A visual guide for crystal and magnetic structure refinement using Mag2Pol

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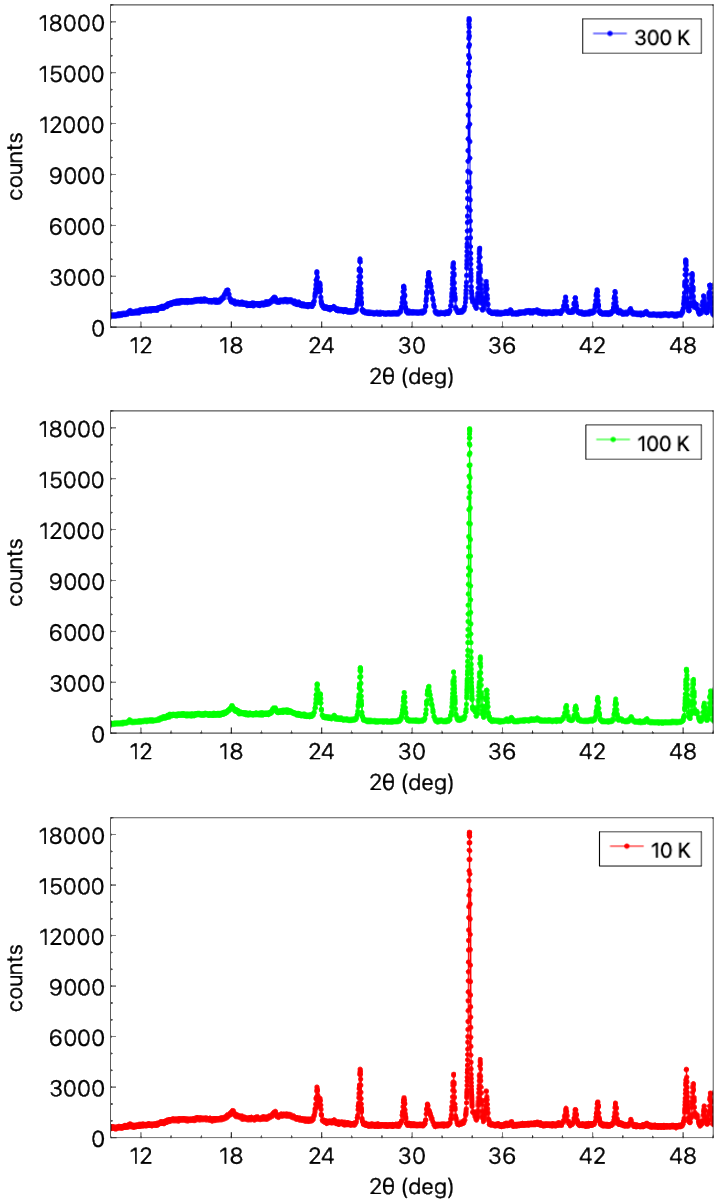
From Neutrons to Structure in Materials: Hands-On Introduction to Neutron Data Analysis
IFJ-PAN Krakow, 17-18 November 2025

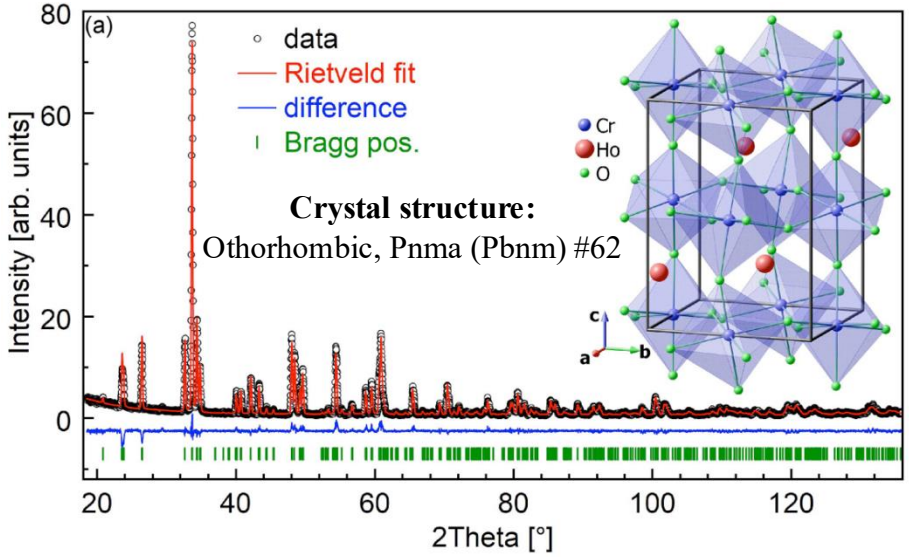
Session 2

Refinement of crystal structure from NPD data

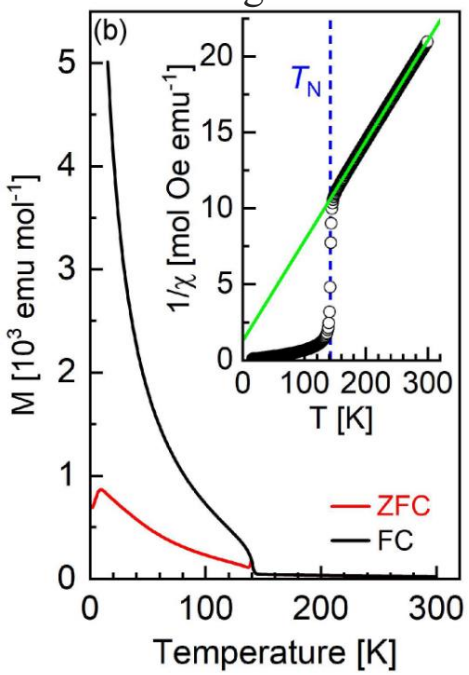
Inhouse and Macroscopic characterizations of $HoCrO_3$

X-ray powder diffraction

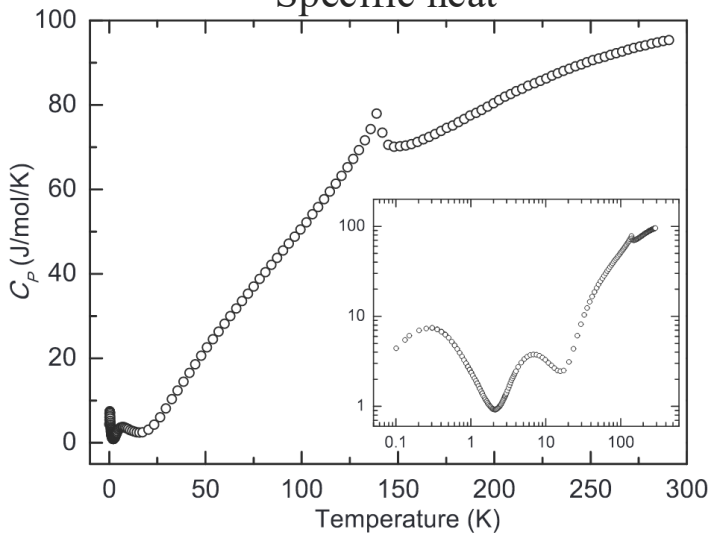




DC magnetization



Specific heat



$T_N \approx 142$ K

From Curie-Weiss fit:

$\mu_{eff} = 11.01 \pm 0.02\ \mu_B$

This corresponds to magnetic ions:

Cr^{3+} (spin only S = 3/2)

Ho^{3+} (strong spin–orbit, J=8)

Neutrons can tell us if both ions order simultaneously. The moment size and directions for each magnetic site in the lattice.

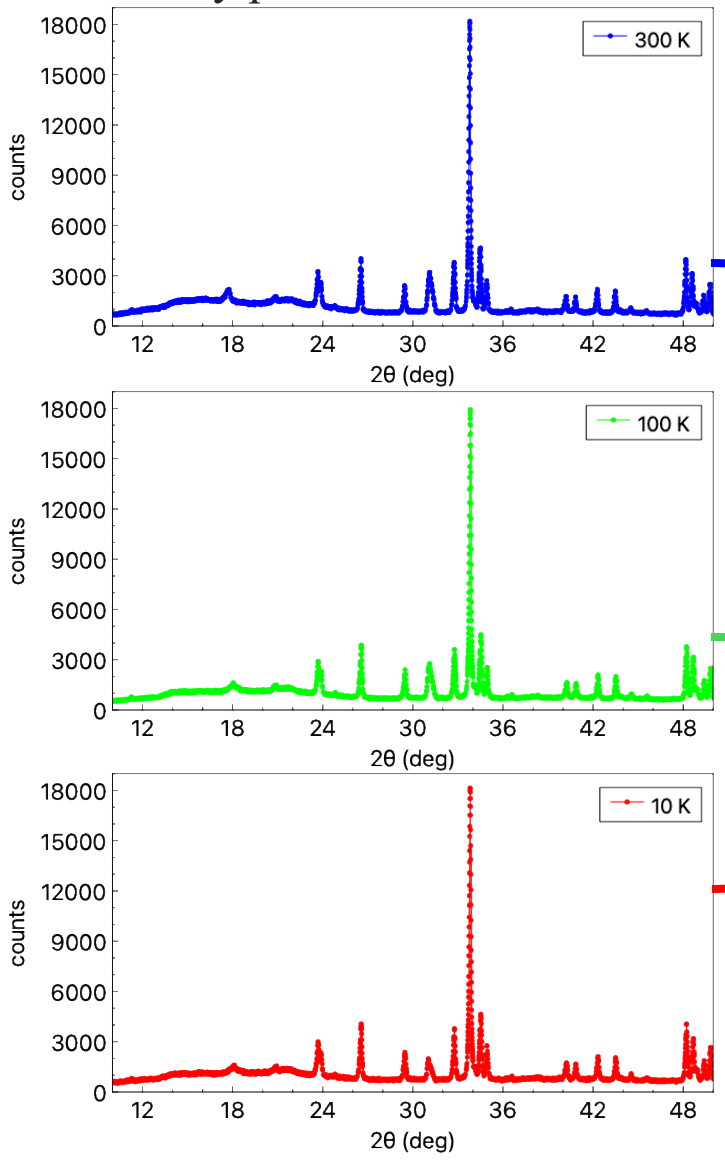
C. M. N. Kumar, PhD Thesis (2012), K. Komędera N. K. Chogondahalli Muniraju, Acta Phys. Pol. A, 145 (2024)

X-ray vs. Neutron powder diffraction

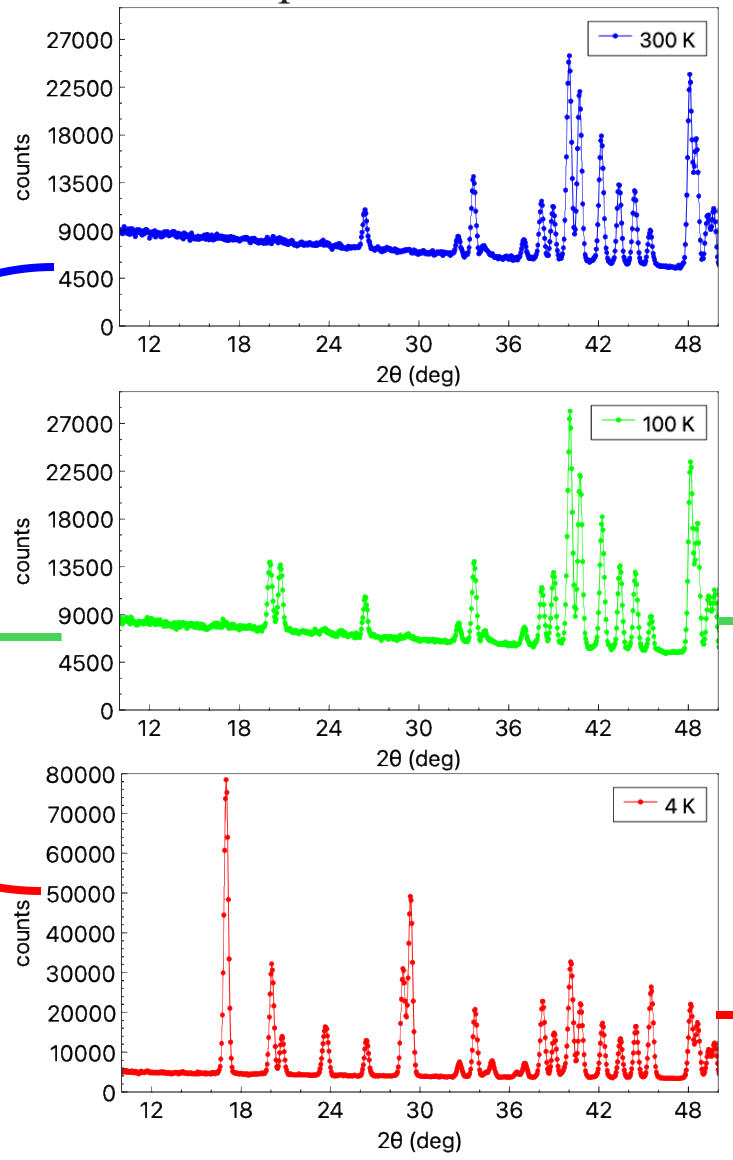
$HoCrO_3$

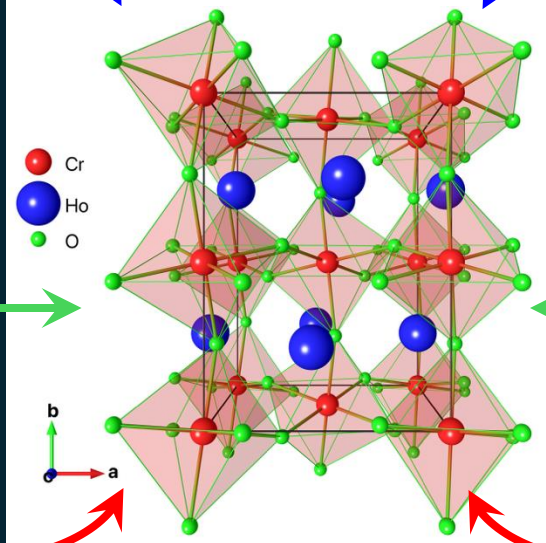
Orthorhombic space group:
Pnma (*Pbnm*) #62

X-ray powder diffraction



Neutron powder diffraction

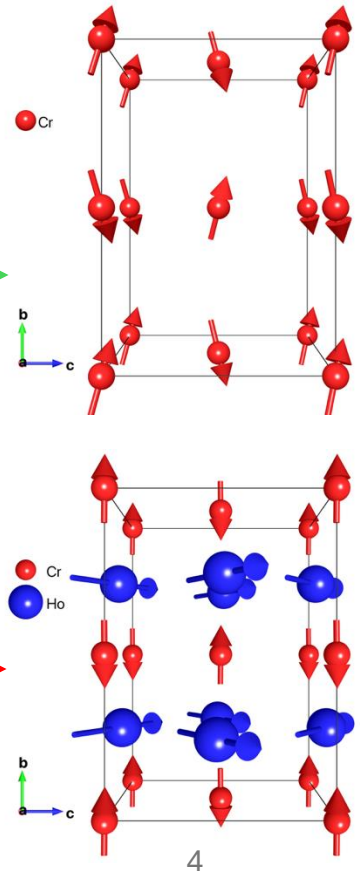




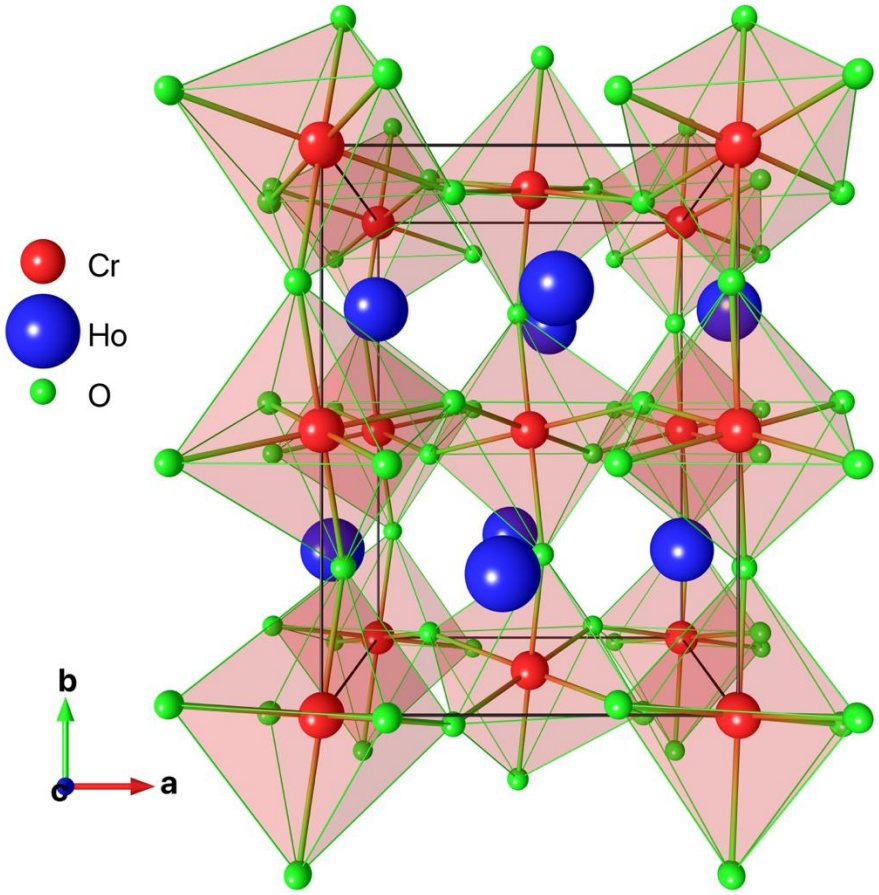
Atom	x	y	z
Cr (4a)	0.0000	0.0000	0.0000
Ho(4c)	0.43449	0.25000	0.00670
O1(4c)	0.53340	0.25000	0.58885
O2(8d)	0.19680	0.05420	0.30450

X-ray diffraction:
Lattice structure

Neutron diffraction:
Lattice + Magnetic



Measurement and data details



Orthorhombic structure: *Pnma* (No. 62)

Instrument: Neutron powder diffractometer SPODI at FRM II research reactor, Garching, Germany

Wavelength: 1.5482 Å

Data files:

Data collected at 300 K: HCO_300K.dat

Data collected at 100 K: HCO_100K.dat (→ HCO_100K.m2p)

Data collected at 4 K: HCO_4K.dat

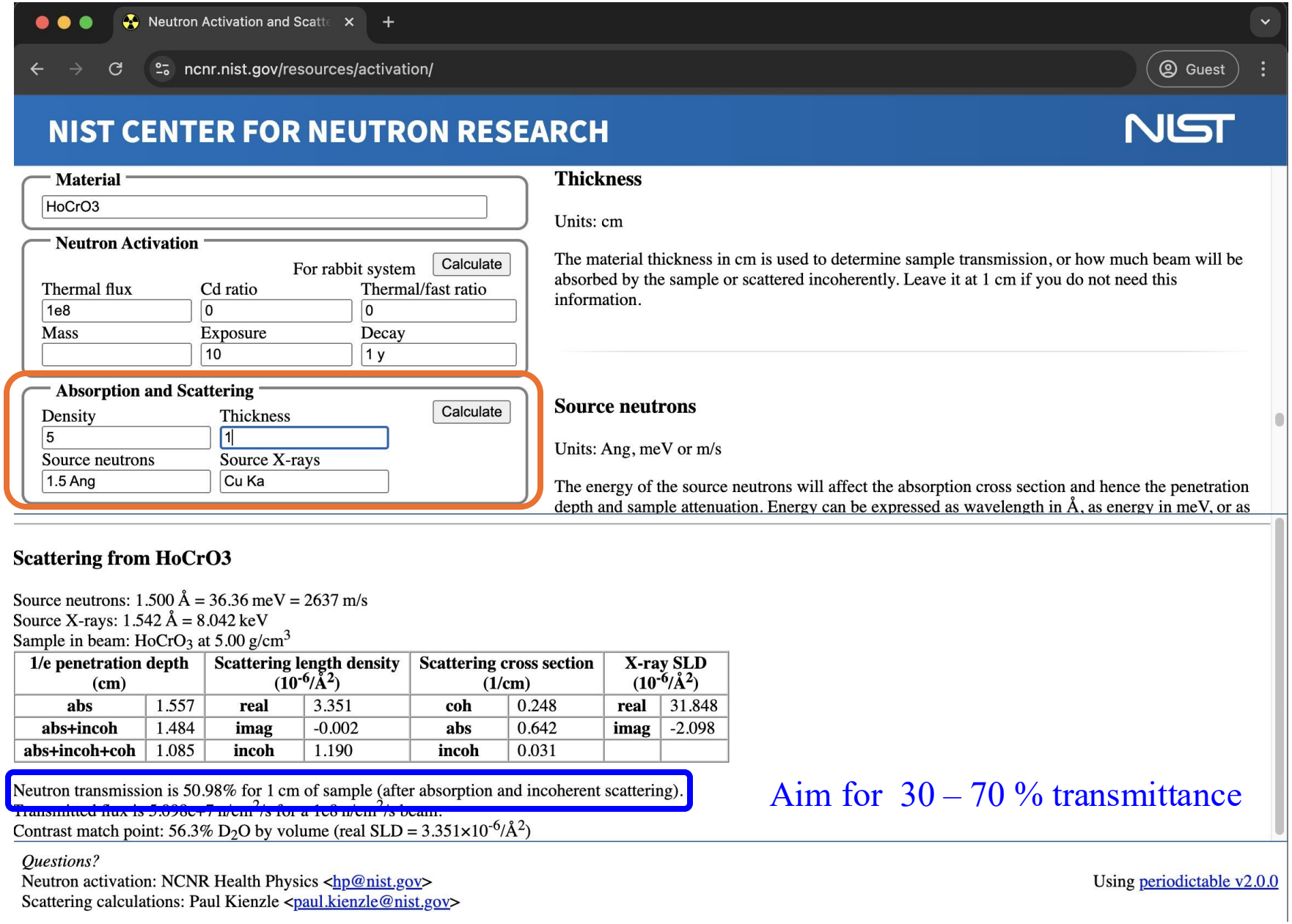
Crystal Information File for crystal structure:

HoCrO3_300K.cif

Mag2Pol version used: 7.2.2

The main interface of Mag2Pol

Is the sample a good scatterer or an absorber?



Aim for 30 – 70 % transmittance

Low transmittance (<20%):
Heavy absorption → distorted intensities, surface scattering, need strong absorption corrections

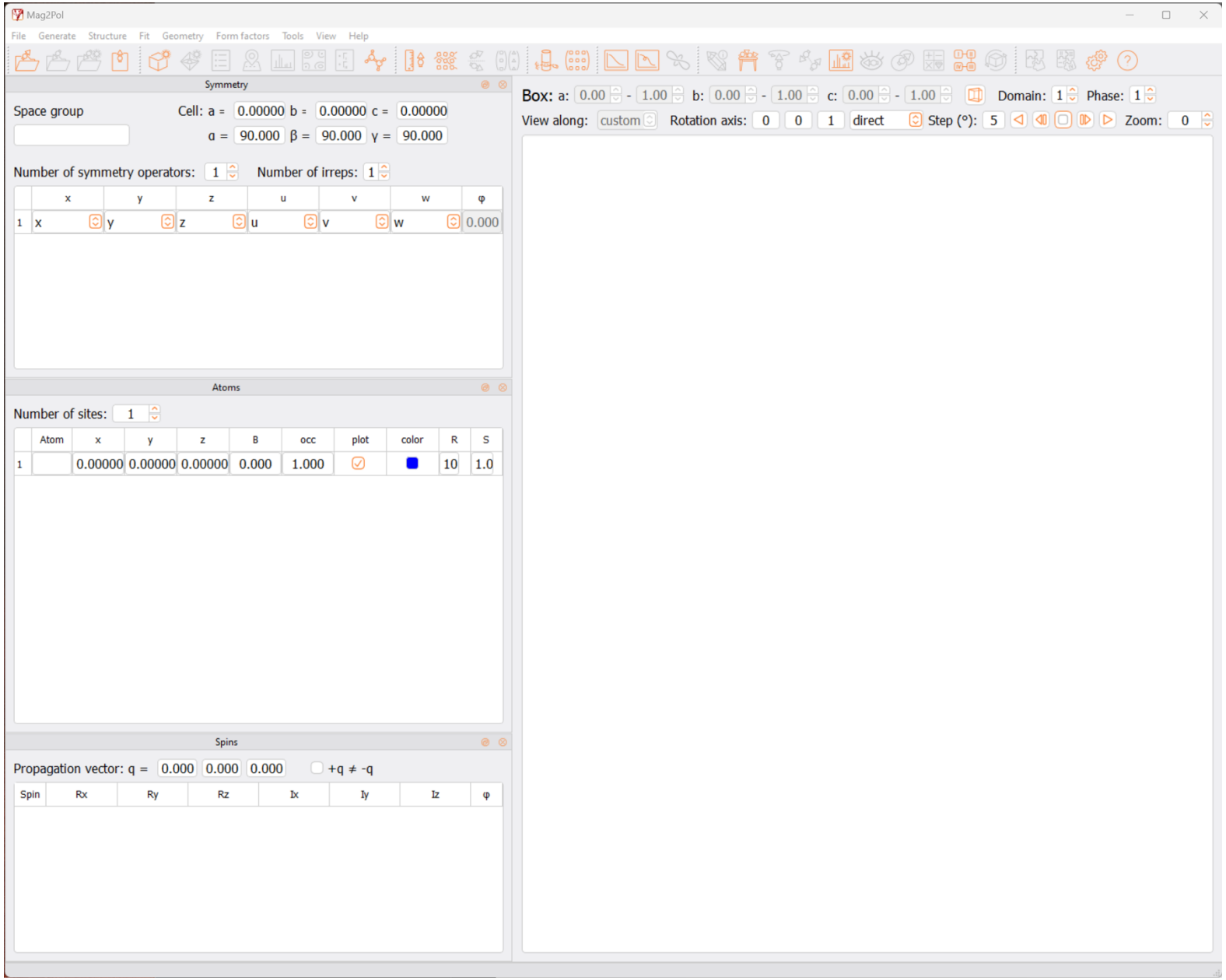
High transmittance (>90%):
Weak scattering → poor S/N, long counting time, weak magnetic peaks

The main interface of Mag2Pol

The main window of Mag2Pol - GUI

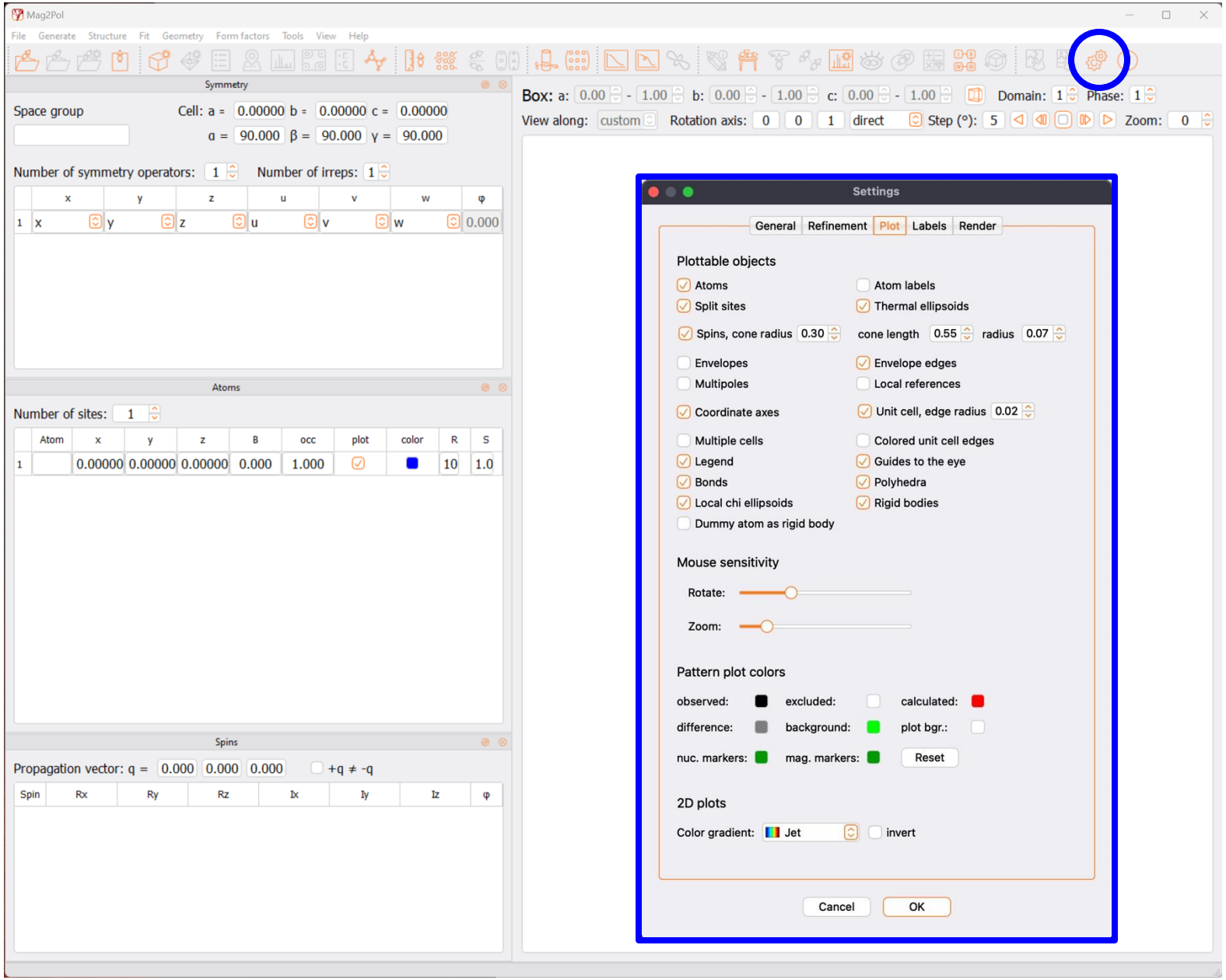
Running the Mag2Pol application, the main window ‘Mag2Pol’ will open. By default, the interface may look slightly different. You can modify this under the menu ‘View’

This window should remain open as long as any other Mag2Pol utility or window is running. The current version of Mag2Pol will not save the project automatically, so make sure to save any project before closing this window.

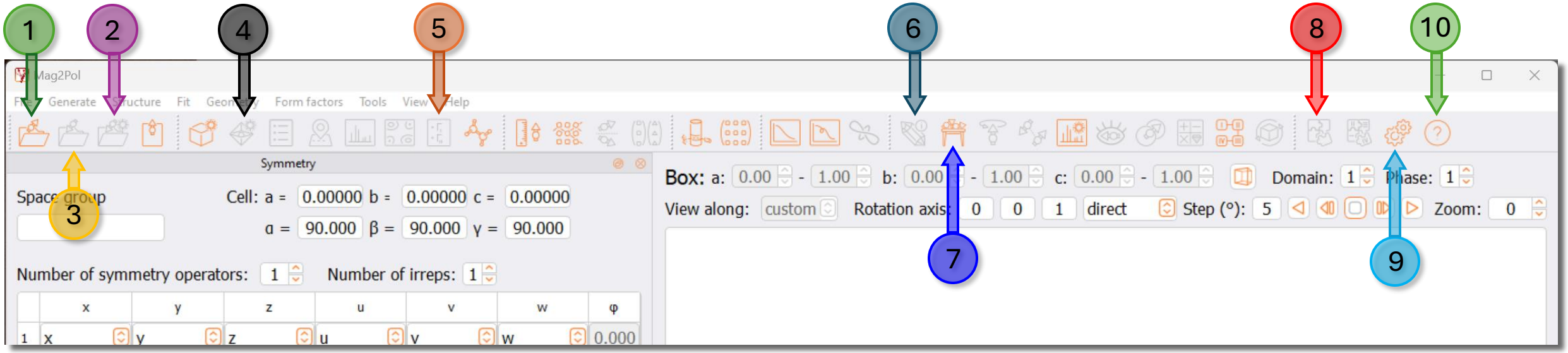


The main window of Mag2Pol - GUI

Some key plot elements that needs to be activated under settings for the purpose of this hands-on. (*For purely aesthetic reasons*)



Menu items (icons) on the main window relevant for this tutorial



1 **Open**: Load a structure file (cif) or an existing Mag2Pol project (xml).

2 **Save as**: Save as a new Mag2Pol project (as *.xml).

3 **Save**: Append the currently working project with the same name.

4 **Bonds**: Opens a new window that is used to modify the visuals of the structural model [*active after loading the structure file, if it is not active even after loading the cif file, press Ctrl+U (on Windows) or Cmd+U (on Mac)*].

5 **Irreducible representations:** Opens a new window that provides details of irreps and lets you select one or more irreps for the refinement of magnetic structure (active when at least one magnetic ion is defined)

6 **Sample info:** Opens a new window that provides details of, lattice and magnetic structures and the results of the last refinements.

7 **Spacegroup tables:** Opens a window on which you can select magnetic space group (Shubnikov group) instead of irreps, to refine the magnetic structure.

8 **Fit:** Opens the window where you can choose the data file, and instrument resolution file, and perform the refinements.

9 **Settings:** Opens a new window that lets you modify a few important settings about GUI, a graphical rendering of plots and figures, and also the refinement procedure.

10 **Manual:** Opens Mag2Pol manual in pdf format.

Refining high temperature NPD data, collected in paramagnetic phase

Try to find a reported structure (Crystal Information File)

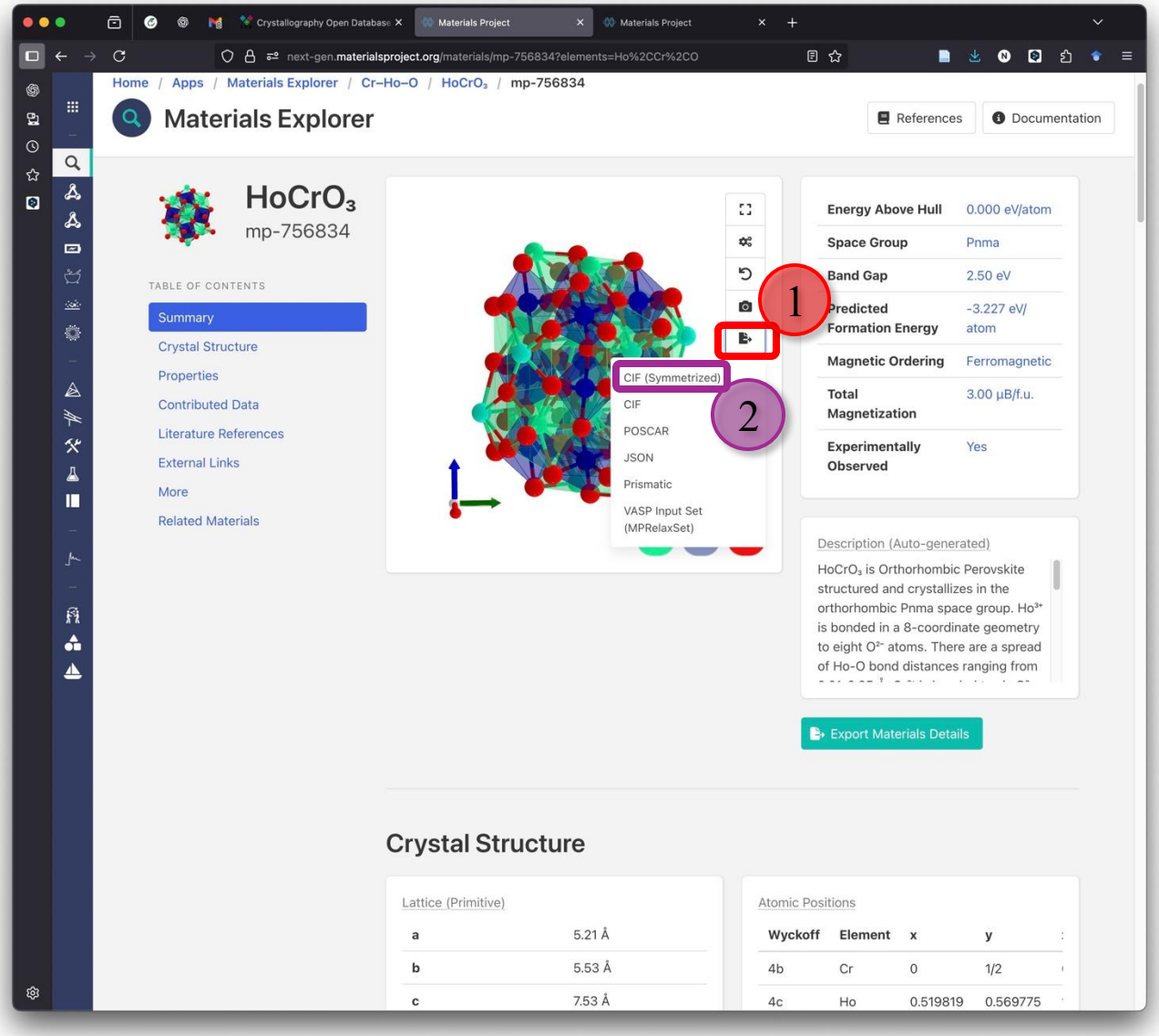
https://next-gen.materialsproject.org/

Search for the structure in multiple free and subscription-based sources. For example, ***COD*** or in the ***Materials Project*** database.

$HoCrO_3$ is not available in COD but can be found in Materials Project (you can sign in by linking your Google, GitHub, and many more accounts, or sign up using your email account).

Download symmetrized CIF and save as:

HoCrO3_mp-756834.cif

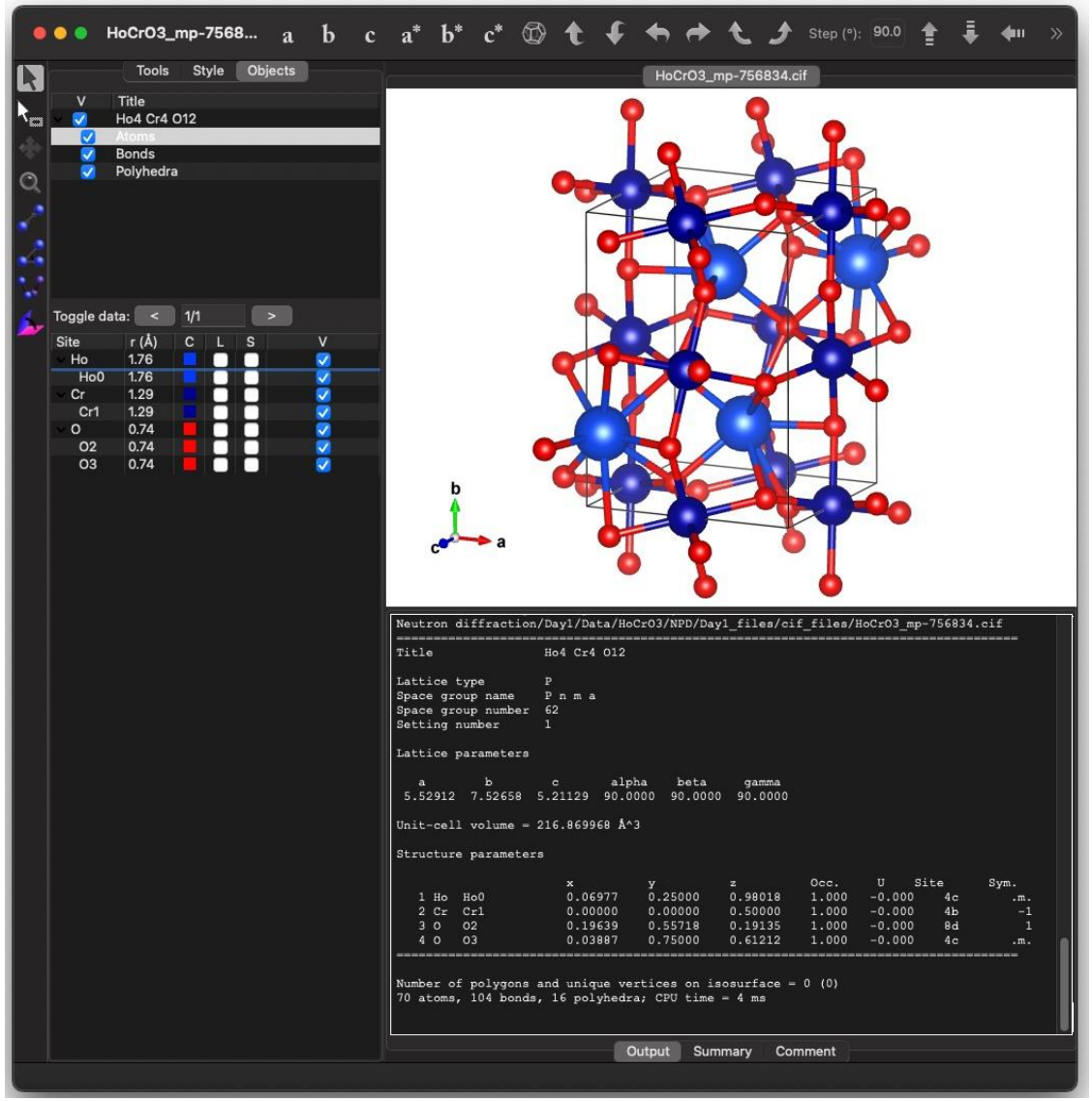


Other free structure databases:

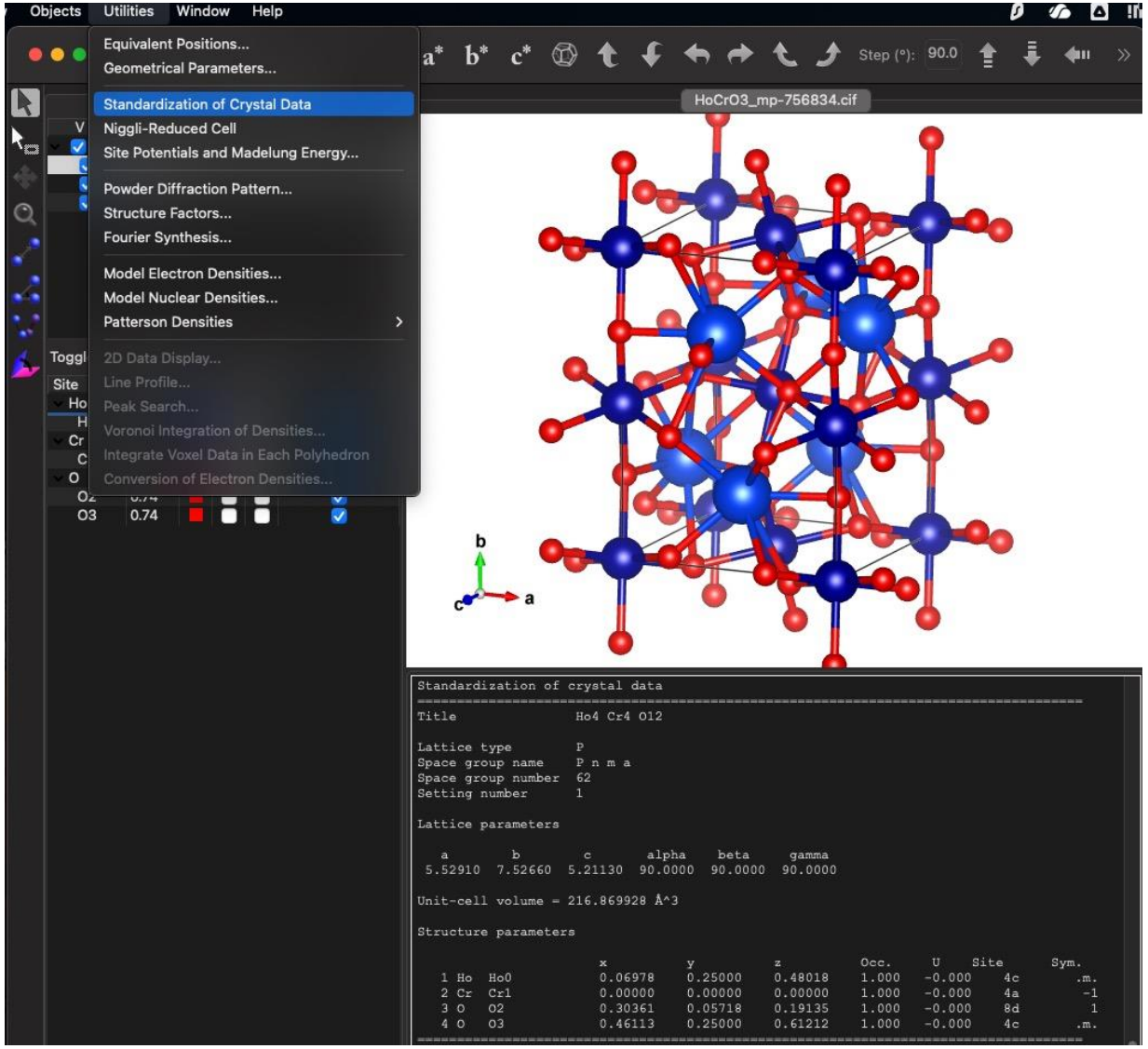
https://www.rruff.net/
https://www.crystallography.net/cod/search.php

Open the CIF file in VESTA and standardize (optional)

HoCrO3_mp-756834.cif



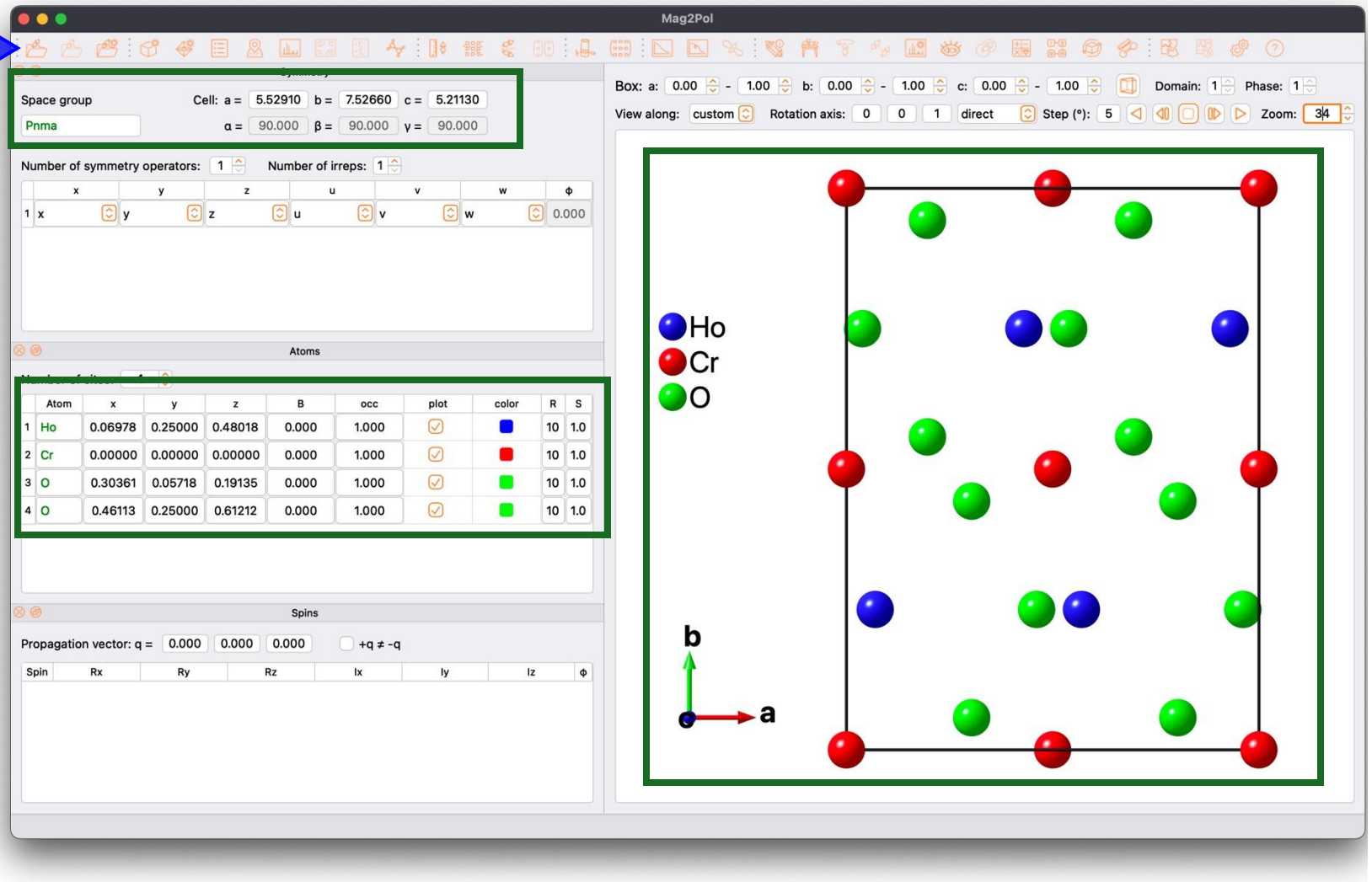
Utilities→Standardization of Crystal Data…
File→ Export Data → HoCrO3_mp-756834_Standardized.cif



Step 1: Loading the structure or an existing project in Mag2Pol

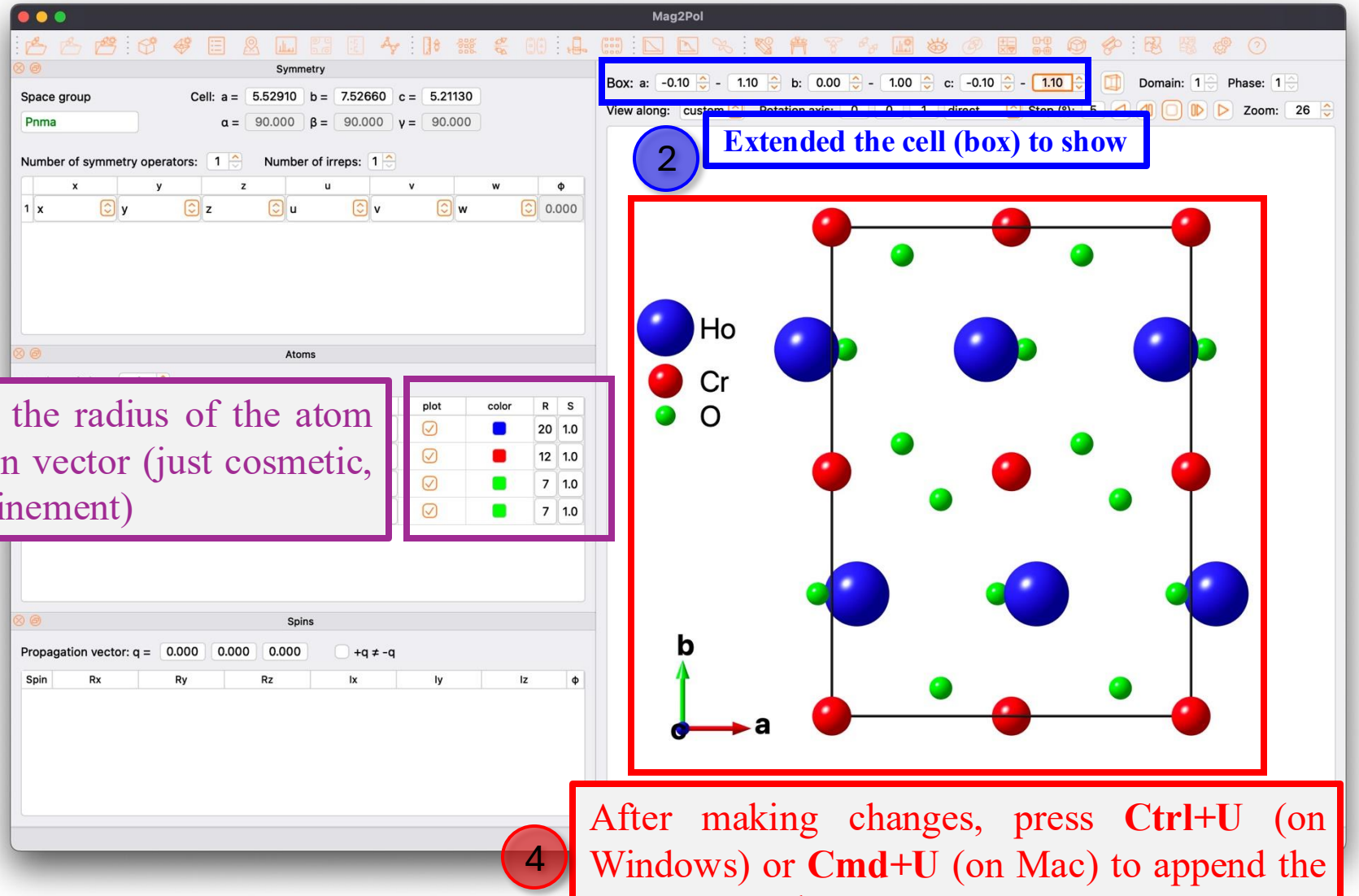
The structure file **(*.cif, *.mcif) or existing Mag2Pol project file (*.xml)** can be loaded by clicking this icon or through the menu tree: File→Open

In this case:
HoCrO3_mp-756834_Standardized.cif



If the structure file is loaded properly, several sections in the main window are populated automatically.

Step 1: Editing structure, plot it your way!

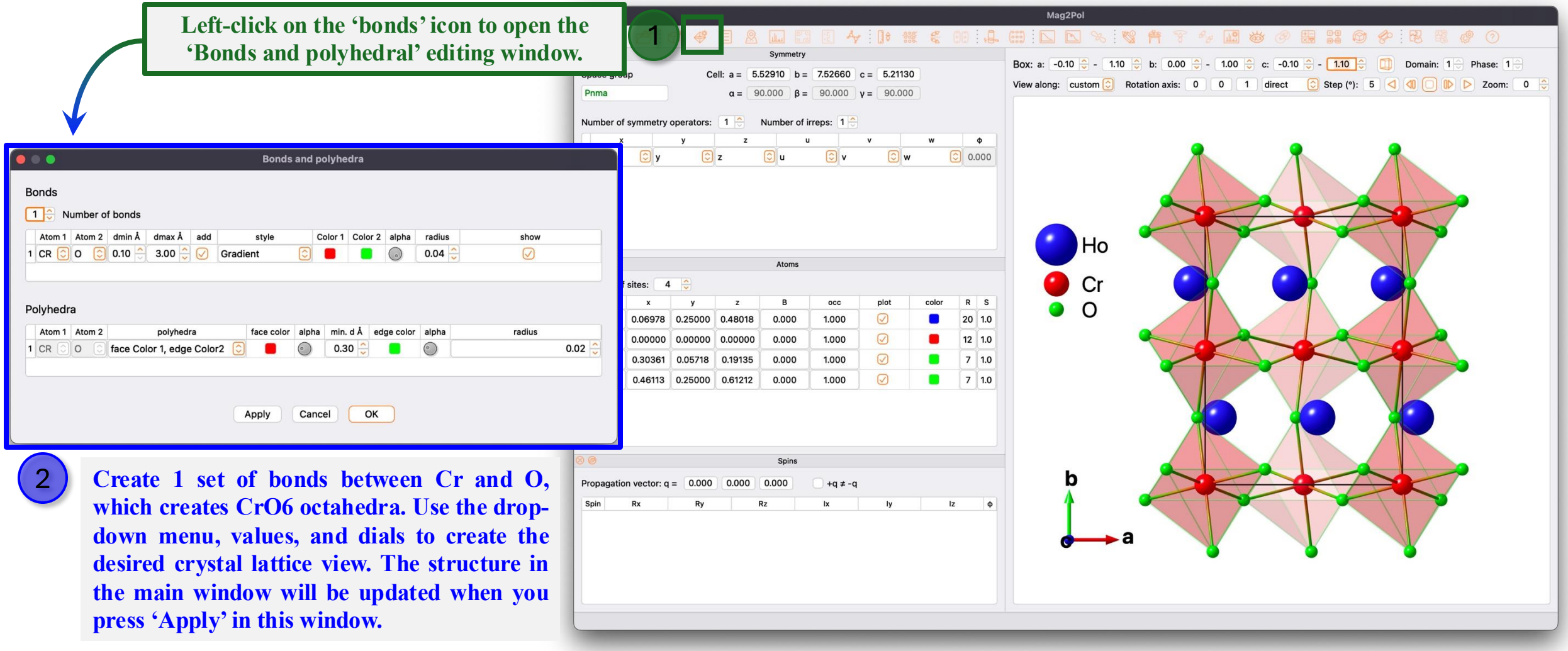


Here, R and S change the radius of the atom and S length of the spin vector (just cosmetic, has no effect on the refinement)

After making changes, press **Ctrl+U** (on Windows) or **Cmd+U** (on Mac) to append the structure on the screen.

Step 1: Editing structure - bond and the octahedra

Left-click on the ‘bonds’ icon to open the ‘Bonds and polyhedral’ editing window.

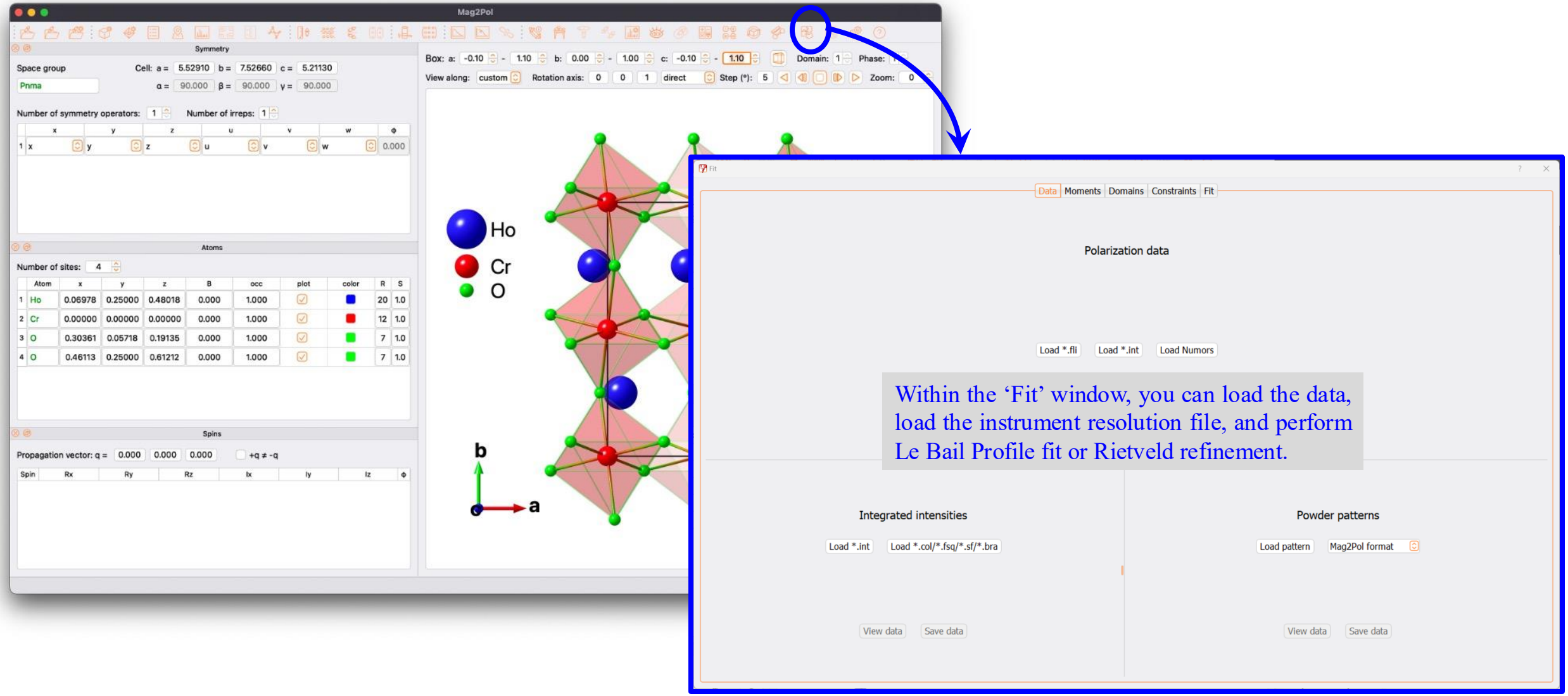


2

Create 1 set of bonds between Cr and O, which creates CrO6 octahedra. Use the drop-down menu, values, and dials to create the desired crystal lattice view. The structure in the main window will be updated when you press ‘Apply’ in this window.

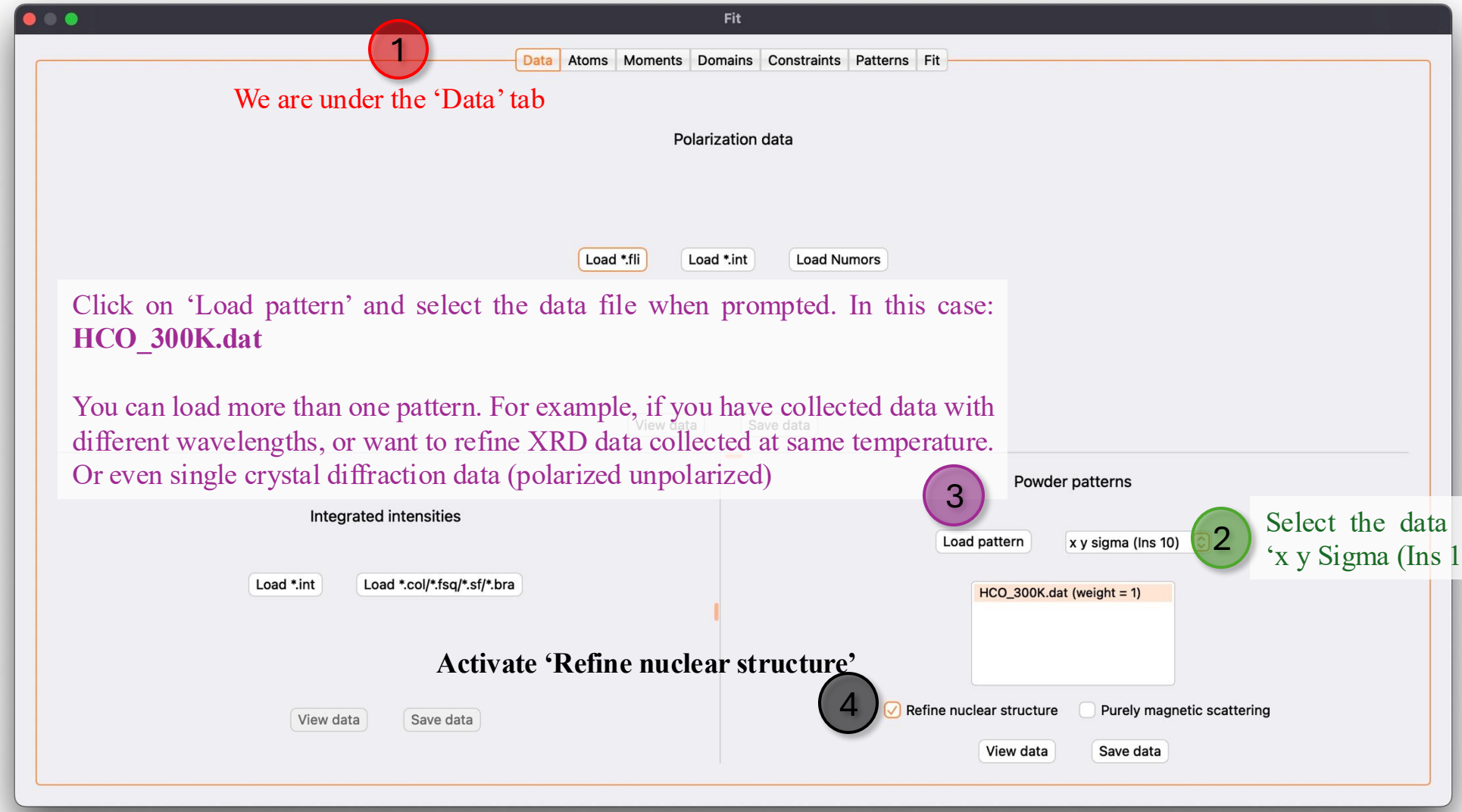
Step 2: Enter the refinement (Fit) window and load experimental data

To load the experimental data we need to open the ‘Fit’ window, by clicking on the icon as shown in the screenshot below (or the keyboard shortcut ‘Ctrl+F’).



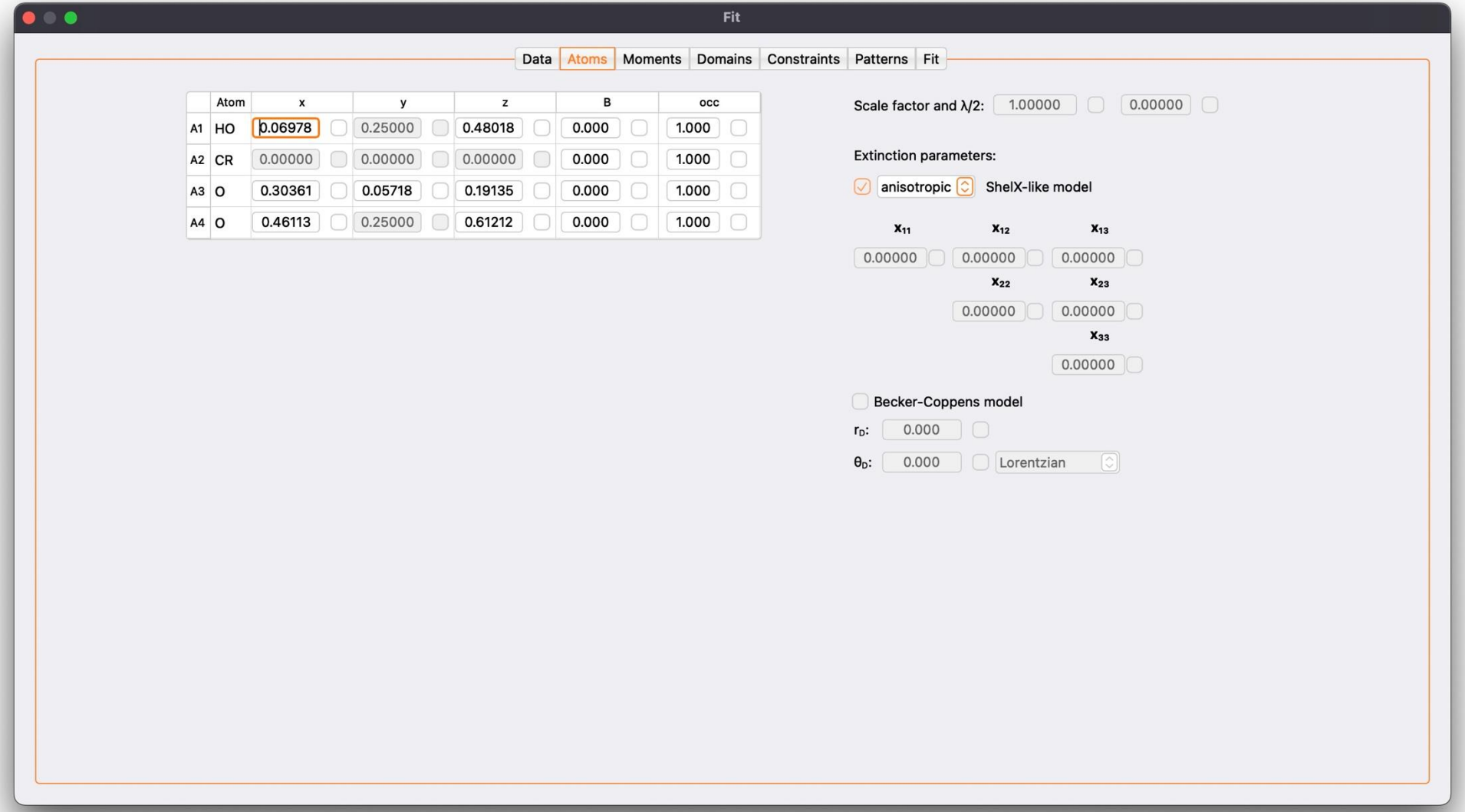
Load the experimental data collected at 300 K

The data can be loaded under the 'Data' tab of the 'Fit' window. Mag2Pol can accept three kinds of data sets. In this example, we will start with 'Powder diffraction data' of $HoCrO_3$ collected at 300 K (Paramagnetic phase). **Follow the numerical order indicated below.**



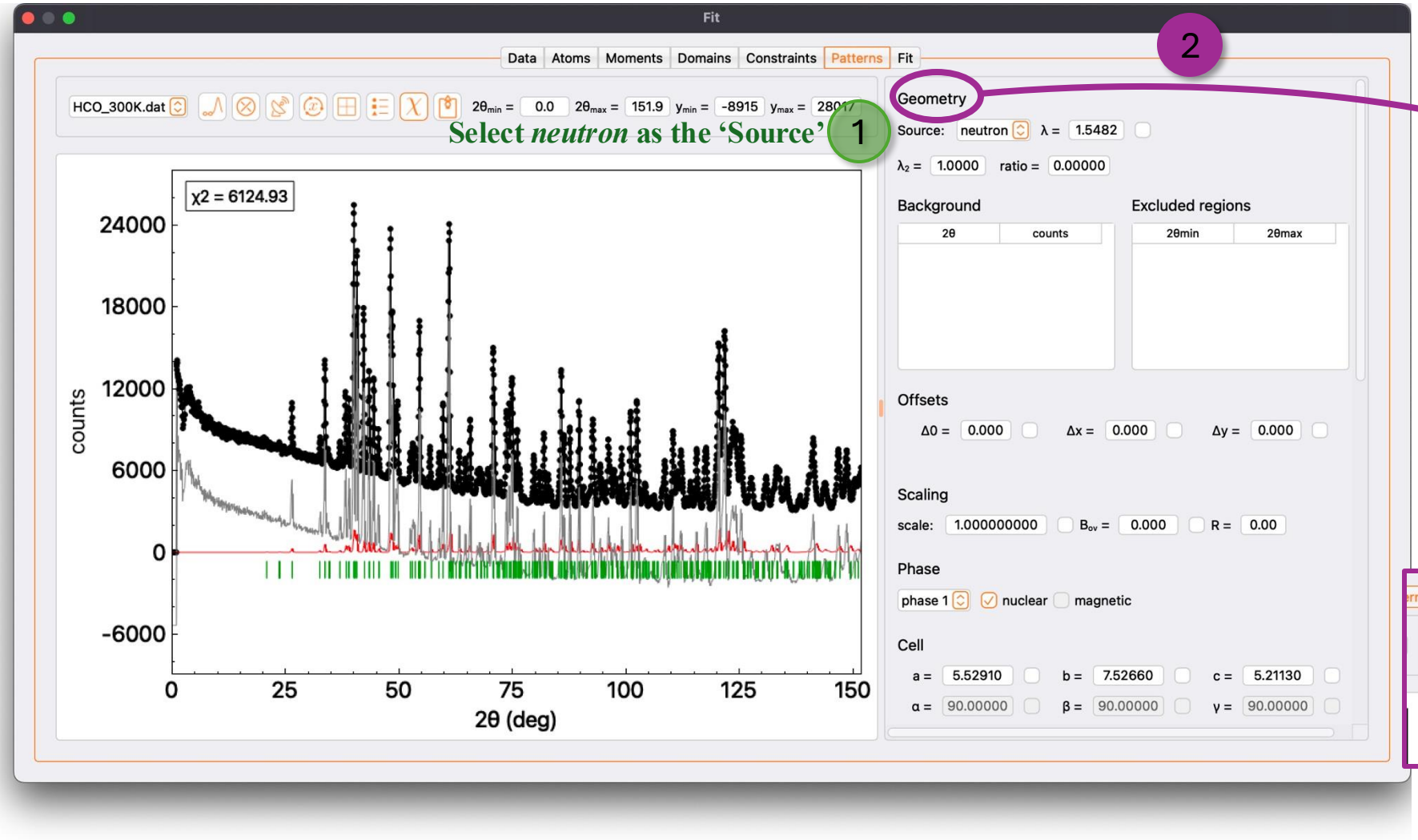
Step 2b: Confirm all the atoms are loaded properly

Activate ‘Atoms’ tab: Check if all the atoms are loaded properly and the occupancies nominally follow the chemical formula. Do not select any of these parameters for refinement at this stage.



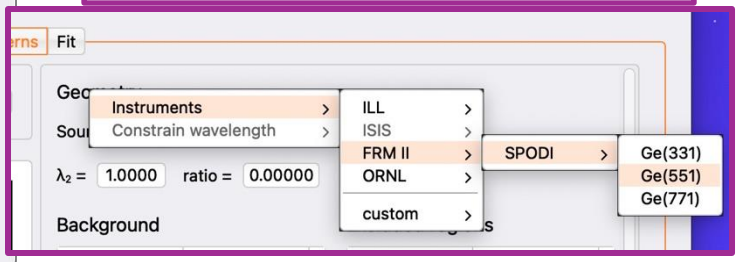
Step 2c: Edit source, instrument, and wavelength parameters

Activate 'Patterns' tab

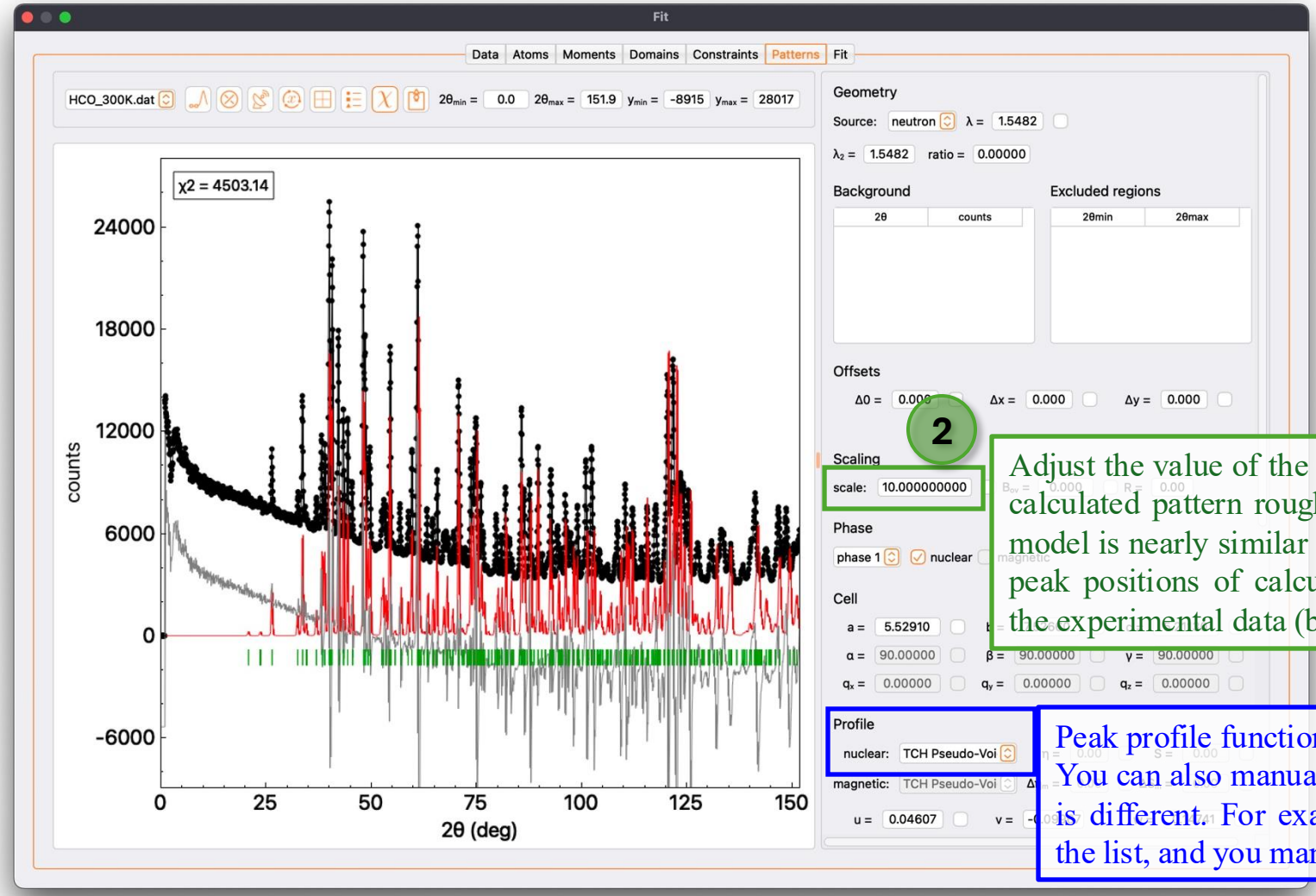


Right-click on the text 'Geometry' and follow the menu items as shown in the screenshot below, to select the neutron facility, instrument, and the appropriate monochromator (which is used for the experiment).

In this case, it is Ge(551) was is used to select neutrons of wavelength 1.5482 Å



Step 2d: Check the peak function and set an appropriate scale factor



Adjust the value of the 'scale' factor so that the intensities of the calculated pattern roughly match those of observed data. If the model is nearly similar and the wavelength choice is correct then peak positions of calculated patterns (red curve) should match the experimental data (black curve)

Peak profile function is automatically updated here. You can also manually select it if, for any reason, it is different. For example, the instrument is not in the list, and you manually entered the wavelength.

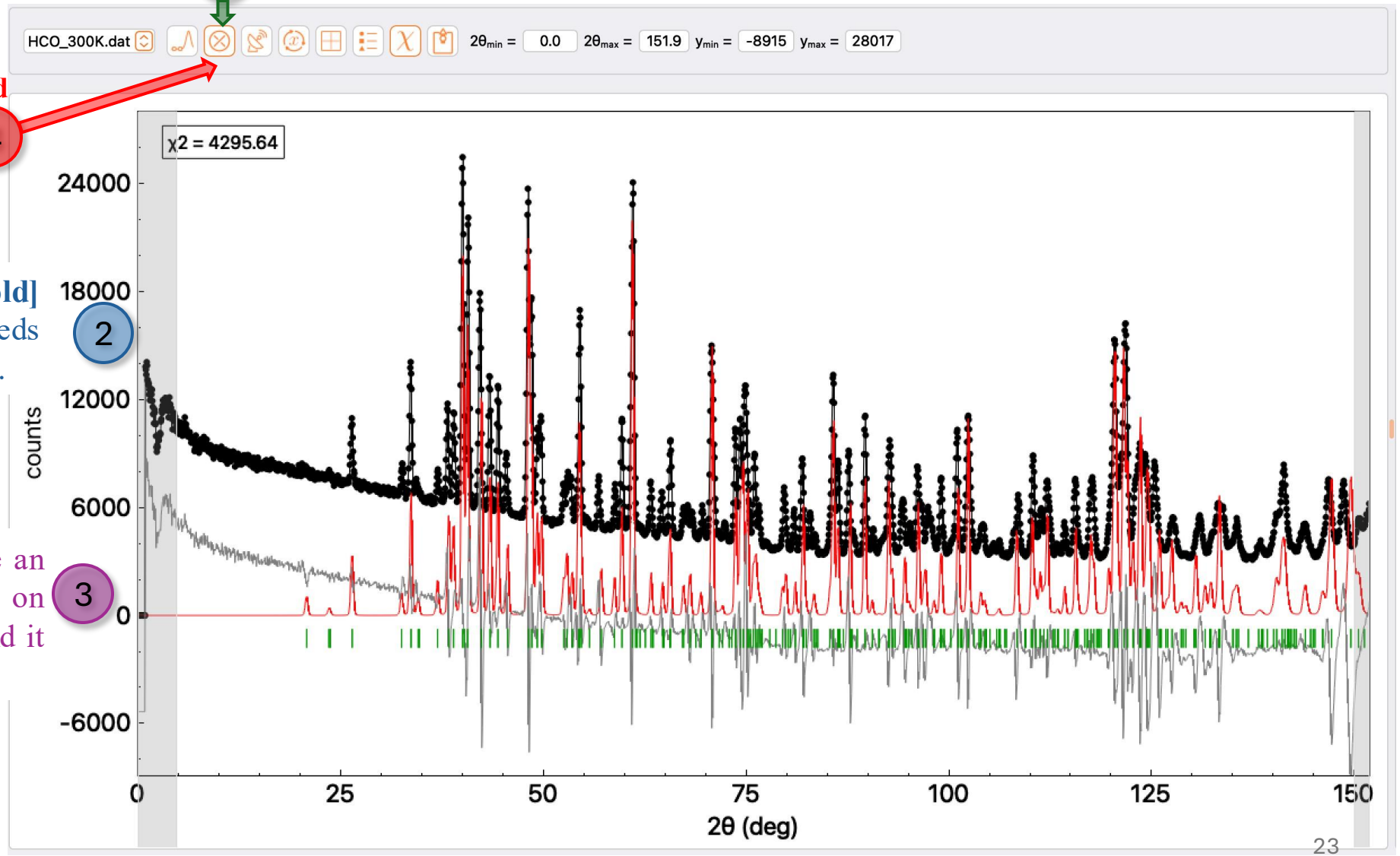
Step 2e: Select the region to exclude from the fit ‘Excluded region’

Toggle the ‘excluded regions’ ON

1

Toggle the ‘excluded regions’ OFF

4



On the plot, **[Left click + hold]** and drag on the region that needs to be excluded. Grey area here.

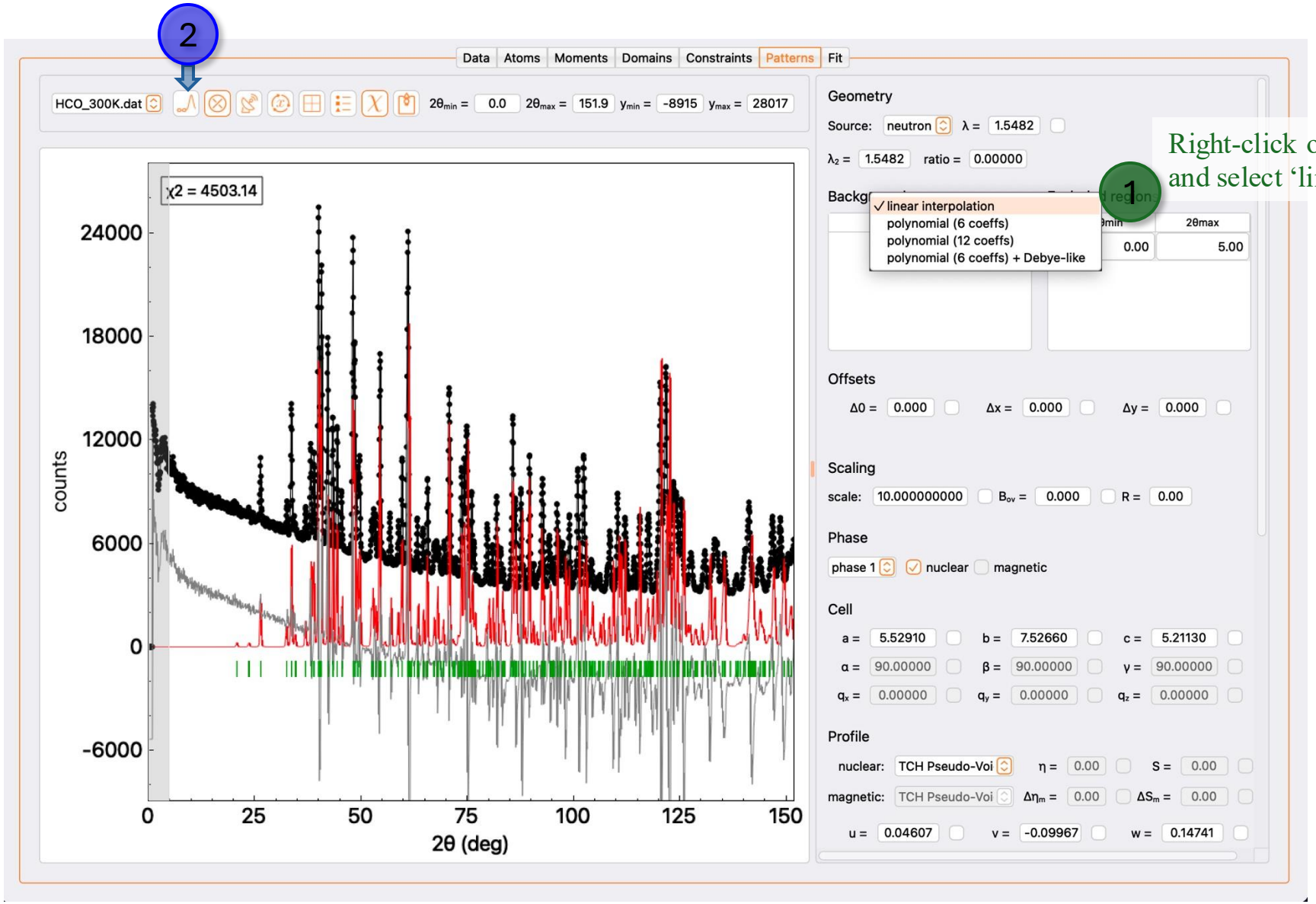
2

If it is necessary to remove an excluded region, right-click on the (grey) excluded area, and it will be removed

3

Step 2f: Select the background type

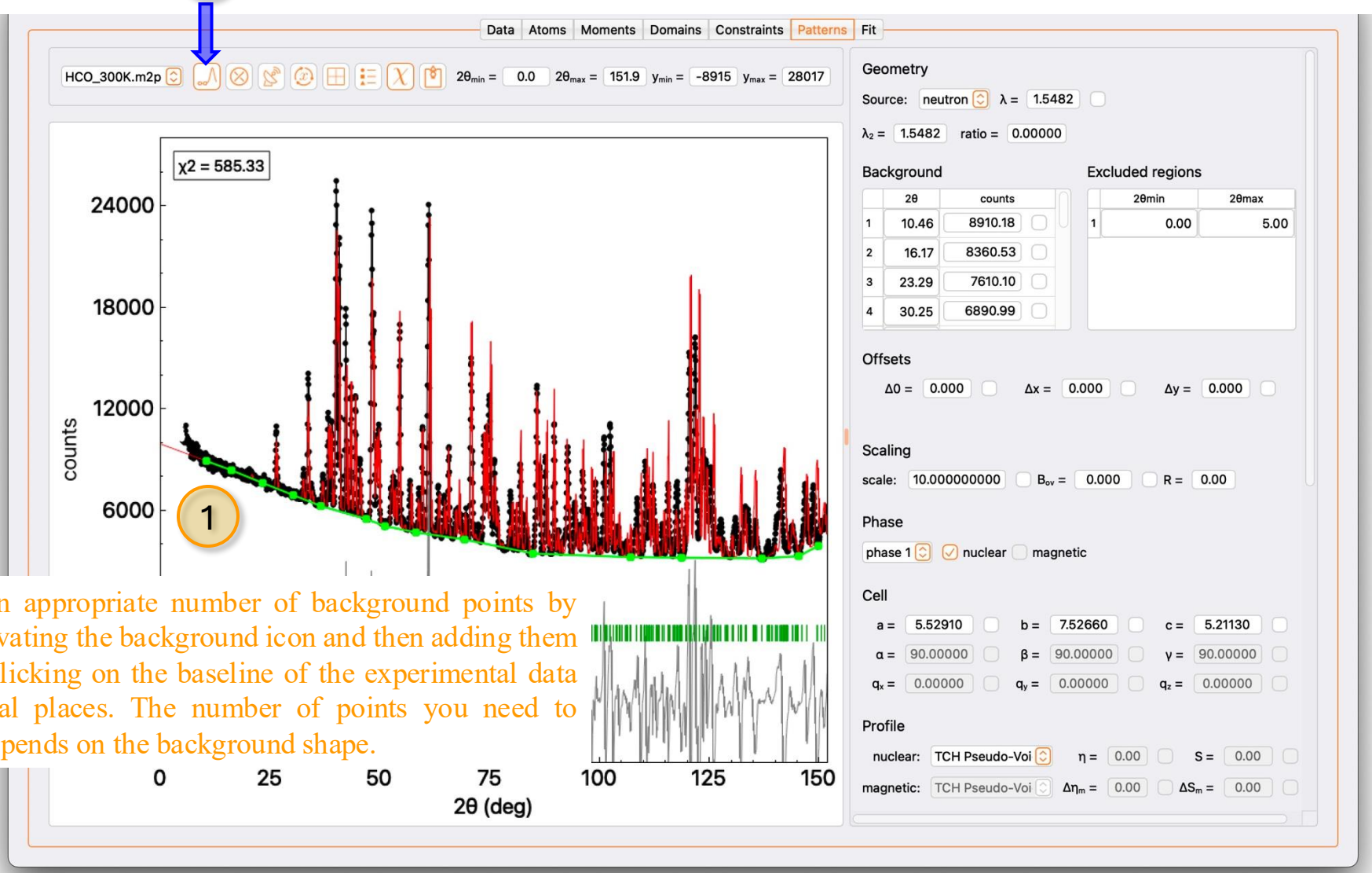
For manual background selection, toggle this ON



Right-click on the text 'Background' and select 'linear interpolation'

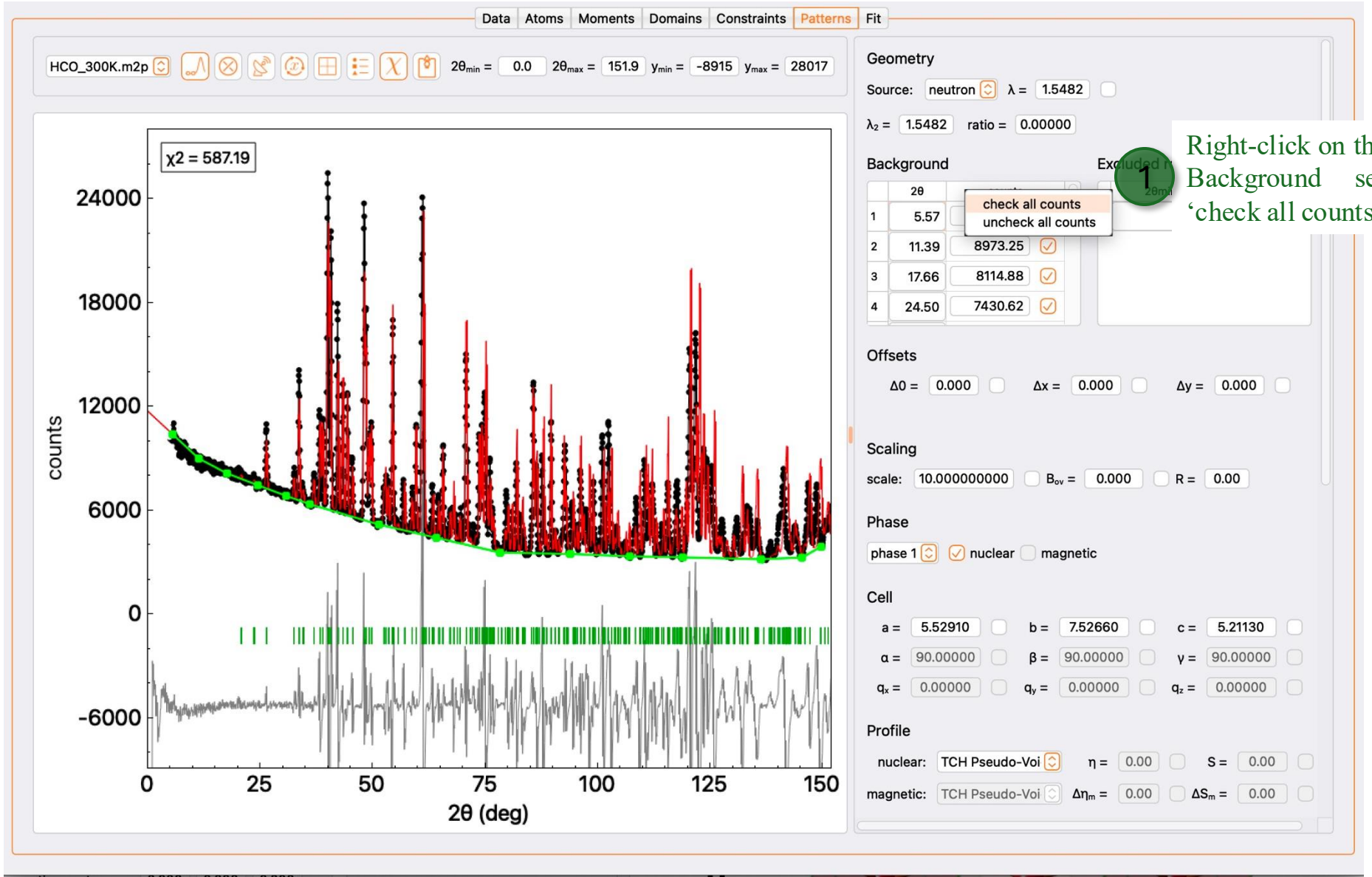
Step 2f: Select the background

Toggle it off after selecting all the background points



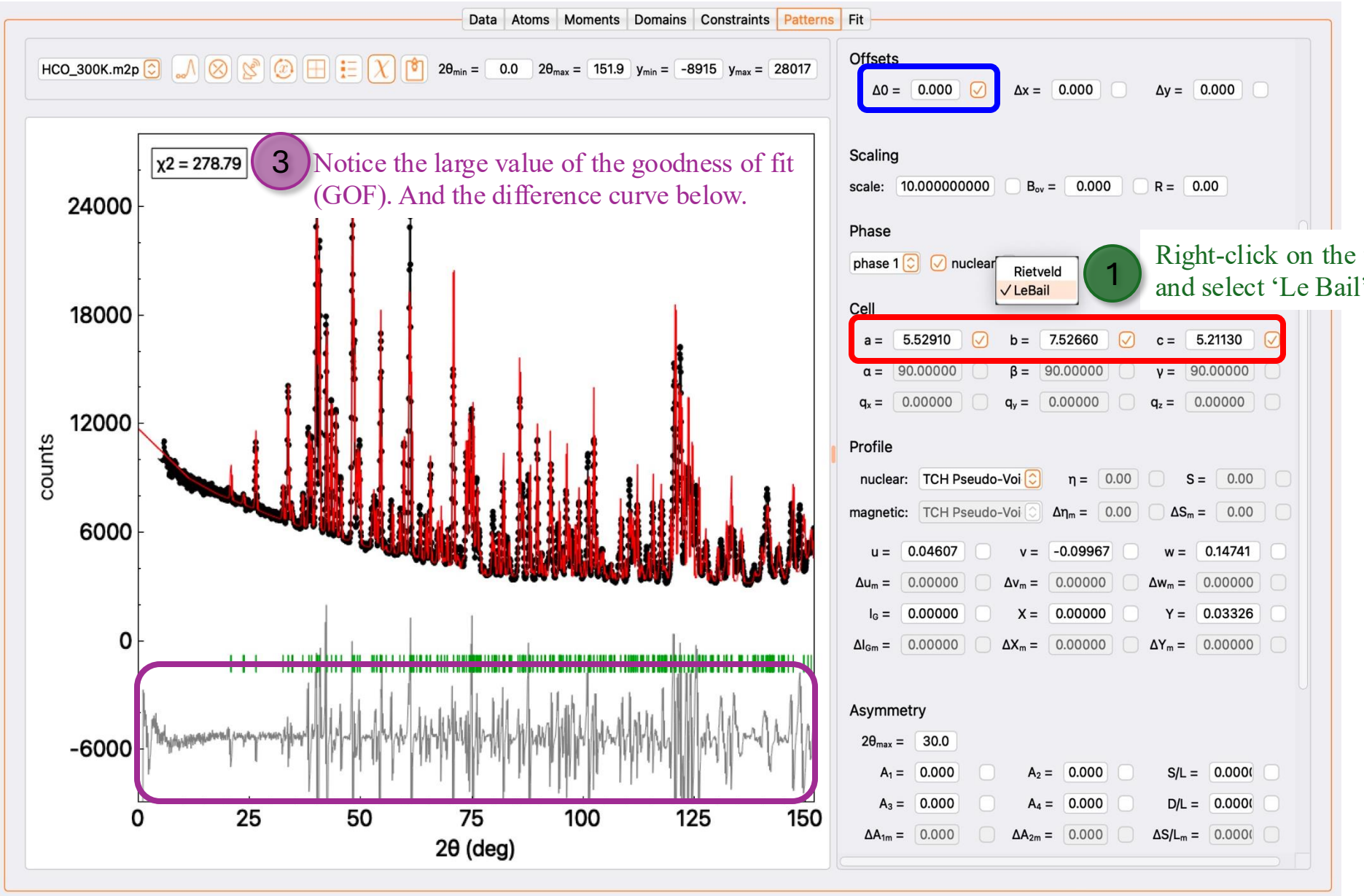
Select an appropriate number of background points by first activating the background icon and then adding them by left-clicking on the baseline of the experimental data at several places. The number of points you need to select depends on the background shape.

Activate the refinement of background



Right-click on the text ‘counts’ in the Background section and select ‘check all counts’

Le Bail fit



Activate the refinement of:

2

Δ0, A constant shift added to all calculated Bragg angles:

$$2\theta_{\text{calc}} \rightarrow 2\theta_{\text{calc}} + \Delta 0$$

Influenced by:

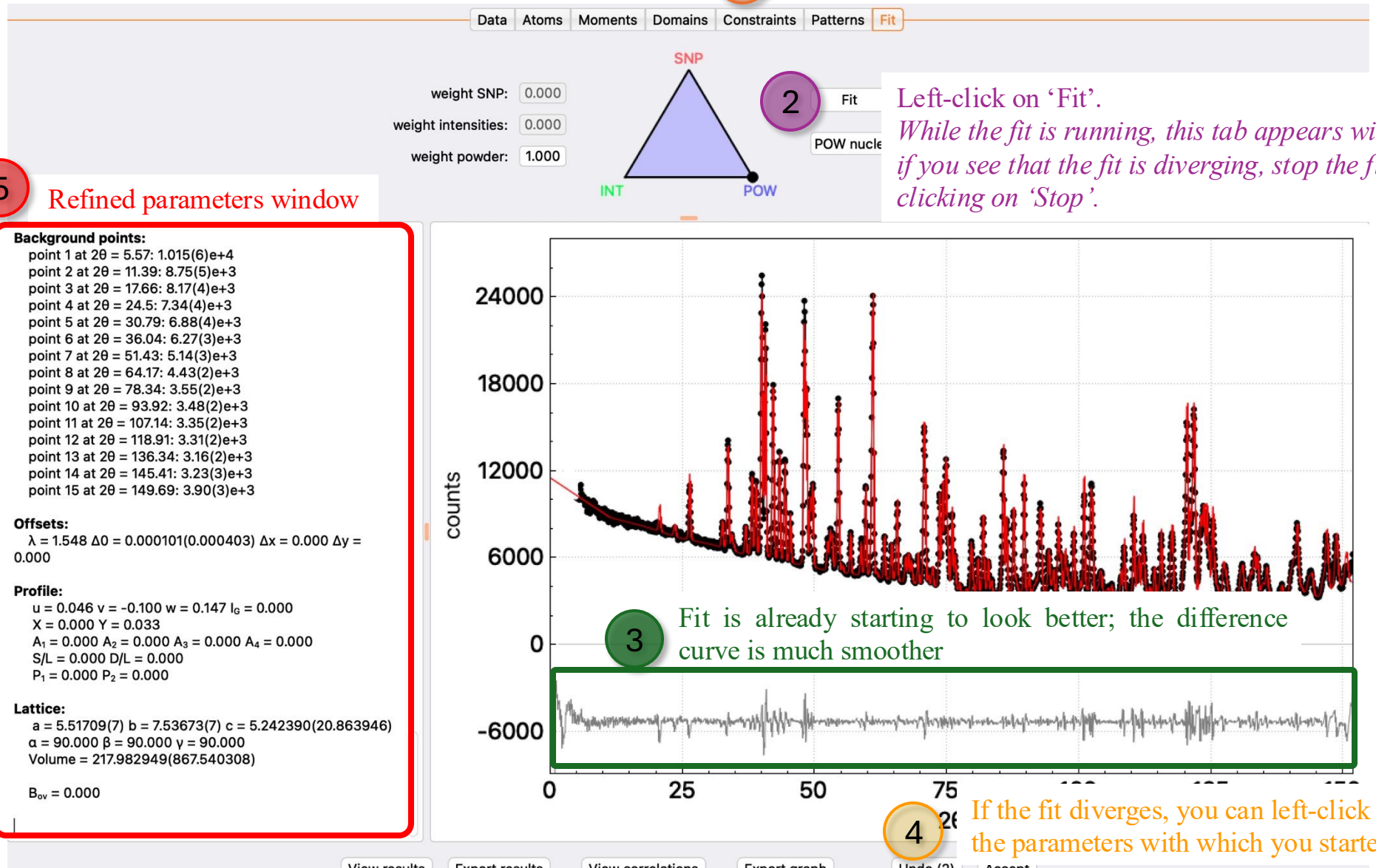
- mis-calibration of detector zero
- instrument alignment

4

a, b, c: Lattice parameters

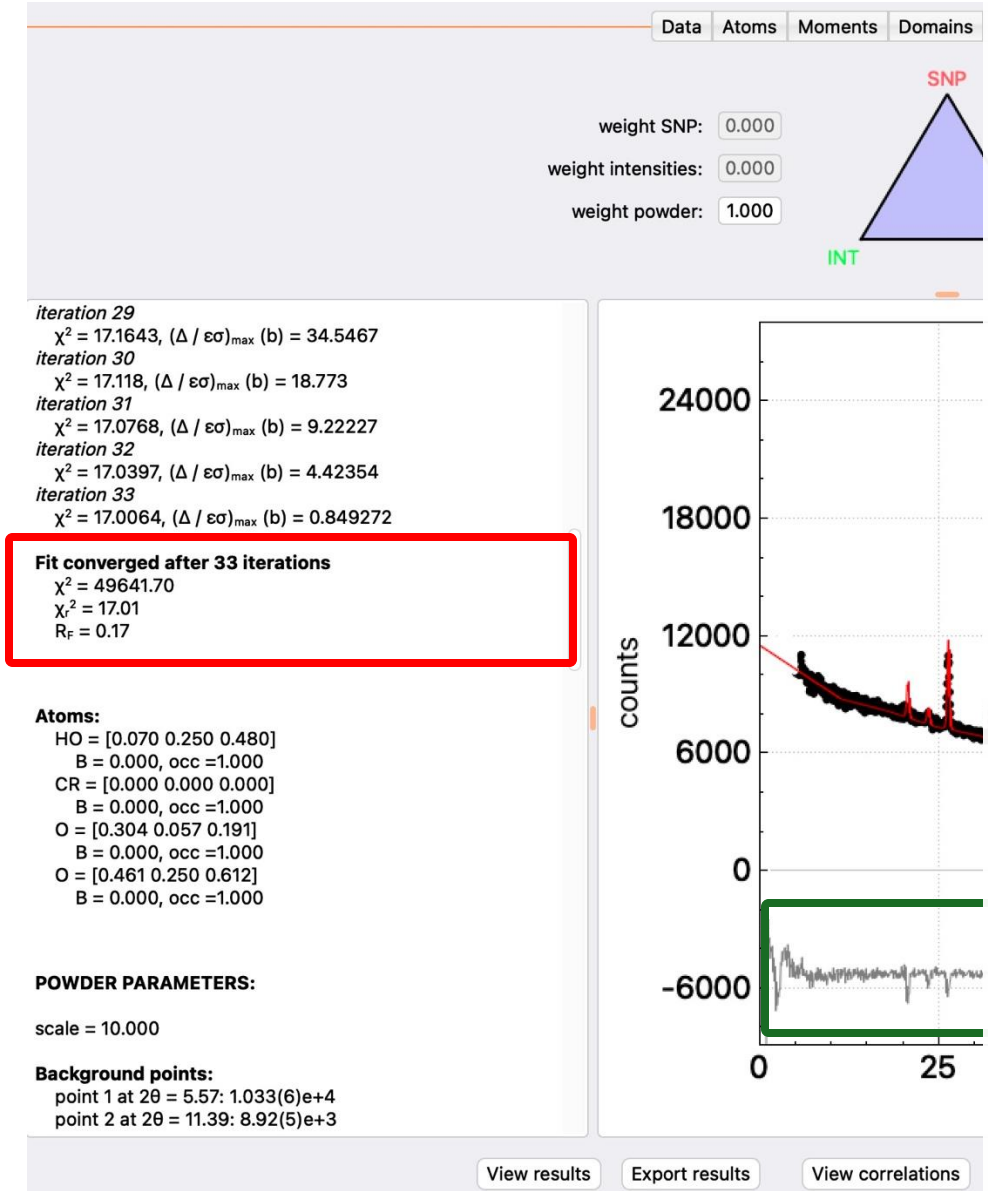
Run Le Bail fit

1 Activate ‘Fit’ tab



Left-click on ‘Fit’.
While the fit is running, this tab appears with the text ‘Stop’, *if you see that the fit is diverging, stop the fitting by right-clicking on ‘Stop’.*

Calculated diffraction intensity: Le Bail fit



Least square minimization via:

$$\chi^2 = \sum_i \left[\frac{y_{\mathrm{obs},i} - y_{\mathrm{cal},i}}{\sigma_i} \right]^2$$

$$\chi_r^2 = \chi^2/(m-n)$$

σ_i = statistical counting uncertainty of each observed point.
m = number of observations (data points),
n = number of refined parameters

Agreement is evaluated via:

$$R_F = 100 \cdot \frac{\sum_Q |F_{\mathrm{obs},Q} - F_{\mathrm{cal},Q}|}{\sum_Q F_{\mathrm{obs},Q}}$$

$$F_{\mathrm{obs},Q} = \sqrt{\frac{I_{\mathrm{obs},Q}}{m_Q L_Q}}$$

Here $I_{\mathrm{obs,Q}} \approx I_{\mathrm{Q}}$ after convergence

Calculated diffraction intensity Le Bail:

$$y_{\mathrm{cal}}(2\theta_i) = \sum_{Q=(hkl)} \underbrace{I_Q}_{\text{refined reflection intensity}} \underbrace{\Phi(2\theta_i - 2\theta_Q - \Delta(2\theta)_{\mathrm{disp}})}_{\text{profile function}} + \underbrace{b_i}_{\text{background}}$$

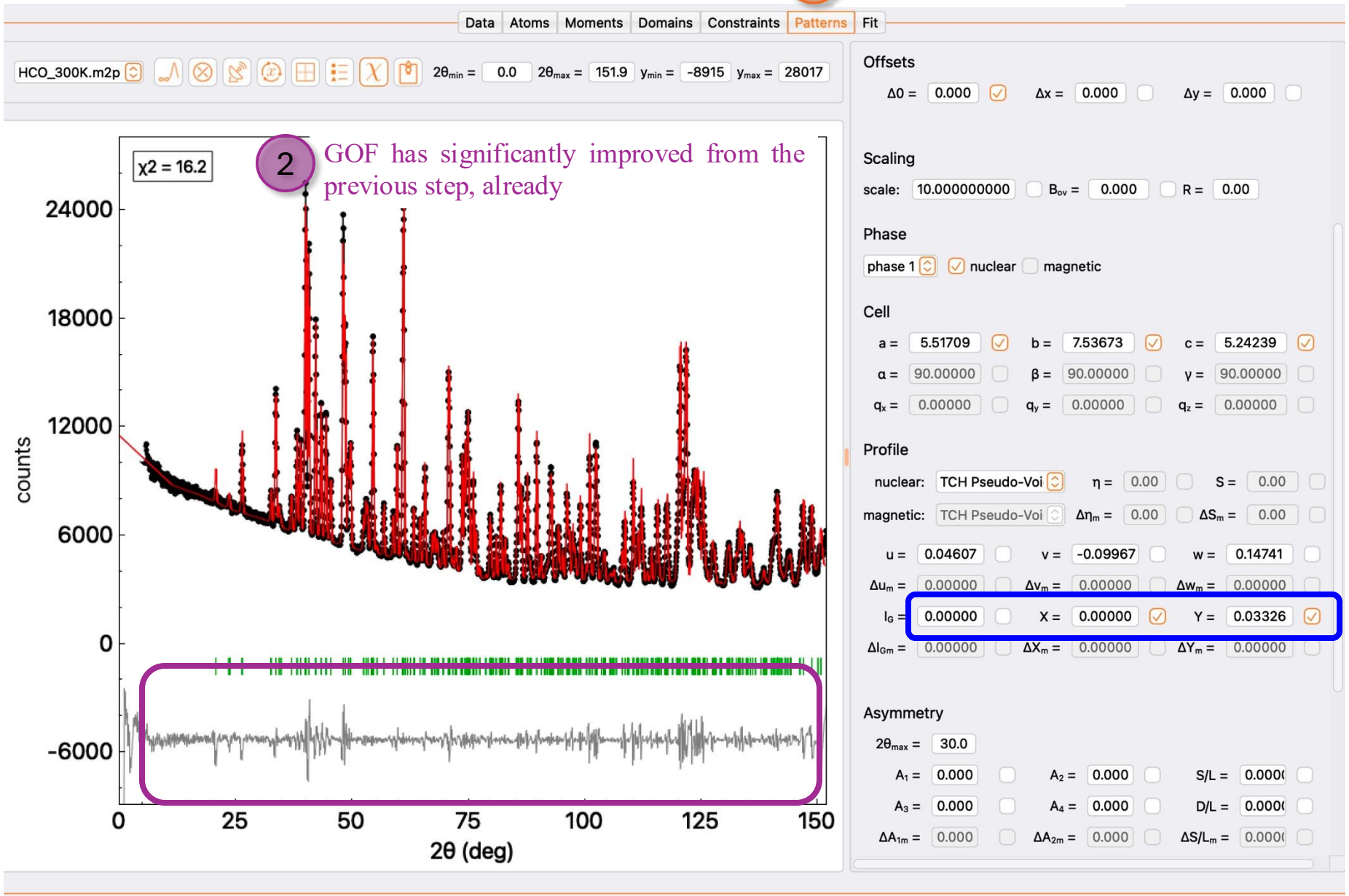
$Q = (hkl)$: reflection index
I_Q : refined integrated intensity of each reflection (free parameter; no structure factor)
Φ(...): peak profile (U, V, W, X, Y, asymmetry, sample displacement, etc.
$\boldsymbol{B_i}$**:** background intensity at point ***i*** (polynomial / point-interpolated / Debye-like), modeling all non-Bragg contributions.
$2\theta_Q$: Bragg angle obtained from lattice parameters via

$$2*d*\sin\theta_Q = \lambda$$

In Le Bail fit, $F_{cal,Q}$ is not computed from structure factors, instead, refined reflection intensities serve as effective F^2

Refine more profile parameters

1 Activate ‘Patterns’ tab



3 Activate refinement of X and Y

X and **Y** control the Lorentzian component of peak width:

$$H_L = X \tan\theta + Y / \cos\theta$$

Influenced by:

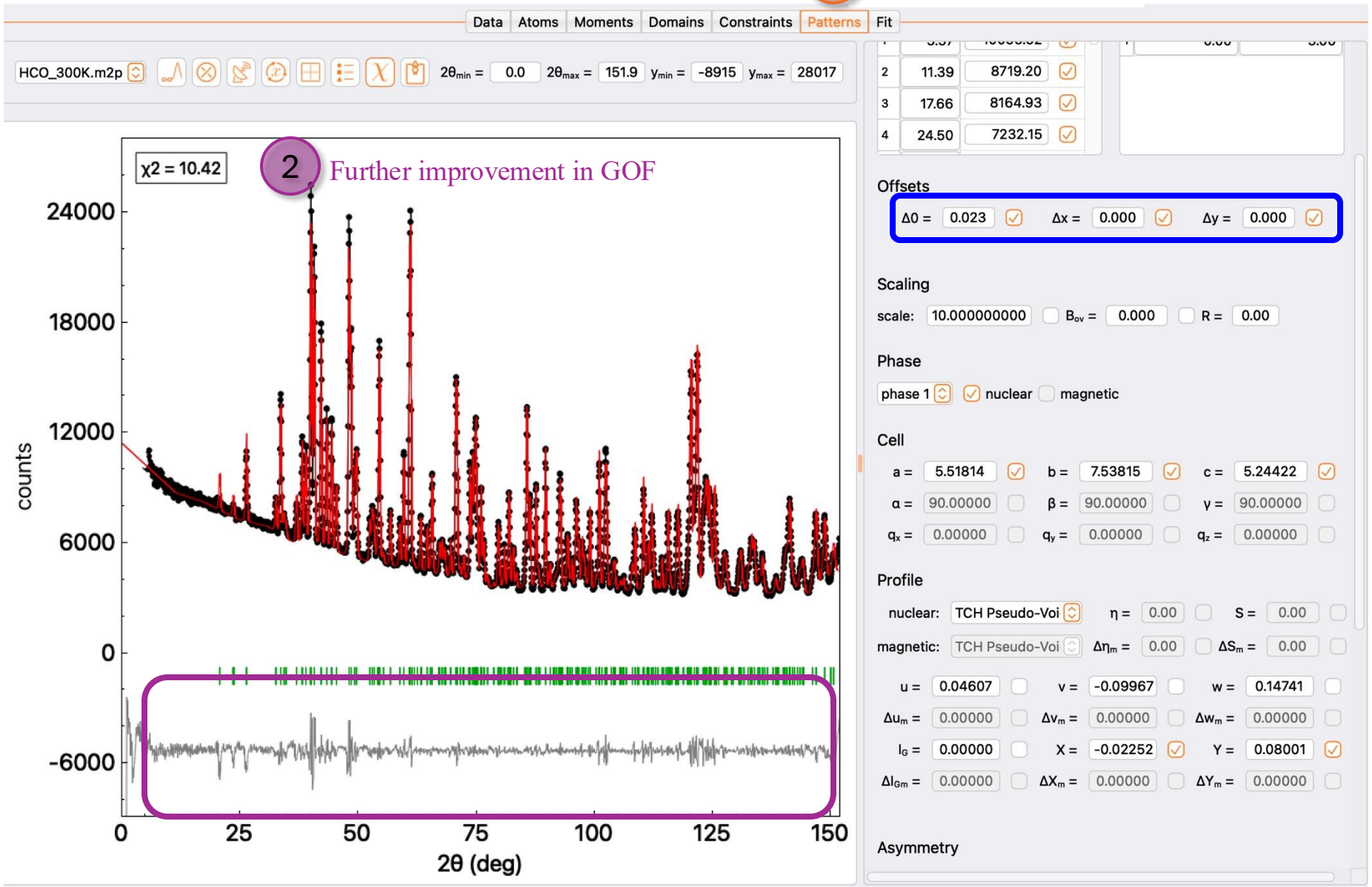
- crystallite size (Lorentzian term)
- strain broadening (high-angle broadening)
- instrument axial divergence (partially)

4

Go to the ‘Fit’ tab and run the refinement by left-clicking on the ‘Fit’ button. Then return back to ‘Patterns’ tab.

Refine more profile parameters

1 Activate ‘Patterns’ tab



3 **Activate refinement of ΔX and ΔY**

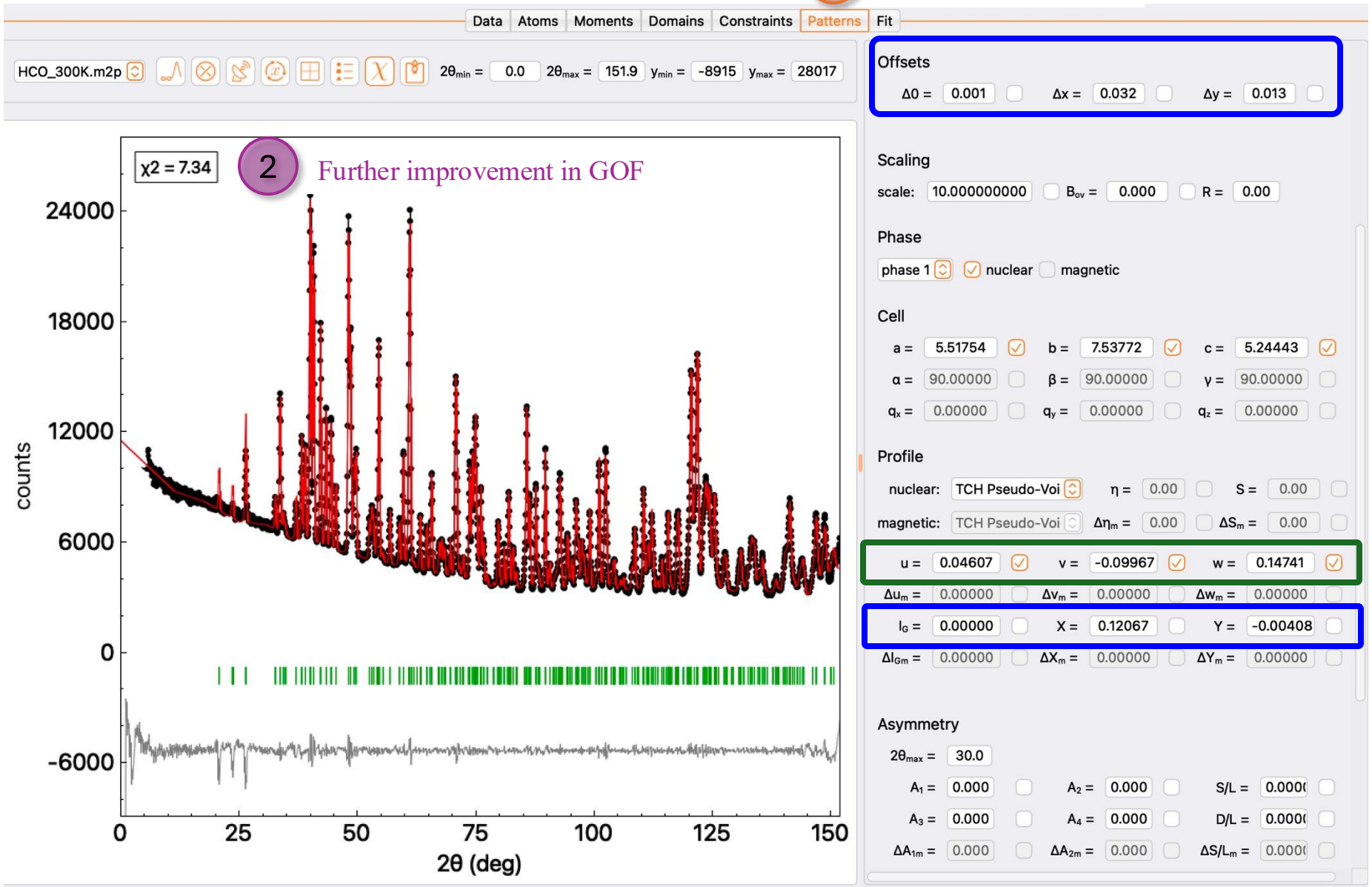
ΔX: Sample height deviation

ΔY: lateral mis-positioning along the beam direction

4 Go to the ‘Fit’ tab and run the refinement by left-clicking on the ‘Fit’ button. Then return back to ‘Patterns’ tab.

Refine more profile parameters

1 Activate ‘Patterns’ tab



3

Fix: Offsets, ΔX and ΔY

4

Refine: ***u, v, w,*** which control the Gaussian component of the peak function

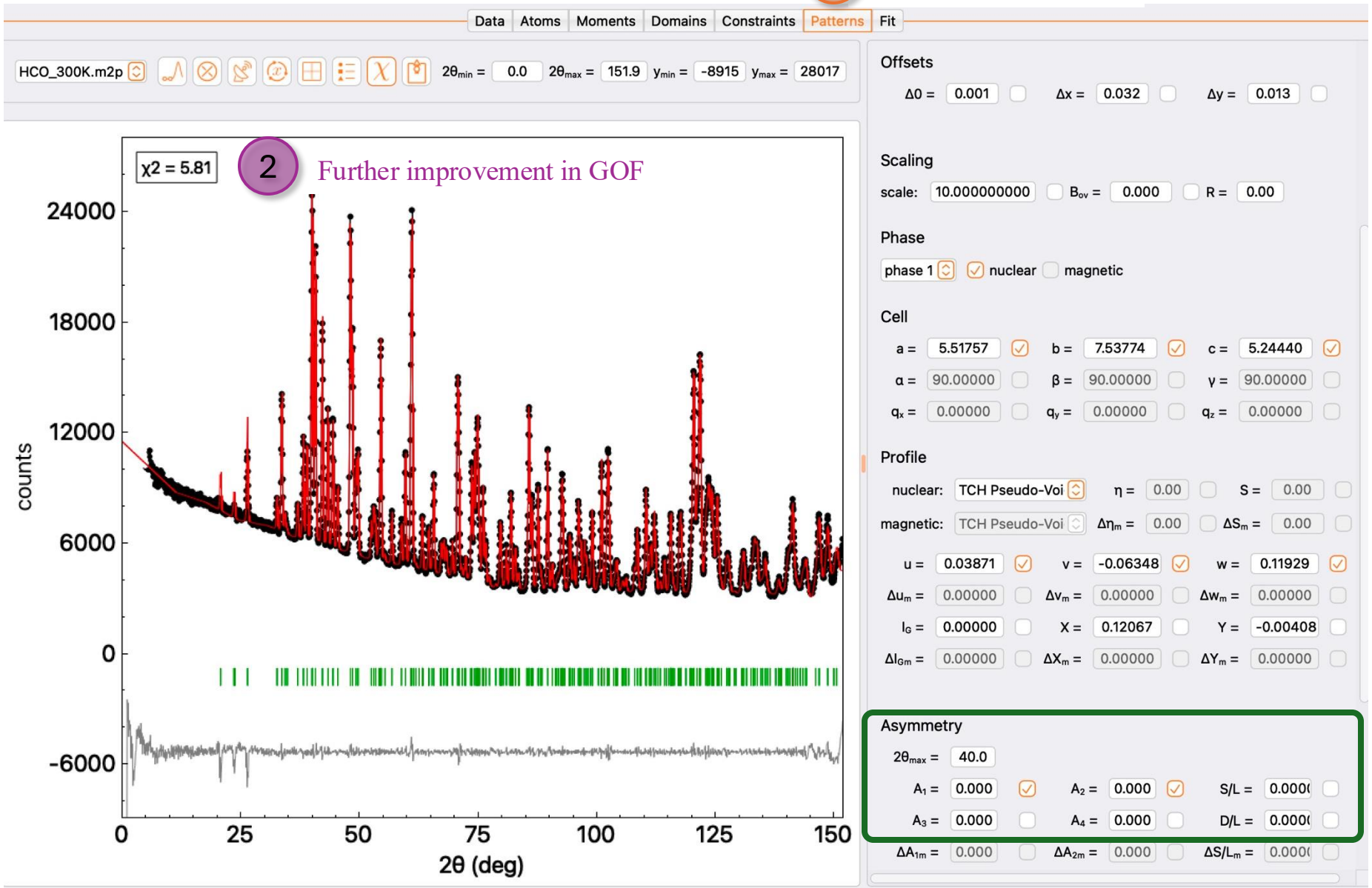
$$H_G^2 = u\tan^2\theta + v\tan\theta + w$$

5

Go to the ‘Fit’ tab and run the refinement by left-clicking on the ‘Fit’ button. Then return back to ‘Patterns’ tab.

Refine more profile parameters

1 Activate ‘Patterns’ tab



3

Refine: A_1, A_2 asymmetry correction coefficients describing peak tails on the low-angle side

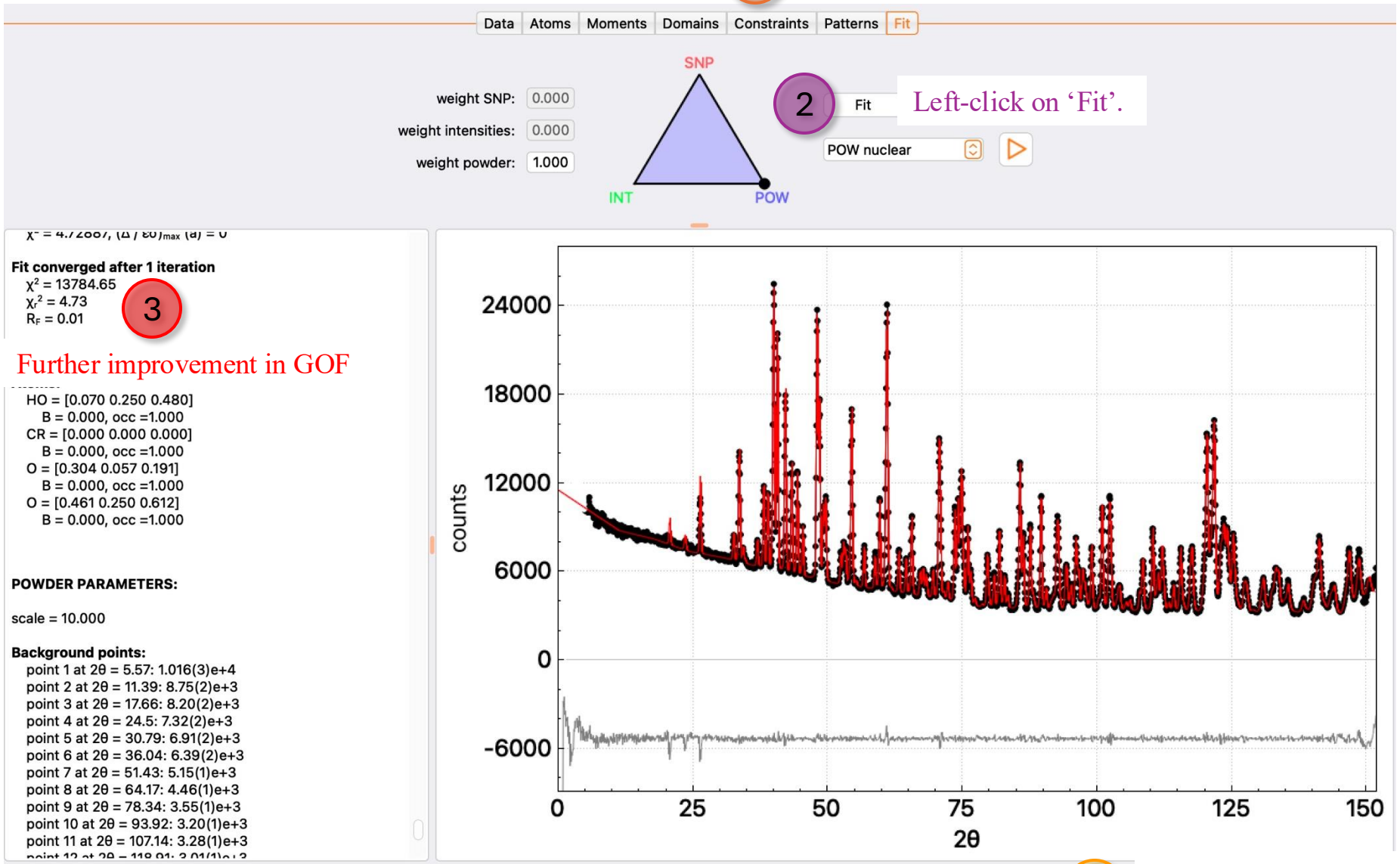
$$\text{Asymmetry} = A_1 f_1(\theta) + A_2 f_2(\theta) + A_3 f_3(\theta) + A_4 f_4(\theta)$$

4

Go to the ‘Fit’ tab and run the refinement by left-clicking on the ‘Fit’ button.

Run final Le Bail fit

1 Activate ‘Fit’ tab



Save a copy of the project file

1 Go to the main Mag2Pol window

2 Save a copy of the project as ‘HCO_300K_LeBail.xml’

Save as

Symmetry

Space group: Pnma

Cell: a = 5.52910 b = 7.52660 c = 5.21130

α = 90.000 β = 90.000 γ = 90.000

Number of symmetry operators: 1 Number of irreps: 1

	x	y	z	u	v	w	ϕ
1	x	y	z	u	v	w	0.000

Atoms

Number of sites: 4

	Atom	x	y	z	B	occ	plot	color	R	S
1	HO	0.06978	0.25000	0.48018	0.000	1.000	☑		20	1.0
2	CR	0.00000	0.00000	0.00000	0.000	1.000	☑		12	1.0
3	O	0.30361	0.05718	0.19135	0.000	1.000	☑		7	1.0
4	O	0.46113	0.25000	0.61212	0.000	1.000	☑		7	1.0

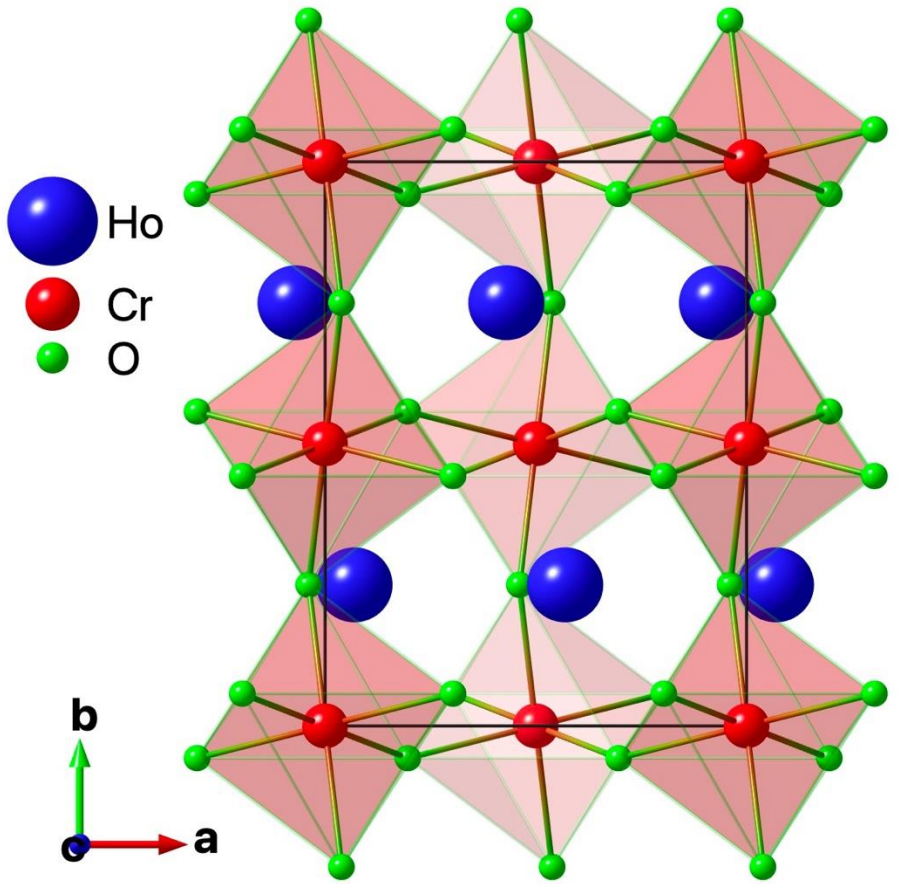
Spins

Propagation vector: q = 0.000 0.000 0.000 +q ≠ -q

Spin	Rx	Ry	Rz	Ix	Iy	Iz	ϕ

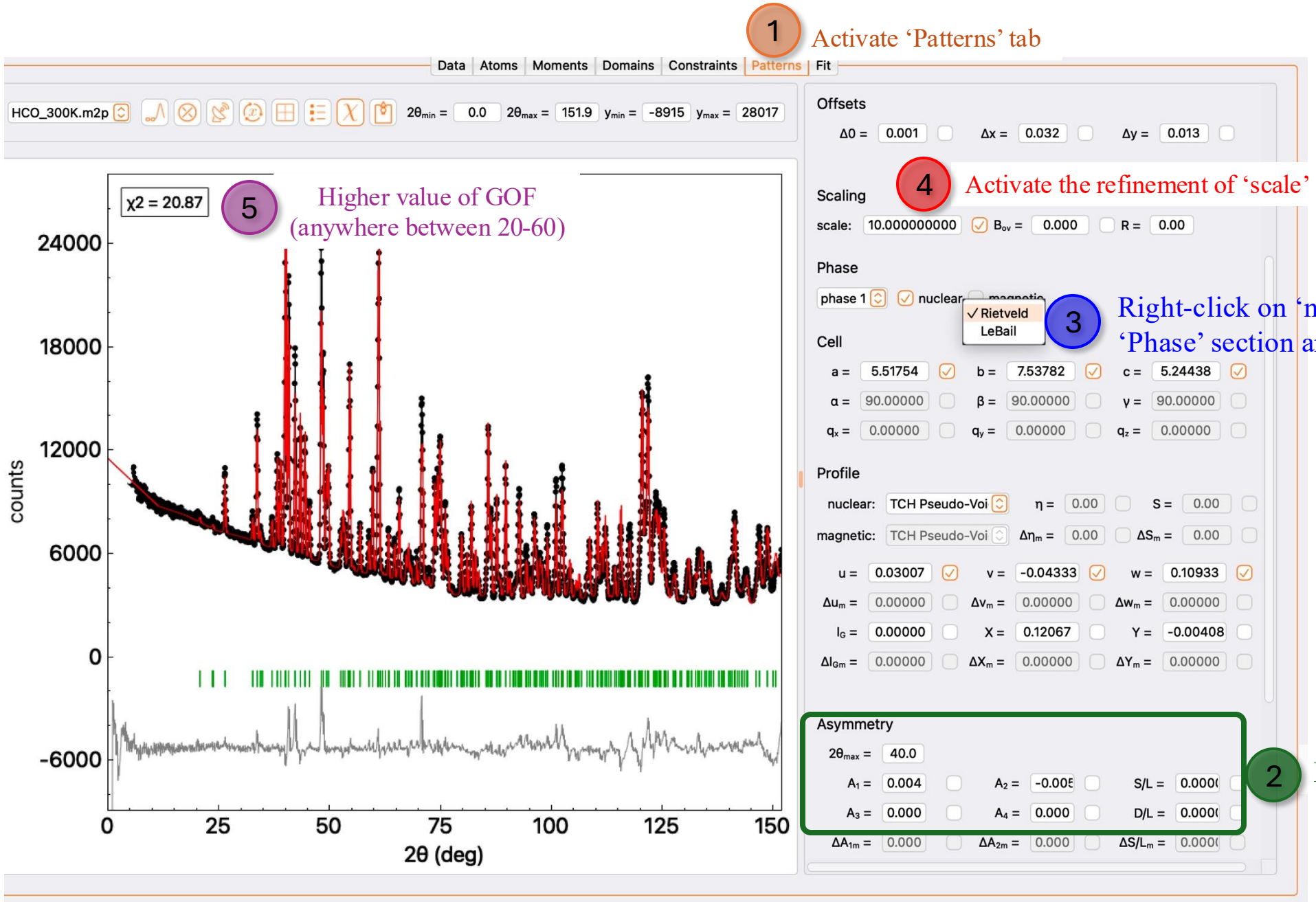
Box: a: -0.10 - 1.10 b: 0.00 - 1.00 c: -0.10 - 1.10 Domain: 1 Phase: 1

View along: custom Rotation axis: 0 0 1 direct Step (°): 5 Zoom: 0

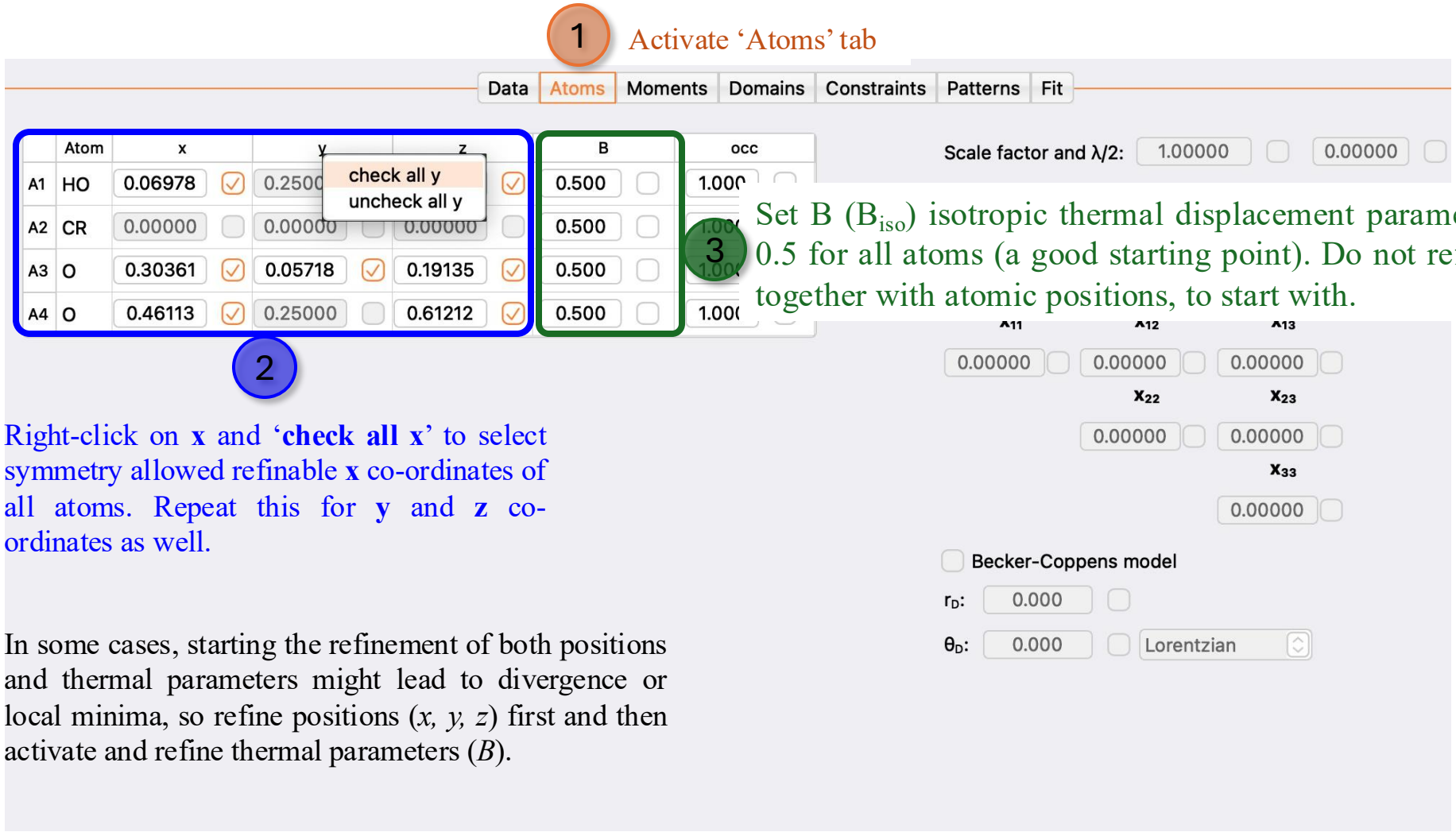


3 Go back to the ‘Fit’ window

Activate Rietveld refinement of lattice structure



Rietveld refinement of lattice structure



Set B (B_{iso}) isotropic thermal displacement parameter to be 0.5 for all atoms (a good starting point). Do not refine them together with atomic positions, to start with.

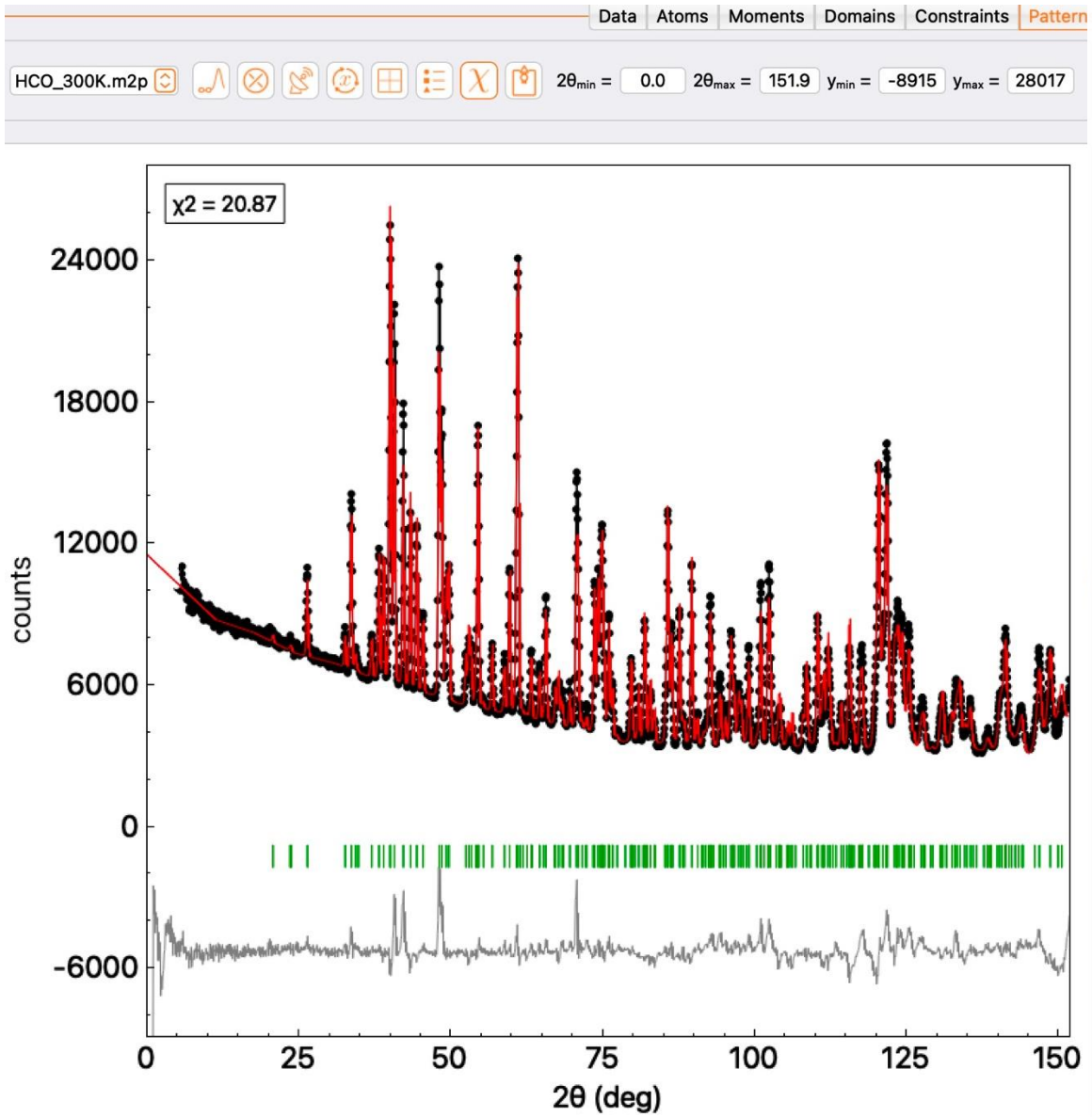
Right-click on **x** and '**check all x**' to select symmetry allowed refinable **x** co-ordinates of all atoms. Repeat this for **y** and **z** co-ordinates as well.

In some cases, starting the refinement of both positions and thermal parameters might lead to divergence or local minima, so refine positions (*x, y, z*) first and then activate and refine thermal parameters (*B*).

4

Go to the 'Fit' tab and run the refinement by left-clicking on the 'Fit' button. Then return to the 'Patterns' tab.

Calculated diffraction intensity: Rietveld fit



Least square minimization via:

$$\chi^2 = \sum_i \left[\frac{y_{\mathrm{obs},i} - y_{\mathrm{cal},i}}{\sigma_i} \right]^2$$

$$\chi^2_r = \chi^2/(m-n)$$

σ_i = statistical counting uncertainty of each observed point.
m = number of observations (data points),
n = number of refined parameters

Agreement is evaluated via:

$$R_F = 100 \cdot \frac{\sum_Q |F_{\mathrm{obs},Q} - F_{\mathrm{cal},Q}|}{\sum_Q F_{\mathrm{obs},Q}}$$

$$F_{\mathrm{obs},Q} = \sqrt{\frac{I_{\mathrm{obs},Q}}{m_Q L_Q}}$$

Calculated diffraction intensity Rietveld:

$$y_{\mathrm{cal}}(2\theta_i) = \sum_{Q=(hkl)} \underbrace{m_Q\, L_Q\, |F_{\mathrm{cal},Q}|^2}_{\text{structure-factor term}} \underbrace{\Phi\big(2\theta_i - 2\theta_Q - \Delta(2\theta)_{\mathrm{disp}}\big)}_{\text{profile function}} + \underbrace{b_i}_{\text{background}}$$

$Q = (hkl)$: reflection index
m_Q: multiplicity
L_Q: Lorentz factor
$|\mathbf{F\_cal,Q}|^2 = |\boldsymbol{N(Q)}|^2 + |\boldsymbol{M_\perp(Q)}|^2$ – from nuclear + magnetic structure factors
$\boldsymbol{\Phi(...)}$ – peak profile (U, V, W, X, Y, asymmetry, sample displacement, etc.
$\boldsymbol{b_i}$ – background intensity at point $\boldsymbol{i}$ (polynomial / point-interpolated / Debye-like), modeling all non-Bragg contributions.

Nuclear structure factor: $$N(Q) = \sum_j o_j b_j e^{-2\pi i(hx_j+ky_j+lz_j)} e^{-B_j \sin^2\theta/\lambda^2}$$

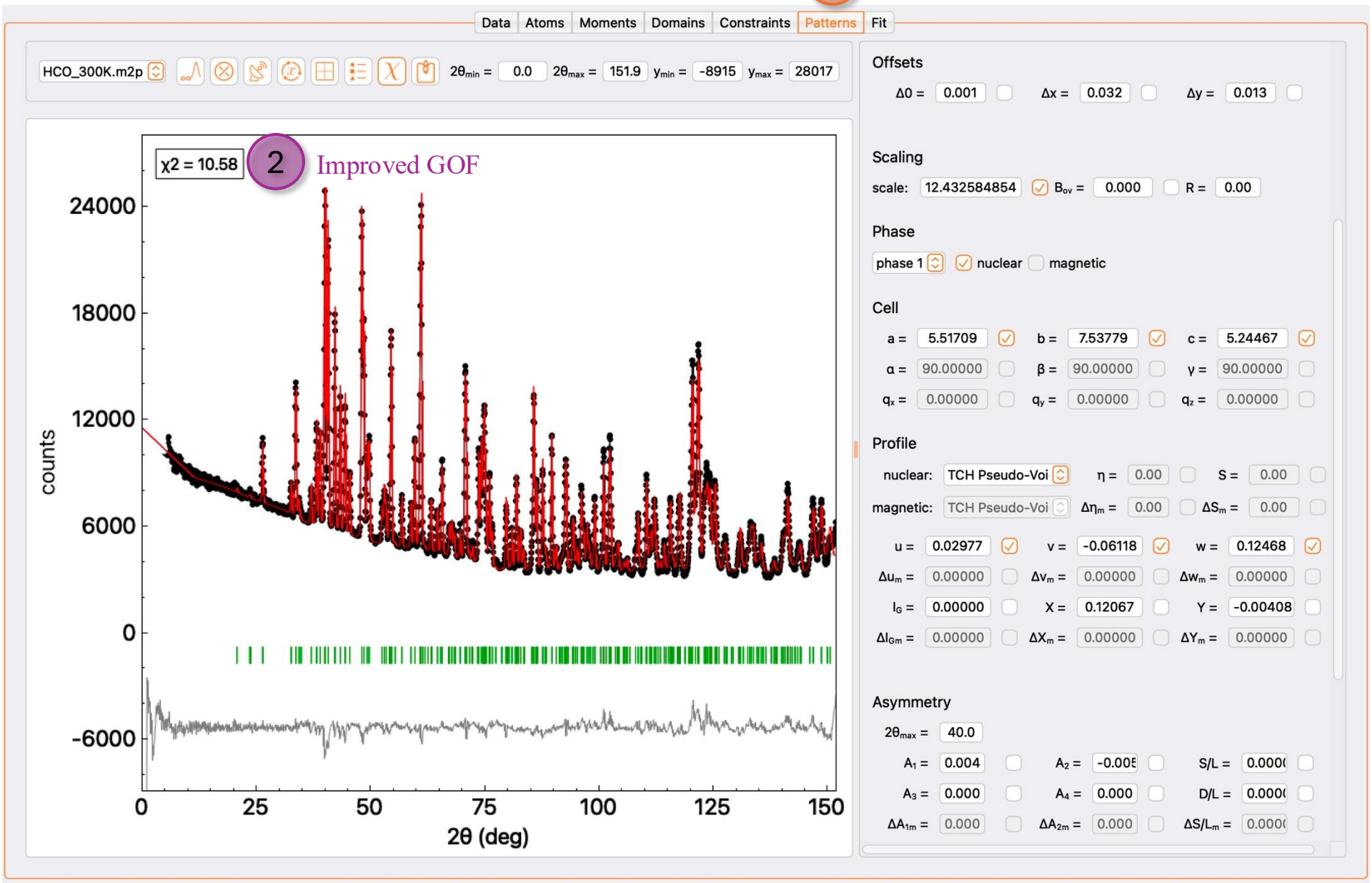
Magnetic structure factor: $$M(Q) = \sum_j p_n o_j S_j f_j(Q) e^{-2\pi i(hx_j+ky_j+lz_j)} e^{-B_j \sin^2\theta/\lambda^2}$$

$$|M_\perp(Q)|^2 = |M(Q)|^2 - |M(Q)\cdot\hat{Q}|^2$$

Only the perpendicular component scatters

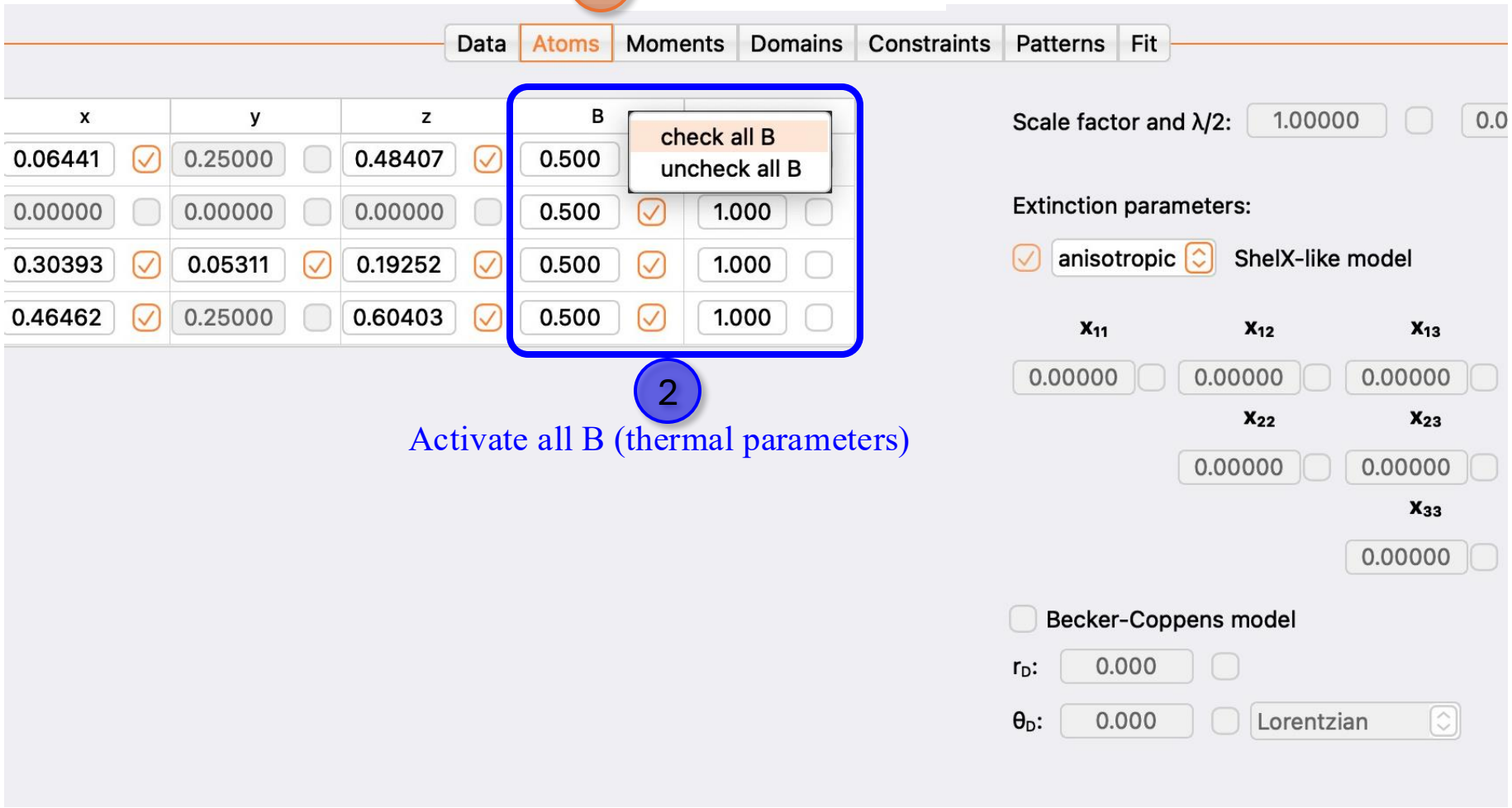
Activate Rietveld refinement of lattice structure

1 Activate ‘Patterns’ tab



Rietveld refinement of lattice structure

1 Activate ‘Atoms’ tab

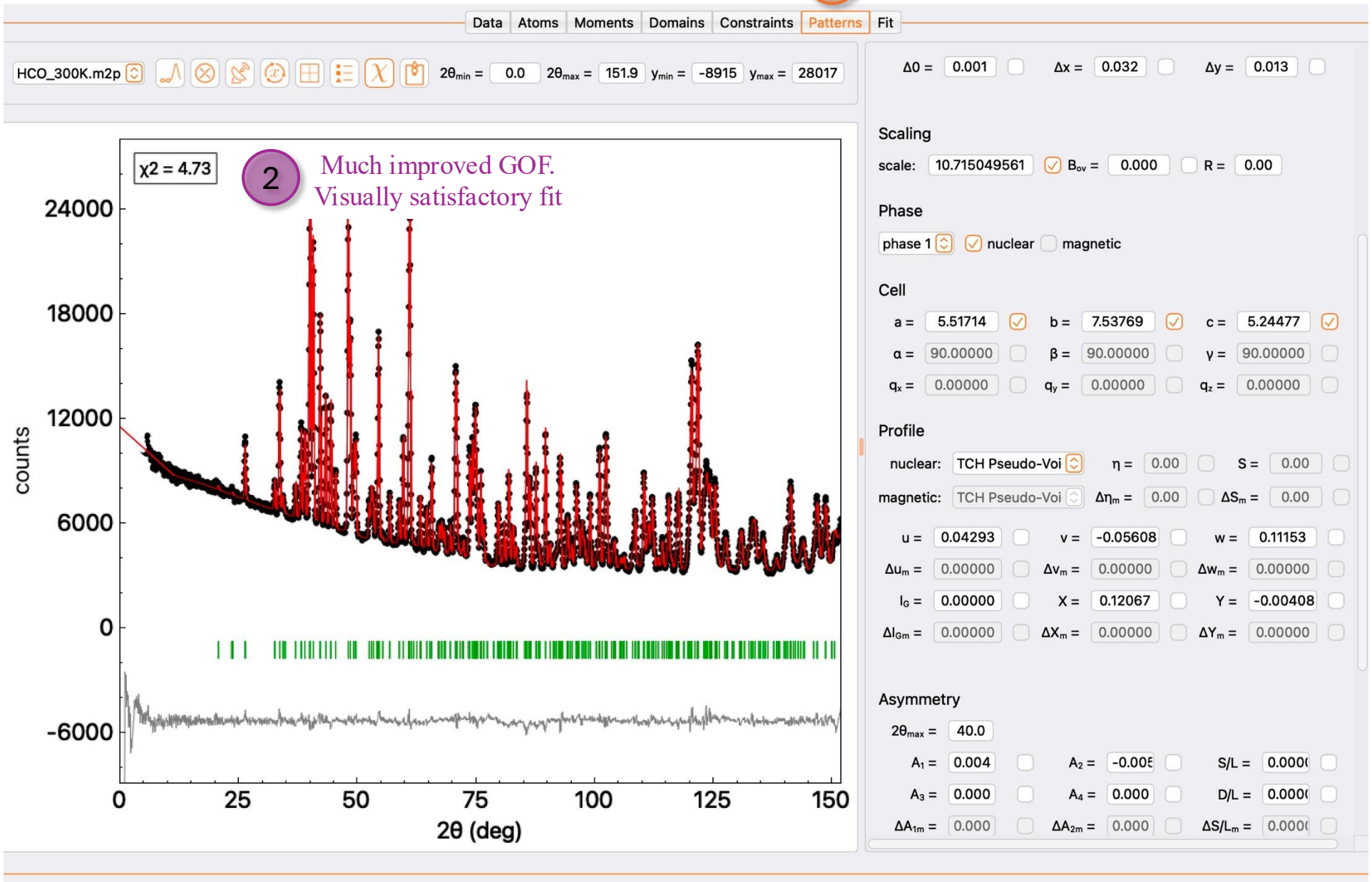


2 Activate all B (thermal parameters)

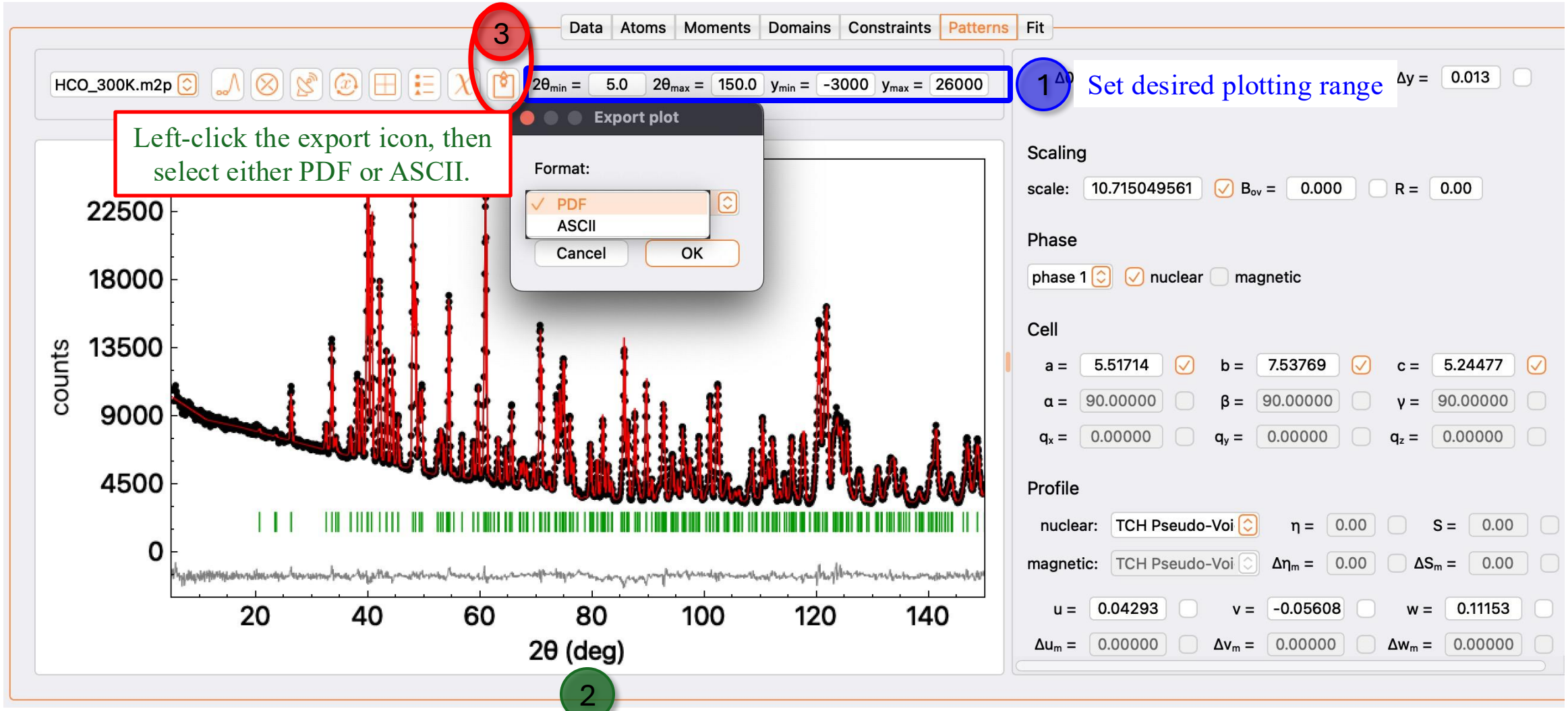
3 Go to the ‘Fit’ tab and run the refinement by left-clicking on the ‘Fit’ button. Then return to the ‘Patterns’ tab.

Rietveld refinement of lattice structure

1 Activate ‘Patterns’ tab



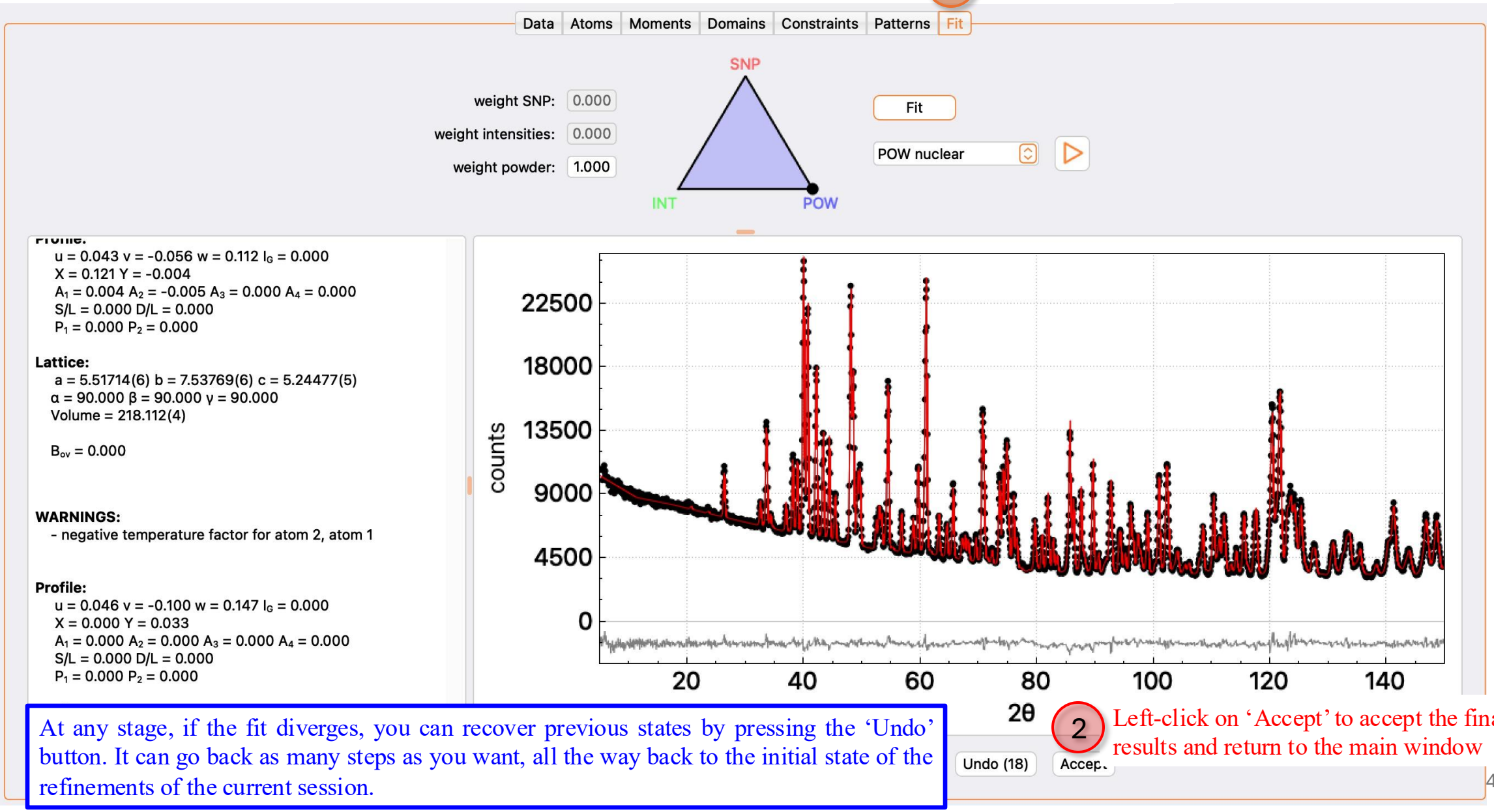
Rietveld refinement of lattice structure



Rearrange the plot window to a desired-looking aspect ratio and font size

Accept the final Rietveld refinement results

1 Activate ‘Fit’ tab

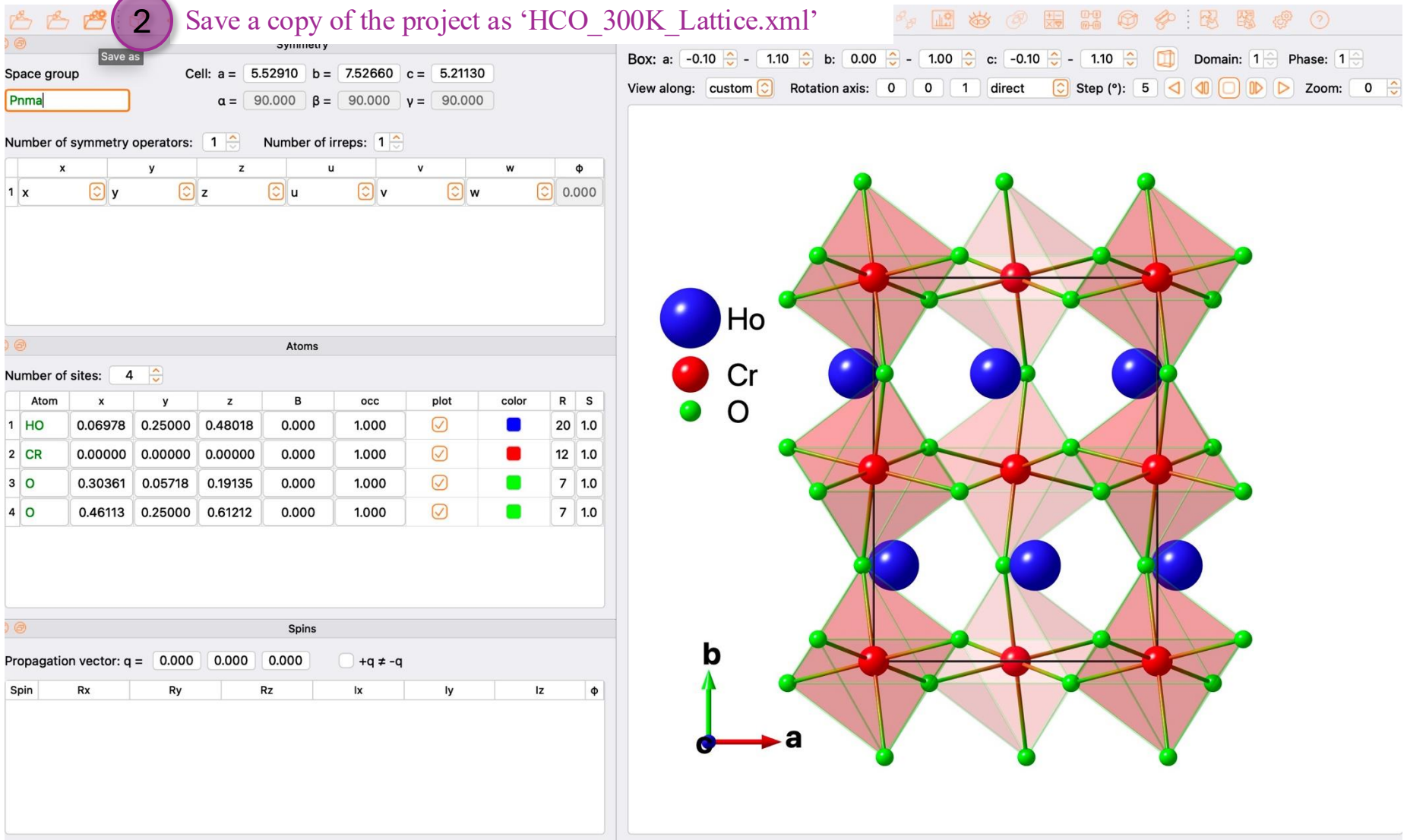


2 Left-click on ‘Accept’ to accept the final refinement results and return to the main window

At any stage, if the fit diverges, you can recover previous states by pressing the ‘Undo’ button. It can go back as many steps as you want, all the way back to the initial state of the refinements of the current session.

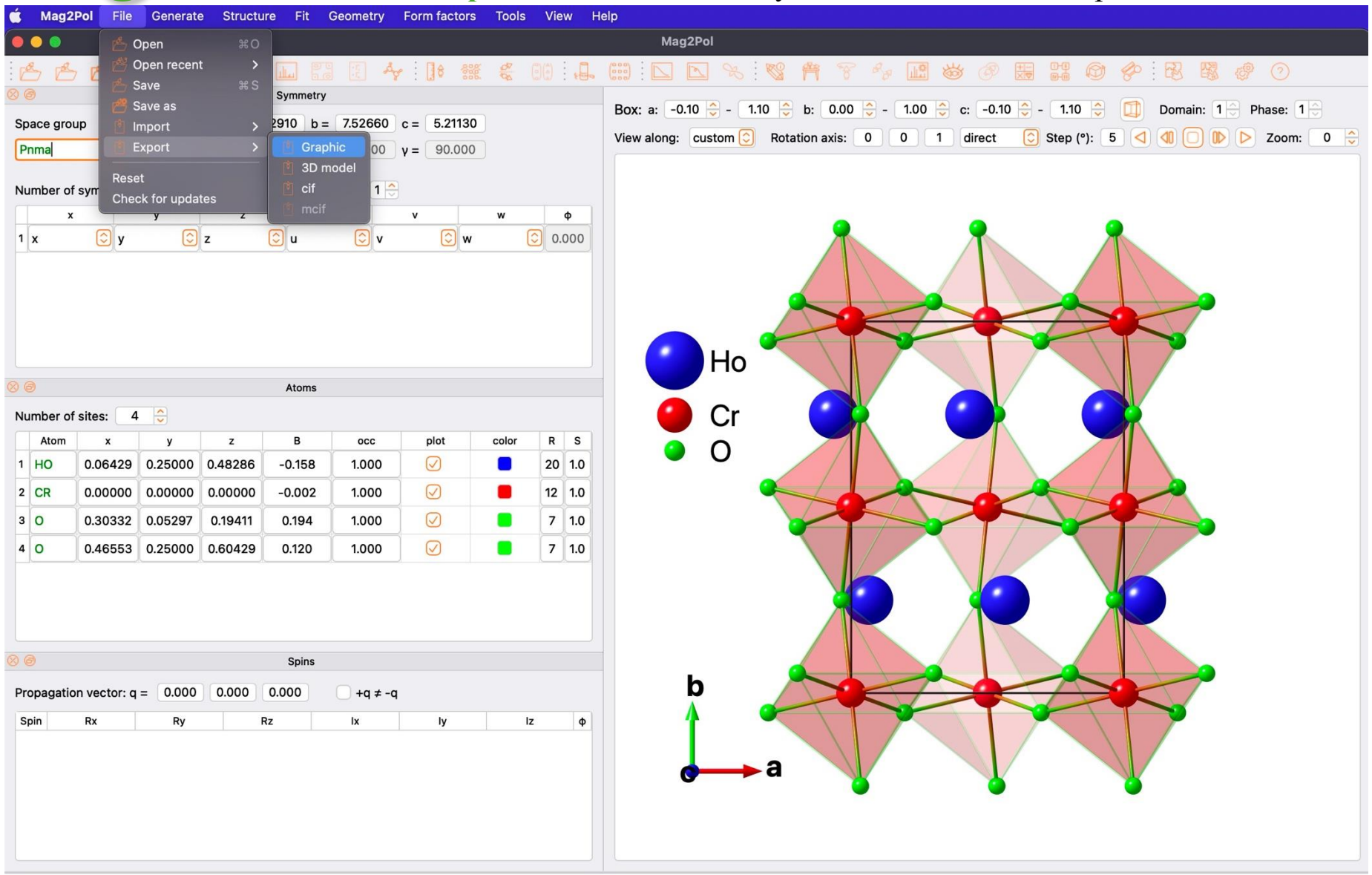
Save a copy of the project file

1. Go to the main Mag2Pol window
2. Save a copy of the project as ‘HCO_300K_Lattice.xml’



Export the structure graphic and cif

1. Use the menu **File→Export→ Graphic** to save the crystal structure exactly as seen on the main window.
Use the menu **File→Export→ cif** to save the crystal information file for publication of or to be plotted in external programs.



Refining low temperature NPD data, collected in the magnetically ordered phase

Save another copy of the project file

We will use the 300 K refinement project as the starting point for the refinement of 4 K data

1 Save a copy of the project as ‘HCO_4K_Lattice.xml’

1 Go to the ‘Fit’ window

Symmetry

Space group: Pnma

Number of symmetry operators: 1 Number of irreps: 1

	x	y	z	u	v	w	ϕ
1	x	y	z	u	v	w	0.000

Atoms

Number of sites: 4

	Atom	x	y	z	B	occ	plot	color	R	S
1	HO	0.06978	0.25000	0.48018	0.000	1.000	☑		20	1.0
2	CR	0.00000	0.00000	0.00000	0.000	1.000	☑		12	1.0
3	O	0.30361	0.05718	0.19135	0.000	1.000	☑		7	1.0
4	O	0.46113	0.25000	0.61212	0.000	1.000	☑		7	1.0

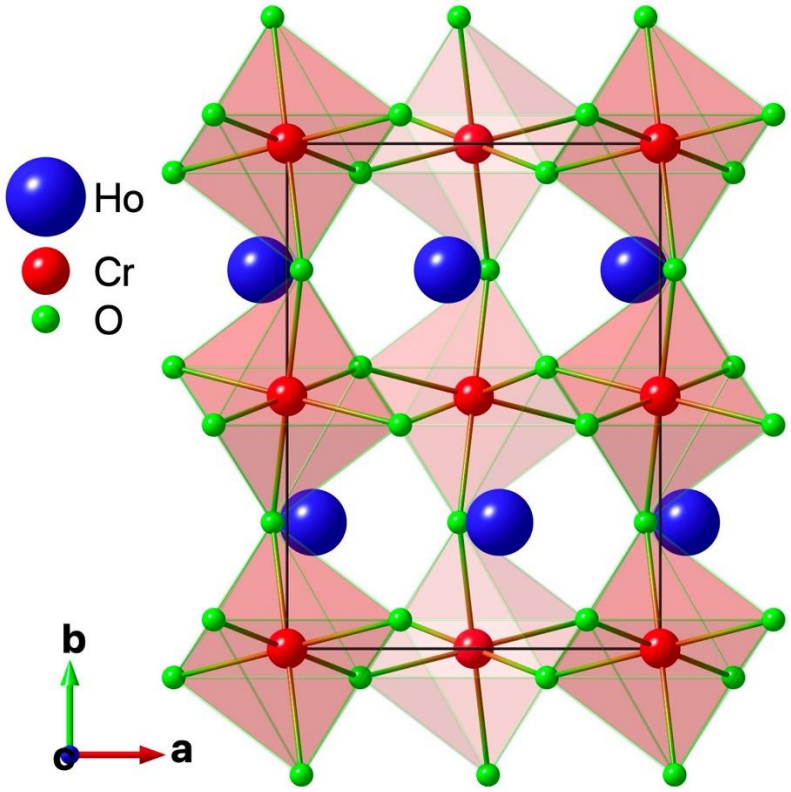
Spins

Propagation vector: q = 0.000 0.000 0.000 +q ≠ -q

Spin	Rx	Ry	Rz	Ix	Iy	Iz	ϕ

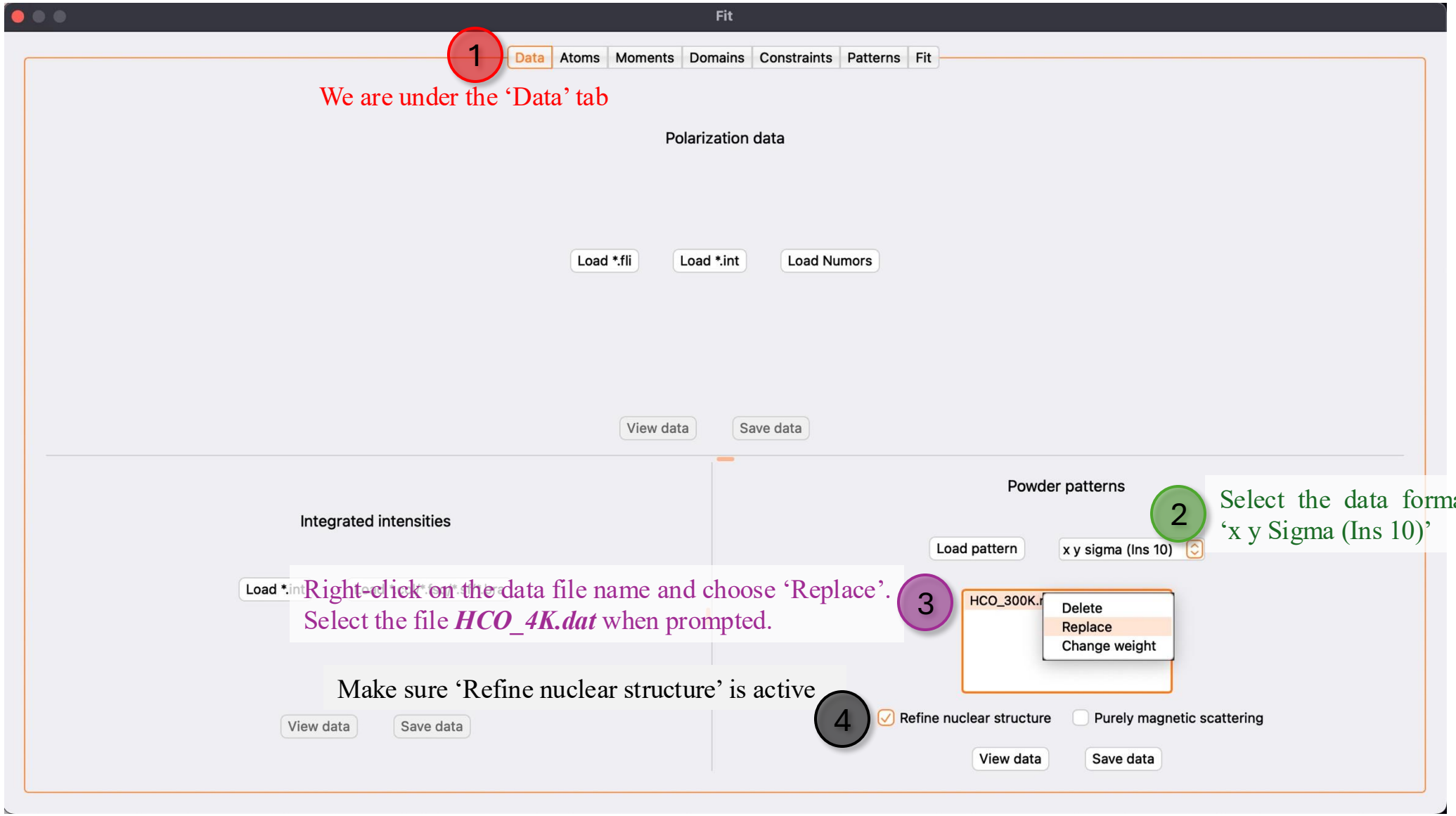
Box: a: -0.10 1.10 b: 0.00 1.00 c: -0.10 1.10 Domain: 1 Phase: 1

dis: 0 0 1 direct Step (°): 5 Zoom: 0

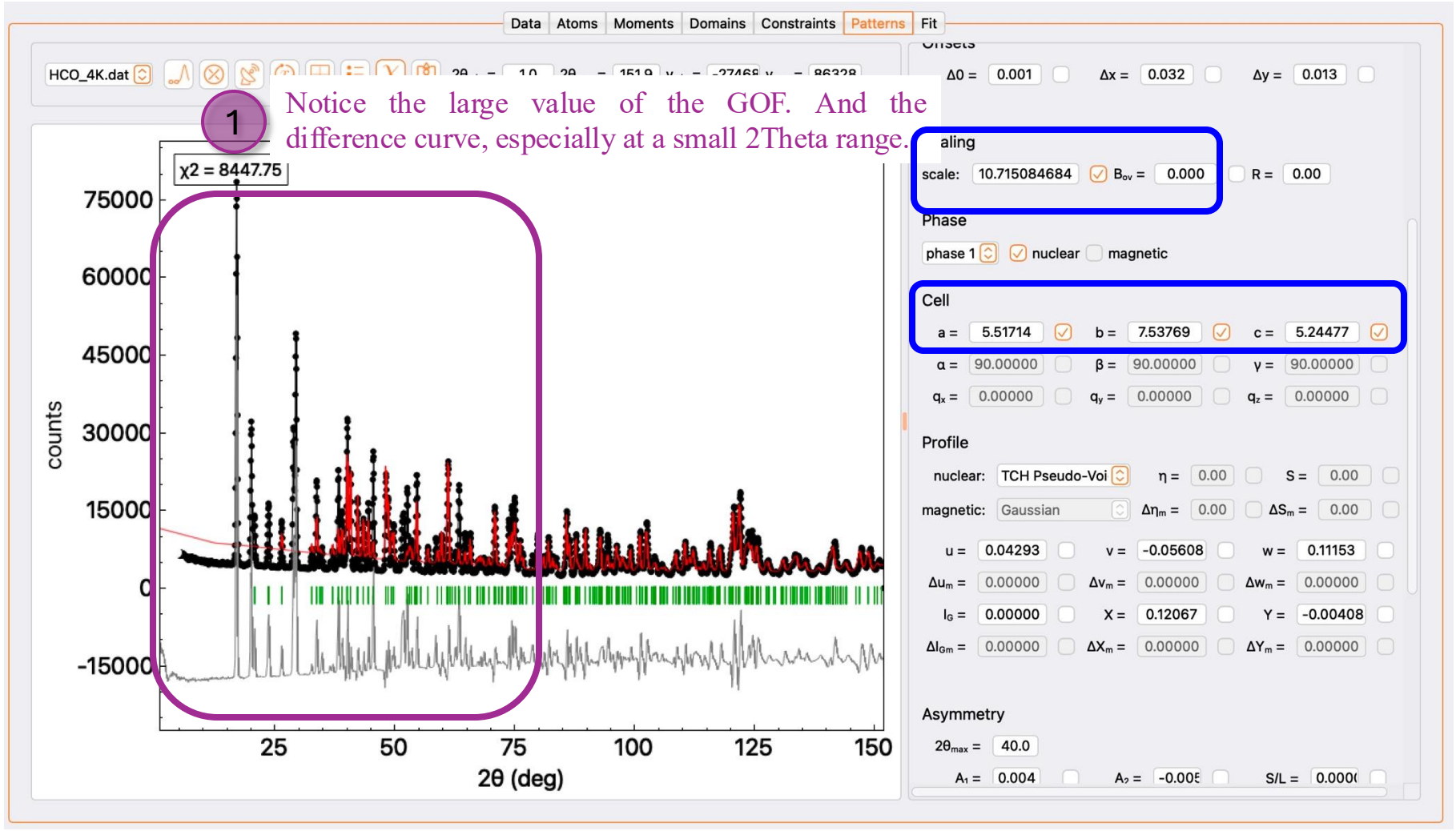


Load the experimental data collected at 300 K

The data can be loaded/replaced under the 'Data' tab of the 'Fit' window. Mag2Pol can accept three kinds of data sets. In this example, we will start with 'Powder diffraction data' of $CrSbSe_3$ collected at 300 K (Paramagnetic phase). **Follow the order indicated below.**



Rietveld fit of Lattice only contribution to 4 K data



2

Refine

Scale and **lattice** parameters (a, *b*, *c*)

4

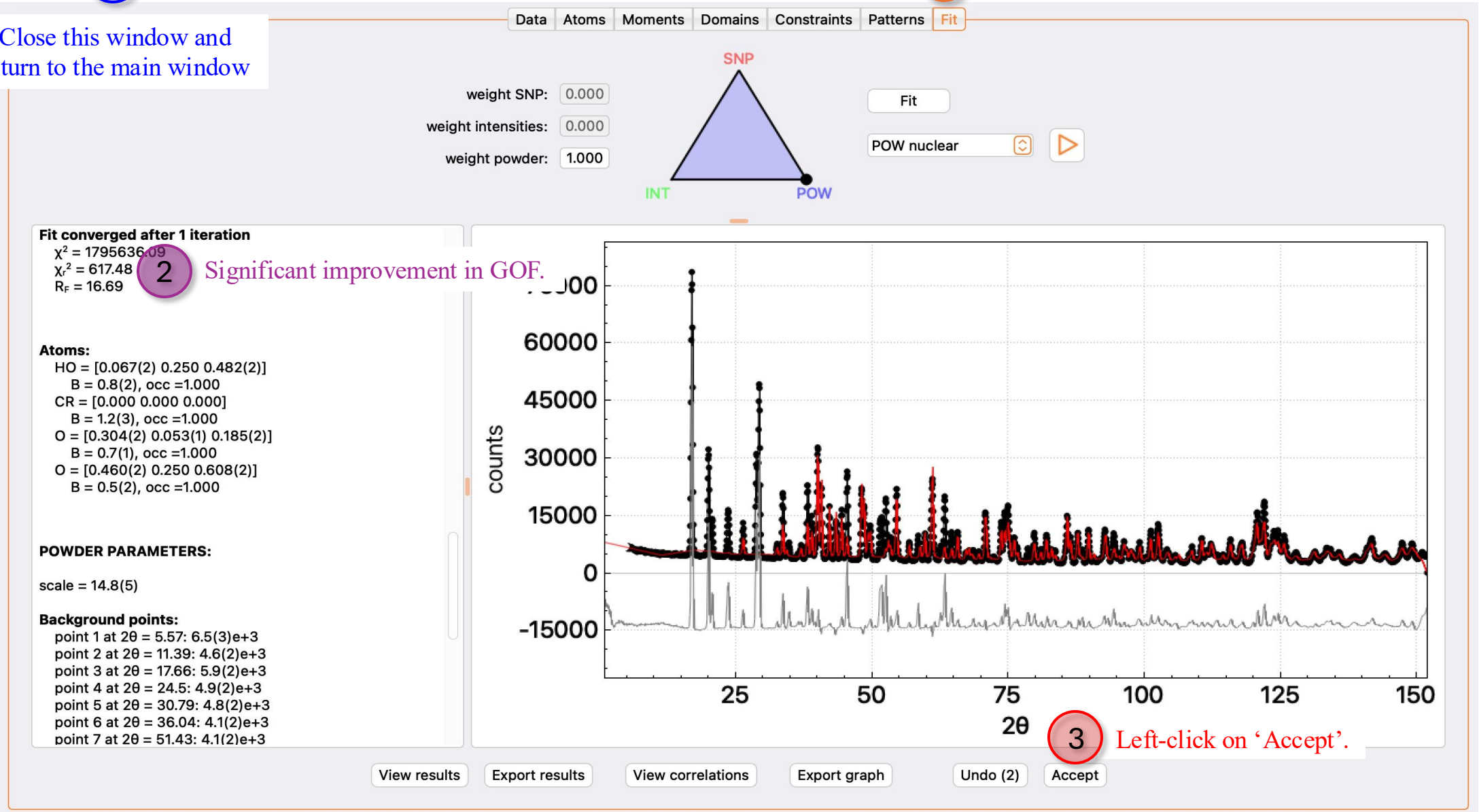
a, b, c: Lattice parameters

Accept the Rietveld refinement results

4

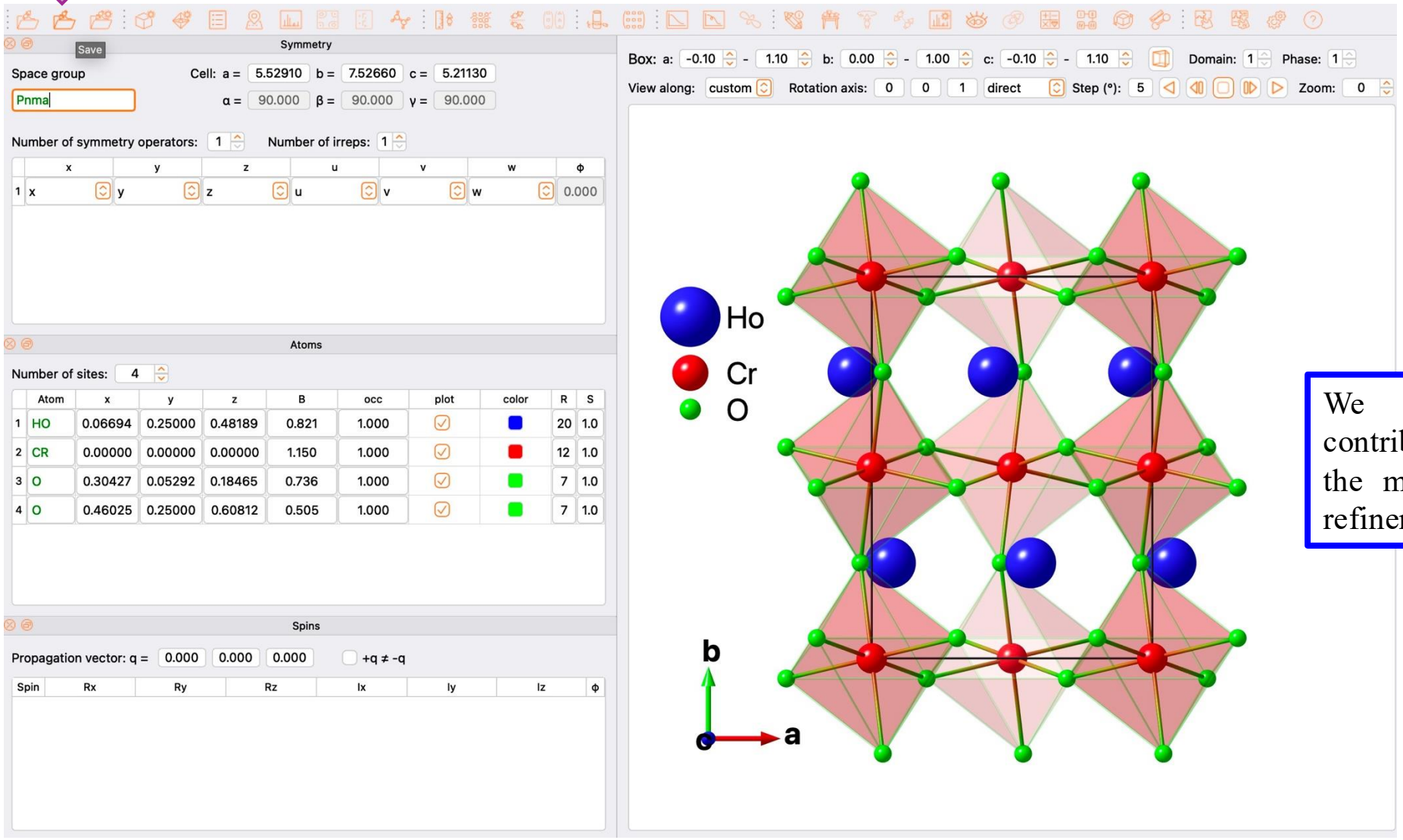
Close this window and return to the main window

1 Activate ‘Fit’ tab



Save Rietveld fit of the Lattice only contribution to 4 K data

1 Left-click on the save icon



We have refined only the lattice contribution so far, and now we can define the magnetic ion and proceed with the refinement of the magnetic structure.

Session 3

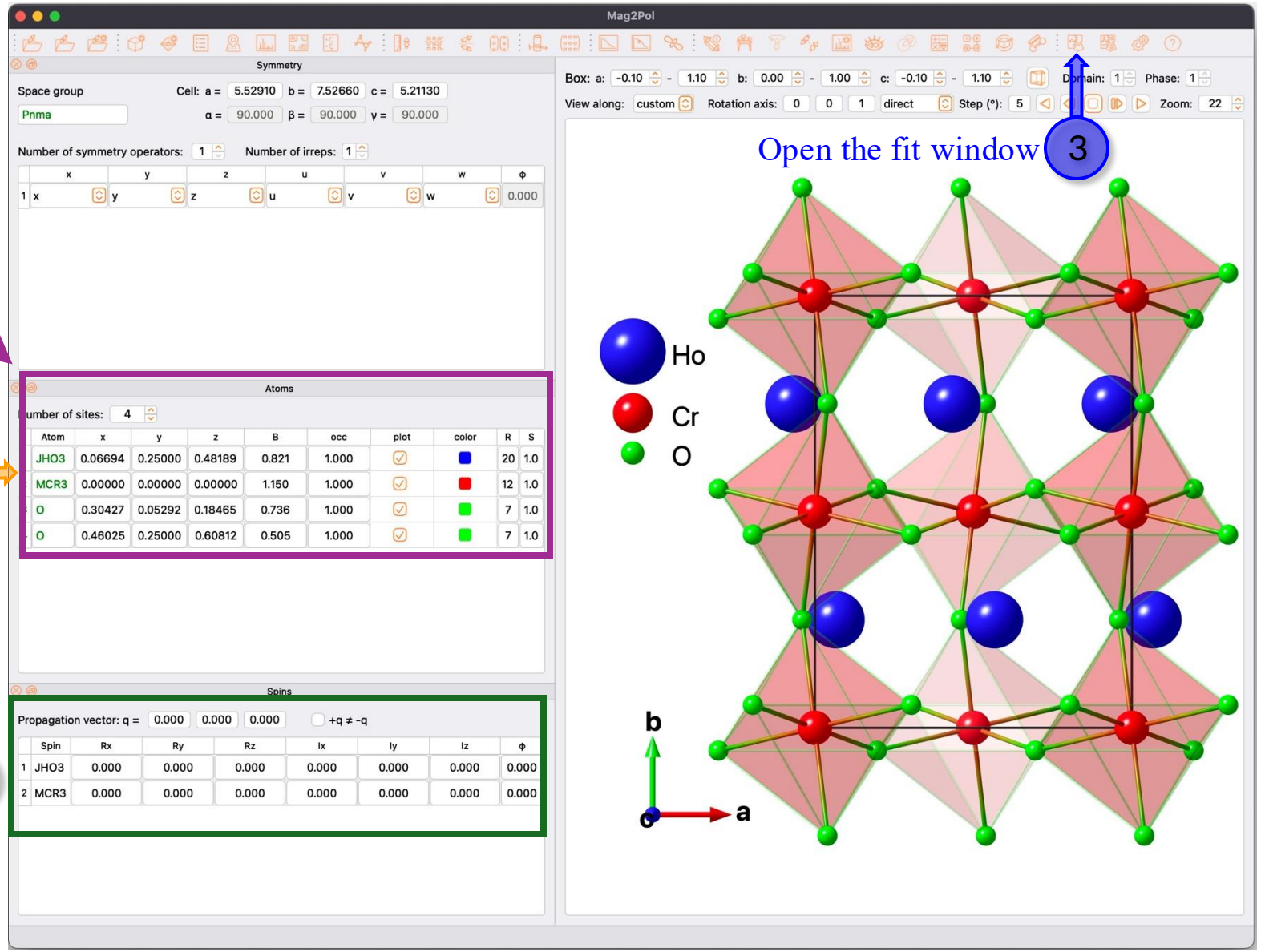
Refinement of magnetic structure

Activate (define) magnetic ions

Ion	Electronic configuration	S	L	J	Notes
Cr^{3+}	$3d^3$ (t_2g^3, ^4A_2g)	3/2	≈ 0	≈ 3/2	3d ion; orbital moment quenched: → spin-only **M**
Ho^{3+}	$4f^{10}$ ($^5I_8$ term)	2	6	8	4f ion; strong spin–orbit: → total J form factor

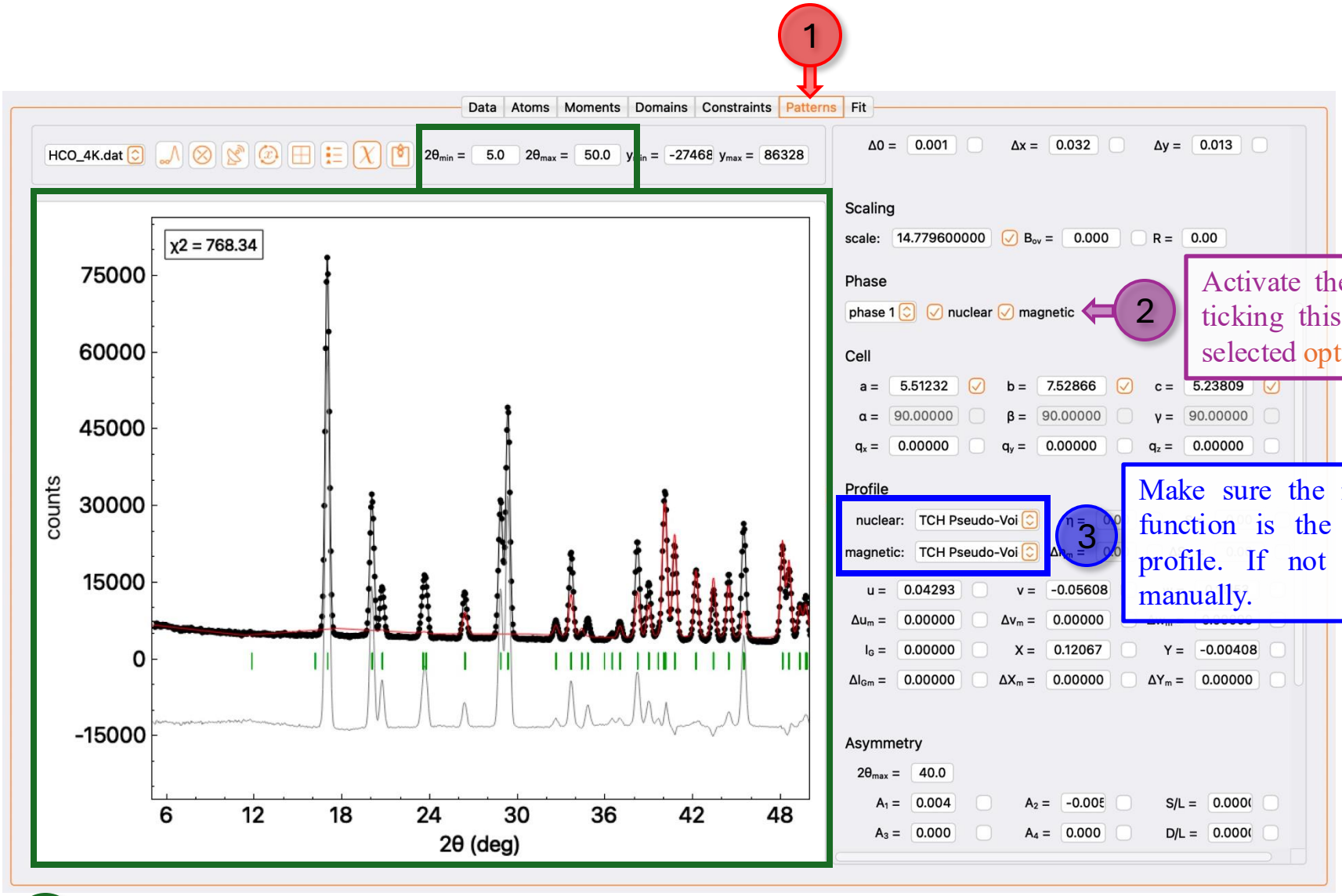
Under the 'Atoms' section:
Depending on the type of magnetic contribution, simply add the prefix **M** or **J** to the magnetic ion, followed by the ionic state. In this case, only Cr^{3+} and Ho^{3+} are magnetic.
After this, update the structure by the keyboard shortcut [Ctrl+U] (on Windows) or [Cmd+U] (on Mac)

As soon as a Magnetic atom is defined under the 'Atoms' section, the 'Spins' section will be populated automatically with the Magnetic ions. By default, the Propagation vector is set to (0, 0, 0). Leave everything default for now. We will determine magnetic propagation vectors soon.



Fix all the atomic parameters

Determination of propagation vector

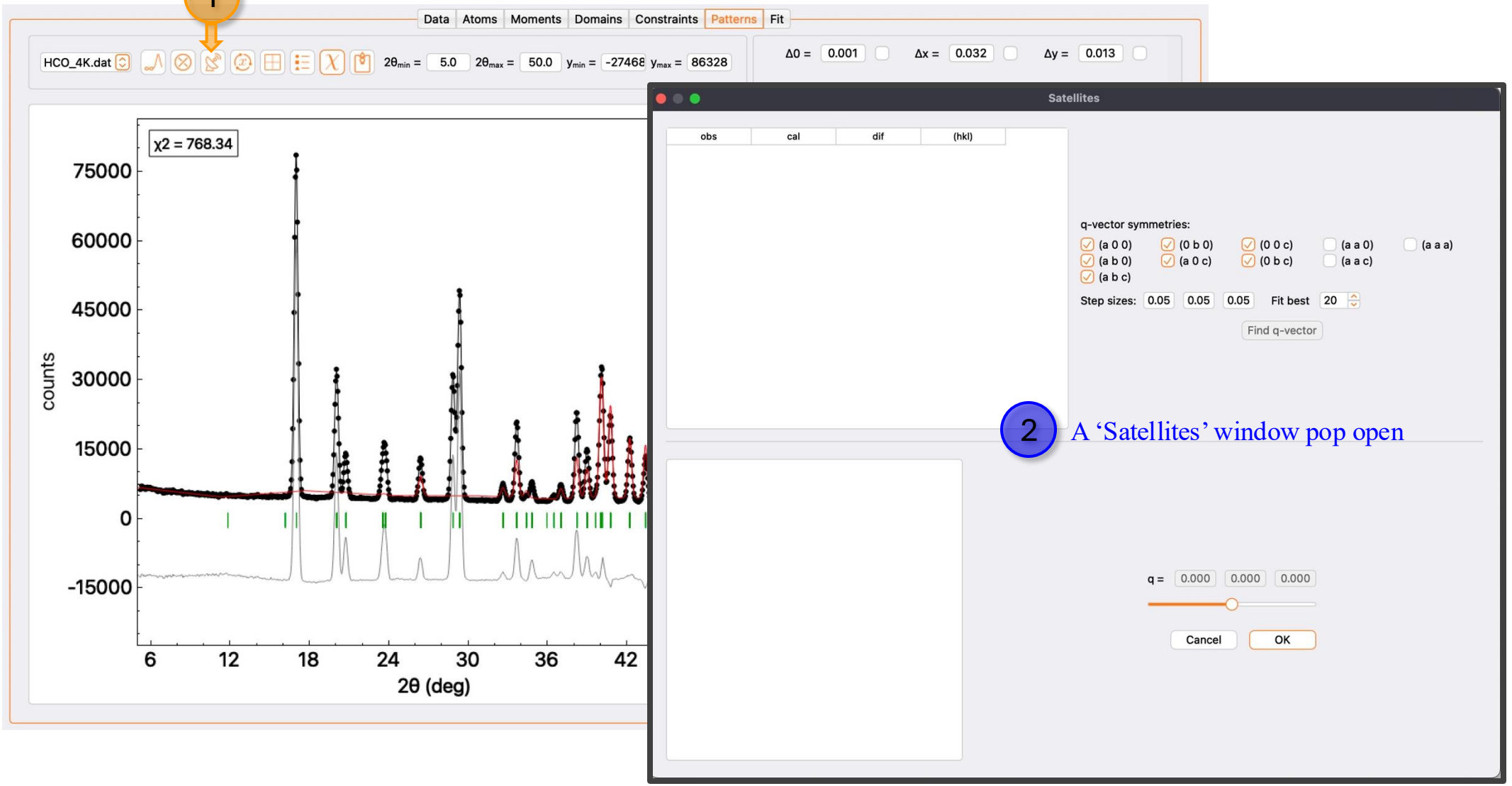


4 Zoom into the section of the diffraction pattern where you see magnetic peaks (at higher *d*-range)

Determination of propagation vector

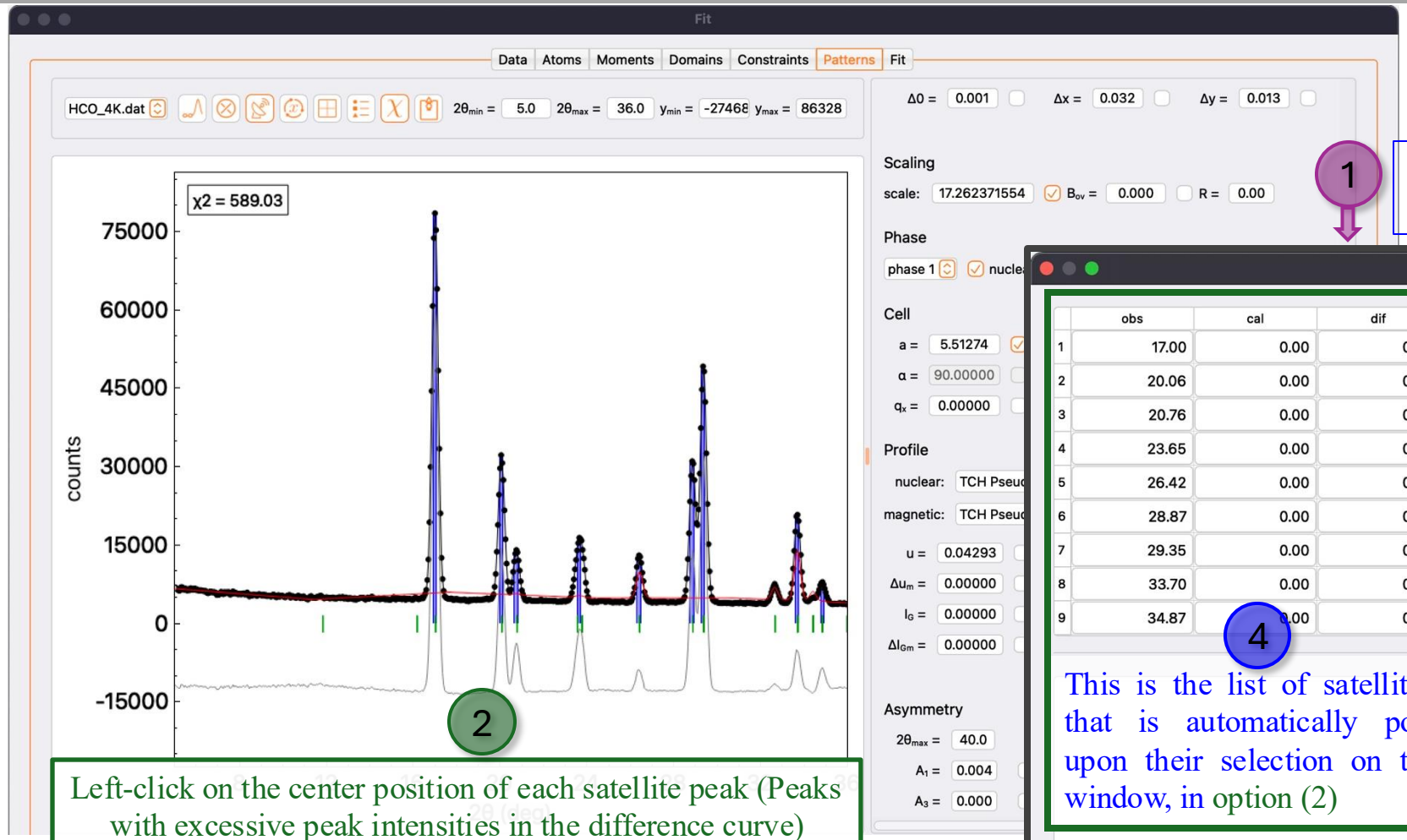
Click on the ‘satellites’ tab which will open a new window

1



2 A ‘Satellites’ window pop open

Select satellite (magnetic) peaks and q-vector type



1

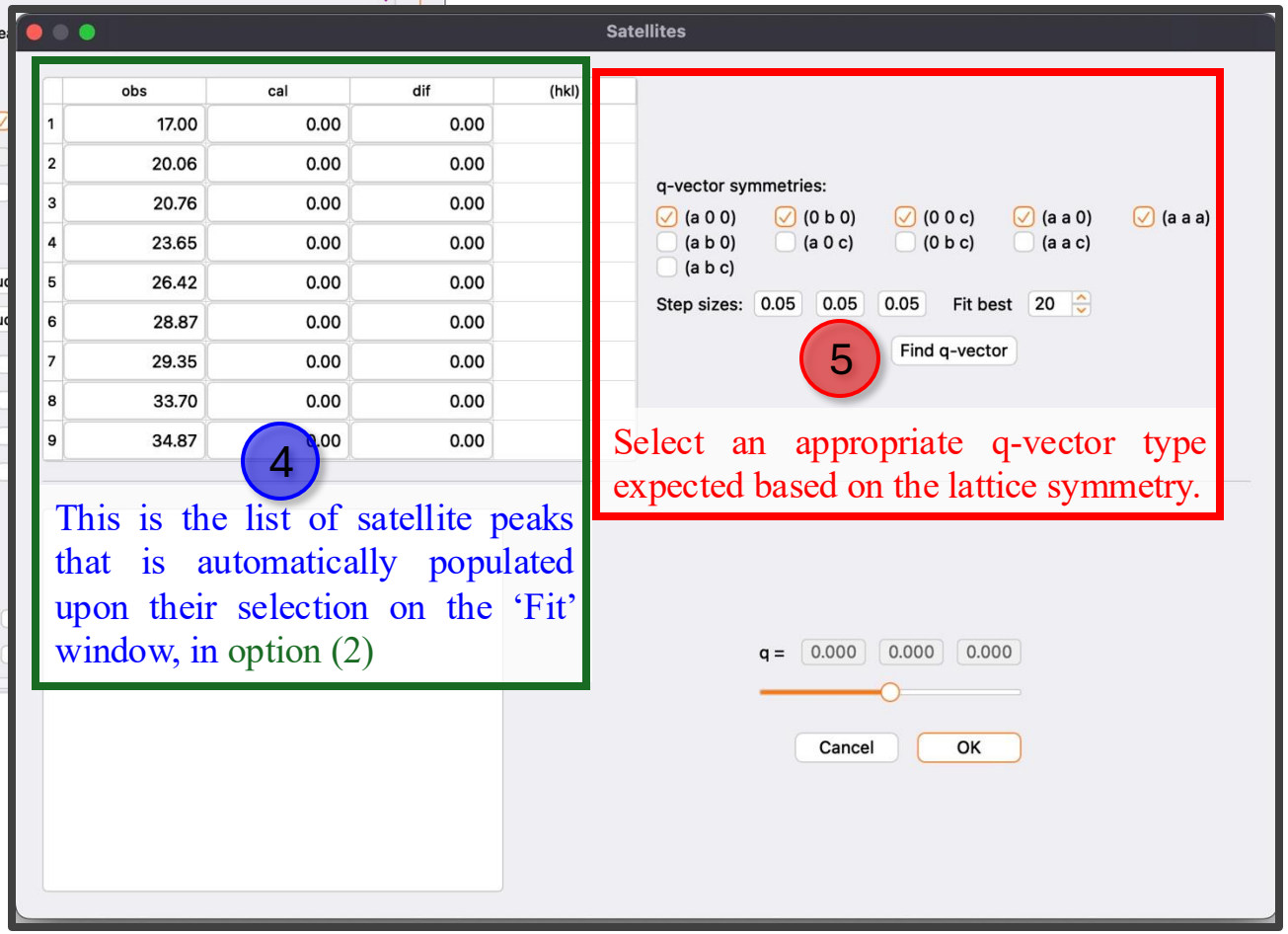
Move this 'Satellites' window away from the 'Fit' window so that the diffraction profile is accessible.

2

Left-click on the center position of each satellite peak (Peaks with excessive peak intensities in the difference curve)

3

Each selected satellite peak will have a blue vertical column. The position of the blue vertical column can be altered using a mouse (left-click + hold + move) and can be removed by right-clicking on it



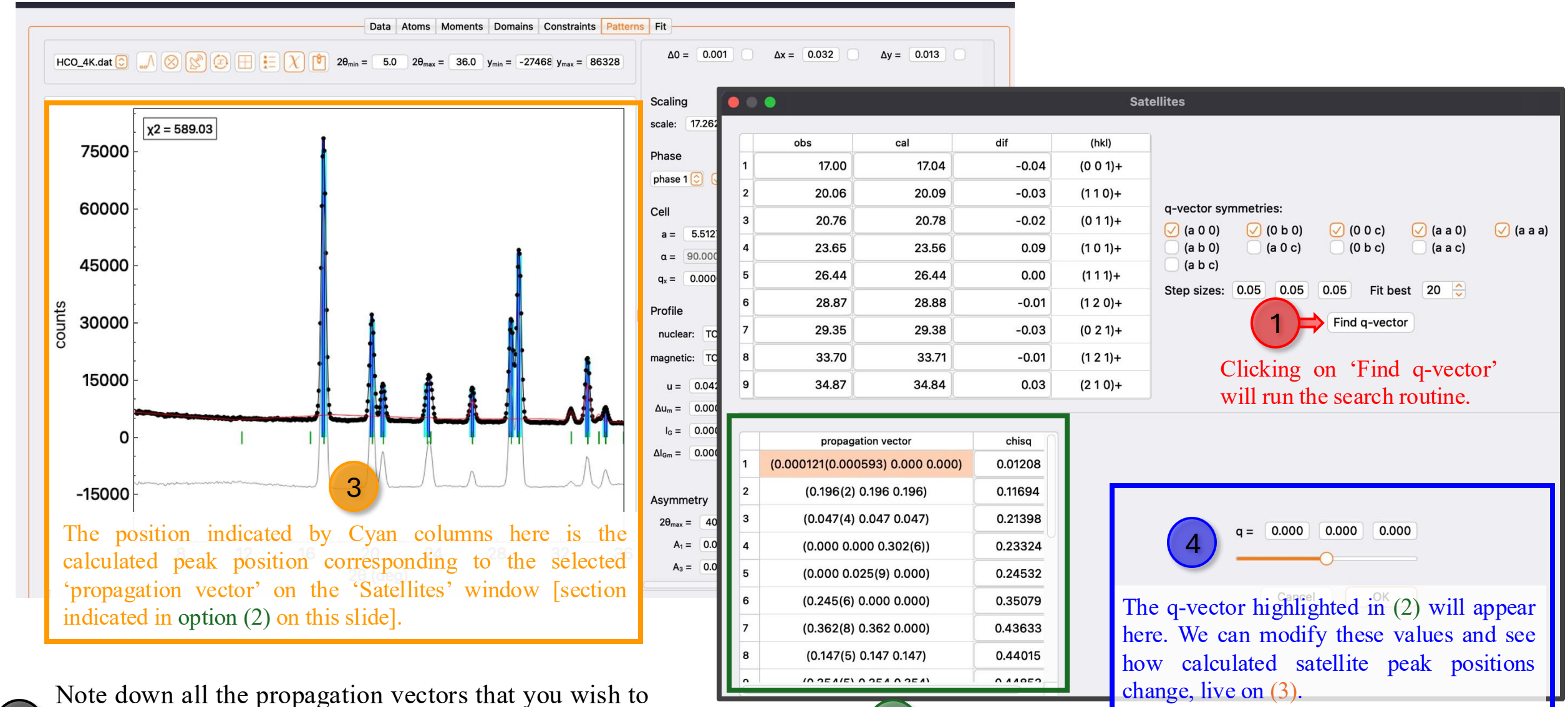
4

This is the list of satellite peaks that is automatically populated upon their selection on the 'Fit' window, in option (2)

5

Select an appropriate q-vector type expected based on the lattice symmetry.

Select satellite (magnetic) peaks



The position indicated by Cyan columns here is the calculated peak position corresponding to the selected 'propagation vector' on the 'Satellites' window [section indicated in option (2) on this slide].

Clicking on 'Find q-vector' will run the search routine.

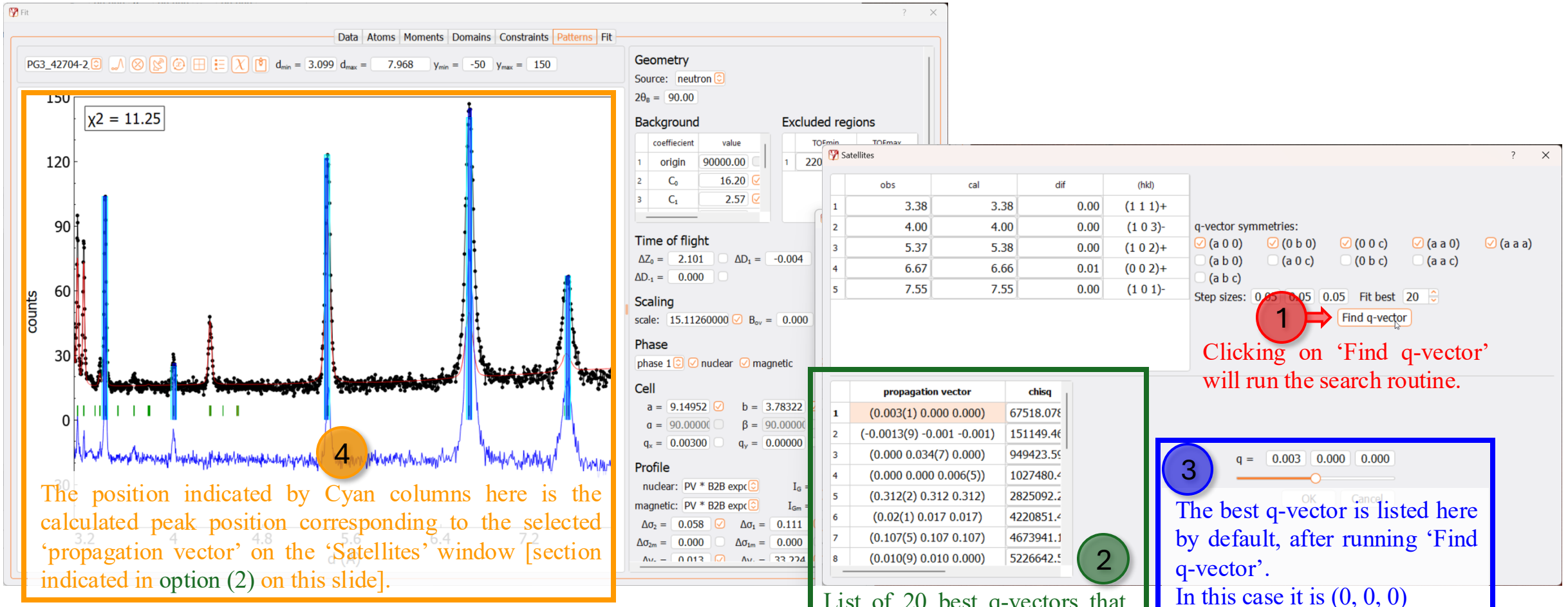
The q-vector highlighted in (2) will appear here. We can modify these values and see how calculated satellite peak positions change, live on (3).

In this case, q-vector is (0, 0, 0)

List of up to 20 best q-vectors that fit the selected satellites, populated automatically after running 'Find q-vector'.

5 Note down all the propagation vectors that you wish to try out (typically is the case for incommensurate structures)

Step 5c: Run the q-vector search, and note down the best q-vectors



The position indicated by Cyan columns here is the calculated peak position corresponding to the selected 'propagation vector' on the 'Satellites' window [section indicated in option (2) on this slide].

List of 20 best q-vectors that fit the selected satellites best, Populated automatically after running 'Find q-vector'.

Clicking on 'Find q-vector' will run the search routine.

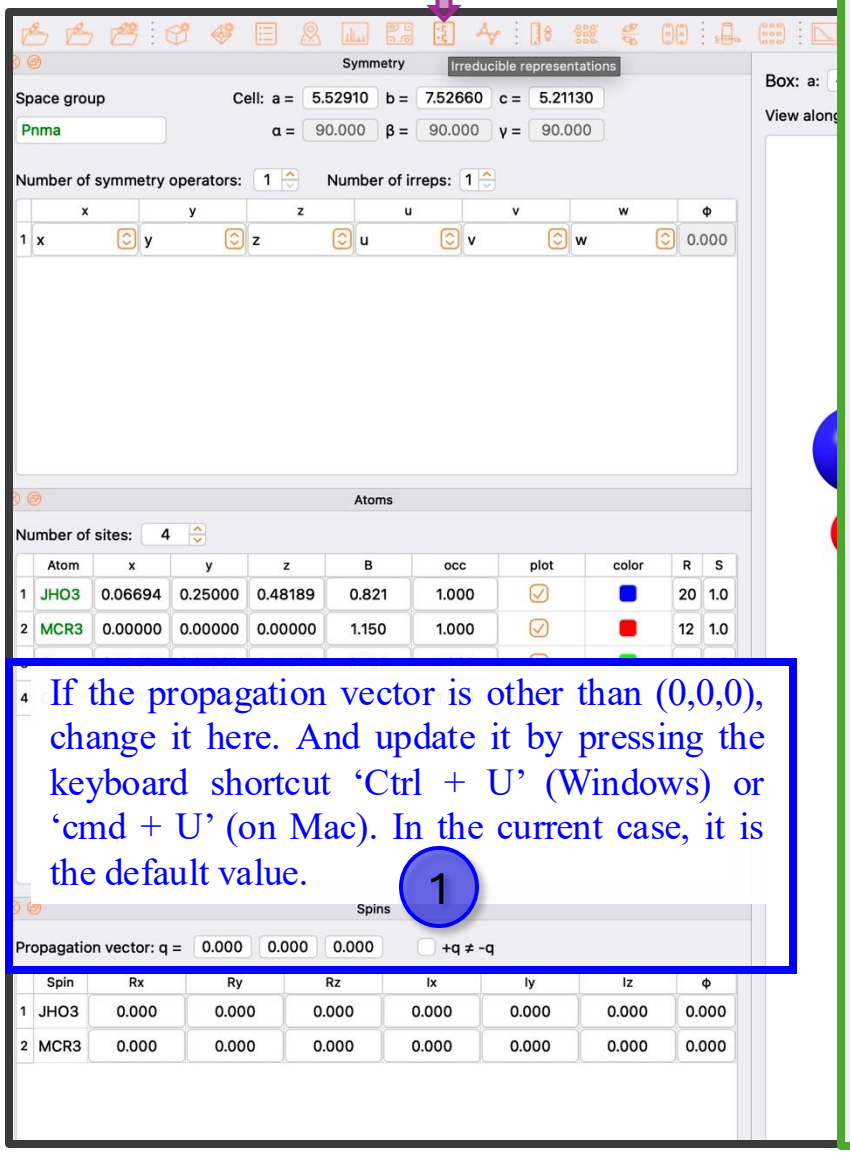
The best q-vector is listed here by default, after running 'Find q-vector'.
In this case it is (0, 0, 0)

5 Note down all the propagation vectors that you wish to try out. Close both the 'Satellites' and 'Fit' windows. And go back to the main window of Mag2Pol.

Refinement of magnetic structure using irreducible representations

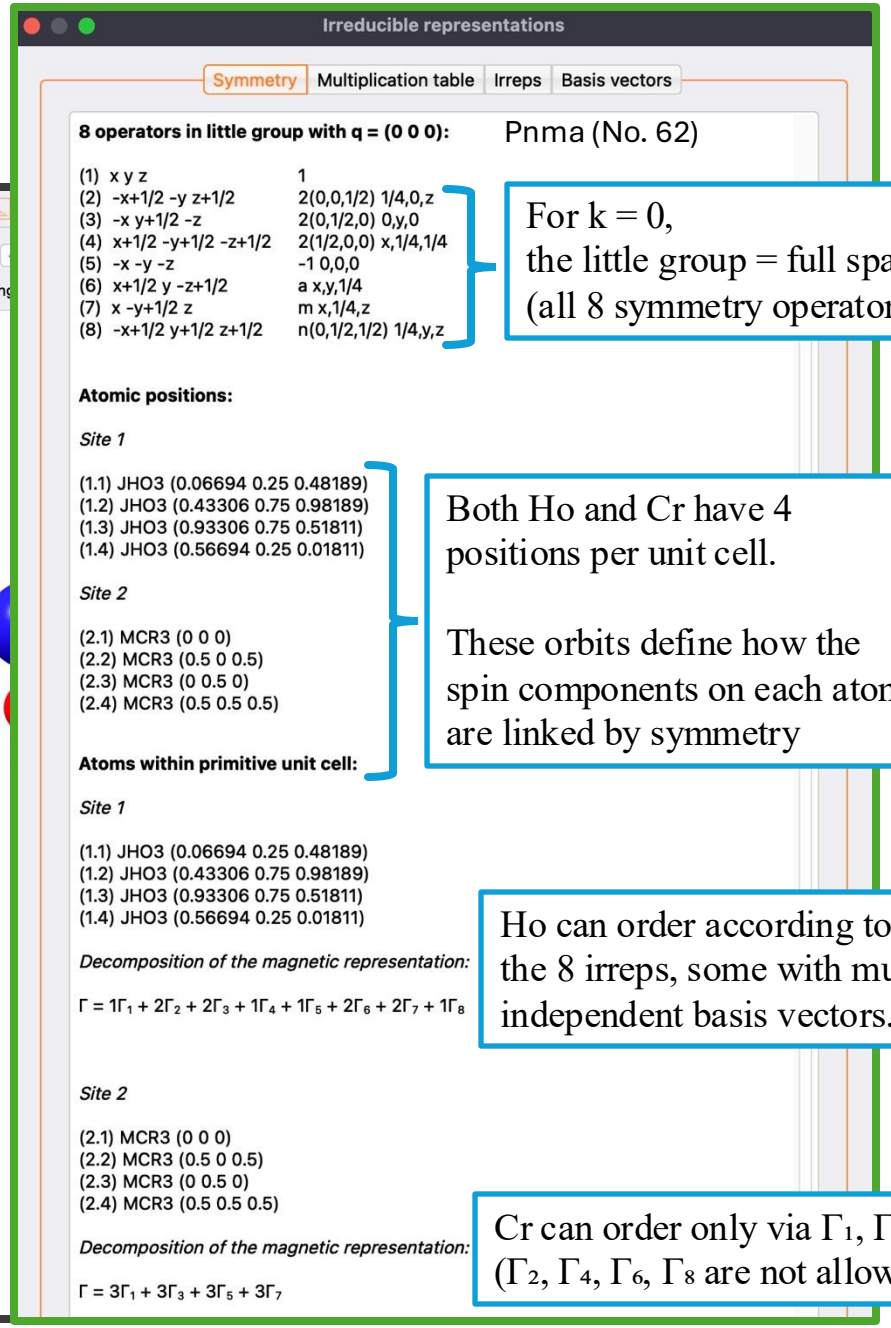
Left-click on ‘irreducible representations’

2



If the propagation vector is other than (0,0,0), change it here. And update it by pressing the keyboard shortcut ‘Ctrl + U’ (Windows) or ‘cmd + U’ (on Mac). In the current case, it is the default value.

1



For k = 0,
the little group = full space group
(all 8 symmetry operators)

Both Ho and Cr have 4 positions per unit cell.

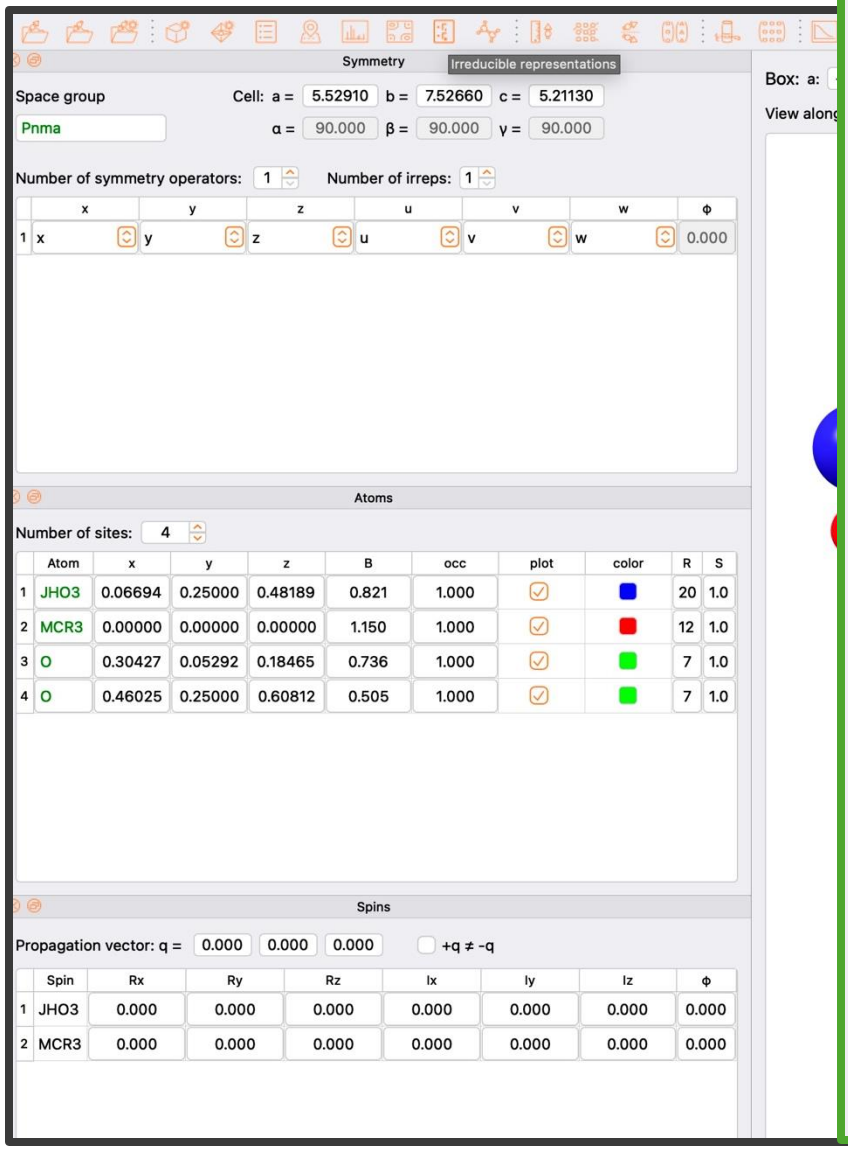
These orbits define how the spin components on each atom are linked by symmetry

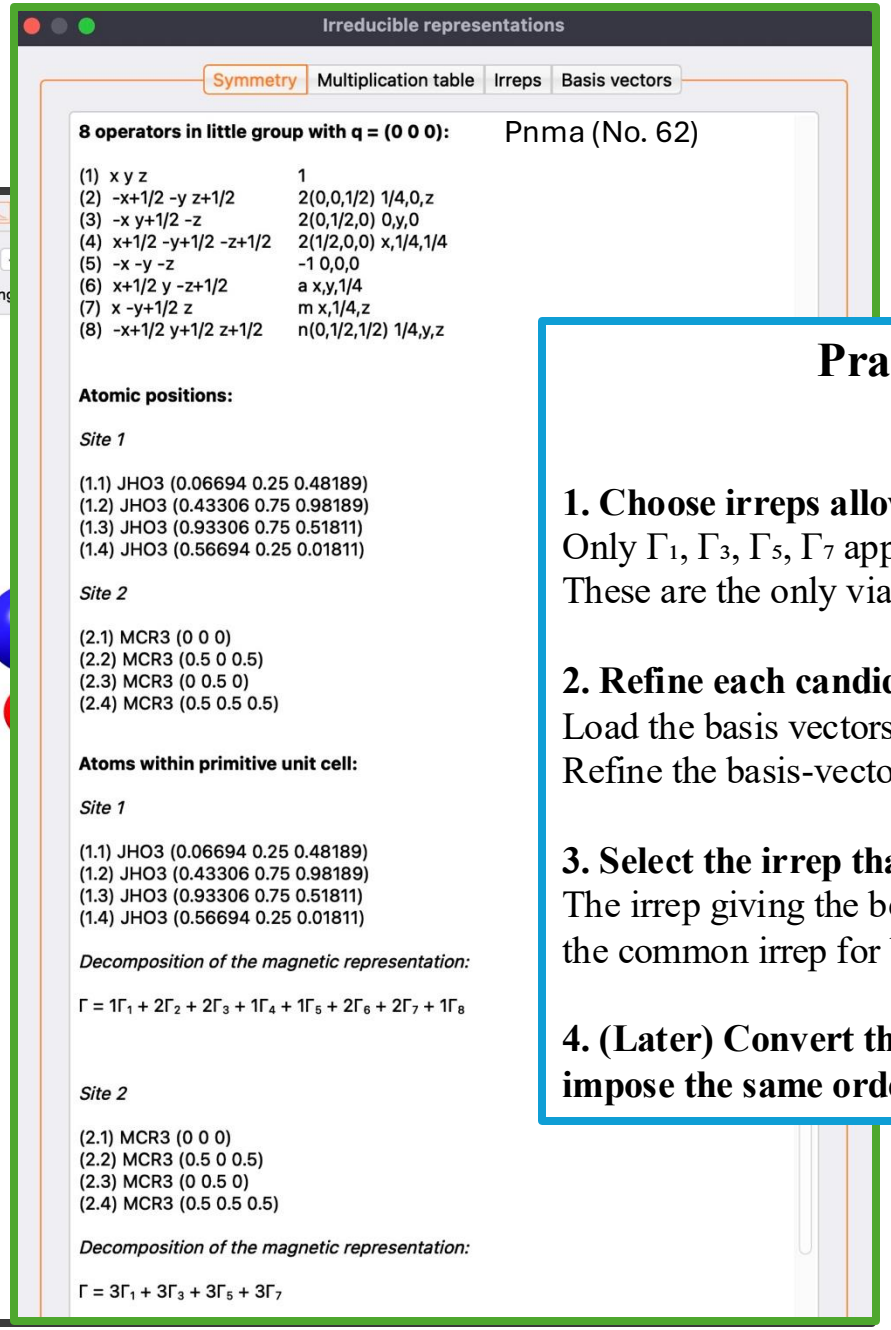
In the newly opened ‘Irreducible representations’ window, the ‘Symmetry’ tab provides details of symmetry analysis and magnetic representation.

Ho can order according to any of the 8 irreps, some with multiple independent basis vectors.

Cr can order only via Γ_1, Γ_3, Γ_5, Γ_7 (Γ_2, Γ_4, Γ_6, Γ_8 are not allowed for Cr).

Refinement of magnetic structure using irreducible representations





Practical refinement strategy

1. Choose irreps allowed for both Ho(4c) and Cr(4a)
Only Γ_1, Γ_3, Γ_5, Γ_7 appear in the Cr decomposition →
These are the only viable magnetic irreps for the whole structure.

2. Refine each candidate irrep
Load the basis vectors for Ho(4c) and Cr(4a) in Mag2Pol.
Refine the basis-vector amplitudes and compare fit quality.

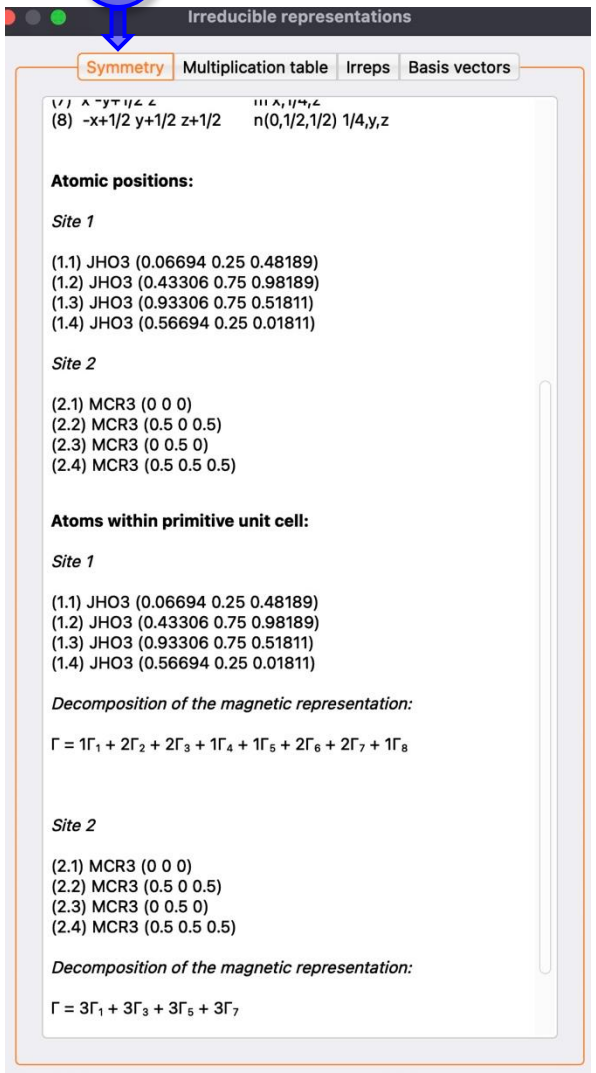
3. Select the irrep that fits best
The irrep giving the best agreement with the magnetic Bragg intensities is the common irrep for both Ho and Cr.

4. (Later) Convert this irrep solution into the corresponding MSG to impose the same ordering via symmetry constraints.

On the ‘Irreducible representation’ window select an irrep (Γn) for the refinement

The **‘Symmetry’** tab provides details of symmetry analysis and magnetic representation

1

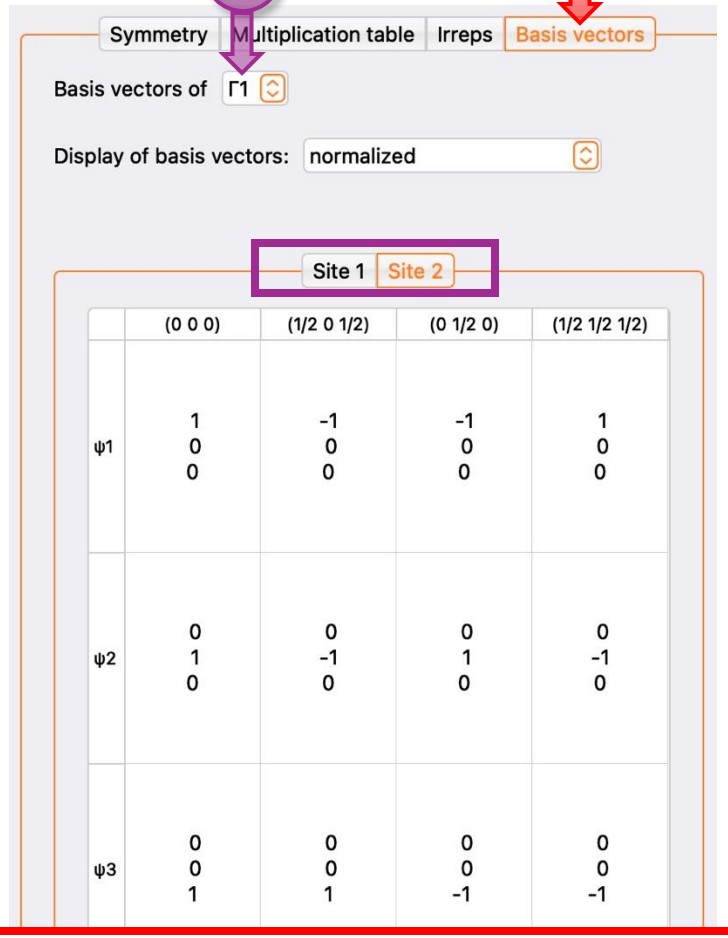


The **‘Basis vectors’** tab provides details of the Basis vectors of all magnetic sites for an irrep.

Choose an irrep to see the Basis vector details for each magnetic ion

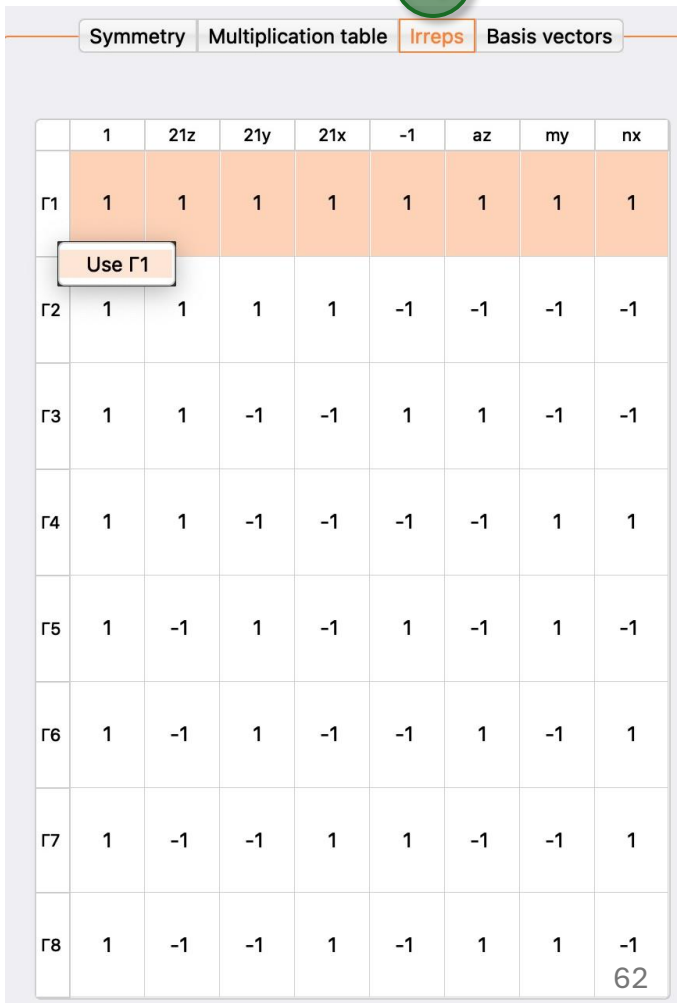
2

3



The **‘Irreps’** tab lists all irreps.
Right-click on one of the irreps symbols (Γ1 in this example), and consequently click on ‘Use Γ1’
Go back to the main window of Mag2Pol

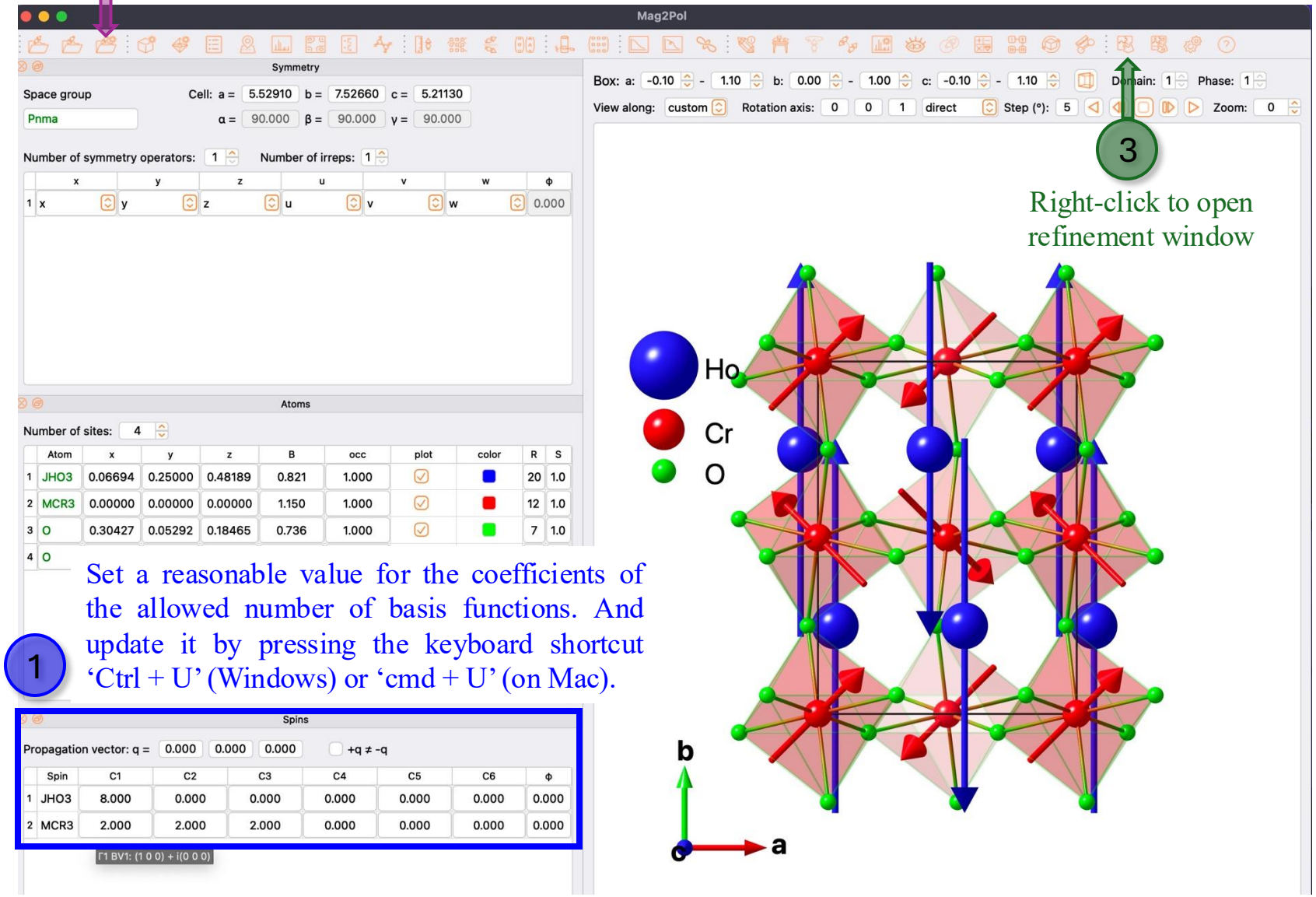
4



	1	21z	21y	21x	-1	az	my	nx
Γ1	1	1	1	1	1	1	1	1
Γ2	1	1	1	1	-1	-1	-1	-1
Γ3	1	1	-1	-1	1	1	-1	-1
Γ4	1	1	-1	-1	-1	-1	1	1
Γ5	1	-1	1	-1	1	-1	1	-1
Γ6	1	-1	1	-1	-1	1	-1	1
Γ7	1	-1	-1	1	1	-1	-1	1
Γ8	1	-1	-1	1	-1	1	1	-1

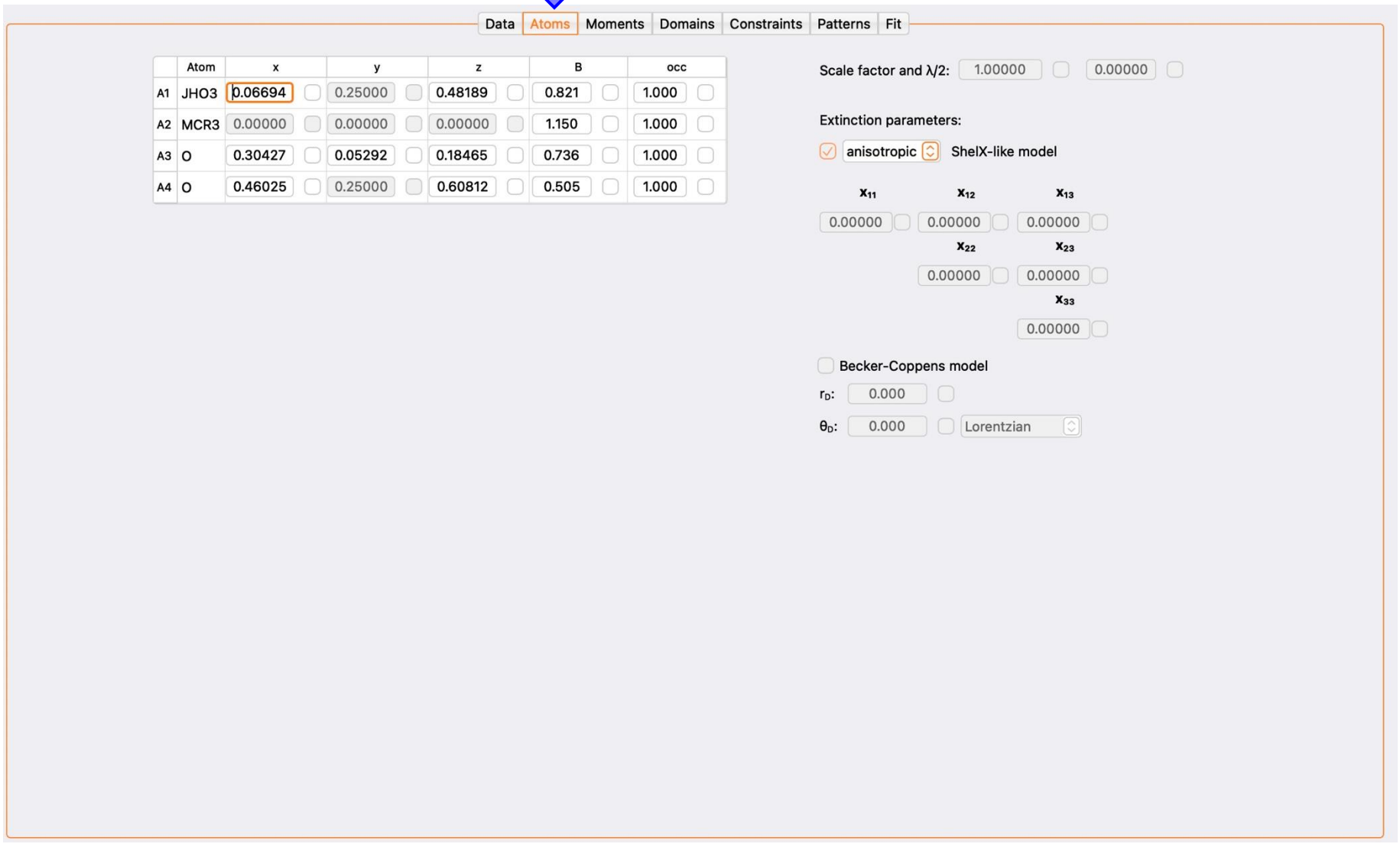
Save the project with an appropriate name that indicates the irrep

2 Save as ‘HCO_4K_Mag-G1.xml’

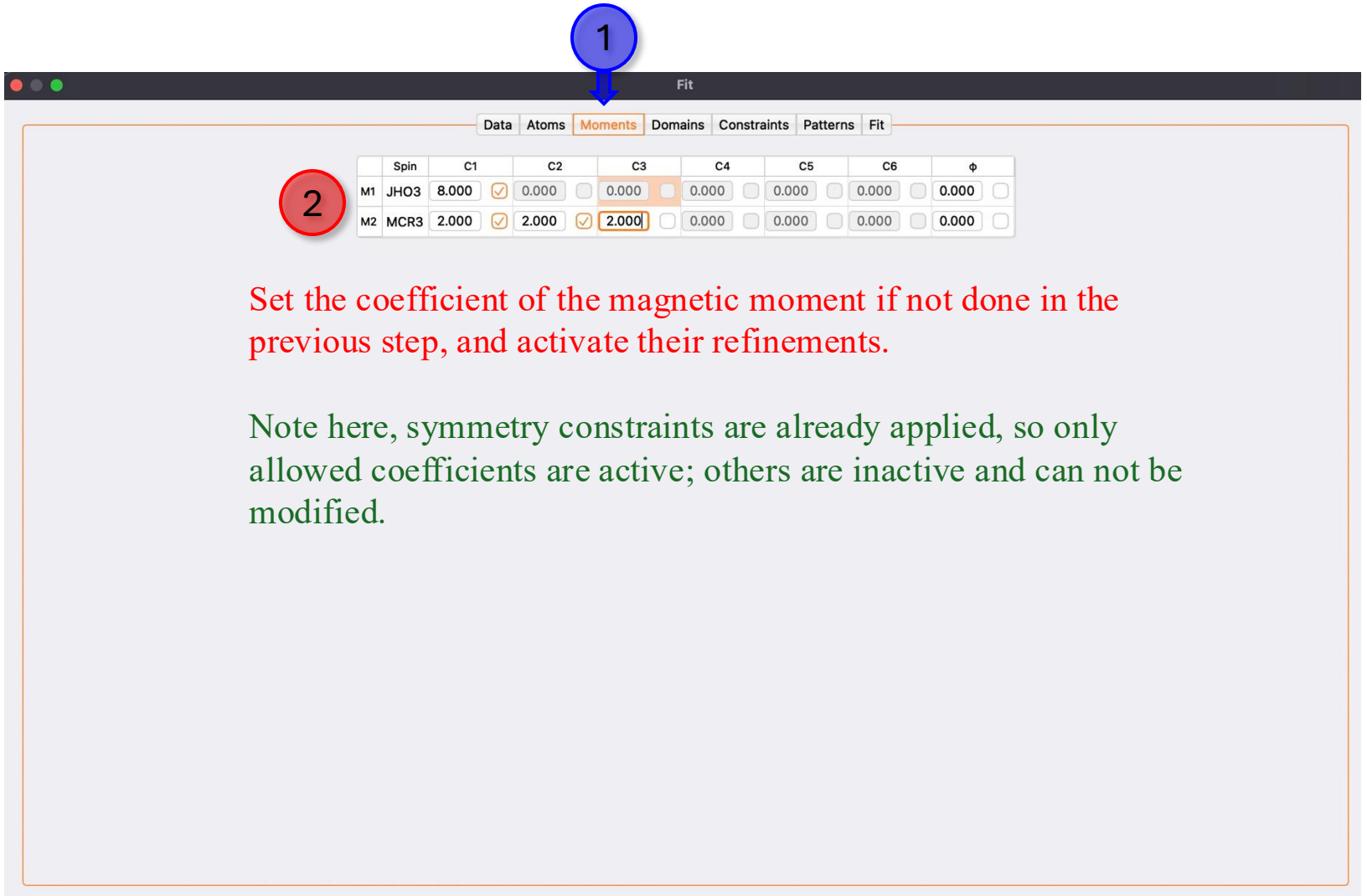


Fix all atomic parameters

On the ‘Fit’ window, under the ‘Atoms’ tab, make sure all the atomic parameters are fixed.



Activate refinement of magnetic moments



Set the coefficient of the magnetic moment if not done in the previous step, and activate their refinements.

Note here, symmetry constraints are already applied, so only allowed coefficients are active; others are inactive and can not be modified.

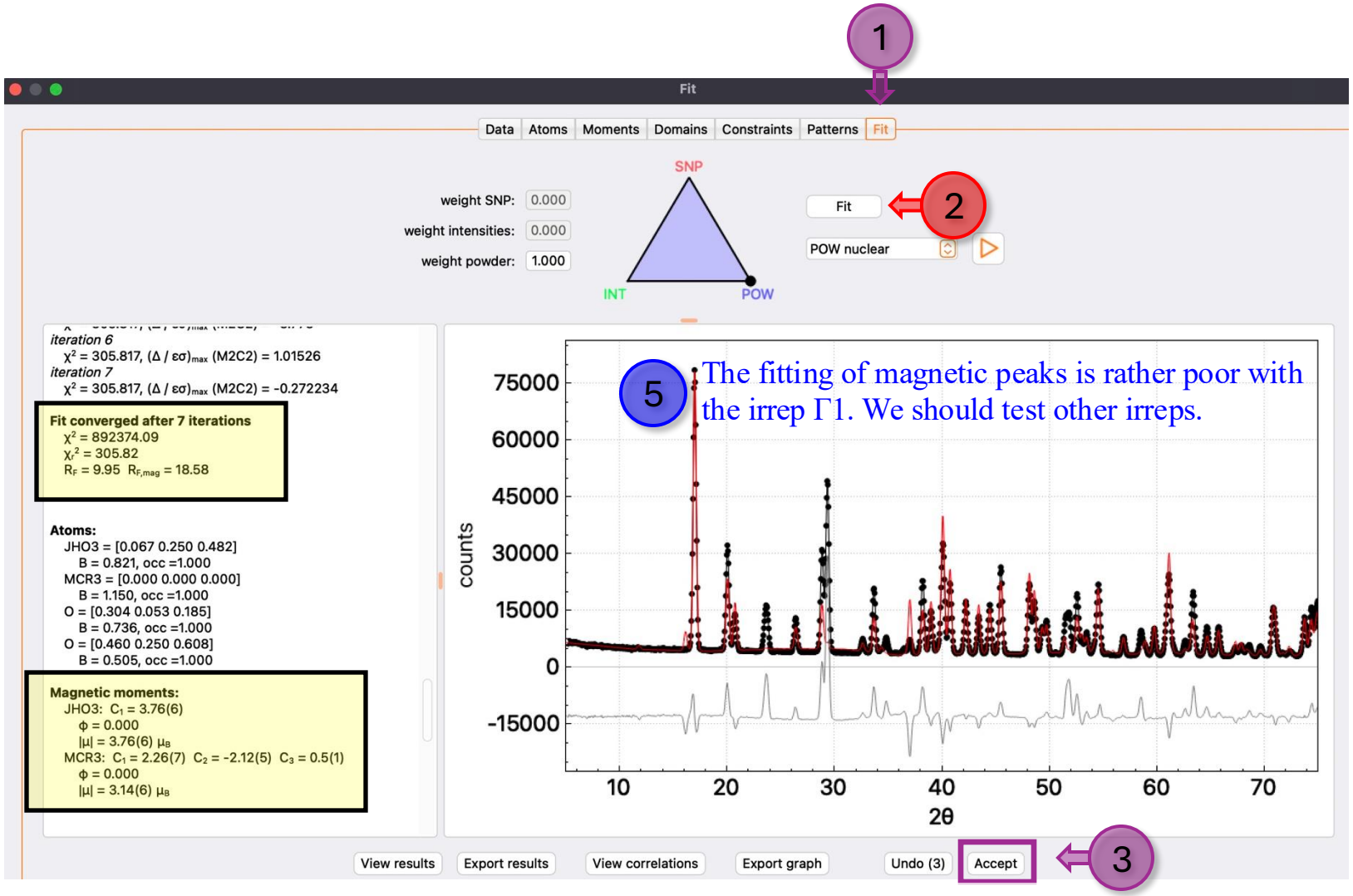
Fix all parameters but the scale

Fix all parameters except the 'scale' parameter.

Also, make sure both 'nuclear' and 'magnetic' phases are active

Nuclear and magnetic phases should have the same Peak functions

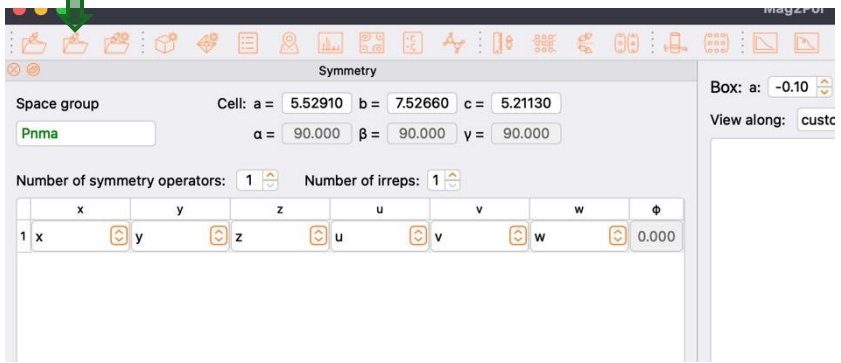
Run the Rietveld fit



Once the convergence is reached, accept the fit results by clicking this button.

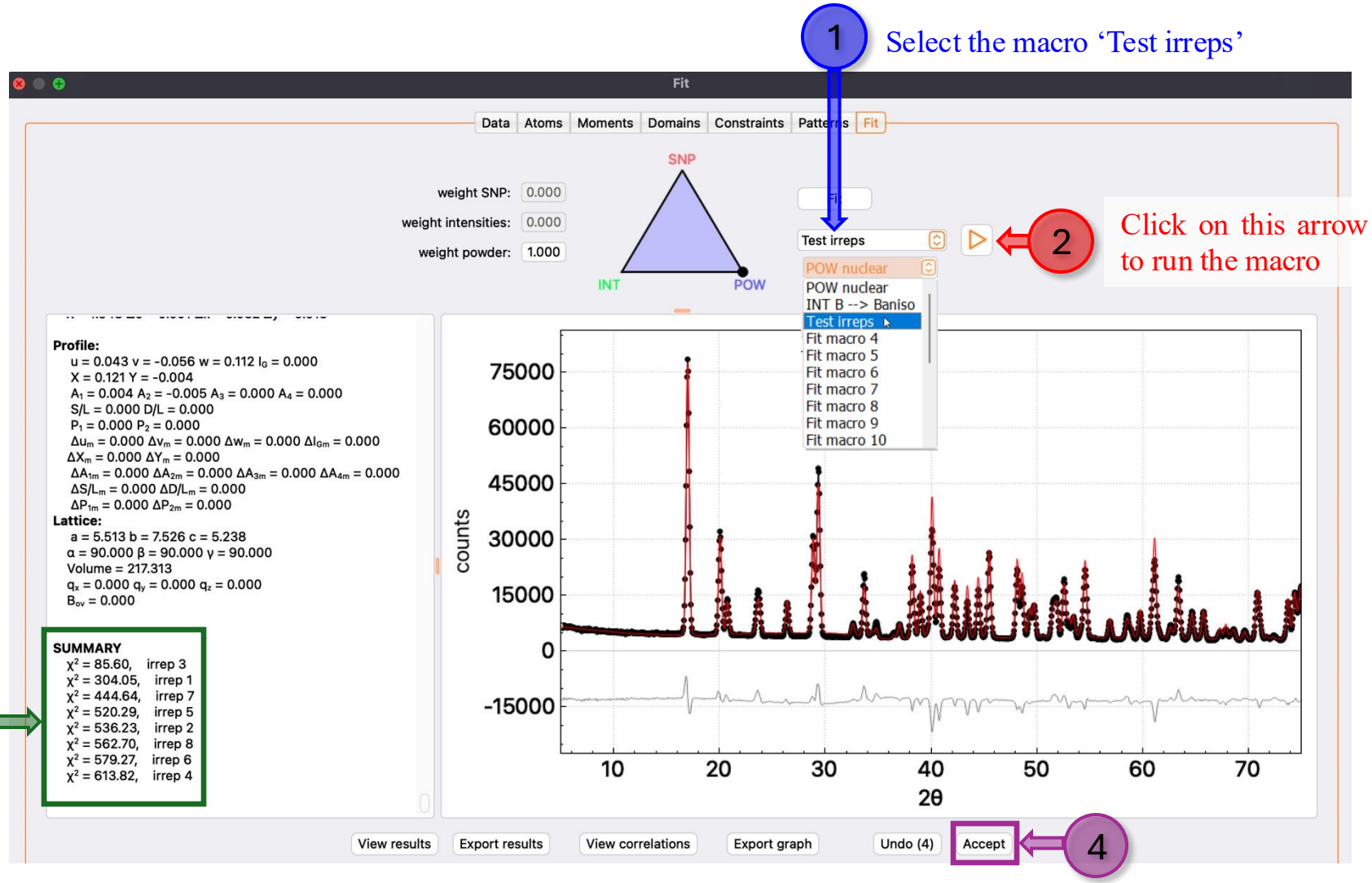
After accepting the refinement results on the 'Fit' window (keep this window open), go to the main window and click on the save button, which appends the previously saved project, **HCO_4K_Mag-G1.xml**

4



Test all irreps by running built-in *Fit macros*

We can test each irrep one by one by selecting them in the main window of the Mag2Pol. But this can also be done much faster using the built-in fit macro ‘Test irreps’.



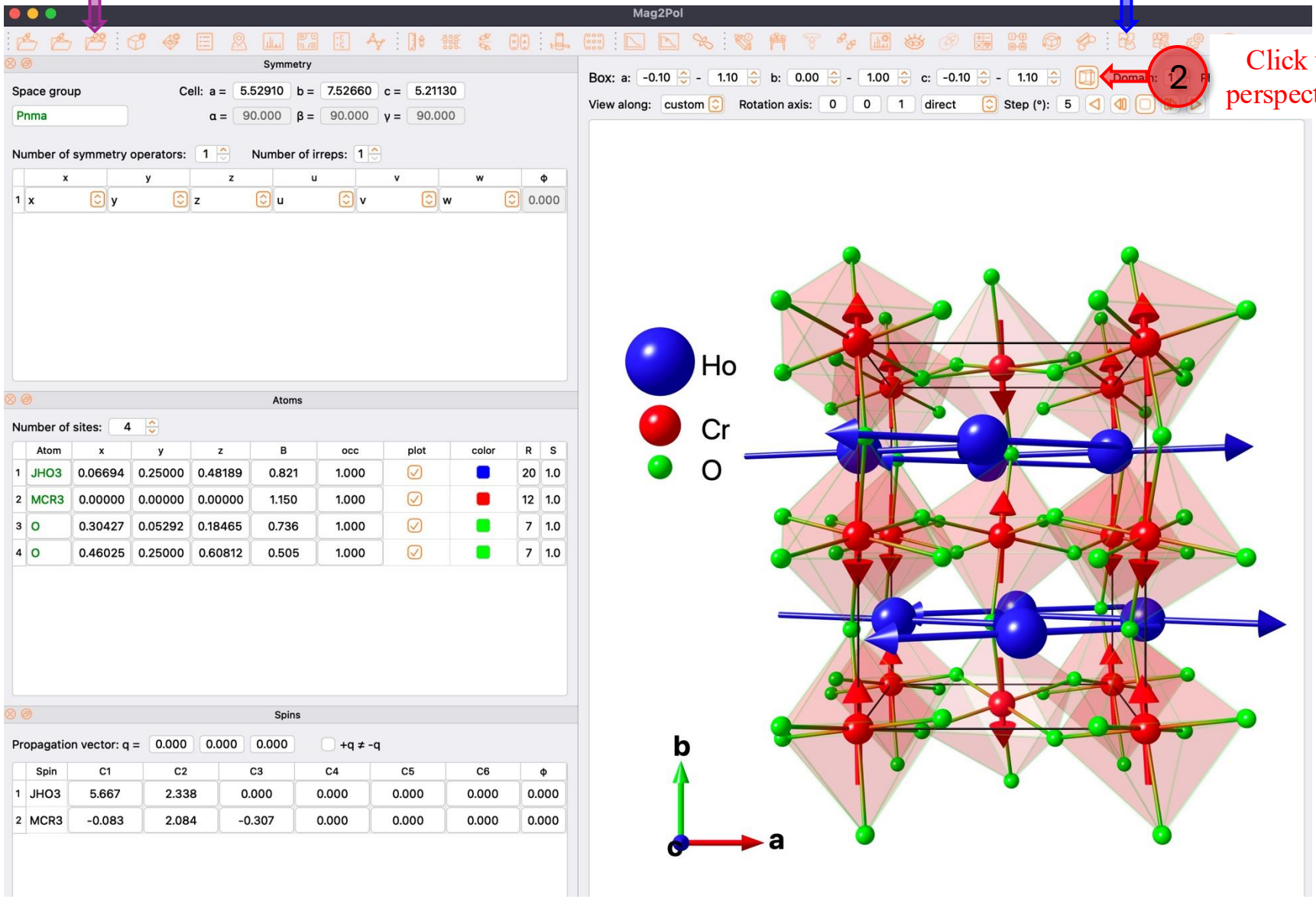
The irrep 3 (Γ3) looks best and is applied to the model at the end of the test. At this stage, we can refine Cell and profile parameters and atomic parameters in steps, which can further improve the refinements.

Save the project with an appropriate name that indicates the new irrep used

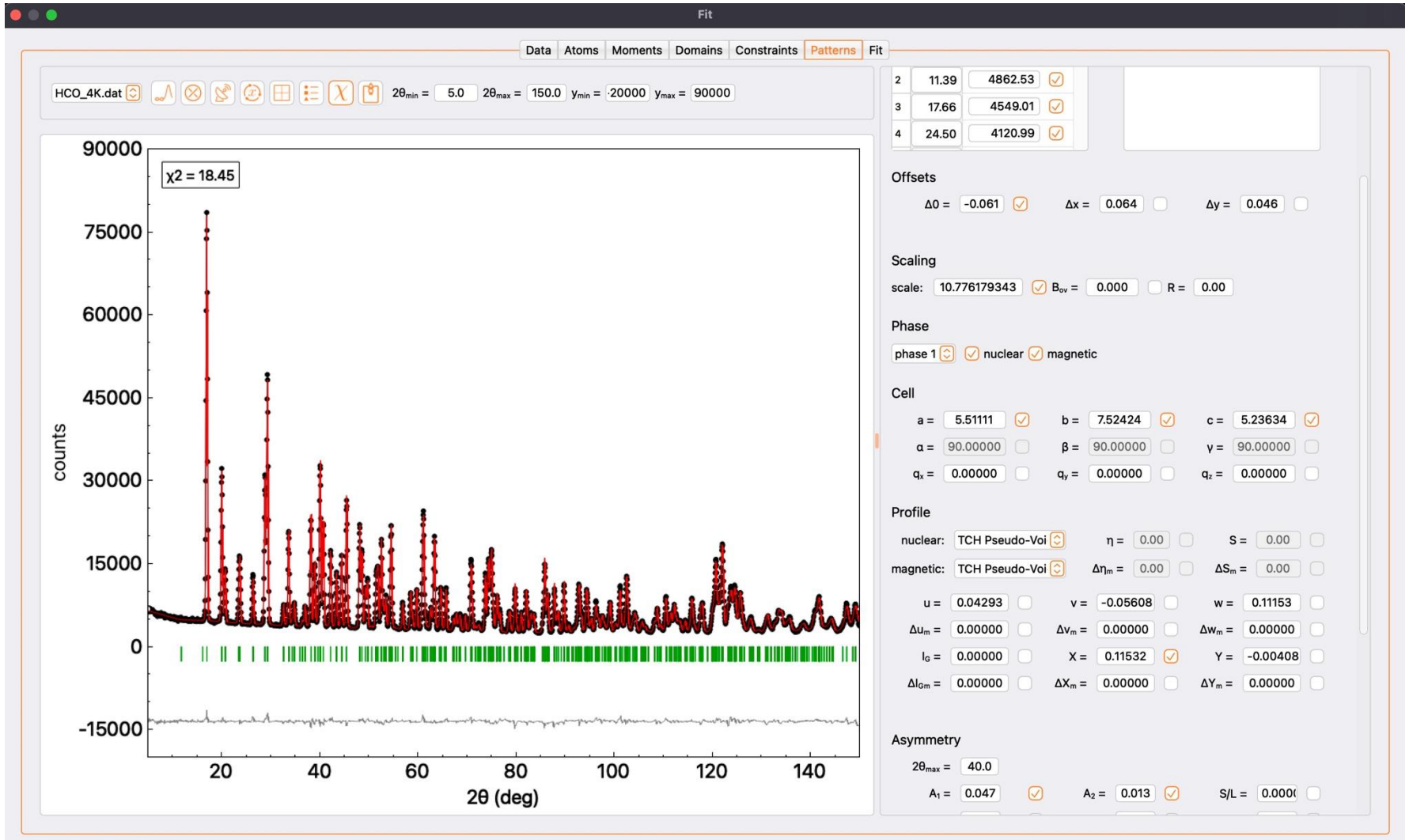
1 Save as 'HCO_4K_Mag-G7.xml'

3 Back to the fit-window

2 Click this to change the perspective of the structure



In steps activate and refine all necessary parameters



Activate refinement of different parameters in steps. Run the fit during each step.

1. BG
2. Offsets and Asymmetry parameters
3. Profile parameters
4. Atomic positions
5. Thermal parameters (B)

Final refinement results for irrep 3

Magnetic R-factor, $R_{F,mag}$ is a good indicator to pick the best irrep (along with the visual fit)

Note that coefficients C_1 and C_3 are quite small and can be fixed to zero during the final refinements

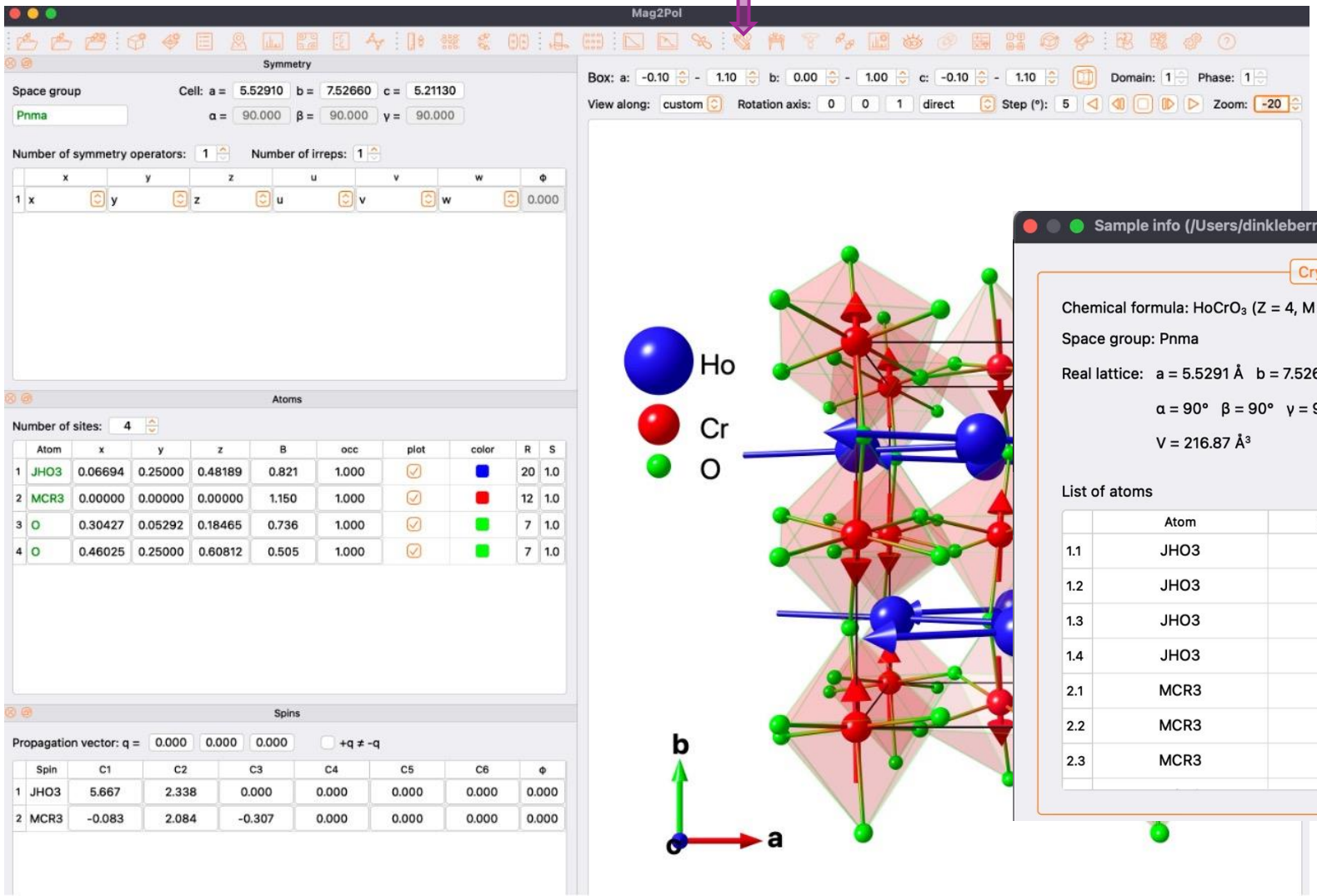
After accepting the refinement results here, do not forget to save the project from the main window!

It is recommended to save versioned copies of the project often. If the program crashes, it is easy to start over from the saved versions.

Inspecting full refinement results

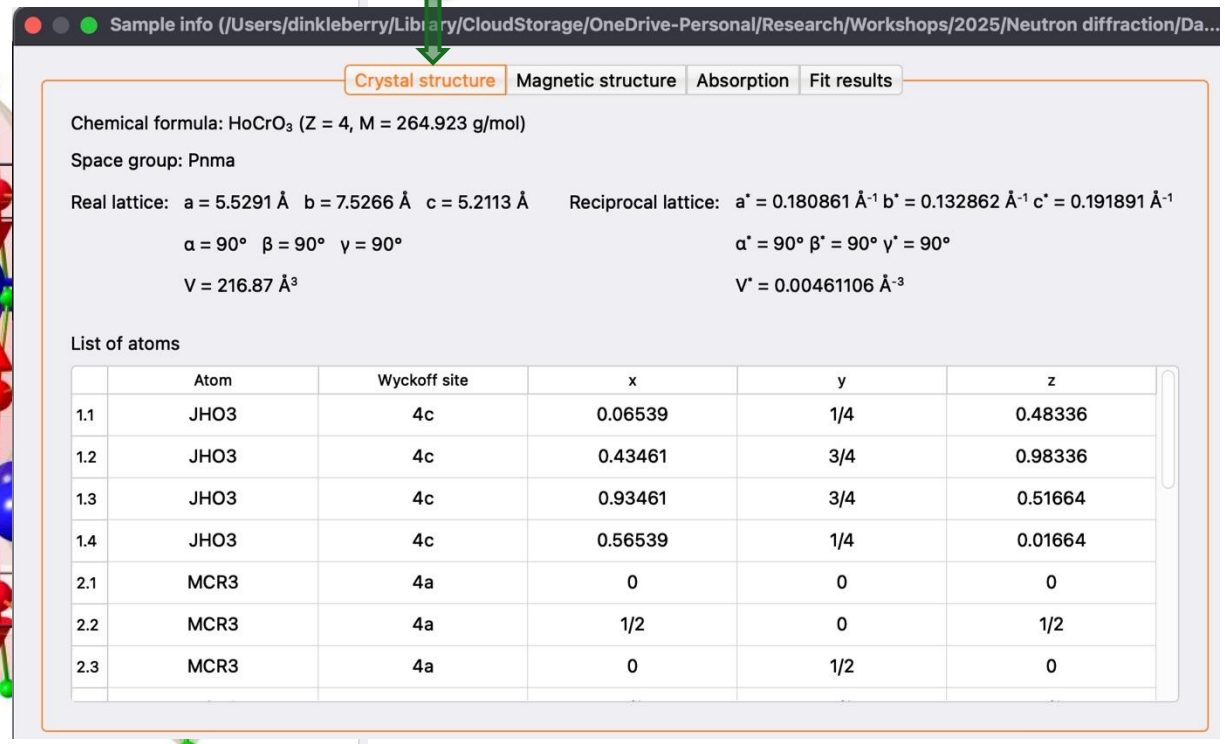
Click this icon to open the ‘Sample info’ window, which contains detailed information on the refined Crystal structure, Magnetic structure, and Fit results. Also indicates the path and name of the current project file.

1



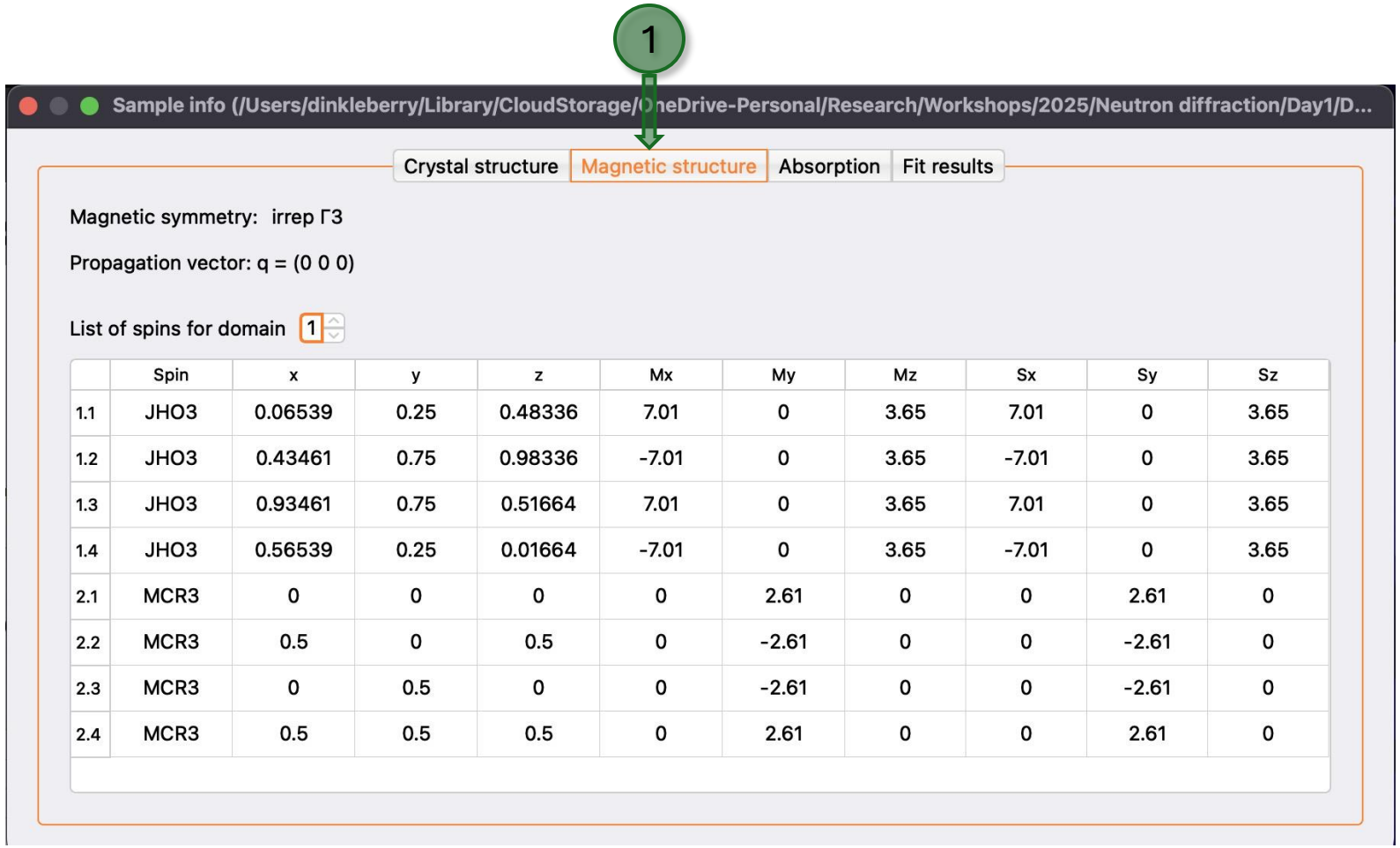
‘Sample info’ window, with the ‘Crystal Structure’ tab active.

2



Inspecting full refinement results

'Magnetic structure' tab information of the magnetic model used in the last fit.



	Spin	x	y	z	Mx	My	Mz	Sx	Sy	Sz
1.1	JHO3	0.06539	0.25	0.48336	7.01	0	3.65	7.01	0	3.65
1.2	JHO3	0.43461	0.75	0.98336	-7.01	0	3.65	-7.01	0	3.65
1.3	JHO3	0.93461	0.75	0.51664	7.01	0	3.65	7.01	0	3.65
1.4	JHO3	0.56539	0.25	0.01664	-7.01	0	3.65	-7.01	0	3.65
2.1	MCR3	0	0	0	0	2.61	0	0	2.61	0
2.2	MCR3	0.5	0	0.5	0	-2.61	0	0	-2.61	0
2.3	MCR3	0	0.5	0	0	-2.61	0	0	-2.61	0
2.4	MCR3	0.5	0.5	0.5	0	2.61	0	0	2.61	0

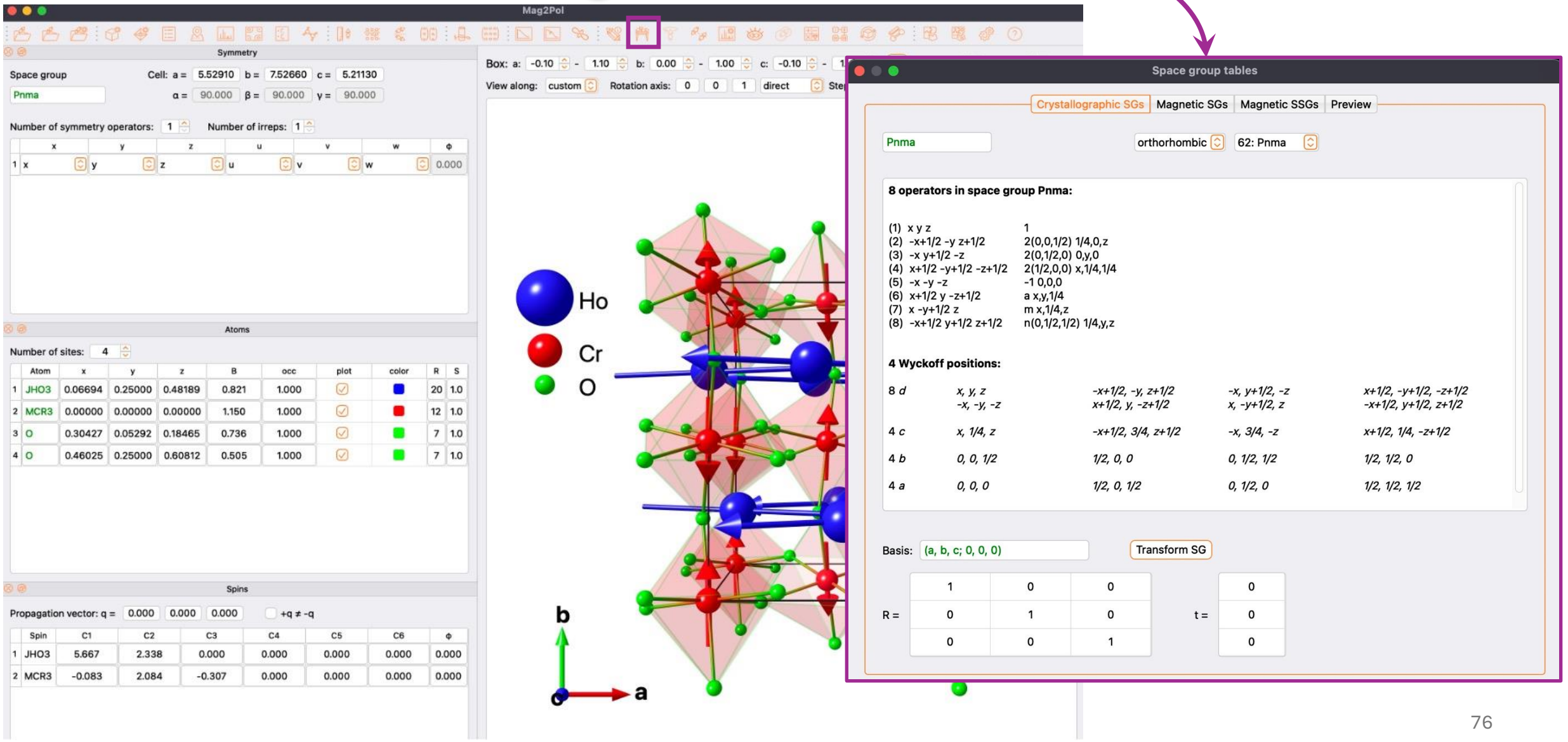
Inspecting full refinement results

The ‘Fit results’ tab contains detailed refinement results of the last fit.

Refining magnetic structure using Shubnikov groups (Magnetic space groups)

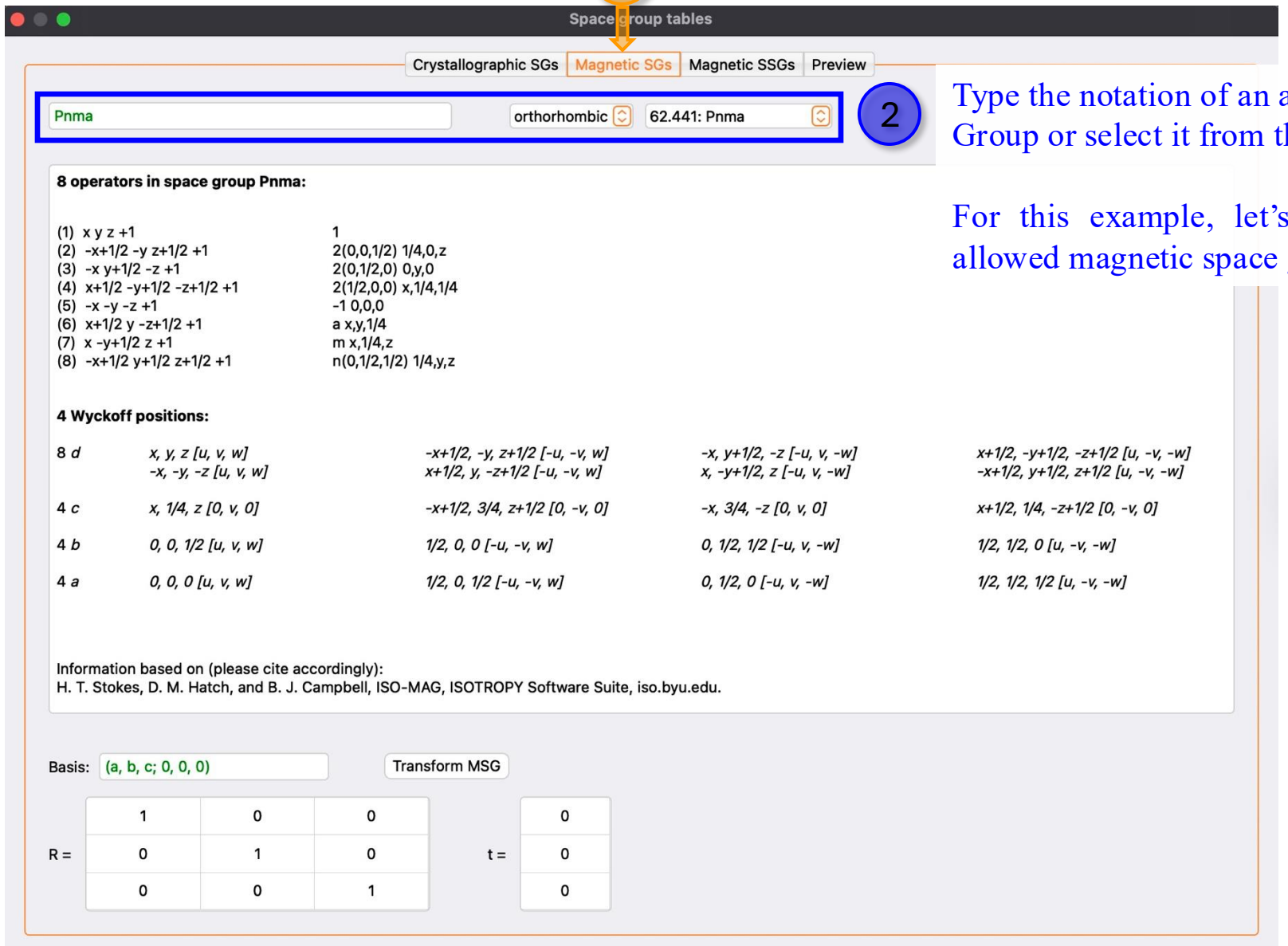
Set up a Magnetic Space Group (MSG)

1 Click on the 'Space group tables' icon to open the new window with the same name.



Set up a Magnetic Space Group

1 Activate the 'Magnetic SGs' tab

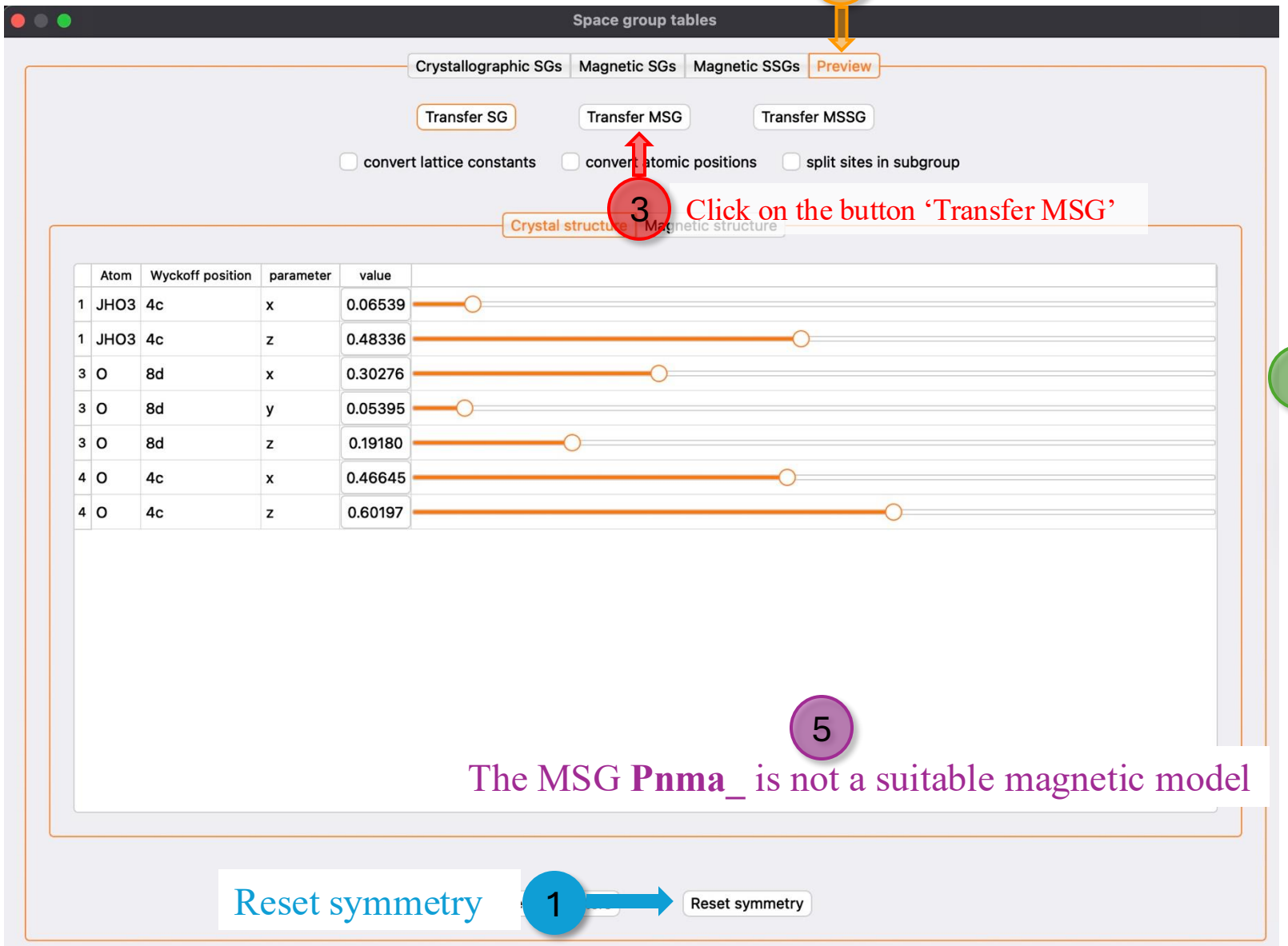


Type the notation of an appropriate Magnetic Space Group or select it from the drop-down menu.

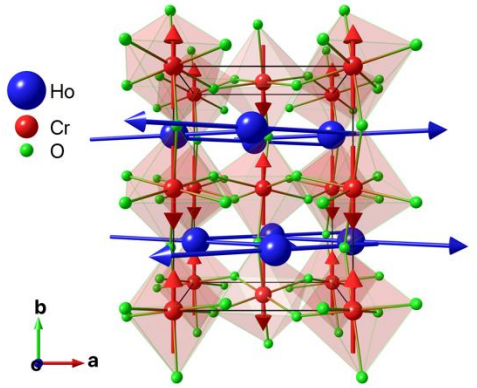
For this example, let's choose one of the first allowed magnetic space groups *Pnma* (62.441).

Set up a Magnetic Space Group

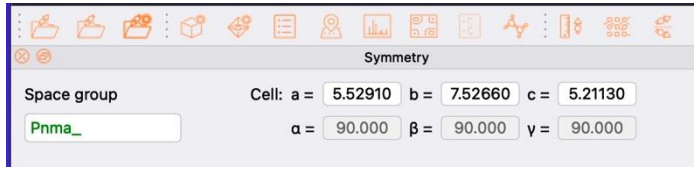
1 Switch to the 'Preview' tab



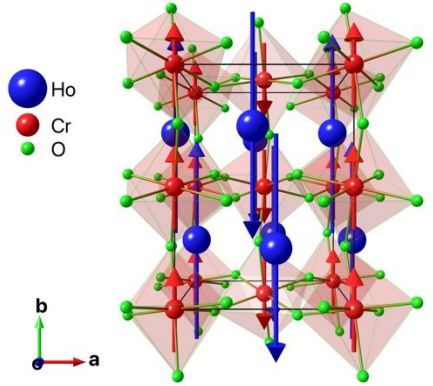
2 Keep an eye on the magnetic structure in the main window



4 In the main window of the Mag2Pol, the space group is appended to **Pnma_** (underscore indicates it is a MSG)



The spin arrangement in MSG **Pnma_** (which is appended now on the main window) looks very different from the irrep 3 above



Set up a Magnetic Space Group

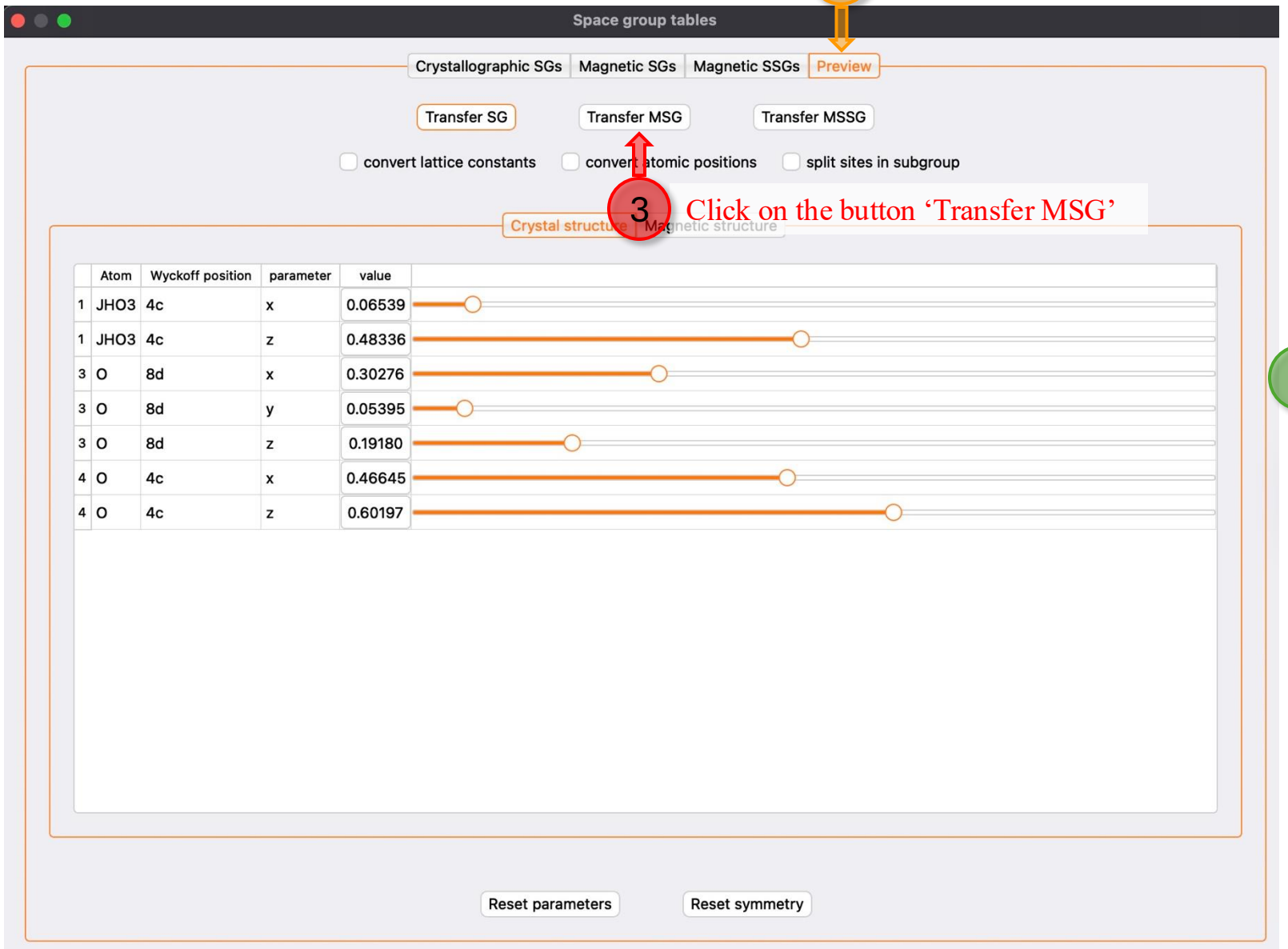
1 Activate the 'Magnetic SGs' tab

Try other MSGs within the Pnma setting of the parent.

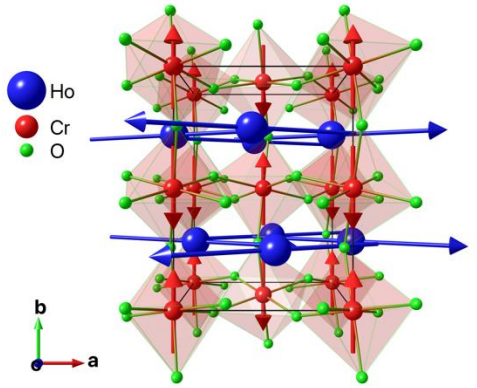
But for the exercise, let's now choose Pn'm'a (62.446).

Set up a Magnetic Space Group

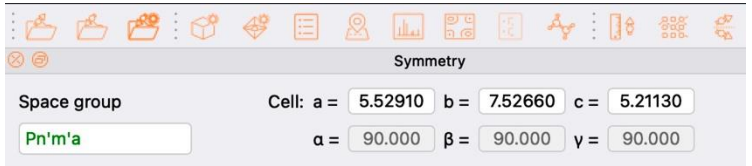
1 Switch to the 'Preview' tab



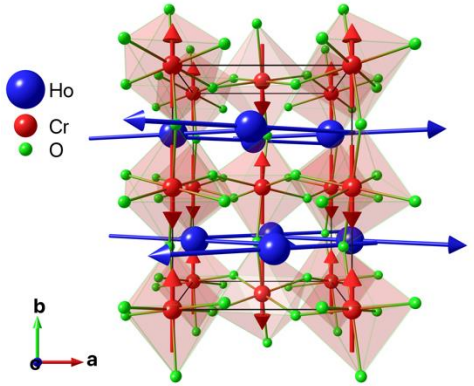
2 Keep an eye on the magnetic structure in the main window



4 In the main window of the Mag2Pol, the space group is appended to **Pn'm'a**



The spin arrangement in MSG **Pn'm'a** is identical to irrep 3. This could be actual MSG



Save the project with MSG Pn’m’a

1 Save the project as ‘**HCO_4K_Mag-Pn’m’a.xml**’

2

Symmetry

Space group: Pn'm'a

Cell: a = 5.52910 b = 7.52660 c = 5.21130

α = 90.000 β = 90.000 γ = 90.000

Number of symmetry operators: 1 Number of irreps: 1

	x	y	z	u	v	w	ϕ
1	x	y	z	u	v	w	0.000

Atoms

Number of sites: 4

	Atom	x	y	z	B	occ	plot	color	R	S
1	JHO3	0.06539	0.25000	0.48336	-0.133	1.000	☑		20	1.0
2	MCR3	0.00000	0.00000	0.00000	-0.067	1.000	☑		12	1.0
3	O	0.30276	0.05395	0.19180	-0.103	1.000	☑		7	1.0
4	O	0.46645	0.25000	0.60197	-0.095	1.000	☑		7	1.0

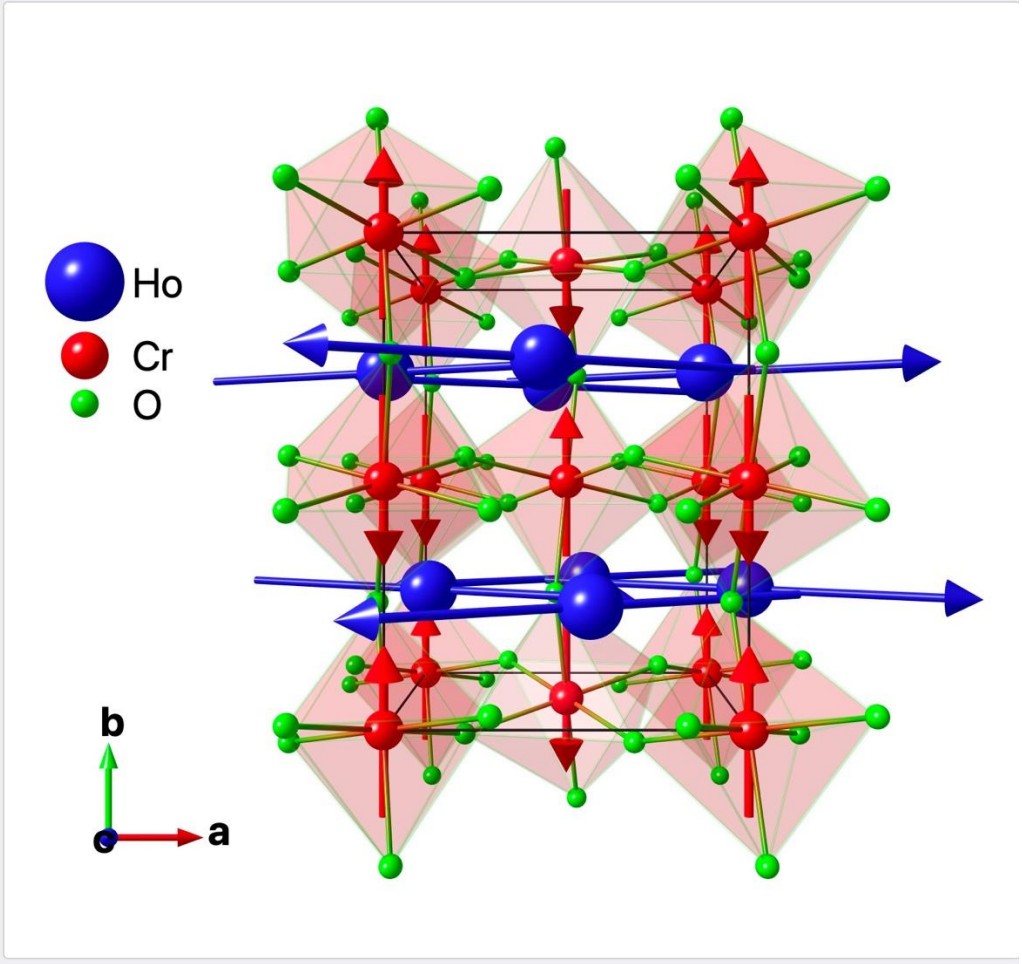
Spins

Propagation vector: q = 0.000 0.000 0.000 +q ≠ -q

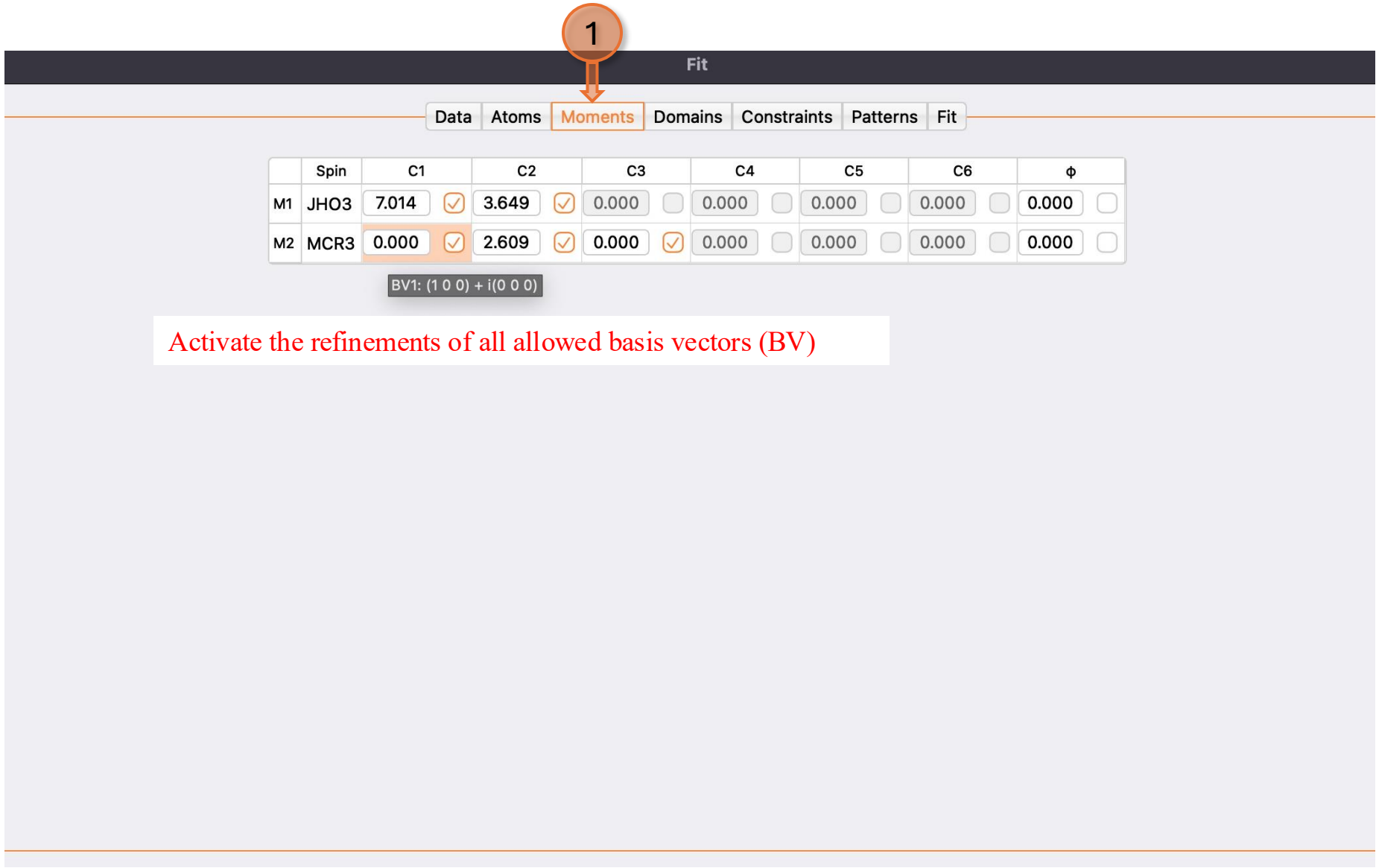
	Spin	C1	C2	C3	C4	C5	C6	ϕ
1	JHO3	7.014	3.649	0.000	0.000	0.000	0.000	0.000
2	MCR3	0.000	2.609	0.000	0.000	0.000	0.000	0.000

Box: a: -0.10 - 1.10 b: 0.00 - 1.00 c: -0.10 - 1.10 Domain: 1 Phase: 1

View along: custom Rotation axis: 0 0 1 direct Step (°): 5 Zoom: -35



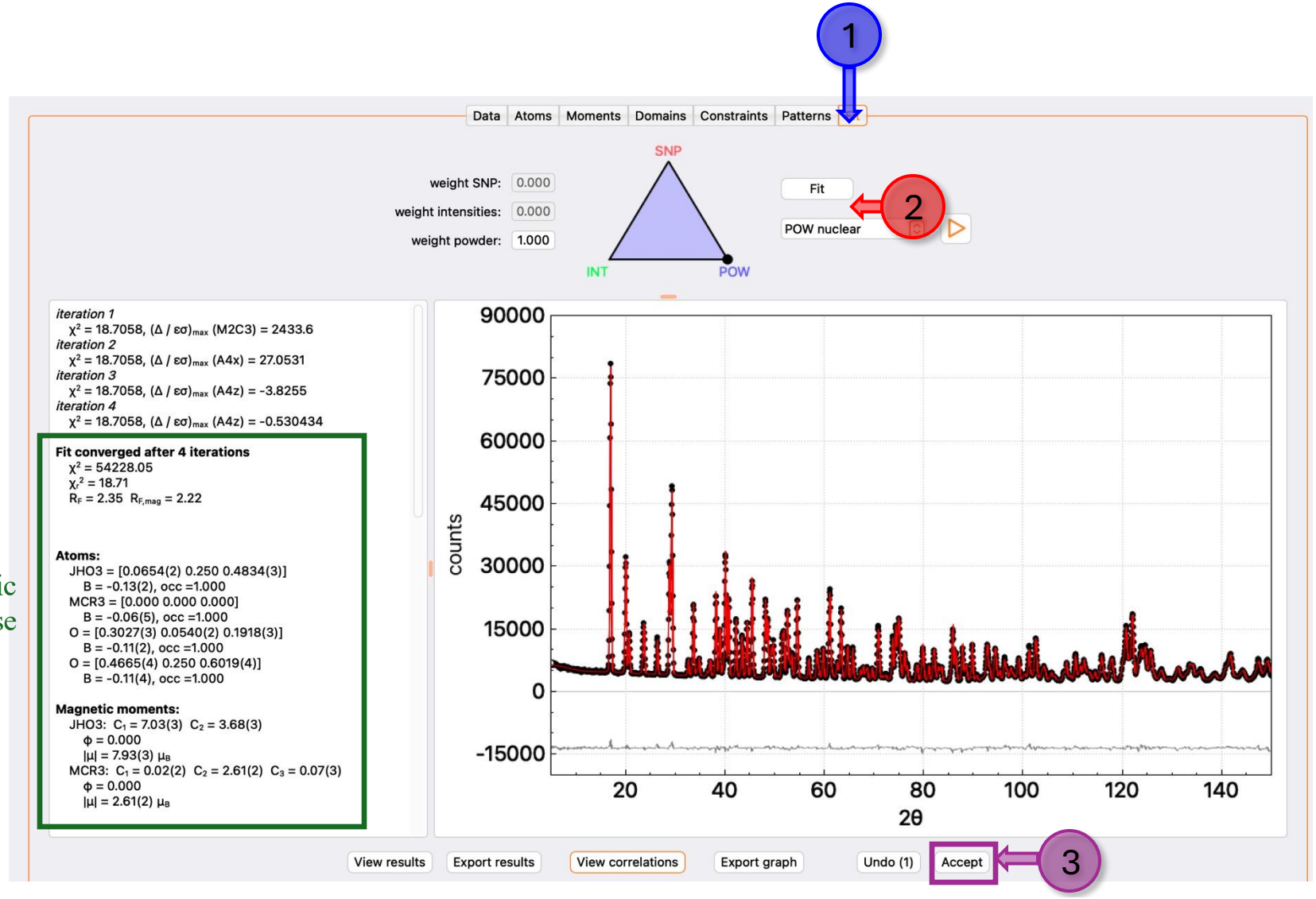
Save the project with MSG Pn’m’a



Activate the refinements of all allowed basis vectors (BV)

Save the project with MSG Pn’m’a

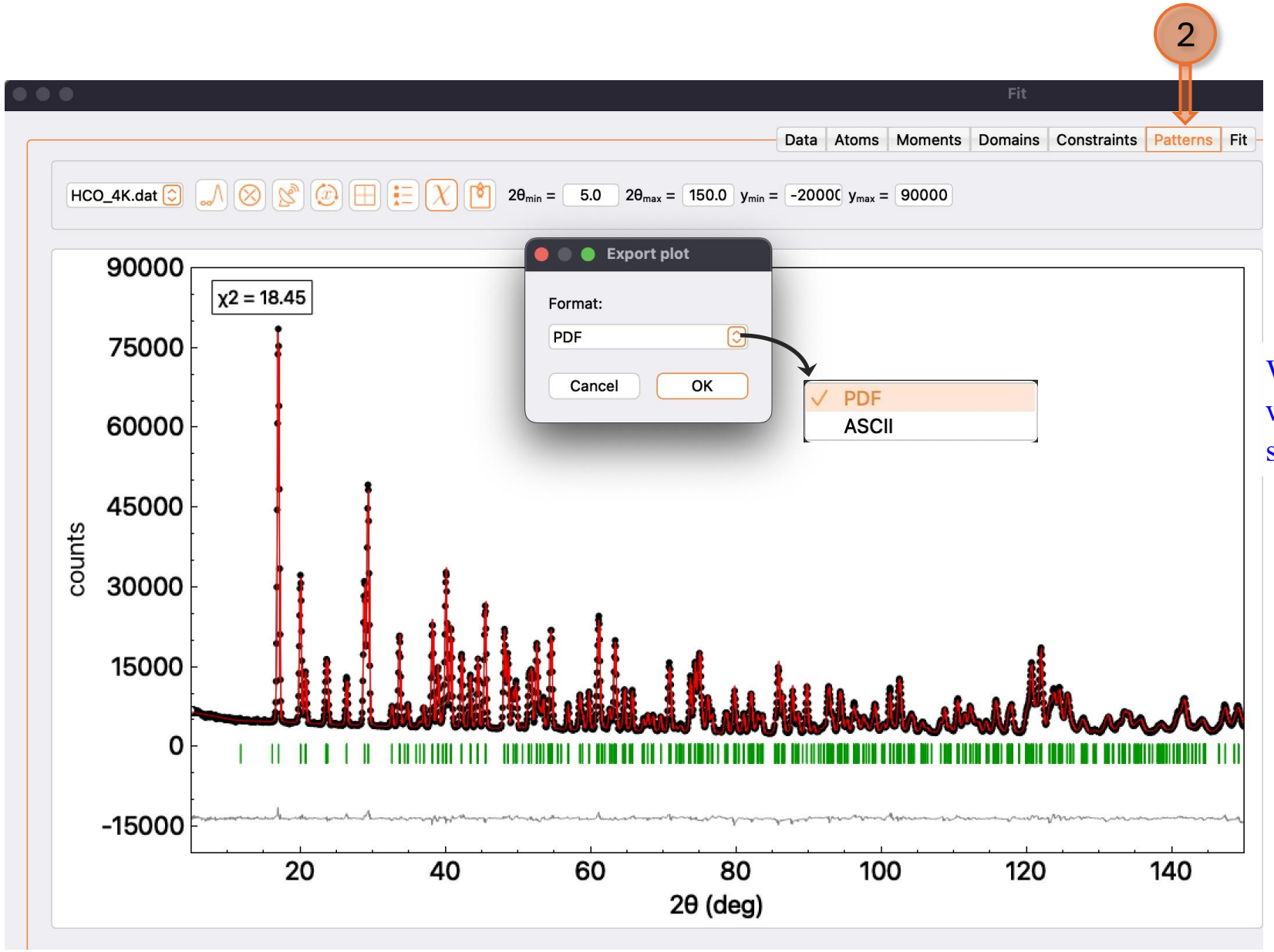
The refinement results using the Magnetic Space Group Pn’m’a are identical to those obtained using irrep Γ3.



After accepting the refinement results here, do not forget to save the project from the main window!

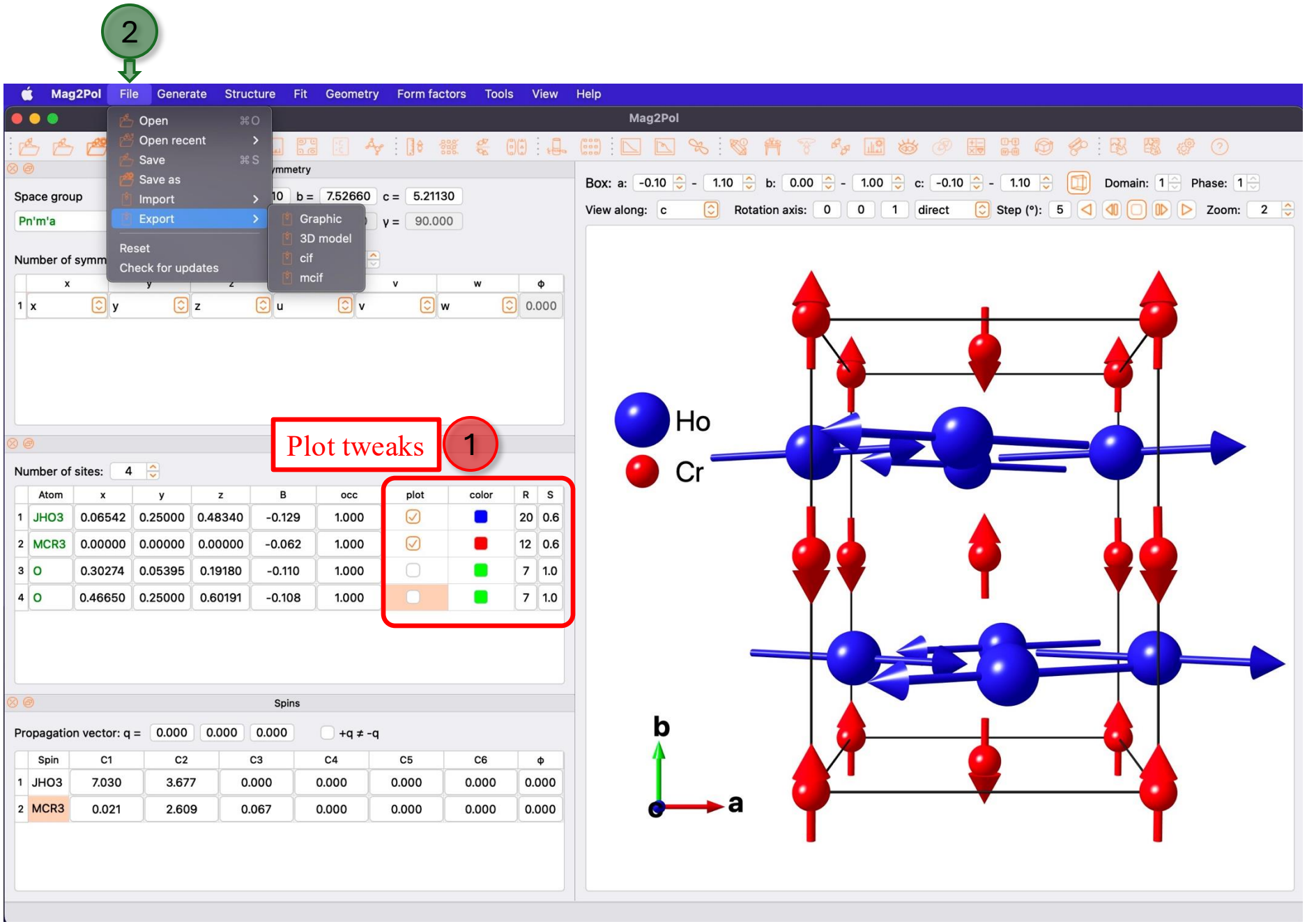
Exporting the refined structures, parameters and patterns

Export refined pattern as PDF or ASCII



While exporting it as a PDF rearrange the window to a desired aspect ratio. What you see is what you get.

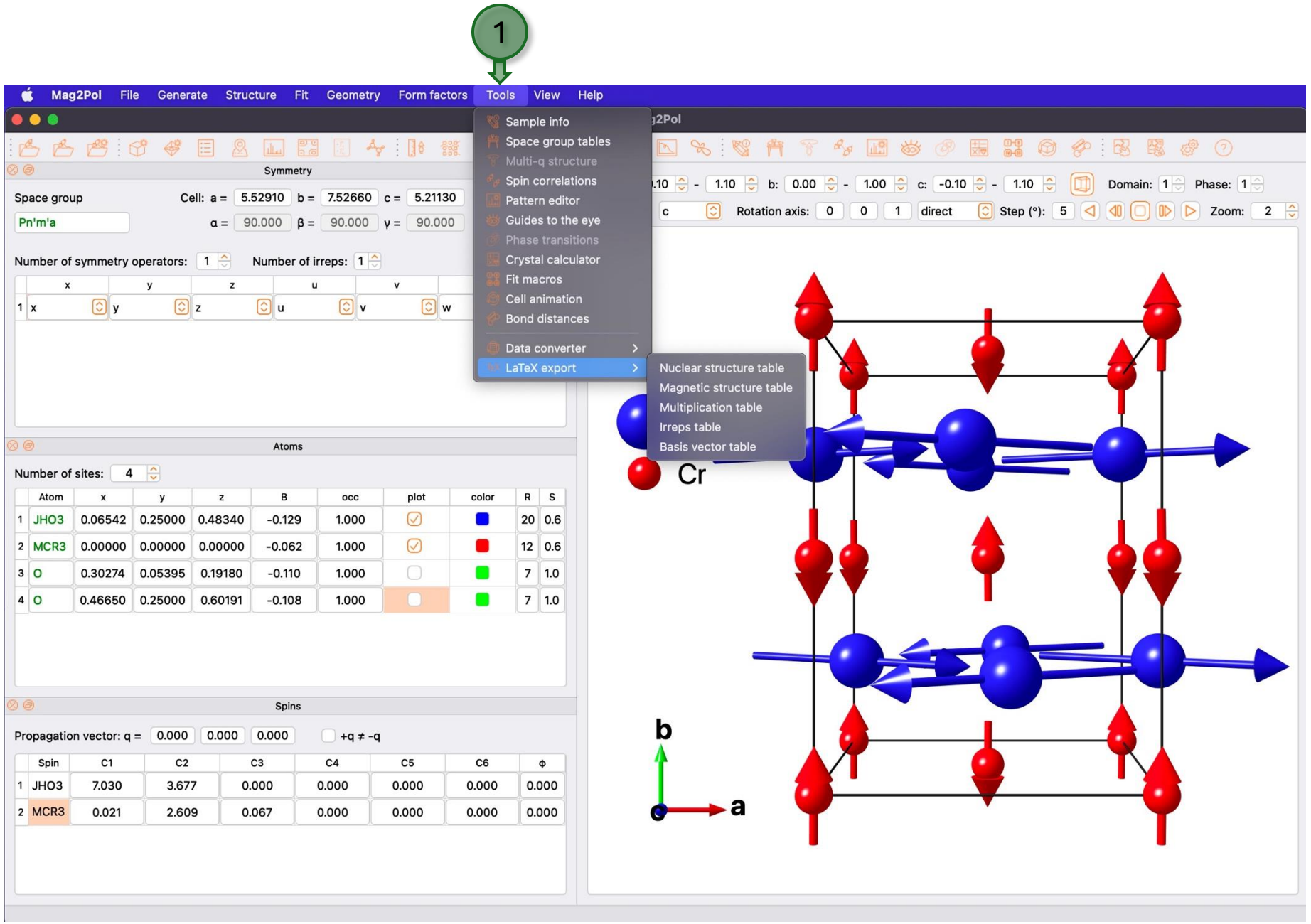
Export the Graphic and structure models



The structure model can be exported as:
*.png (Graphic)
*.cif
*.mcif
by following the menu tree:

File→Export→

Step 7e: Export refinement results as latex tables

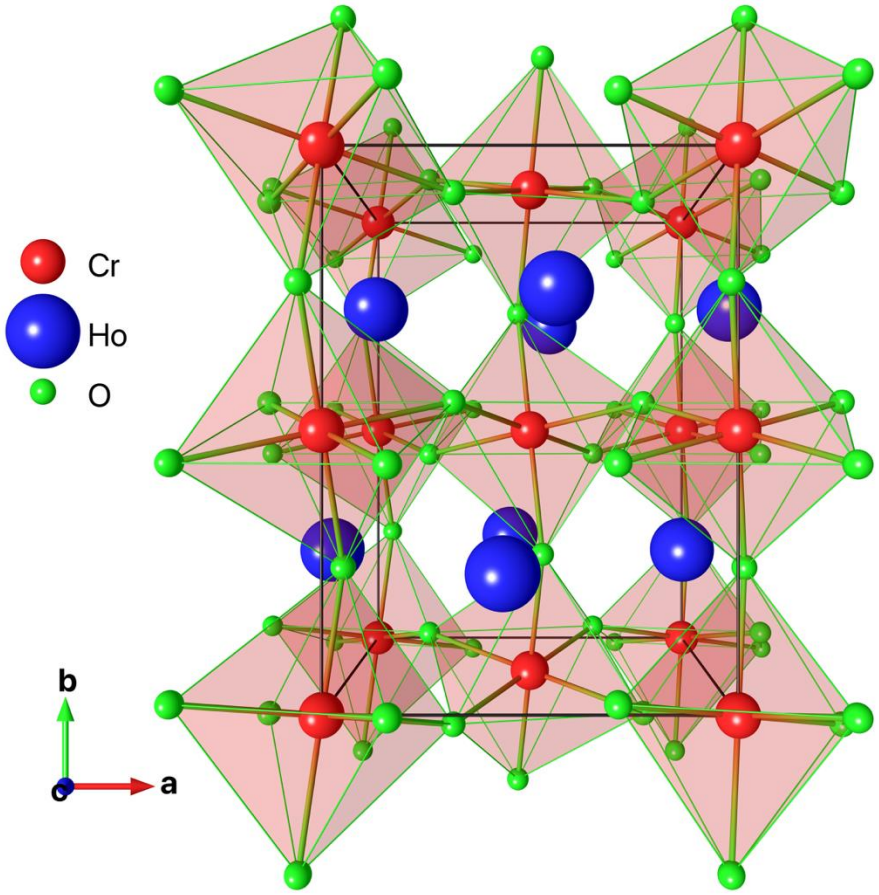


A variety of fit results can be copied directly in LaTeX format by following the menu tree:

Tools→LaTeX export

Exercise

Refine the lattice and magnetic structure of $HoCrO_3$ at 100 K



Orthorhombic structure: *Pnma* (No. 62)

Instrument: Neutron powder diffractometer SPODI at FRM II research reactor, Garching, Germany

Wavelength: 1.5482 Å

Data files:

Data collected at 100 K: HCO_100K.dat (→ HCO_100K.m2p)

Hint:

Only Cr^{3+} is magnetically ordered at this temperature

Before you go!

- Our tutorial on the refinement of time-of-flight data is available on the website for the Tof Neutron Diffractometer POWGEN, Oak Ridge National Laboratory: Link here

- Polish users who performed measurements at ILL, please acknowledge the financing in the publications by including the following statement:

The authors {name of specific author(s) if necessary} acknowledge(s) the Polish Ministry of Education and Science decision no. 2023/WK/08 to fund the scientific membership of Poland at the ILL, which made this research possible.

- Cite the use of Mag2Pol as follows:

Mag2Pol: a program for the analysis of spherical neutron polarimetry, flipping ratio and integrated intensity data, N. Qureshi, J. Appl. Cryst. **52** (2019) 175-185.

If you have any questions:

Regarding Mag2Pol and need to report bugs.

Contact:

Navid Qureshi
Institute Laue Langevin,
Grenoble, France
Email: qureshi@ill.fr

Regarding this tutorial, or need a collaborator on the lattice and magnetic structure refinement.

Contact:

Naveen Kumar Chogondahalli Muniraju
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Krakow, Poland
Email: naveen.chogondahalli@ifj.edu.pl

Good luck with your next refinement project!