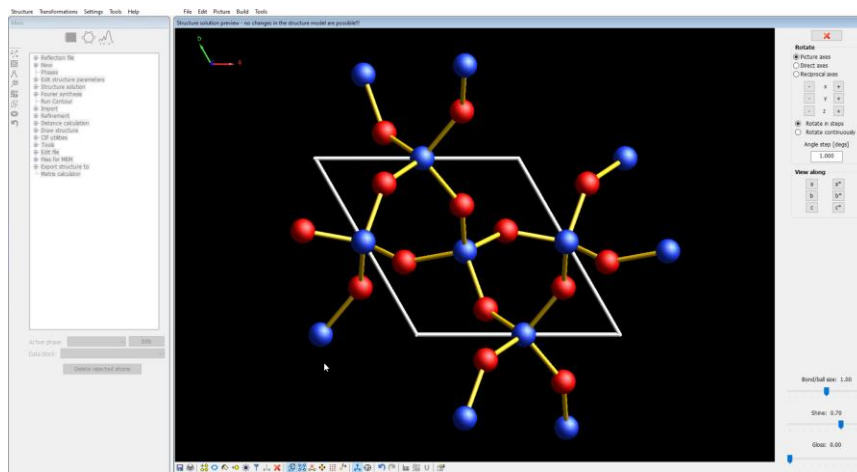
"Draw structure"

Use the "Build molecule" and "draw cell" option at the bottom to get a clearer visualiization.

Structure solution
Run Superflip
Run SIR/Expo

Commands for Superflip

Basic commands	Advanced commands
Formula:	Si O ₂
Formula units:	3 <input type="button" value="Calculate density"/>
Actual space group:	P3121 <input type="button" value="Change"/>
<input type="checkbox"/> use in le Bail decomposition structure information for all	
<input type="checkbox"/> allow manual editing of the command file before start	
<input type="checkbox"/> use previously prepared input file for Superflip	
<input type="checkbox"/> use old solution and reinterpreted	
<input type="checkbox"/> Repeat Superflip: Until the convergence detected	
<input checked="" type="checkbox"/> Repeat Superflip: Number of runs => 5	
<input type="checkbox"/> Use local normalization	
<input type="checkbox"/> Use a specific random seed => 111	
<input checked="" type="checkbox"/> Define explicitly delta value => 0.9	
Iteration scheme: <input checked="" type="radio"/> CF <input type="radio"/> LDE <input type="radio"/> AAR	
Starting model: <input checked="" type="radio"/> Random phases <input type="radio"/> Patterson superposition map	



The tetrahedral network looks like a reasonable structure solution.

Click on the red "X" (top right) to close JanaDraw

"Accept last solution"

[refinements/quartz_4+solution is the current state of the Jana files]

5. Kinematical refinement

Activate "JanaDraw" mode by clicking on the 6-ring above the command tree

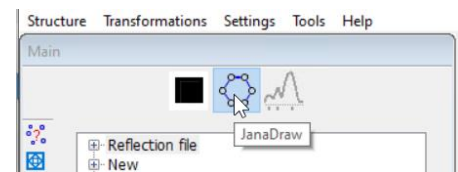
In the command tree expand "Refinement"

Double click "Refinement commands"

Set "Number of cycles" to 20

Uncheck "Refinements on $F(\text{obs})^2$ "

OK; YES+START



The refinement converges with $R(\text{obs}) = 19.17\%$ and $wR(\text{all}) = 22.27\%$

Right click a red atom in JanaDraw -> "Define/Edit atom parameters"

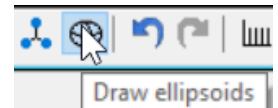
ADP parameter(s): select "harmonic (anisotropic)"

Click "Select atom(s) from list"

Select "Si1"; OK

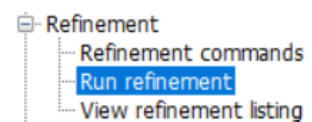
ADP parameter(s): select "harmonic (anisotropic)"; OK

In JanaDraw click the icon "Draw ellipsoids"



During the next refinement cycles you can follow the structural changes after each refinement cycle.

Double click "Run refinement"



Alternatively, you can use the quick button on the left



$R(\text{obs}) = 21.85$, $wR(\text{all}) = 20.46$

The ADP tensors of O1 and Si1 are non-positive definite. This is most likely a result of the systematic errors introduced by the kinematical approximation.

Expand "Edit structure parameters" in the Command tree

Double click "Edit extinction parameters"

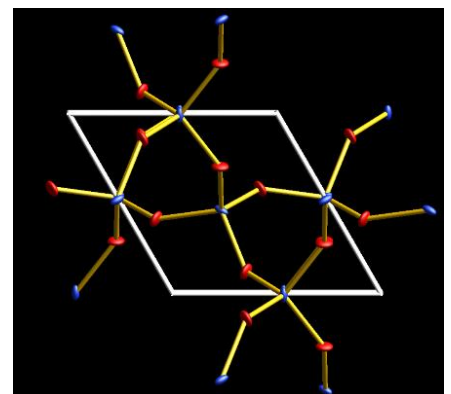
Select radio button "SHELX model"

Activate the checkbox next to "EXT1"

OK; YES

Run refinement

The refinement converges with $R(\text{obs}) = 16.90\%$ and $wR(\text{all}) = 17.39\%$



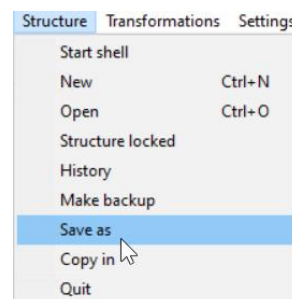
In the literature, kinematical refinements are frequently published with applied extinction correction, which improves the resulting model in terms of R factors and displacement parameters. However, a systematic and thorough investigation of this aspect is not reported in the literature (as far as we know). Thus, this correction should be applied with care. For example, the resulting anisotropic displacement parameters (ADPs) are not necessarily representative for the thermal motion of the atoms. [refinements/quartz_5+adp_exti is the final state of the kinematical refinement]

In the top menu bar: "Structure" → "Save as"

File name: "quartz_dyn"; SAVE

[On screen: Do you want to continue with the new structure?]

YES



PART 3 – Dynamical refinement

Important! If you want to be sure that you can reproduce the following part of the tutorial, use the file from the folder "reference_cif_pets" provided with the example files. Otherwise there may be minor differences. You may also continue with a copy of "refinements/quartz_5+adp_exti"

6. Import reflection file for dynamical refinement

Copy "Qtz_dyn.cif_pets" from the folder of the data reduction with PETS to the folder "Jana"

In Jana2020: Expand "Reflection file" -> "Import/modify reflection file" (double click)

Click "Delete"; OK;

"Reflection file" -> "Import/modify reflection file" (double click)

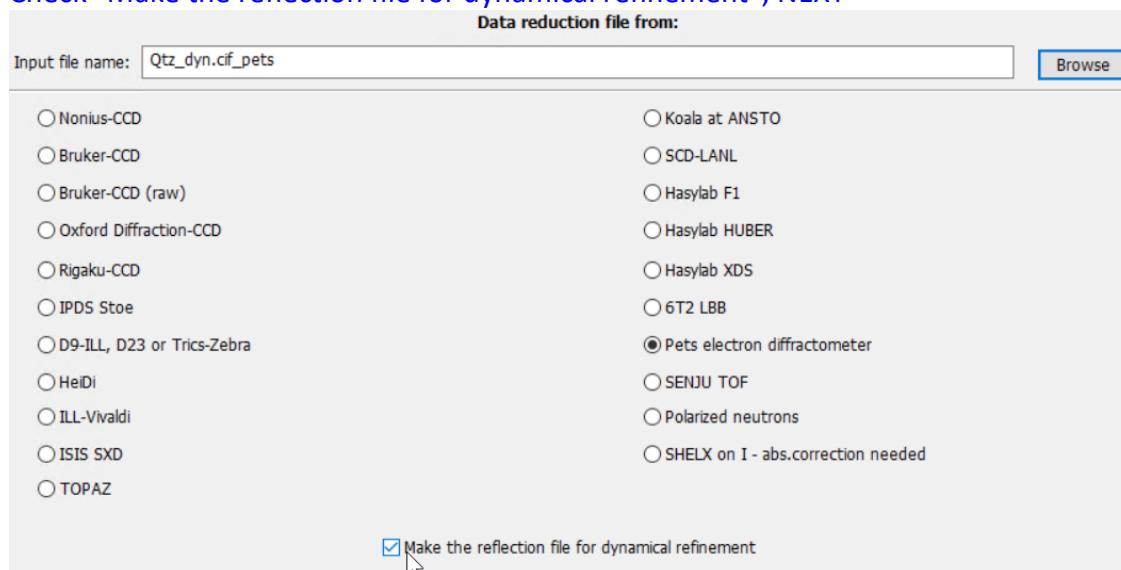


Select "Single crystal": "known diffractometer formats"; NEXT

Select "Pets electron diffractometer"

Click "Browse"; Locate "Qtz_dyn.cif_pets"; OPEN

Check "Make the reflection file for dynamical refinement"; NEXT



[On the screen: Complete/correct experimental parameters]

Leave the default settings unchanged.

NEXT; NEXT;

All 6131 input reflections were properly handled