

7. Generate output file for structure determination

In the "Finalize integration" options, check the checkbox "kinematical"

Select the radio button "fit profile" as mode of intensity estimation

Activate the checkbox "frame scaling" with Laue class "2/m"

Interframe correlation range: 8

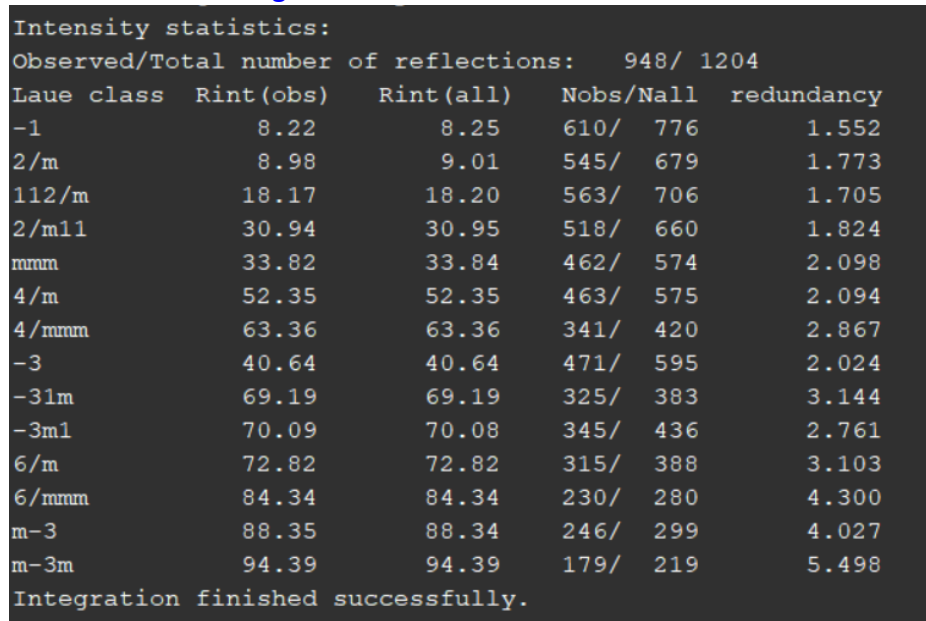
Interframe correlation weight: 0.4

Activate the checkbox "dynamical" with virtual frame settings:

number of frames: 7

step between frames: 5

Click on "Finalize integration"



Intensity statistics:				
Observed/Total number of reflections: 948/ 1204				
Laue class	Rint(obs)	Rint(all)	Nobs/Nall	redundancy
-1	8.22	8.25	610/ 776	1.552
2/m	8.98	9.01	545/ 679	1.773
112/m	18.17	18.20	563/ 706	1.705
2/m11	30.94	30.95	518/ 660	1.824
mmm	33.82	33.84	462/ 574	2.098
4/m	52.35	52.35	463/ 575	2.094
4/mmm	63.36	63.36	341/ 420	2.867
-3	40.64	40.64	471/ 595	2.024
-31m	69.19	69.19	325/ 383	3.144
-3m1	70.09	70.08	345/ 436	2.761
6/m	72.82	72.82	315/ 388	3.103
6/mmm	84.34	84.34	230/ 280	4.300
m-3	88.35	88.34	246/ 299	4.027
m-3m	94.39	94.39	179/ 219	5.498
Integration finished successfully.				

These stats confirm the point group 2/m.

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

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

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

Click on "File" → "Save"

The state of the data reduction is saved in the folder "reference_data_reduction"

Close PETS2.0

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, it is recommended to use the file "glycine.cif_pets" in the folder "reference_cif_pets" provided with the tutorial files. Using your own cif_pets file is also possible, but your results may slightly differ from the results described in this tutorial.

Start Jana2020

Main menu bar: "Structure" → "New"

Enter "glycine" as filename; "open"

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 "Pets electron diffractometer"

Make sure that "Make the reflection file for dynamical refinement" is NOT checked

"Browse" for the file glycine.cif_pets; "Open"; NEXT

Data reduction file from:

Input file name:

<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 E1
<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"/> G2 LBB
<input type="radio"/> D9-ILL, D23 or Trics-Zebra	<input checked="" type="radio"/> Pets electron diffractometer

[On the screen: Complete/correct experimental parameters]

The unit cell parameters, radiation type and wavelength are correctly set. The sample was measured at T = 100 K. The temperature has no effect on the structure solution or refinement.

NEXT

[On the screen: Define the reference cell]

We do not want to change anything.

NEXT

1204 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. You may alternatively start the symmetry Wizard by expanding "Reflection file" in the Command tree. There, double click on "Make space group test".

NEXT

"Maximal deviation for cell angles in degs": 1.5

Uncheck "Search for higher symmetrical supercell"

NEXT;

If the monoclinic point group is not shown it means that in the previous step the allowed deviations were too strict.

Select the point group "2/m"; NEXT

[On the screen: Select cell centering]

We assume a primitive unit cell.

Select the radio button "P"

NEXT

[On the screen: Select space group]

Space group	#obs/#all	ave(I/sig(I))	Figure of merit
P21/m	0/0	-----/-----	1.00000
P2/m	0/0	-----/-----	1.00000
Pm	0/0	-----/-----	1.00000
P21	0/0	-----/-----	1.00000
P2	0/0	-----/-----	1.00000
P21/n	26/82	8.211/3.138	2.41007
P2/n	26/82	8.211/3.138	2.41007
Pn	26/82	8.211/3.138	2.41007

The space group should be selected on the basis of the analysis of reciprocal space. You may do so in PETS2.0 with the option "Reciprocal-space sections". From the reciprocal-space sections, we could derive the space group P21/n. Space group determination is not part of this tutorial.

Choose the space group "P21/n"; NEXT

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

"accept the space group transformed into the original cell"; 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;

944/1204 reflections read from input file

918/1122 reflections written to output file

OK; OK;

(At the bottom) "Sigma(I(ave)) from": select "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) = 7.68/8.98 for 478/630 reflections ...

OK; FINISH;

OK to start the structure solution wizard

4. Structure solution

The structure solution setup windows opens automatically after the space group determination. You may alternatively start the structure solution by expanding "Structure solution" in the Command tree. There, double click on "Run Superflip".

Enter the chemical formula: C2 H5 N O2

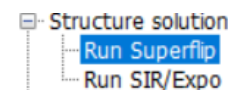
Formula units: 4

Activate "Use local normalization"

Iteration scheme: CF

Starting model: Random phases

For peak search use: EDMA – fixed composition



Commands for Superflip

Basic commands

Advanced commands

Formula:

C2 H5 N O2

Phase:

Formula units:

4

Calculate density

Sum formula from structure model

Actual space group: P21/n

Change the space group

☐ use in le Bail decomposition structure information for already identified phases

☐ allow manual editing of the command file before start

☐ use previously prepared input file for Superflip

☐ use old solution and reinterpreted

☐ Repeat Superflip: Until the convergence detected

☒ Repeat Superflip: Number of runs => 10

☒ Use local normalization

☐ Use a specific random seed => 111

☒ Define explicitly delta value => 0.9

Iteration scheme:

☒ CF
 ☐ LDE
 ☐ AAR

For peak search use:

☒ EDMA - fixed composition
 ☐ EDMA - fixed number of atoms => 0
 ☐ EDMA - peak interpretation by Jana2020
 ☐ Peaks from Jana2020
 ☐ Peaks from Jana2020 but first run Fourier

Starting model:

☒ Random phases
 ☐ Patterson superposition map

Biso:

0

Maxcycles:

2000

Run Superflip

Open the listing

Draw structure

Draw 3d map

Accept last solution

Quit

"Run superflip"

The structure is solved by superflip.

OK

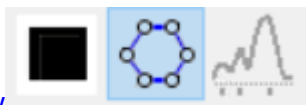
"Draw structure"

"View along": b

Show atom labels:

Click on the red "X" to close JanaDraw

"Accept last solution"



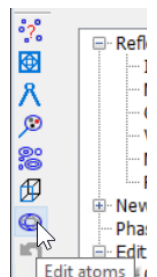
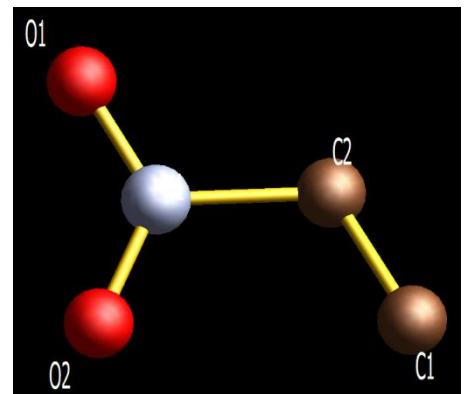
Open JanaDraw

Possibly, like in the shown picture, atom types were not correctly assigned. The oxygen atoms should be bonded to a carbon (sp³) atom, and the terminating C1 atom is nitrogen.

To correct the atom types:

Click the quick button "Edit atoms" on the left part of the main window

Double click C1 atom

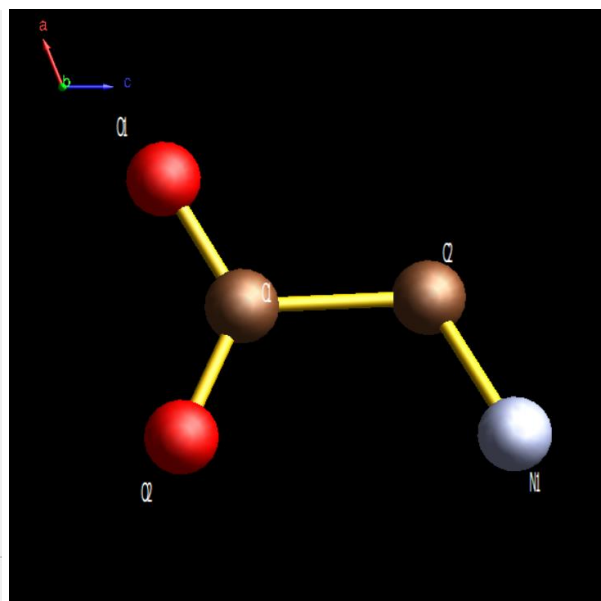
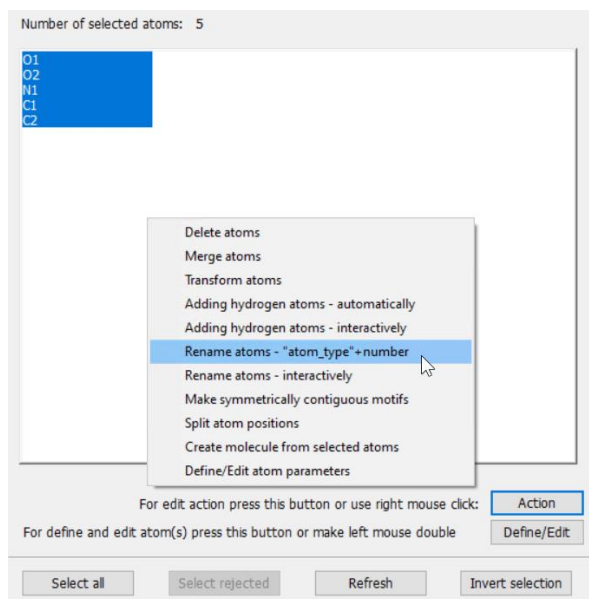
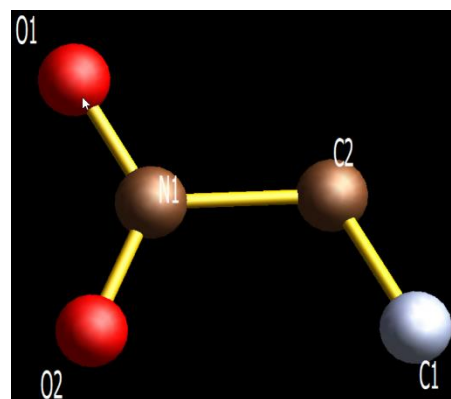


Select correct "Atomic type"; N
 Select the second atom to change (N1) with "select atom(s) from list"
 Select N1; OK;
 Change the atom type and select "C"
 OK

The atom types are corrected, but not the labels.

Click "Select all" to select all atoms of the list
 Click "Action" -> "Rename atoms - atom_type+number"
 OK; YES

Now the structure should look like the one shown below.



[KIN_4+solution is the current state of the Jana files. You may use those files for comparison or use them for the subsequent kinematical refinement.]

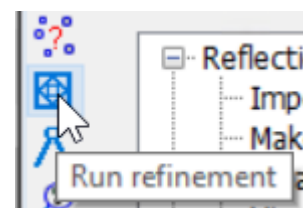
5. Kinematical refinement

Right-click the quick button "refinement" (on the left panel) to access the "Refinement commands" window

Uncheck the checkbox "Refinement on F(obs)**2"

OK; YES+START

The refinement converges with R(obs) = 22.53% and wR(all)= 29.32 %



Click JanaDraw

Right-click the carbon atom C2 (which is bonded to another C atom and the nitrogen atom) -> "Adding hydrogen atoms – automatically"; OK;

"Run refinement"

The refinement converges with wR(all)= 32.52

[glycine_1+refinementH is the current state of the Jana files]

The structure is not charge balanced yet and three hydrogen sites must be identified. One C-O distance is a bit shorter. If there is a hydroxyl group, there must be 2 hydrogens bonded to the terminal N1. It is well known that the hydrogen of OH of glycine molecules in the solid state migrate to the nitrogen atom, so that the terminating nitrogen is part of (NH₃)⁺

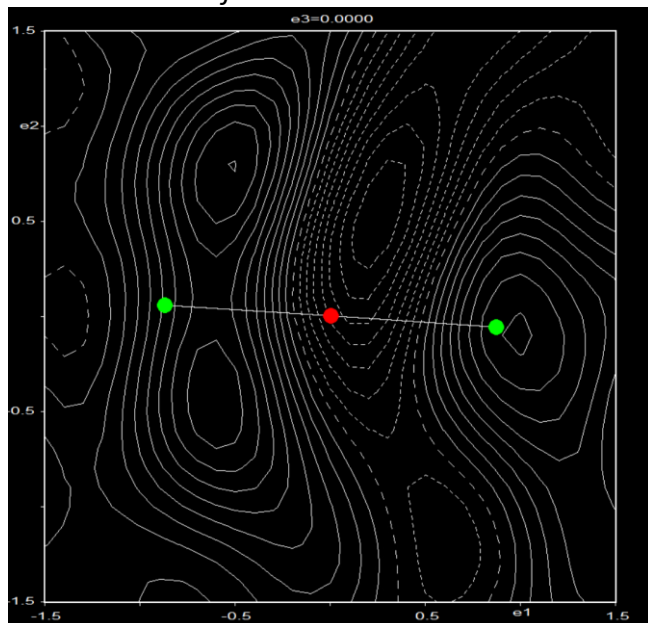
In JanaDraw, click on N1 so that only this atom is selected

Right-click the terminal N1 -> "Adding hydrogen atoms – interactively"; OK;

By default, Jana expects N to be bonded to one non-H neighbor (C2) and two hydrogen sites

Click "Locate positions in map"

The difference Fourier map shows the nitrogen site (red) and the two calculated hydrogen sites (green). The map only fits well to the calculated hydrogen site on the right, but not so well to the site on the left.



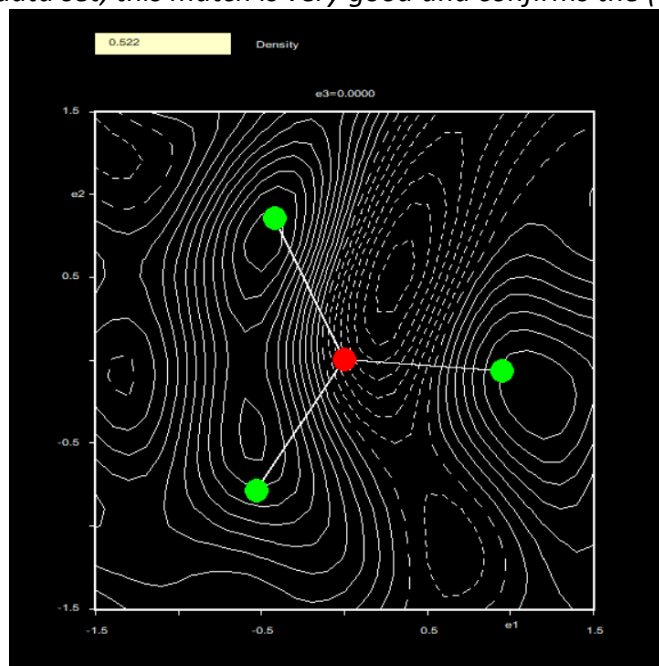
Close the Fourier map by clicking the red X

[On the screen: Adding "hydrogen" atoms for "N1"]

Select the radio button "Tetrahedral"

Click "Locate positions in map"

The difference Fourier map shows the nitrogen site (red) and the 3 calculated hydrogen sites (green). This map fits much better to the expected hydrogen coordinates. The bottom left H site is a bit shifted, but given that it is a kinematical refinement based on one (rather incomplete) data set, this match is very good and confirms the (expected) $(\text{NH}_3)^+$ terminal group.



Close the Fourier map by clicking the red X

[On the screen: Adding "hydrogen" atoms for "N1"]

Adding "hydrogen" atoms for "N1"

Hydrogens	
H1N1	1st
H2N1	2nd
H3N1	3rd

APPLY

Constraints were automatically written. You can see them by opening the M50 file with an editor or in Refinement -> Refinement commands -> Restraints/Constraints -> Keep commands

Run refinement

The refinement converges with $wR_{all} = 25.10$

[KIN_5+refinementAllH is the current state of the Jana files]

With the left CTRL key pressed, select all C, N and O atoms

Right click on one of the atoms -> Define/Edit atom parameters

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

At the bottom menu of JanaDraw click on "Draw Ellipsoids" button

"Run refinement"

The ADPs are not acceptable.

Edit Structure parameters -> Edit extinction parameters (double click)

Select radio button "SHELX model"

Check "EXTI"; OK; YES

Run refinement

The refinement converges with $wR_{all} = 19.97\%$.

The ADPs are now physically allowed as all ADP tensors are positive definite, but their shapes do not look as if they correctly represented the thermal motion of the atoms. This problem can be partly "solved" by removing the worst fitting reflections using the "Skip reflections" option in "Select/Listing" of the "Refinement commands". However, the resulting ADPs are still not reliable and should not be considered representative.

[KIN_5+ADP+EXTI is the final (kinematical) state of the Jana files.]

In the top menu bar: "Structure" -> "Save as"

File name: "glycine_dyn"; SAVE

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

YES

6. Data import for dynamical refinement

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

Click "Delete"; OK;

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

Specify type of the file to be imported

Single crystal: ☒ known diffractometer formats

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

Select "Pets electron diffractometer"

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