# Example: 13.3 α-glycine

Last update 29.06.2023

Data Electron diffraction (continuous rotation) Topic Data reduction, structure solution, refinement Level Basic

# Input data

## Data

Electron diffraction data were measured on a transmission electron microscope JEOL JEM-2100-LaB<sub>6</sub> (hybrid pixel detector from ASI, 200 kV acceleration voltage,  $\lambda = 0.02508$  Å). 164 frames, each covering a goniometer rotation of  $\Delta \alpha = 0.3558^{\circ}$ , exposure time = 0.3 s Every 20<sup>th</sup> frame was recorded with a defocus to check for the crystal position.

## Input files

Folder "tiff": contains measured diffraction patterns in TIF format Folder "reference\_cif\_pets": reference PETS output files for structure determination

## Additional information

glycine.pts as input file to PETS2:

lambda 0.025080	# wavelength
geometry continuous	# diffraction geometry: continuous-rotation
omega 230	# orientation of goniometer axis
phi 0.18000	# semi-angle of the covered angular range $\Delta \alpha$ of one frame
virtualframes 751	

# The idea of virtual frames is to assure that the dynamical calculation of rocking curves avoids that incomplete or partial intensities are determined. Only fully integrated intensities should contribute to the refinement. 7 frames are (virtually) combined to form one virtual frame. Two subsequent virtual frames have an offset of 5 frames.

aperpixel 0.005340 noiseparameters 2.5000 1.0000

center 234.87 233.17 beamstop no

dstarmax 1.800 dstarmaxps 2.0 i/sigma 7.00 5.00 reflectionsize 8.0

referencecell 5.08760 11.80920 5.46150 90.00000 111.99200 90.00000 1 #reference lattice parameters

# **Keywords**

Continuous rotation, in-situ crystallization

# References

For further information about the data processing in PETS2, see:

- L. Palatinus et al. Specifics of the data processing of precession electron diffraction tomography data and their implementation in the program PETS2.0. Acta Cryst. B 75: 512-522 (2019).

-PETS2 manual pets.fzu.cz/download/

For further information about the dynamical refinement and associated parameters:
- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: theory and implementation. Acta Cryst. A71: 235–244 (2015).
- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data. Acta Crystallogr B71: 740–751 (2015)

# **Highlights**

None

# PART 1 - Data reduction in PETS2

## 1. Peak search

Start PETS2 by double clicking the executable. Main menu bar: "File"→"Open" Open the file glycine.pts Open the "Parameters" menu by clicking on the arrow ("v") All important parameters are already set in the input file. Click the action button "Peak search".

Notes

The progress can be followed in the "Image data" panel.

In the console, for each frame the estimated position of the primary beam, the number of Friedel pairs and the number of significant peaks is given. If you cannot see the console, activate View  $\rightarrow$  Panels  $\rightarrow$  Console.

The process ends with the following message in the console:

```
Minimum, average and maximum position of the primary beam: 234.74
236.10 236.63 | 232.08 233.24 233.65
Finished reading 3274 peaks from the file glycine petsdata/glycine.rpl.
```

# 2. Tilt axis and peak analysis

Click on the action button "Tilt axis".

Notes

In the "Cylindric projection" panel, a cylindrical projection of the difference space of the extracted peak positions is displayed. A correct azimuthal angle refinement results in the image containing sharp peaks aligned on sinusoidal curves. This step provides a first estimation of data quality. The refined omega angle is 230.301 degrees.

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Click the action button "Peak analysis". Click "Peak analysis (continue)" Click "Peak analysis (continue)"

# 3. Find unit cell and orientation matrix

Click "Find unit cell and orientation matrix" Click on "Find possible cells automatically"

Notes

A triclinic unit cell is found with  $\alpha$  and  $\beta$  close to 90 degrees, and  $\gamma$  =11.946°. PETS did not suggest a monoclinic unit cell because the tolerance settings are too tight.

Find possible cells automatically     from difference space O from triplets												
Maximal d* difference for indexing (rec.Å): 0.018												
Angular tolerance for rec. direction (deg):												
Maxim	al volume (	Å^3):			5000							
Symme	try search:	tolerance t	for lengths for angles (i	(%): deg):	3							
	а	b	c	α		β	۱	<b>,</b>	v	Bravais	ind/all	
1	4.926	5.380	11.433	91.156	j	90.014	111.9	46 2	280.973	aP	661/673	
2	4.927	5.380	16.102	83.459	)	87.103	68.05	50 3	393.284	aP	407/673	
3	4.926	5.377	11.678	88.276	5	84.533	68.07	71 2	285.613	aP	312/673	

#### Increase "tolerance for angles" to 2 degrees

Click on "Find possible cells automatically"

#### Notes

Now the found unit cell is recognized as monoclinic.

However,  $\alpha$ -glycine is usually described in a cell with a  $\approx$  5 Å, b = 11.4 Å, c = 5.4 Å.

Open the "Modify cell" menu by clicking on the arrow ("v")

Define the following transformation matrix:

a\*' = c\*, b\*' = -b\*, c\*' = a\*

## And Click "Transform by matrix"

^				Modify ce	11							
Reduce cell	Check	centering	Go to	supercell								
Transformation matrix: O direct space												
a*' = 0	*a* -	+ 0	*b* +	1	*c*							
b*' = 0	*a* ·	+ -1	*b* +	0	*c*							
c*' = 1	*a* -	+ 0	*b* +	0	*c*							
transfo	orm centering	g for change	ed cell vol	ume								
Transform b	y matrix	Reset to u	init matrix	:								

## Go back to the indexing menu and Click "Refine Cell"

	а	b	с	α	β	Ŷ
cell:	4.9262	11.4306	5.3792	88.830	111.945	89.995
s.u.:	0.0009	0.0052	0.0011	0.028	0.015	0.027

Leave the indexing panel by clicking "Finish"

## 4. Integrate intensities

In the options of "Process frames for integration" accessible by clicking one of the side-arrows of the action button, increase RC width to 0.004 (rec. Å) and the apparent mosaicity to 0.25 (degrees).



^	Proc	ess frames for integr	ation	^
In	tensity determination method:			
pr	ofile parameters used for integration:			
	rocking curve width (rec. Å)	0.004		
	apparent mosaicity (deg.)	0.25		
	shift integration mask to maximum intensity	,		

We will assume an extremely broad rocking curve to make sure that reflections are integrated on all frames and later adapt the profile parameters.



#### Notes

The expected profile (red) is too broad. We first look at the lowest-resolution shell at the bottom. Reflections at this resolution are hardly affected by the mosaicity and thus are suitable to estimate the RC width.

Open options for "Optimize reflection profile". Run the refinement.

Click "Finalize integration" keeping all the parameters by default.

#### Notes

In the console, a list appears summary of the integration. PETS2 automatically recognized Luae class 2/m, performed frame scaling using this symmetry and for kinematical integration it refined error model and rejected reflections with intensities too far from the group of the symmetrically equivalent reflections (5%). Inspect the "Frame scales" graph. The last frames have very low scale – remove them.



Rerined pa.	rameters of	the e	rror n	nodel:	s fac =	0.3973,	s_b =	1.4556,	s_ad	d = 0.3989		
info: 51 c	out of 130	7 refi	ection	s ide	ntified a	s outli	ers at tr	te reje		limit of	1.50 an	d rejected
Intensity	statistics:											
Observed/T	otal number	of in	tegrat	ed re	flections		/ 1256					
cwl Laue c	lass Rint (c	(obs) Rint (all			Rmeas (obs	) Rmea	Rmeas(all)		Nall	redundancy		
112/m					21.5							
2/m11					36.3					1.842		
menon												
	54		54		62.9							
4/mmm												
-3m1												
6/mmm	84		84									
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0.83-1.05	1.20-0.95				0.52	2.23	5.80			8.83		
1.05-1.20	0.95-0.83				0.50	2.21				13.49		
1.20-1.32	0.83-0.76				0.52	1.99	5.02			17.96		
1.32-1.43	0.76-0.70				0.51							
	0.66-0.63			176			4.44			20.91		
	0.60-0.58						4.52					
				198								
			688	1807								
*** Report	from dynam	ical i	ntegra									

Open the frame dialog by clicking on the "Frame dialog.

Image options	Indexing displ	ay options							
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Display cut o	ff:	24	Reset						
Image type:	nage ssed image ation Paralle	el images	~						
Show overlay: resolution rings peaks-search integration beam stop									
use for ca	lculation								
Center: x		236.5881							
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Angles: α		44.2164							
β		0							
Δω		0							
intensity scal	e	1							
Frame dial	og								
Oistortic	ons (in %)								

## Mark frames 154 to 159 Deactivate the checkbox "use for calculation"

ame dialo	g						;
is dialog	provides optio	n to edit several images at	once.				
Index	File name	File path	^	General	Distortions (in %)		
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151	00151	tiff/00151.tiff		File na	me:	-	
2 152	00152	tiff/00152.tiff		File pa	itn:		
153	00153	tiff/00153.tiff		use	for calculation		
154	00154	tiff/00154.tiff		Displa	y cut off:		
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# 5. Optimize frame orientation

Open the "Optimize frame geometry" menu by clicking on the arrow ("v"). Set the options to match the image below.

^	Optimize frame geometry
for simulation use: ouniform intensities frame orientation angles center of the diffraction patterns RC width apparent mosaicity distortions Reliptical parabolic Replace global distortions	Reset to default Smoothing of correction angles: Onone Oplynomial Omoving average Order: 3

The frame orientation (alpha-tilt, beta-tilt and omega angle) and DP centers are optimized. You can evaluate the success and the progess of this optimization by looking at the following curves in the Graph tab:

-Tilt corrections: with  $\Delta$ alpha and  $\Delta$ beta-tilt frame by frame. You should see a trend. -Origin corrections: corrections applied on x and y coordinates of the center frame by frame.



Open the "Find unit cell and orientation matrix" menu by clicking on the arrow ("v") At the top "Data used for indexing": select "cor" Click "Refine cell"

This refined the orientation matrix taking the new frame orientations into account.

	а	b	с	α	β	γ
cell:	4.9226	11.4504	5.3809	88.998	111.942	90.123
s.u.:	0.0003	0.0070	0.0005	0.018	0.006	0.016

Click "Finish"

## 6. Remove suspicious frames

We only want to use reflections from frames that were properly measured. Go to the "Image data" panel Select radio button "processed" At the bottom menu activate "si" to see the integration masks Set the "Display cut off" to 10 Set the "Frame" to 1, i.e. 1 of 159

We can clearly see that on Frame 1/159 the bottom has a dark area with ~0 counts suggesting that something, probably a part of the TEM grid, blocked the electron path between the sample and the detector in this area. Though a part of the frame, it is easier to remove the complete frame.

Set the "Frame" to 2, i.e. 2 of 159

#### Notes

The not-illuminated area has moved a bit downwards, but still a significant fraction of the detector was not illuminated and thus the reflection intensities are not properly determined in this area. The same holds for frames 3, 4, 5, 6 and 7. On Frame 8 the bottom left corner shows still shadow, but the area is very small.

Open the "Frame dialog" In the "Image options" tab Mark frames 1 to 7 (using the SHIFT key and the mouse or up/down keys) Deactivate the checkbox "use for calculation" OK

Run again "Process frames for integration".

## 7. Generate output file for structure determination

Run "Optimize reflection profile". Click on "Finalize integration"

Ref:	ined pa:	rameters (	of the	error i	model:	: s_fac =	0.3587,	s_b =	3.4505,	s_ad	ld = 0.1782		
Inf		out of 12	10 rei	Election	ns ide	ntified as	s outli	ers at t	he reje¢		limit of	and 1	rejected.
Inte	ensity :	statistic:	5:										
Obse	erved/Te	otal numbo	er of	integra	ted re	eflections	: 785	/ 1155					
cwl	Laue c	lass Rint	(obs)	Rint (	all)	Rmeas(obs	) Rmea	ıs(all)	Nobs/	Nall	redundancy		
*	-1		6.99		7.04	9.7	0	9.78	531/	748	1.544		
*	2/m		8.48		8.54	11.0	0	11.08	485/	662	1.745		
	112/m		15.98	1	6.02	20.1	2	20.17	502/	687	1.681		
	2/m11		29.51		9.52	35.5	6	35.59	465/	641	1.802		
	mmm		31.99		2.01	37.6	5	37.68	424/	563	2.052		
	4/m		51.36		1.34	59.6	9	59.68	420/	557	2.074		
	4/mmm		60.88		0.86		0	68.09	318/	414	2.790		
			38.53		8.53	45.6		45.70	424/	575	2.009		
	-31m		67.51		7.49			75.12	300/	376	3.072		
	-3m1		68.56		8.54	77.0	8	77.07	318/	425	2.718		
	6/m		71.89		1.86	79.9		79.91	290/	378	3.056		
	6/mmm		84.01		4.00					276	4.185		
	m-3		84.91		4.91		8		242/	296	3.902		
	m-3m		91.57		1.56	96.8	9	96.88	176/	216	5.347		
Inf	o: Esti	mated Lau	e clas	ss: 2/m									
Stat	tistics	for Laue	class	2/m									
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0.83	3-1.05	1.20-0.9		2 88	178	8 0.49	2.11	12.07	14.	86	14.87		
1.0	5-1.20	0.95-0.8		3 87	172	2 0.51		8.79		05	9.16		
1.20	0-1.32	0.83-0.7	67	0 96	187	7 0.51	1.85	7.20	12.	00	12.39		
1.33	2-1.43			6 85	172	2 0.49		6.89		08	10.40		
1.43	3-1.52	0.70-0.6	64	9 77		0.43							
1.5		0.66-0.6		4 52	175		1.44	4.36		89			
1.60		0.63-0.6		0 34	167								
1.6	7-1.74	0.60-0.5		5 26	188	3 0.14	1.27	4.09			32.62		
1.74	4-1.80	0.58-0.5	61		197		1.17	4.32					
0.00		Inf0.5	6 48	5 662	1806		1.74	11.41		48	8.54		
***	Report	from dyna	amical	integr	ation	***							

#### Notes

The Rint(obs) is now 8.48%. 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"  $\rightarrow$  "Save"

All the refined parameters and graphs are saved also in the Log file, which are accessible after the job is again opened or you can access them in glycine\_petsdata\log folder. Close PETS2

# PART 2 – Structure solution and kinematical refinement

## 1. Create new structure

## Notes

*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"  $\rightarrow$  "New" Enter "glycine" as filename; "open"

## 2. Import Wizard

## Notes

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:	Browse						
O Nonius-CCD	○ Koala at ANSTO						
O Bruker-CCD	⊖ SCD-L <u>A</u> NL						
⊖ Br <u>u</u> ker-CCD (raw)	O Hasylab <u>E</u> 1						
O Oxford Diffraction-CCD	🔿 Hasylab <u>H</u> UBER						
○ Rigaku-CCD	◯ Hasylab X <u>D</u> S						
○ IPDS Stoe	<u>○6</u> T2 LBB						
O D9-ILL, D23 or Trics-Zebra	<u> <u> P</u>ets electron diffractometer </u>						

## Notes

[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

Notes

[On the screen: Define the reference cell]

We do not want to change anything.

## NEXT

1155 input reflections were properly handled. OK [On the screen: Define parameters for absorption and scaling procedure] NEXT

Notes

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

FINISH; OK

## 3. Symmetry wizard

#### Notes

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

NEXT;

Continue with basic cell

×.			×						
	Sel	ect the supercell							
Cell parameters Volume Crystal system									
5.381 11.450 13.6	99 90.26 90.57 91	00 3*281.28	Orthorhombic						
	continue	with basic cell							
	A	1 0 0 3							
	8 -	0 1 0 - 6							
	c	0 0 1 c							
	for a the metric f	a fata a su ta mata ministra							
	Save the matrix h	or ruture use in matrix calculator							
	Bac	k Next	Cancel						

#### Notes

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

Notes

[On the screen: Select cell centering]

We assume a primitive unit cell.

Select the radio button "P"

NEXT

Notes

[On the screen: Select space group]

	Select space	e group						
	Characteristics for systematically absent reflections							
Space group	#obs/#all	ave(I/sig(I))	Figure of merit					
P21/n	9/81	3.858/1.273	0.11363					
P2/n	9/81	3.858/1.273	0.11363					
Pn	9/81	3.858/1.273	0.11363					
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/a	37/79	6.801/3.834	2.25652					
P2/a	37/79	6.801/3.834	2.25652					
Pa	37/79	6.801/3.834	2.25652					
P21/c	32/80	7.330/3.563	2.32005					
P2/c	32/80	7.330/3.563	2.32005					
Pc	32/80	7.330/3.563	2.32005					

The space group should be selected based on the analysis of reciprocal space. You may do so in PETS2 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]

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

Notes

[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;

Notes

784/1155 reflections read from input file

775/1074 reflections written to output file

OK; OK;

(At the bottom) "Sigma(I(ave)) from": leave "Poisson"

Notes

PETS2 provides uncertainties based on detector and counting statistics, which can be modified by error model refinement and outliers will be rejected. This was done during data processing so the expected sigmas should be close to normal distribution.

NEXT

Notes

Summary after averaging

Rint(obs/all) = 6.40/6.45 for 476/616 reflections ...

OK; FINISH;

OK to start the structure solution wizard

## 4. Structure solution

#### Notes

The structure solution setup windows open 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 "Rund 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

sic commands Ad	vanced comman	ds						
ormula:	C2 H5 N O2						Phase:	
ormula units:	4	<u>C</u> alculate (	structure model	Show Periodic Table				
Actual space group:	: P21/n		Change	e the space	group			
use in le Bail dece	omposition struct	ture informat	tion for alrea	ady identifie	d phases			
<u>a</u> llow manual edit	ting of the comm	and file befo	ore start					
use pre <u>v</u> iously pr	epared input file	for Superflip						
use old solution a	and r <u>e</u> interpret it							
Repeat Superflip:	: Until the conve	rgence dete	cted			<u>B</u> iso: 0		
Repeat Superflip:	Number of runs	=> 10				Maxcycles: 20	00	
Use local normaliz	ation							
Use a specific ran	dom seed =>	111						
Define explicitly of	delta value =>	0.9						
Iteration scheme:	CF			For peak set	arch use:	O EDMA - fixed	composition	
	OLDE					O EDMA - fixed	number of atoms =>	0
	AAR					O EDMA - peak	interpretation by Jana2020	
	o					• Peaks from J	ana2020	
Starting model:	Random pha	ises				O Peaks from J	ana2020 but first run Fourie	er -
	O Patterson su	perposition	map					
	Run Sup	erfip	Open the	e listing	Draw s	structure	Draw 3d map	

"Run superflip"



The structure is solved by superflip. The solution is never the same in superflip as it starts from random phases. Therefore, there are files, where the solution for this tutorial is saved. For identical solution, click on the button "Replace the result with tutorial files".

ormula units:			Phase:			
	4	<u>Calculate</u> density	Sum formula from s	tructure model	Show Periodic Table	
ctual space group	: P21/n	C <u>h</u> ang	e the space group			
use in le Bail deo	omposition struct	ture information for alre	ady identified phases			
allow manual ed	ting of the comp	nand file before start				
use previously p	repared input file	for Superfin				
use old solution	and reinterpret it					
Repeat Superfir	: Until the conve	ergence detected		Biso; 0		
Reneat Superfir	Number of runs	s => 10		Maxcycles: 20	00	
Use local normal	zation					
Use a specific ra	ndom seed =>	111				
Define explicitly	delta value =>	0.9				
the section of the se	OF		For peak search use:	○ E <u>D</u> MA - fixed	composition	
ceracion scheme:	OLDE			O EDMA - fixed	number of atoms =>	0
ceración scheme:	OLDE					
ceración scheme:	⊖ AAR			○ EDMA - peak	interpretation by Jana <u>2</u> 02	0
Starting model:	AAR     Random pha	ases		<ul> <li>EDMA - peak</li> <li>Peaks from Ja</li> </ul>	interpretation by Jana <u>2</u> 02 ina2020	0

ОК

"Draw structure" "View along": b Click on the red "X" to close Vesta "Accept last solution"

**Open JanaDraw** 



Notes

All the atoms in the solution (tutorial) are correctly assigned. If you did not use provided solution, assign the atoms correctly. The oxygen atoms should be bonded to a carbon (sp2) atom, and the terminating atom is nitrogen. To correct the atom types



Double click an atom and change the "Atomic type". Do not forget to correct also the labels. OK.



OK; YES

Now the structure should look like the one shown below.



## 5. Kinematical refinement

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



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

OK; YES+START

The refinement converges with R(obs) = 19.45% and wR(all)= 26.38 % 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"

## Notes

The refinement converges with wR(all)= 26.14

The structure is not charge balanced yet and three hydrogen sites must be identified. The C-O distances are rather similar. 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  $(NH_3)^+$ 

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"

#### Notes

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 at the bottom, but not so well in the upper part.



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"

Notes (Cookbook normal)

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. Given that it is a kinematical refinement based on one (rather incomplete) data set, this match is very good and confirms the (expected) ( $NH_3$ )<sup>+</sup> terminal group.



## Close the Fourier map by clicking the red X [On the screen: Adding "hydrogen" atoms for "N1"]

Adding hydrogen atom	STOP INT			
Tetrahedral	1.01	H distance	1.2	ADP expansion factor
○ Trigonal	Use typical neutron di	stance		
○ Apical	1 Number of ne	ighbors	Hydrogens	
	C2	1st	H1N1	1st
		-	H2N1	2nd
			H3N1	3rd
				_
Use anchoring =>		Arichor		Torsion angle
	Locato positions in man		Falact	
	Locace posicions in map		Jelecc	
	Avoid	Quit	Apply	

## APPLY

## Notes

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
Notes
The refinement converges with wRall= 21.91
With the left CTRL key pressed, select all C, N and O atoms
Right click anywhere except on atoms -> Define/Edit atoms
ADP parameter(s): "harmonic (anisotropic)"; OK;
At the bottom menu of JanaDraw click on "Draw Ellipsoids" button "Run refinement" The ADPs are not acceptable. This is due to the dynamical effects.

Reset the ADPs to isotropic In the top menu bar: "Structure" → "Save as" File name: "gylcine\_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 • known diffractometer formats

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

Click "Browse"; Locate "glycine dyn.cif pets"; OPEN

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

Notes

Single crystal:

[On the screen: Complete/correct experimental parameters]

You may set the Temperature to 100 (Kelvin). This has no influence on the refinement.

NEXT; NEXT;

All 3134 input reflections were properly handled.

OK; NEXT; FINISH; OK

Notes

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

Note that "Make the reflection file for dynamical refinement" is checked.

NEXT;

2424/3134 reflections read from input file ... OK; OK; FINISH

## 7. Dynamical refinement setup

In the command tree (left), expand "Edit structure parameters"  $\rightarrow$  "Edit parameters for electron diffraction" (double click)

Notes

[On the screen: many options starting with orientation matrix] In the top section on the left:

Set "Maximal diffraction vector g(max)" to 1.8

Notes

This g(max) is related to the resolution of the dynamical calculations, which should in general be a bit higher than the resolution of the highest reflections used in the refinement.

Set "RSg(max)" to 0.7

Leave "DSg(min)" at 0 Å<sup>-1</sup>

## Notes

RSg(max) and DSg(min) filter out reflections for which an incomplete or unreliable rocking curve integration is expected, e.g. reflections that lie on or are close to the goniometer rotation axis.

Set "Number of threads" to the number of physical cores of your processor In the top section on the right: Set "Number of integration steps" to 38 Set "Geometry" to "rotation" In the section in the middle: "Select zones for refinement" Unselect zones 1, 2, 4, 8, 12, 16, 20, 24, 28, 31 (using the CTRL key + mouse click)

		<b>N</b>								
Zone#1	Zone#23	Orientation matrix: 011	0.19381	012	0.00372	013	0.15226			
Zone#2	Zone#24	1121	-0.1013	8 1122	0.00019	1123	0.13008			
Zone#3	Zone#25	021				1 020				
Zone#4	Zone#26	U31	0.00967	U32	-0.08741	U33	0.01106			
Zone#5	Zone#27					1			7	
Zone#6	Zone#28	Maximal diffraction vector	g(max):	1.8	Numbe	er of inte	egration steps:	38	Geometry:	O precession
Zone#7	Zone#29	Maximal excitation error (h	(atriv)	0.01	_					rotation
Zone#8	Zone#30	Maximal excitation error (in	Maximal excitation error (Matrix):		Use dy	namic a	pproach:	$\leq$		Crocacion
Zone#9	Zone#31	Maximal excitation error (F	Maximal excitation error (Refine):			vin version:				
Zone#10	and the second se									
Zone#11		RSg(max):	RSg(max):		0.7 Apply correction for crystal tilt: 🔽					
Zone#12		DSa(min):	DSa(min):							
Zone#13		0000(1111).	b3g(min).		For Fourier rescale to Fcalc:					
Zone#14		Number of threads:		2	<u> </u>					
Zone#15		Dun en en mander								
Zone#16		Dyngo commands:								
Zone#17				- la ab an a s	· · · · · · · · · · · · · · · · · · ·	-	Deferrer		h talan ang a	
Zone#18			5	elect zone	s for refineme	ent	Define zor	ies of equal t	nicknesses	
Zone#19										
Zone#20		Run optimizations	excent	of scale of	ntimize also.		hickness	Show thick	mess plots	
Zone#21		Run optimizations	except	or scale, o	pennie abo.	2	inchiro ad	Show chick	areas pieca.	
Zone#22						0	rientation			

Notes

These are the virtual frames of which at least one frame was deactivated during the data reduction.

"except of scale, optimize also": check "Thickness" Click "Run optimizations"

Notes

The dependence of R factors of individual virtual frames on the thickness is determined. The initial thickness estimation is necessary to get a stable starting point for the dynamical refinement and to avoid getting stuck in a local minimum.

Click "Show thickness plots"

Notes

You can look at the dependence of the R factor on the thickness for the different frames. The best R factors are typically found with a thickness of about 400 Å or in the range 200 to 600 Å.

Close the plot (red X)

Click on "Select zones for editing"; "Select all"; OK.

Notes

The EDThick box is yellow and locked because the parameter is not the same for all selected zones.



Click EDThick box; click Unlock and change the value to 400.

This changes EDThick for all zones to 400 Å.

Click on "Select zones for editing"; "Refresh"; OK

By changing number of zone by "Zone#" textbox, we can see that all zones have EDThick 400 Å Uncheck the checkbox "Thickness"

Click on "Run optimizations"

This will optimize the scale factor for each frame based on a thickness of 400 Å.

OK; YES to save m42 file.

## 8. Dynamical refinement

"Refinement" -> Refinement commands Check for convergence: stop if 0.5 in 1 consecutive cycles. Deactivate "Refinements on F(obs)\*\*2 OK; YES+START Notes

The refinement converges with wR(all) of 12.11%.

## 9. Refinement with anisotropic ADPs

Set ADPs of the non-hydrogen atoms to be harmonic. In Edit atoms choose the atoms of interest. Press Define/Edit button. Choose harmonic (anisotropic) for ADP parameter(s). Edit atoms Phase: Phase#1 Number of selected atoms: 5 01 02 NI H222 H101 H201 H301

OK; OK; Yes. Open JanaDraw. Refine the structure. *Refinement converged to wR(all) equal to 10.90%.* 

## 10.Refinement without constraints

In the Command tree, expand "Refinement " "Refinement commands" (double click) -> Restraints/Constraints Click "Keep commands"; "Refresh"; "Select all"; "Disable"; OK;

Notes

Note that the button "Keep commands" is now labelled "Keep commands [0+4!]", indicating that there are 0 active "keep" commands and 4 disabled "keep" commands.

OK; YES+START

Notes

Refinement converges to R(obs) 7.72% and wR(all) 10.17%.

All hydrogen atoms refined freely while maintained the expected geometry.

For comparison, you may also try an unconstrained kinematical refinement as continuation of step 5. Or you may try to locate the hydrogen sites from the difference Fourier map in a dynamical refinement after removing the hydrogen sites.

