

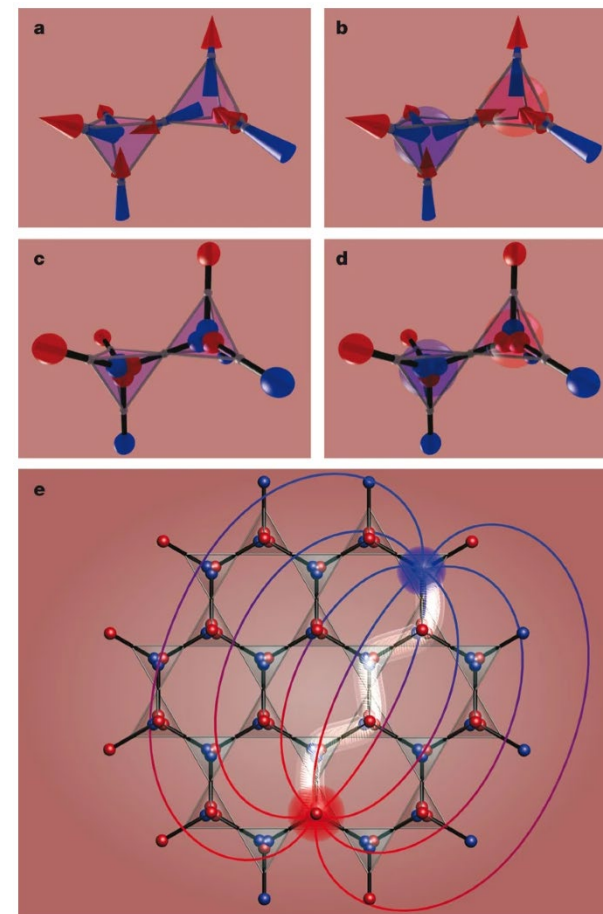
Hands-on polarized powder data from POWDER (HB-2A) $\text{Ho}_2\text{Ti}_2\text{O}_7$

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Polarization Workshop 2026, ORNL.

Background: $\text{Ho}_2\text{Ti}_2\text{O}_7$

- The pyrochlore $\text{Ho}_2\text{Ti}_2\text{O}_7$ is characterized by Ising spins along the $\langle 111 \rangle$ axis on a frustrated lattice.
- In zero field there is no long-range order, instead short range “spin-ice” order gives rise to exotic properties, including magnetic monopoles.
- The local anisotropy encodes and drives the underlying physics.
- **This example shows how half-polarized measurements on a powder can provide details on the local anisotropy.**
 - In this case, the powder data yields the same information as a single crystal analysis.
See DOI:10.1103/PhysRevResearch.1.033100 for more details.
 - **The goal of this exercise is to find the site susceptibility tensor**



$$\chi = \frac{m}{H} = \begin{pmatrix} \chi_{11} & \chi_{12} & \chi_{13} \\ \chi_{12} & \chi_{22} & \chi_{23} \\ \chi_{13} & \chi_{23} & \chi_{33} \end{pmatrix}$$

Details of this $\text{Ho}_2\text{Ti}_2\text{O}_7$ example






- Polarized neutron powder diffraction data collected on HB-2A at HFIR in a magnetic field of 1 T at several temperatures from 1.5 K to 100 K using $\lambda = 2.41 \text{ \AA}$.
 - ~ 3 gram $\text{Ho}_2\text{Ti}_2\text{O}_7$ polycrystalline rod
 - Counting time per temperature/field was ~3 hours
 - This example will look at 20 K, 1T data with flipper on and flipper off
 - Other temperatures are included to follow the change in anisotropy

- Files included in example:

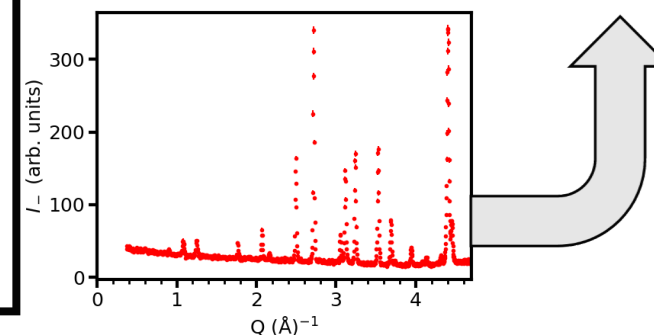
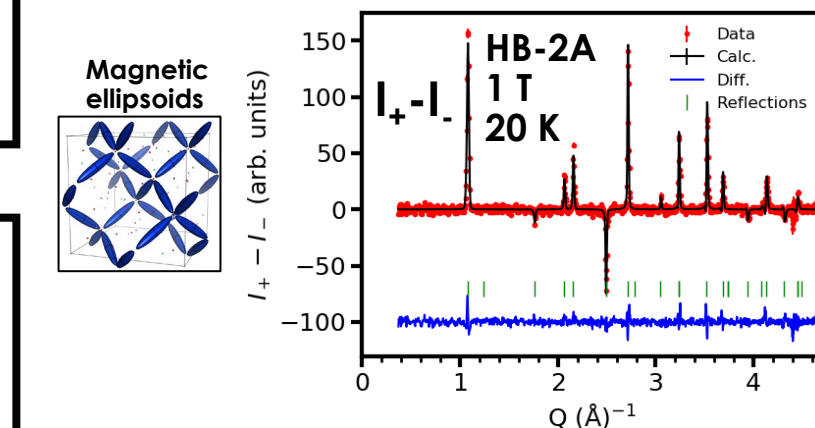
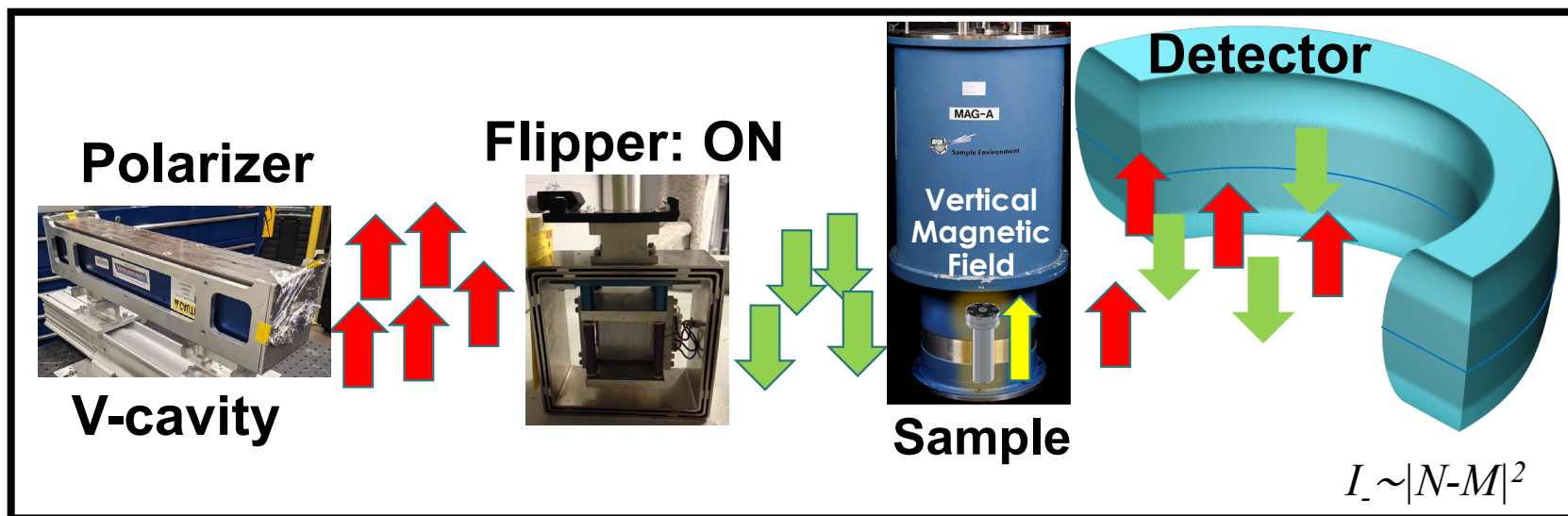
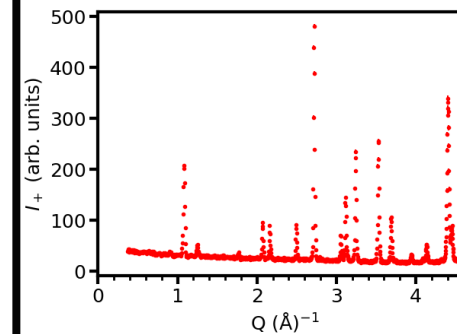
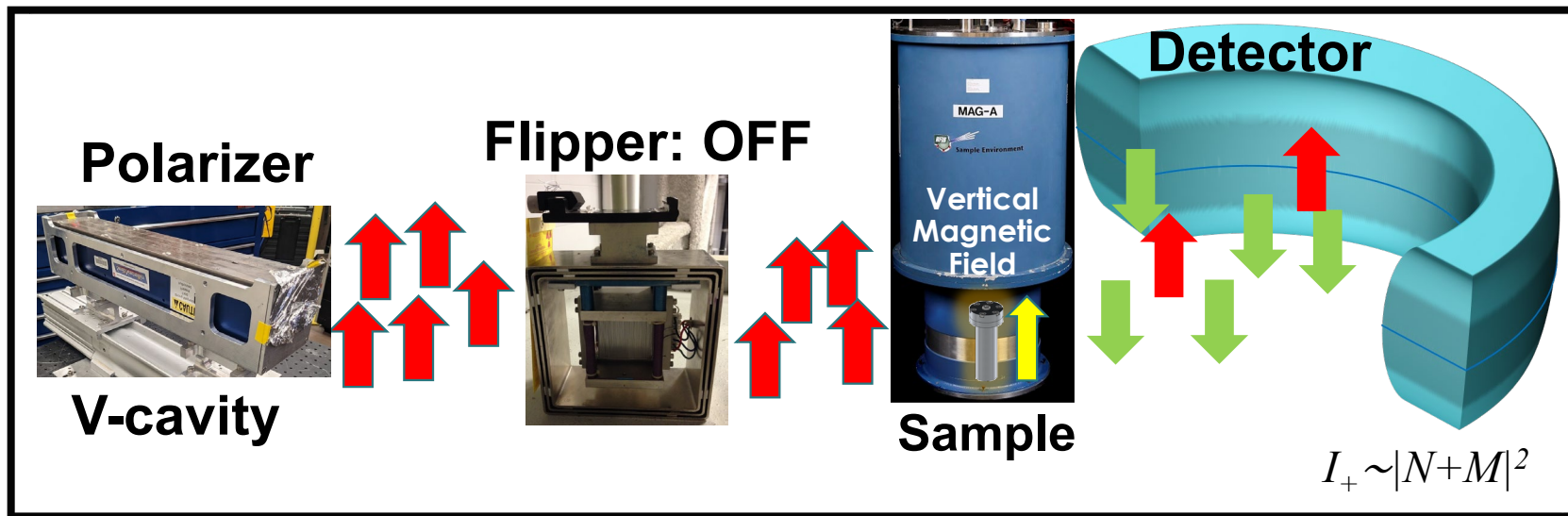
- Datafiles:

- **Ho227_20K_1T_Iplus_Iminus.dat**
 - **Ho227_0T.dat**

- Crystallographic Information File: **Ho227_crystal_structure.cif**

Name	
 Backup	
 Ho227_HB2A_other_temperature_data	
 Ho227_0T_data	
 Ho227_20K_1T_Iplus_Iminus	
 Ho227_crystal_structure	

Half-polarized Neutron Powder Diffraction: HB-2A



Ho₂Ti₂O₇ Powder Example

- This example will use crysPy to determine the local site susceptibility in the following steps:
 - Step 1: Installing and running CrysPy
 - Step 2: Refine unpolarized, non-magnetic Ho₂Ti₂O₇ crystal structure using CrysPy
 - Step 3: Refine the polarized Ho₂Ti₂O₇ data using CrysPy
 - Step 4: Interpreting the results

Ho₂Ti₂O₇ Powder Example

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 - Step 4: Interpreting the results

Step 1: Installing and running CrysPy <https://sites.google.com/view/cryspy/>

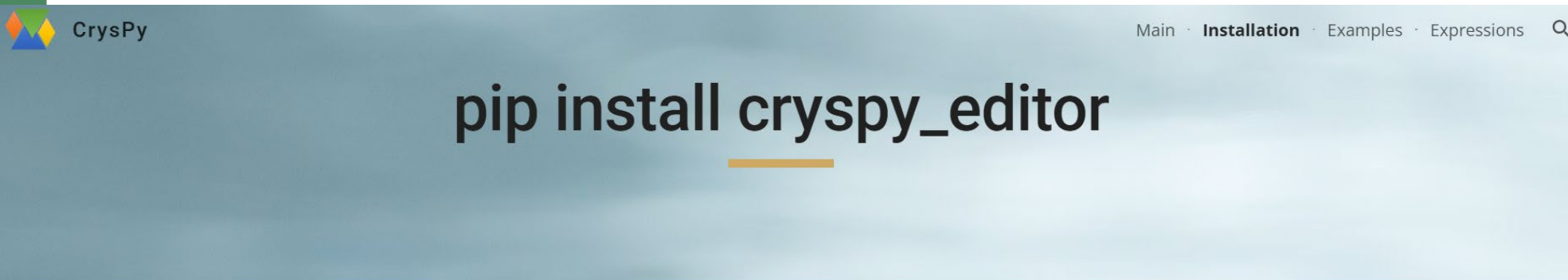


Iurii Kibalin

CrysPy can refine polarized as well as unpolarized data on both powders and single crystals.

Neutron and x-ray data.

Python based with GUI



Stable version (PIP)

The stable version of CrysPy and "CrysPy Editor" can be downloaded through pip (python 3 has to be installed):

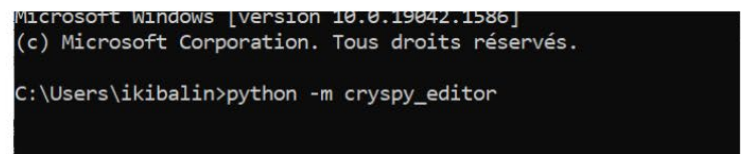
```
python -m pip install cryspy_editor
```

to upgrade the library type:

```
python -m pip install --upgrade cryspy cryspy_editor
```

To run "CrysPy editor" type in console:

```
python -m cryspy_editor
```

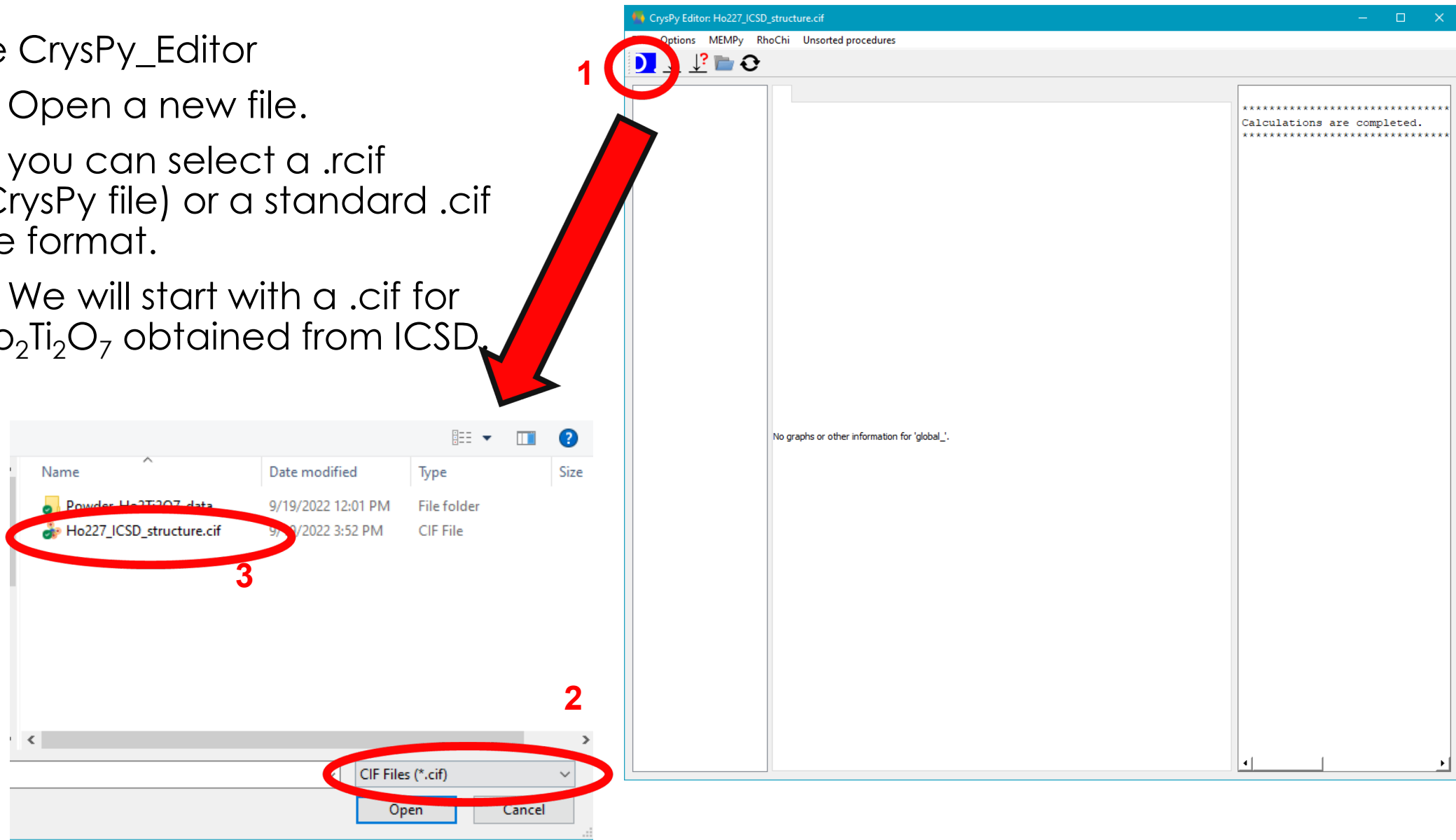


Ho₂Ti₂O₇ Powder Example

- This example will use CrysPy to determine the local site susceptibility in the following steps:
 - Step 1: Installing and running CrysPy
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 - Step 4: Interpreting the results

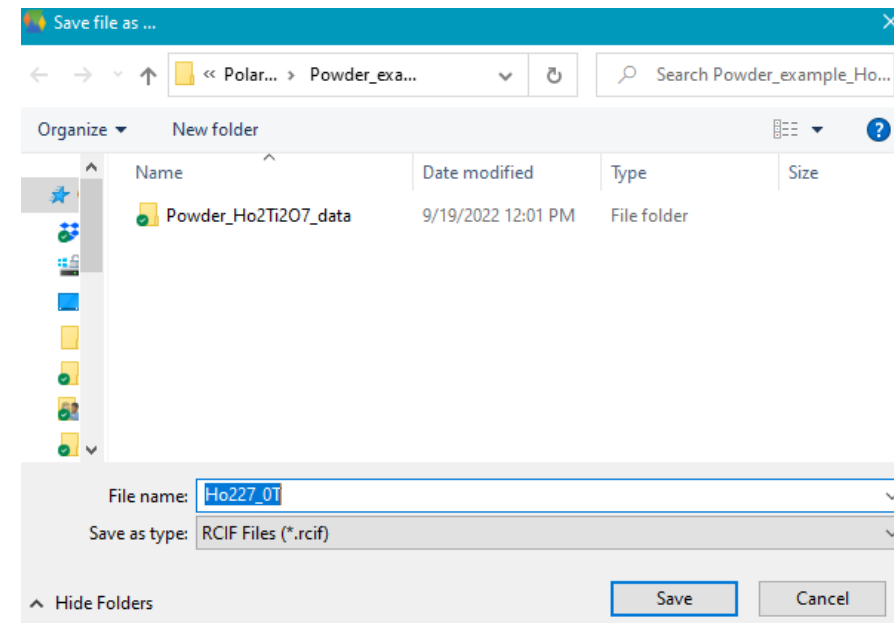
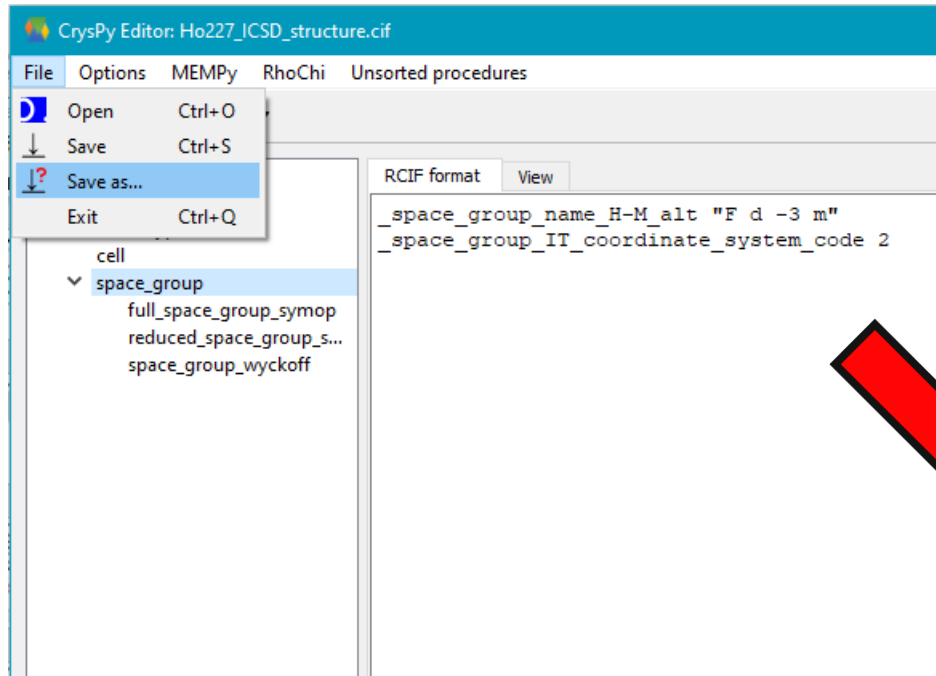
Step 2: Refine the unpolarized, non-magnetic crystal structure using CrysPy

- In the CrysPy_Editor
 - **1.** Open a new file.
 - **2.** you can select a .rcif (CrysPy file) or a standard .cif file format.
 - **3.** We will start with a .cif for $\text{Ho}_2\text{Ti}_2\text{O}_7$ obtained from ICSD.



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- Save this in .rcif format



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- This structure parameters are loaded into the crysPy_Editor from the cif

- Right click on the name "crystal_252236-icsd".
- Renaming this will make things easier later.
- Call it something simple like "Phase 1"

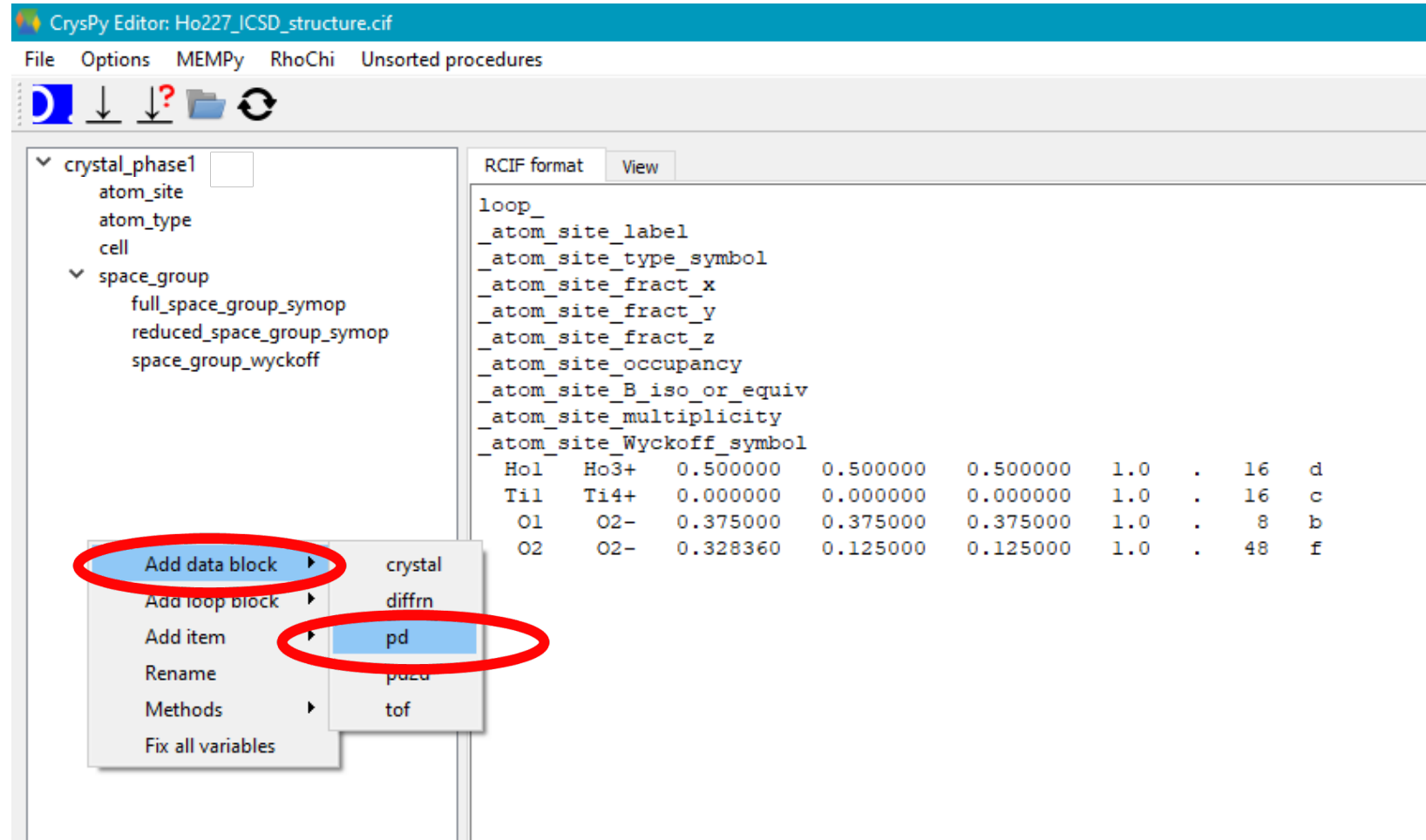
The image shows two screenshots of the crysPy Editor interface. The top screenshot shows the file 'crystal_252236-icsd' selected in the left-hand tree view. A red circle highlights the file name, and a red arrow points to an 'Input dialog' window. The dialog has a text field containing 'Phase 1' and 'OK' and 'Cancel' buttons. The bottom screenshot shows the same interface after the file has been renamed to 'crystal_phase1', which is also circled in red. The right-hand pane displays the RCIF format data for the structure.

loop_	_atom_site_label	_atom_site_type_symbol	_atom_site_fract_x	_atom_site_fract_y	_atom_site_fract_z	_atom_site_occupancy	_atom_site_B_iso_or_equiv	_atom_site_multiplicity	_atom_site_Wyckoff_symbol
	Hol	Ho3+	0.500000	0.500000	0.500000	1.0	.	16	d
	Til	Ti4+	0.000000	0.000000	0.000000	1.0	.	16	c
			0	0.375000	0.375000	1.0	.	8	b
			0	0.125000	0.125000	1.0	.	48	f

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- We now want to add the data and instrument parameters to refine the data.

- 1.** Right click on the left hand panel
→ Add data block
→ pd



CrysPy Editor: Ho227_ICSD_structure.cif

File Options MEMPy RhoChi Unsorted procedures

crystal_phase1

- atom_site
- atom_type
- cell
- space_group
 - full_space_group_symop
 - reduced_space_group_symop
 - space_group_wyckoff

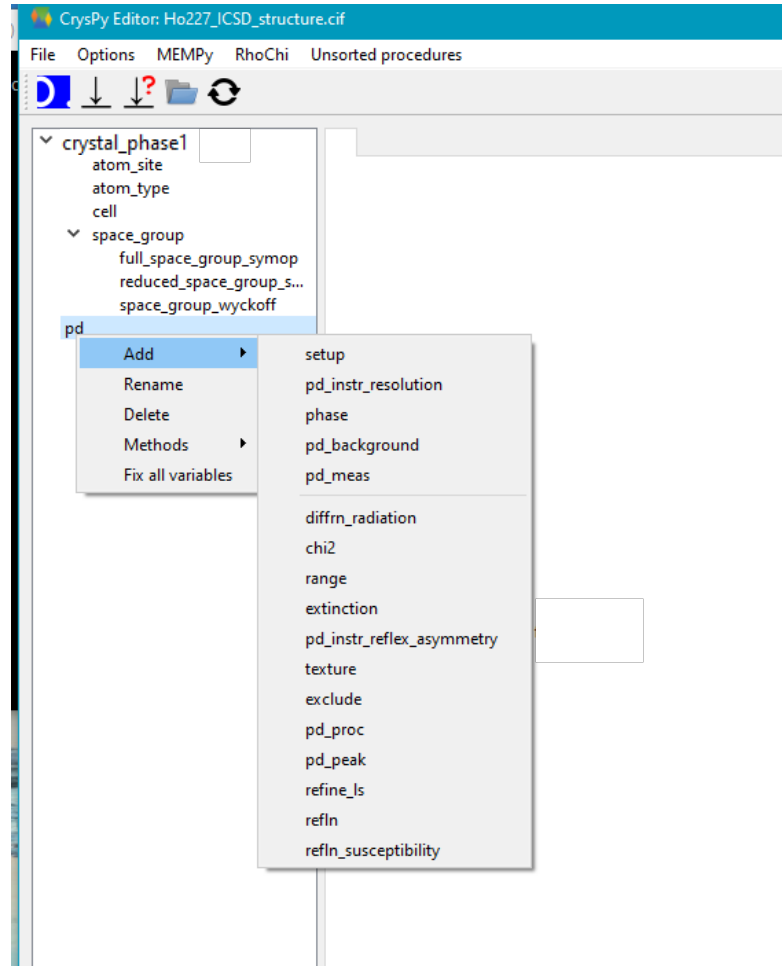
RCIF format View

```
loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_B_iso_or_equiv
_atom_site_multiplicity
_atom_site_Wyckoff_symbol
Ho1 Ho3+ 0.500000 0.500000 0.500000 1.0 . 16 d
Ti1 Ti4+ 0.000000 0.000000 0.000000 1.0 . 16 c
O1 O2- 0.375000 0.375000 0.375000 1.0 . 8 b
O2 O2- 0.328360 0.125000 0.125000 1.0 . 48 f
```

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

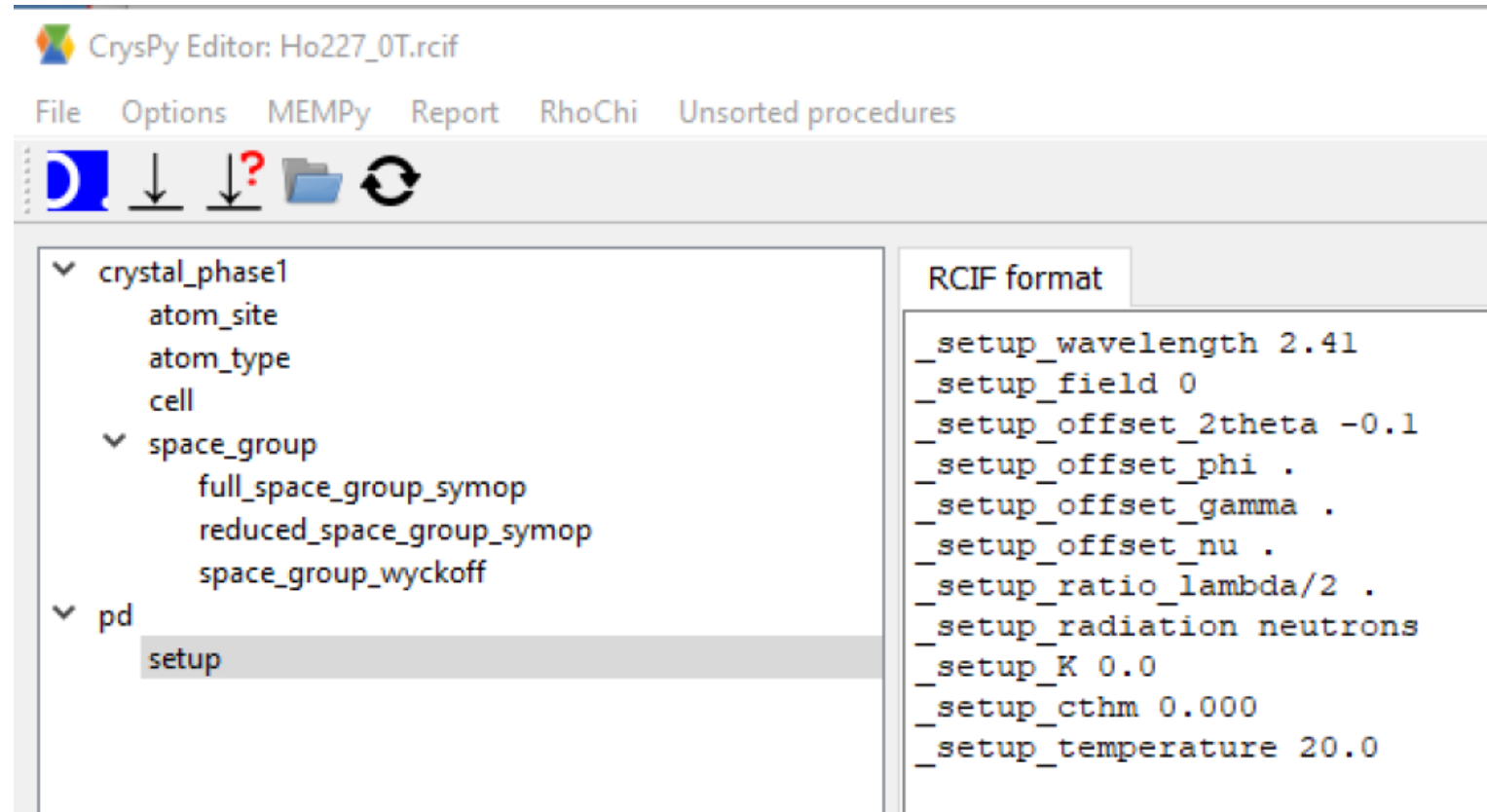
- We now want to add the data and instrument parameters to refine the data.

- Right click on “pd” to select the subcategories.
- We will add these one at a time.



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **setup**
- We will first refine the zero-field data. So set field "0"
- The wavelength used on HB-2A was 2.41
- From calibration measurements the 2theta offset of the detector was -0.1
 - This should be discussed with instrument team



The screenshot shows the CrysPy Editor interface for the file 'Ho227_0T.rcif'. The menu bar includes 'File', 'Options', 'MEMPy', 'Report', 'RhoChi', and 'Unsorted procedures'. The toolbar contains icons for opening, saving, and refreshing. The main window is divided into two panes. The left pane shows a tree view of the configuration with the following structure:

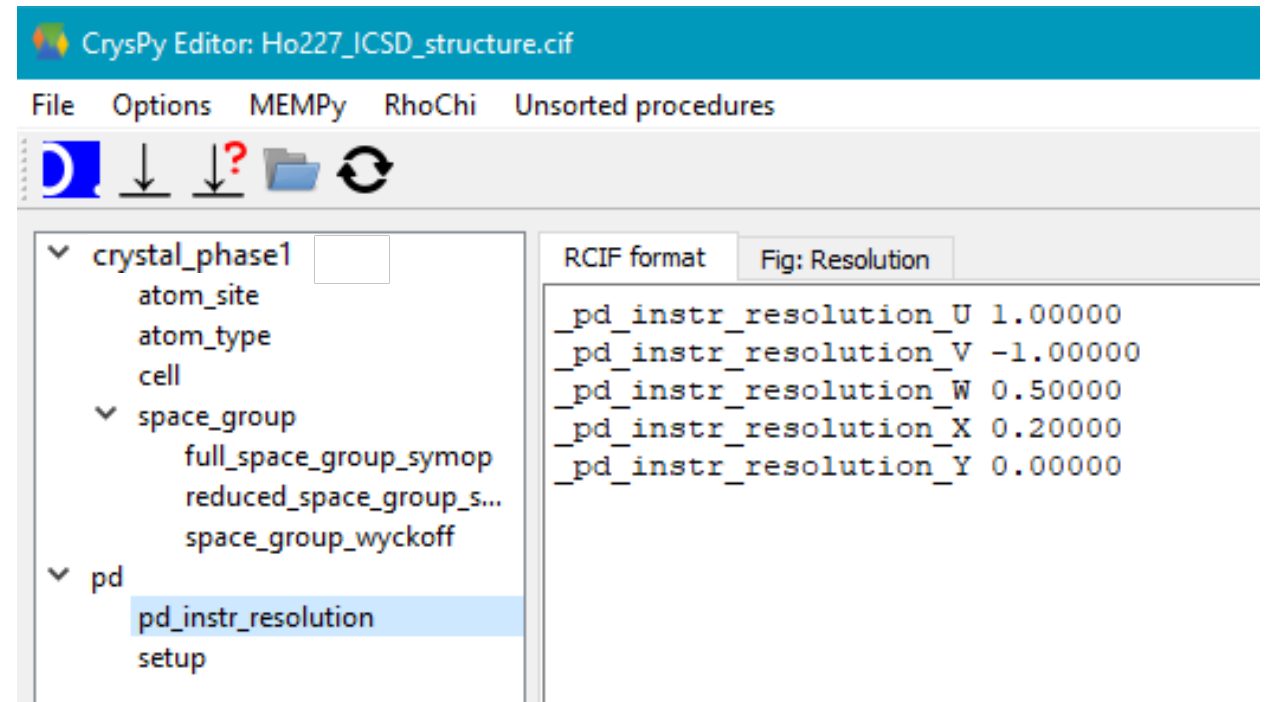
- crystal_phase1
 - atom_site
 - atom_type
 - cell
 - space_group
 - full_space_group_symop
 - reduced_space_group_symop
 - space_group_wyckoff
- pd
 - setup

The right pane, titled 'RCIF format', displays the following configuration parameters:

```
_setup_wavelength 2.41
_setup_field 0
_setup_offset_2theta -0.1
_setup_offset_phi .
_setup_offset_gamma .
_setup_offset_nu .
_setup_ratio_lambda/2 .
_setup_radiation neutrons
_setup_K 0.0
_setup_cthm 0.000
_setup_temperature 20.0
```

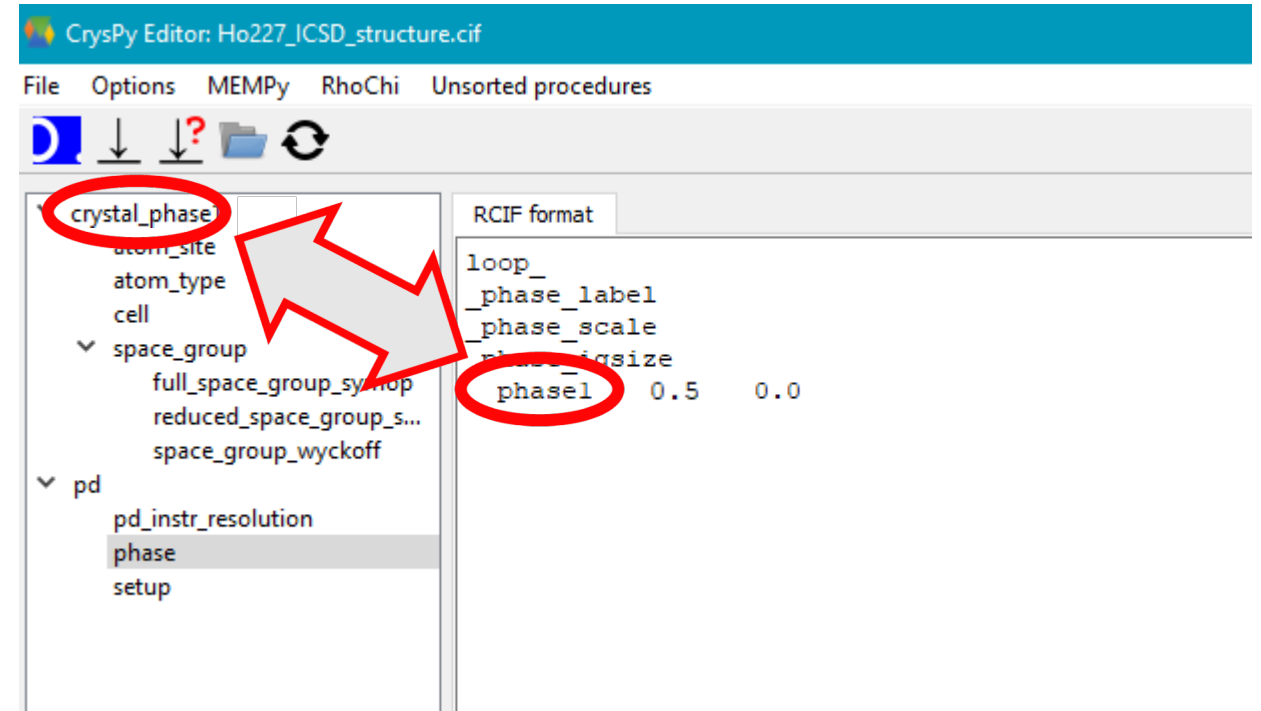
Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **pd_instr_resolution**
- Starting profile peak shape parameters.
- These will depend on instrument.
 - Ask instrument scientist for reasonable values or calibration data



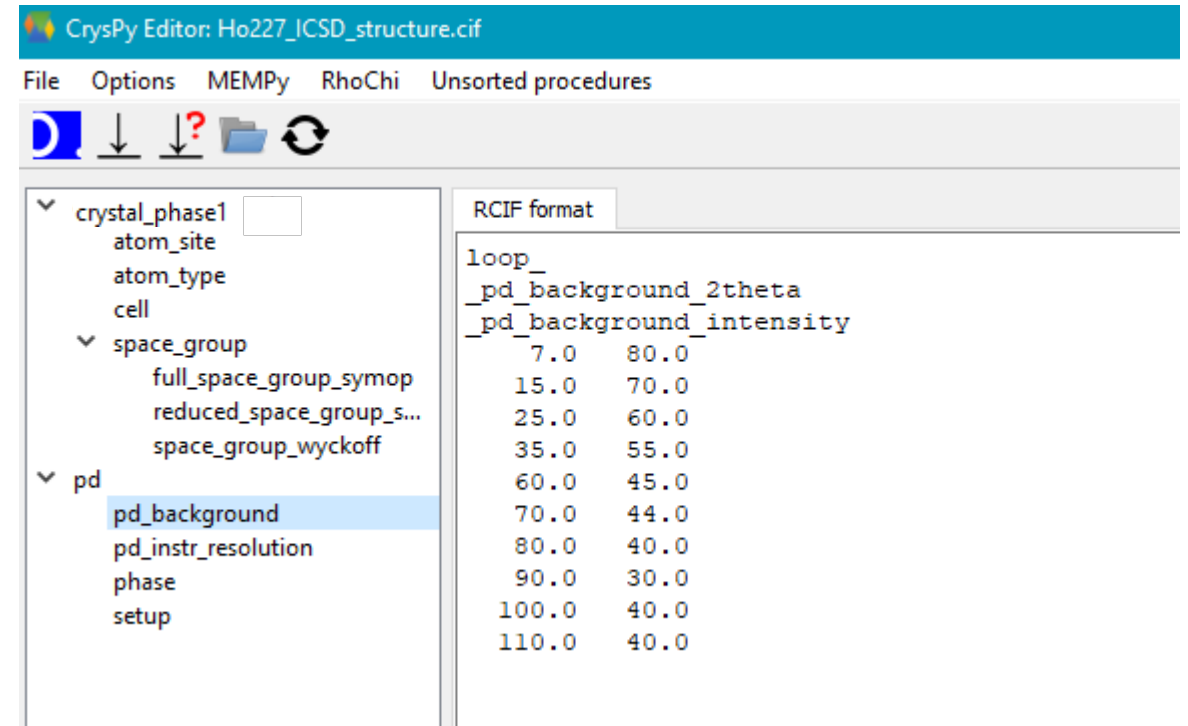
Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **phase**
- Make sure the phase_label matches the crystal name!!!!
 - “phase1”
- The starting value for the scale is based on a best guess. This will be refined.



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **pd_background**
- Check the data file and put in some reasonable background starting values.
- *NOTE: Be careful not to refine a 2theta position that is excluded.*



CrysPy Editor: Ho227_ICSD_structure.cif

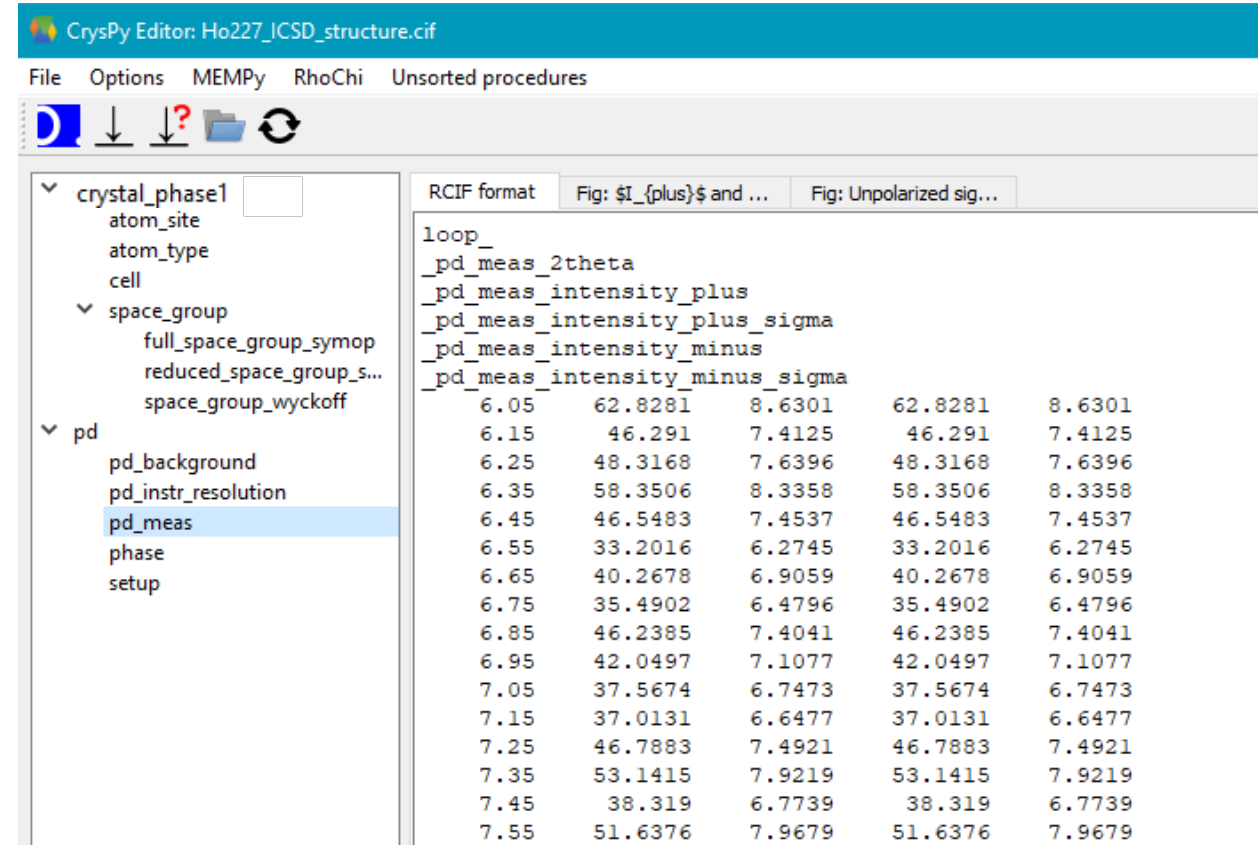
File Options MEMPy RhoChi Unsorted procedures

RCIF format

Parameter	Value 1	Value 2
loop_		
_pd_background_2theta		
_pd_background_intensity		
7.0	80.0	
15.0	70.0	
25.0	60.0	
35.0	55.0	
60.0	45.0	
70.0	44.0	
80.0	40.0	
90.0	30.0	
100.0	40.0	
110.0	40.0	

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **pd_meas**
- Add the data. The 0T data is saved in the example folder and named “Ho227_0T_data.dat”.
- Copy and paste the data over everything there in the “pd_meas” tab
- NOTE: we add both plus and minus data, even though this is 0T. This makes it easier to use this 0T refined phase and background values for the actual polarized data in STEP 3.



CrysPy Editor: Ho227_ICSD_structure.cif

File Options MEMPy RhoChi Unsorted procedures

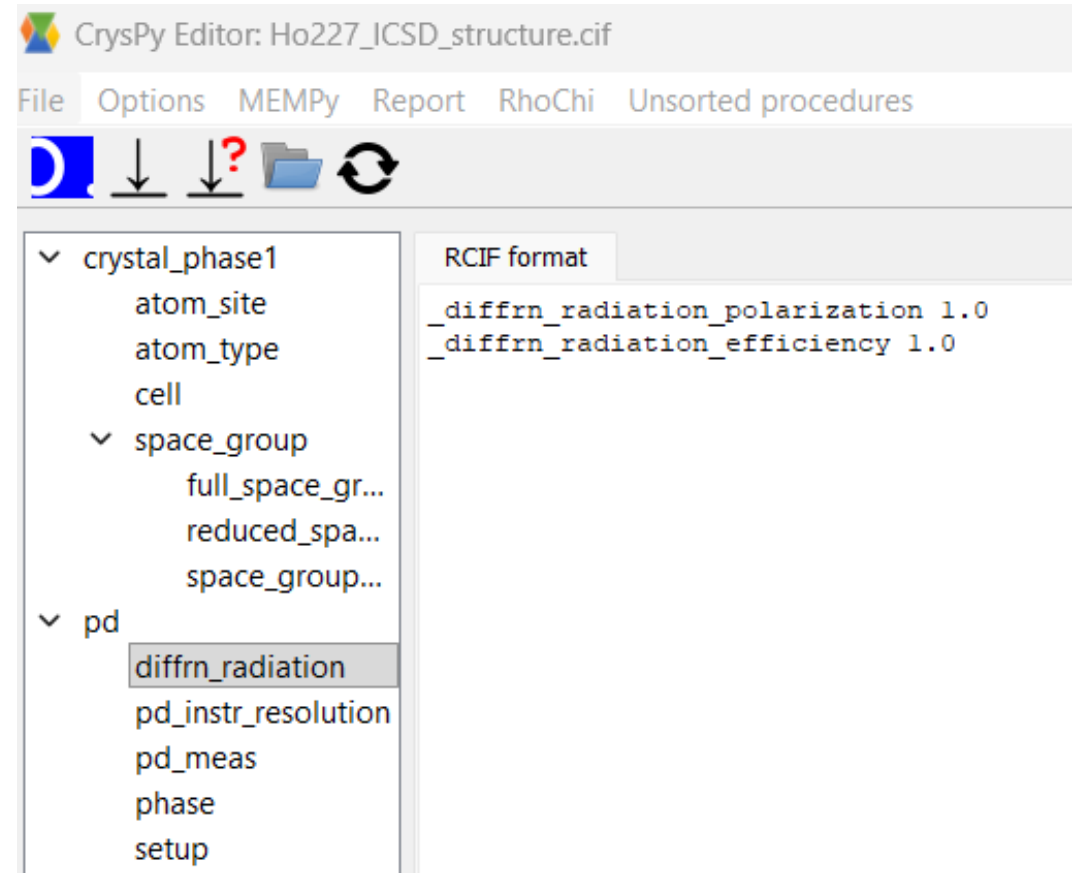
RCIF format Fig: \$I_{plus}\$ and ... Fig: Unpolarized sig...

loop_					
_pd_meas_2theta					
_pd_meas_intensity_plus					
_pd_meas_intensity_plus_sigma					
_pd_meas_intensity_minus					
_pd_meas_intensity_minus_sigma					
	6.05	62.8281	8.6301	62.8281	8.6301
	6.15	46.291	7.4125	46.291	7.4125
pd_background	6.25	48.3168	7.6396	48.3168	7.6396
pd_instr_resolution	6.35	58.3506	8.3358	58.3506	8.3358
pd_meas	6.45	46.5483	7.4537	46.5483	7.4537
phase	6.55	33.2016	6.2745	33.2016	6.2745
setup	6.65	40.2678	6.9059	40.2678	6.9059
	6.75	35.4902	6.4796	35.4902	6.4796
	6.85	46.2385	7.4041	46.2385	7.4041
	6.95	42.0497	7.1077	42.0497	7.1077
	7.05	37.5674	6.7473	37.5674	6.7473
	7.15	37.0131	6.6477	37.0131	6.6477
	7.25	46.7883	7.4921	46.7883	7.4921
	7.35	53.1415	7.9219	53.1415	7.9219
	7.45	38.319	6.7739	38.319	6.7739
	7.55	51.6376	7.9679	51.6376	7.9679

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

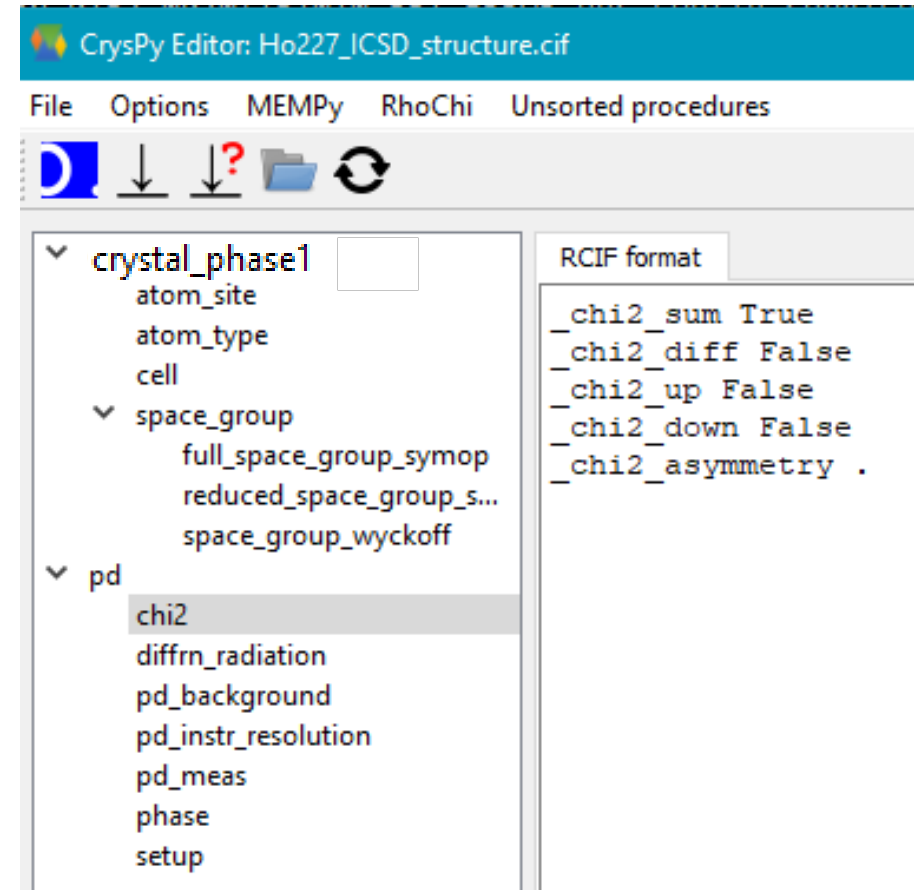
- **pd** → **diffrn_radiation**

- This is for the beam polarization.
- The incident beam polarization should be known for the specific instrument.
- *NOTE: for cases where the sample depolarizes the beam this can be refined or even better experimentally determined.*
- The data in step 2 data is 0 T and polarization is not being considered. So set to standard values shown.
 - We will discuss this further when looking at the polarized data in Step 3



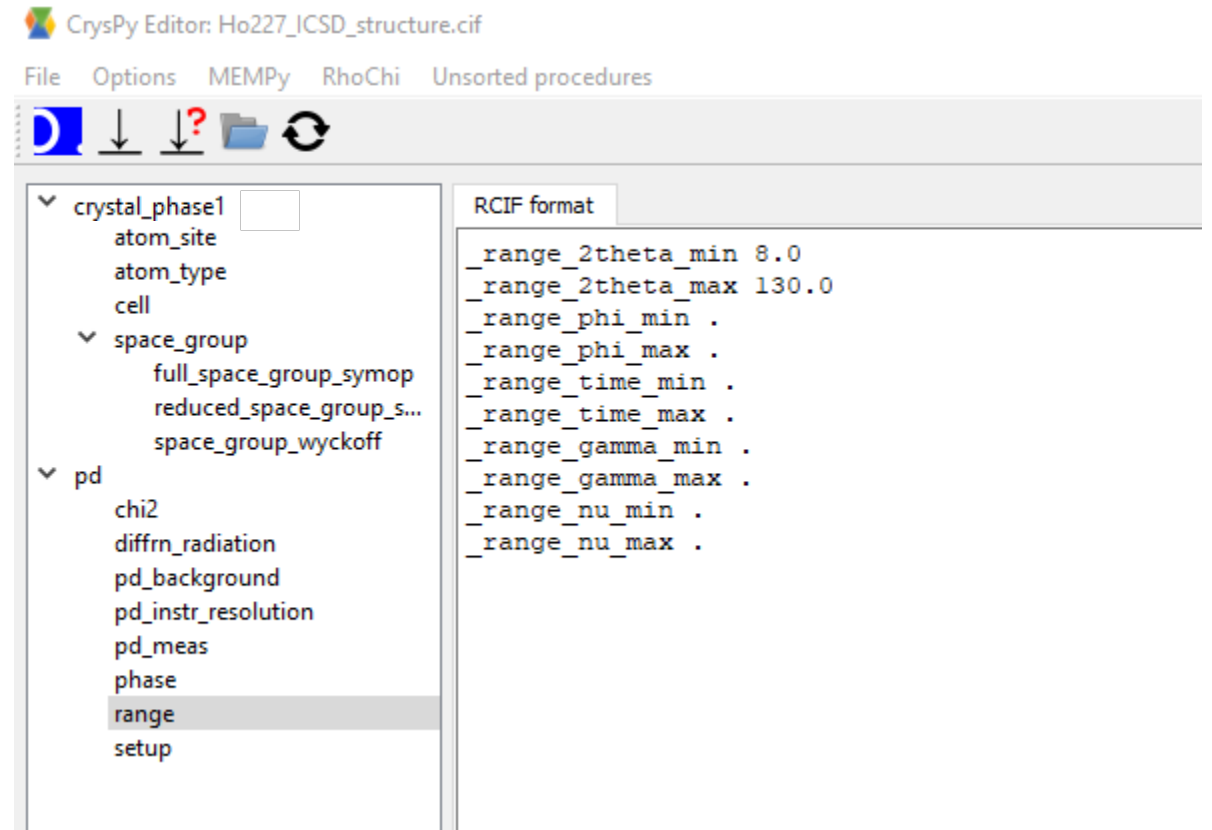
Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **chi2**
- This allows you to flag which data is refined.
 - `chi2_sum` is minus + plus (so recovers the unpolarized data)
 - `chi2_diff` is the difference. This will be used in STEP 3 when refining the polarized data.



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → range
- Range of data to consider



CrysPy Editor: Ho227_ICSD_structure.cif

File Options MEMPy RhoChi Unsorted procedures

Crystal structure parameters:

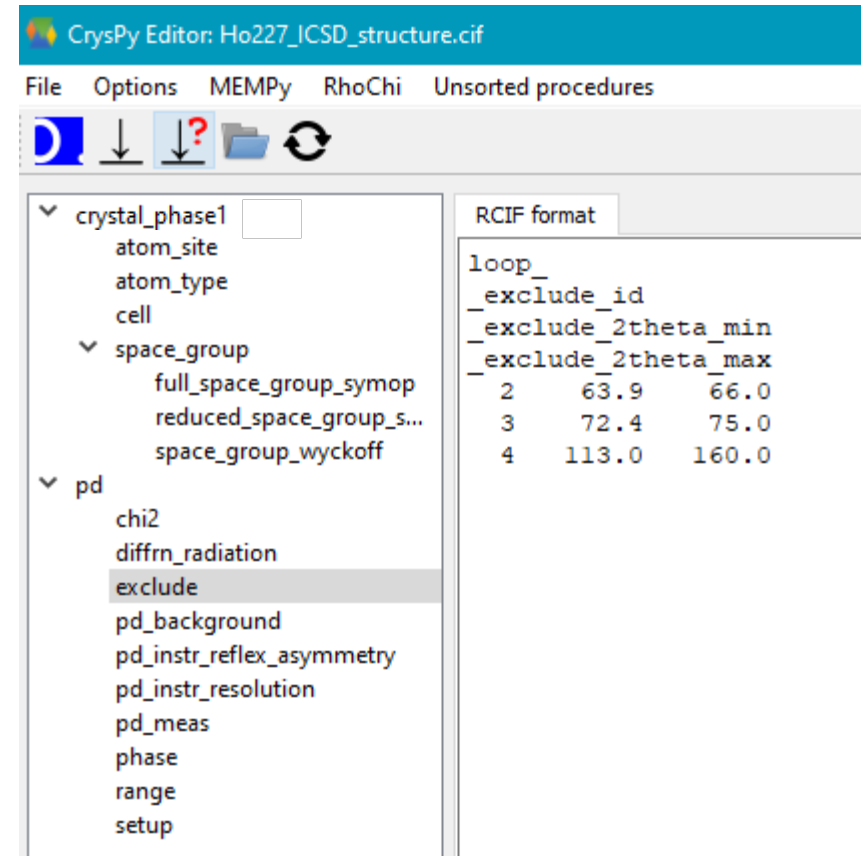
- crystal_phase1
- atom_site
- atom_type
- cell
- space_group
 - full_space_group_symop
 - reduced_space_group_s...
 - space_group_wyckoff
- pd
 - chi2
 - diffrn_radiation
 - pd_background
 - pd_instr_resolution
 - pd_meas
 - phase
 - range**
 - setup

RCIF format

```
_range_2theta_min 8.0
_range_2theta_max 130.0
_range_phi_min .
_range_phi_max .
_range_time_min .
_range_time_max .
_range_gamma_min .
_range_gamma_max .
_range_nu_min .
_range_nu_max .
```

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- **pd** → **exclude**
- If there are regions of the data that you want to exclude, this is added here.
- *NOTE: it is best to refine all the data. But in this case we will remove the peaks from the Al sample can*



The screenshot shows the CrysPy Editor interface for the file 'Ho227_ICSD_structure.cif'. The 'pd' section is expanded, and the 'exclude' option is selected. The 'RCIF format' panel on the right displays the following data:

loop_	_exclude_id	_exclude_2theta_min	_exclude_2theta_max
	2	63.9	66.0
	3	72.4	75.0
	4	113.0	160.0

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- This is the end of the subcategories that you need to add.

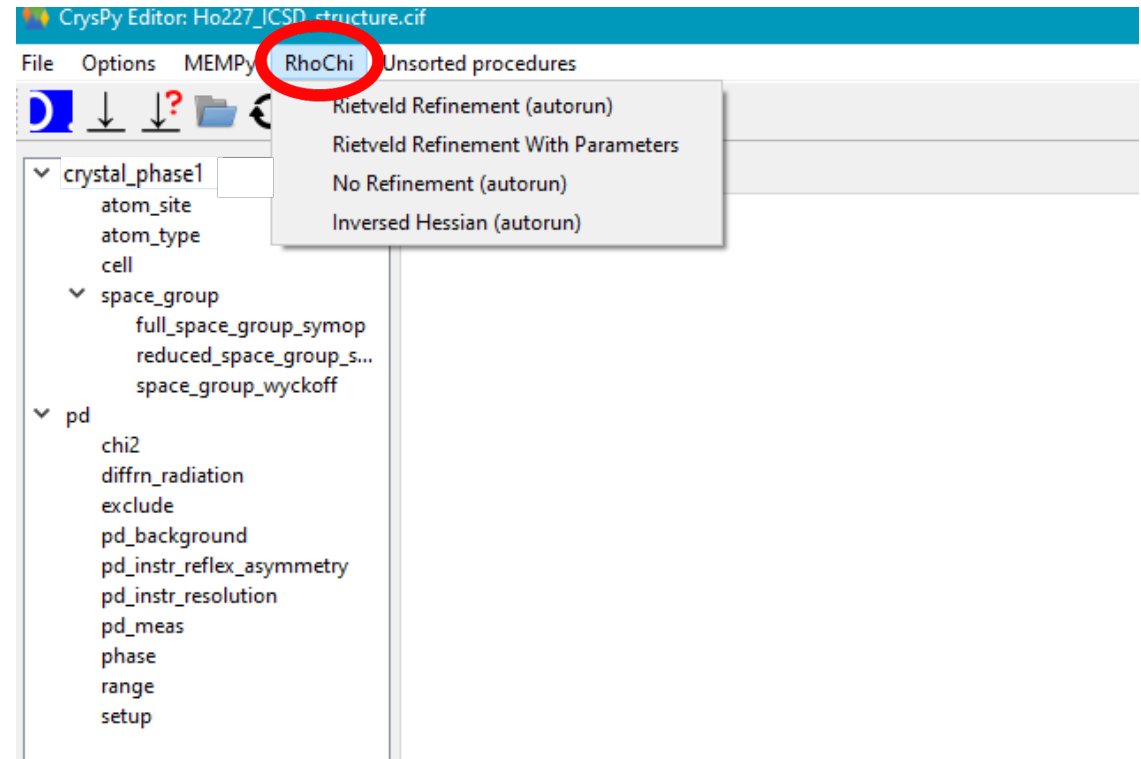
- Hit the save button



- When you run crysPy more subcategories will appear.
- These contain information on the model (reflection list, goodness of fit, etc).

Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

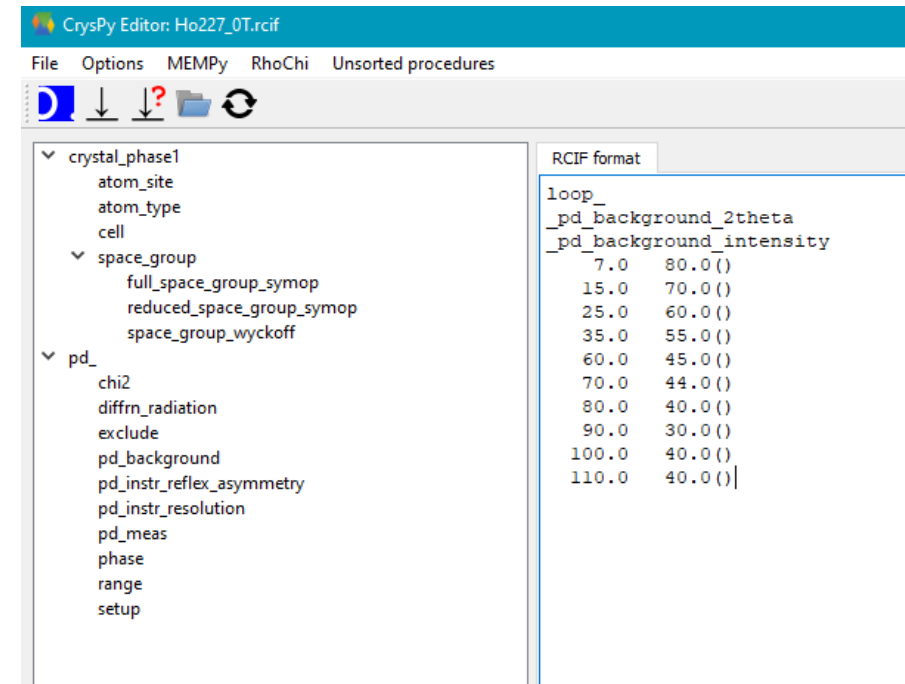
- The refinement is run using the “RhoChi” drop-down menu
- First run “No Refinement (autorun)” to check if the initial parameters are close.



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- We're now ready to refine the parameters.
- To set a variable to refine put a () after it.
- For categories with lots of values you can right click and select "refine all variables"
 - Do this for the background

NOTE: There might be an error when you run the refinement. But there is a quick fix!



The screenshot shows the CrysPy Editor interface for a file named 'Ho227_0T.rcif'. The interface includes a menu bar with 'File', 'Options', 'MEMPy', 'RhoChi', and 'Unsorted procedures'. Below the menu bar is a toolbar with icons for file operations. The main area is divided into two panes. The left pane shows a tree view of the crystal structure parameters, including 'crystal_phase1', 'atom_site', 'atom_type', 'cell', 'space_group', 'full_space_group_symop', 'reduced_space_group_symop', 'space_group_wyckoff', 'pd_', 'chi2', 'diffn_radiation', 'exclude', 'pd_background', 'pd_instr_reflex_asymmetry', 'pd_instr_resolution', 'pd_meas', 'phase', 'range', and 'setup'. The right pane shows the 'RCIF format' table, which lists the refined values for the parameters. The table has two columns: the parameter name and its value in parentheses.

Parameter	Value
loop_	
_pd_background_2theta	
_pd_background_intensity	
7.0	80.0 ()
15.0	70.0 ()
25.0	60.0 ()
35.0	55.0 ()
60.0	45.0 ()
70.0	44.0 ()
80.0	40.0 ()
90.0	30.0 ()
100.0	40.0 ()
110.0	40.0 ()

Potential bug in this version of CrysPy (March 2026)

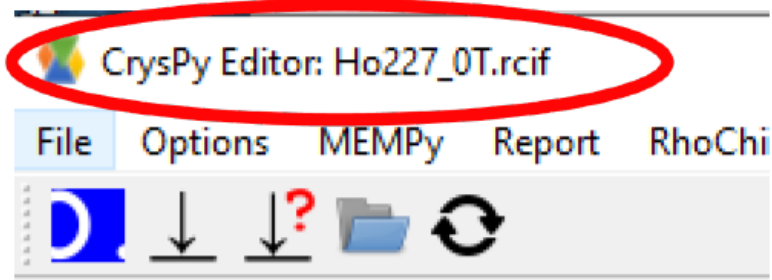
- If you see this error, then reopen the “.rcif” file **[SEE NEXT SLIDE]**

```
*****
Rietveld refinement by CrysPy (module RhoChi)
*****
Derivatives are calculated numerically.
Preliminary calculations...
*****
Calculations are completed.
*****
Result of function is
ERROR DURING PROGRAM EXECUTION

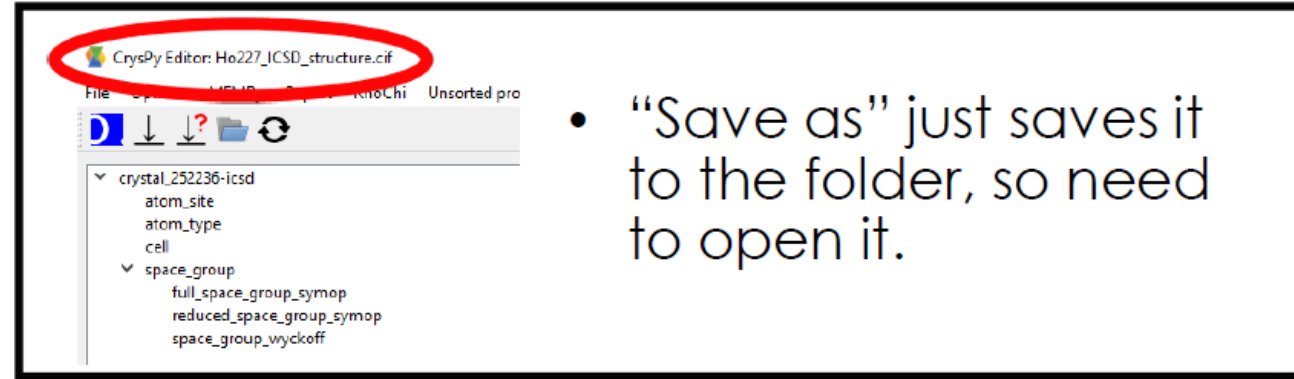
Traceback (most recent call last):
  File "C:\Users\cr9\AppData\Local\anaconda3\lib\site-
packages\crspsy_editor\cl_thread.py", line 127, in run
    out = func(*arg)
  File "C:\Users\cr9\AppData\Local\anaconda3\lib\site-
packages\crspsy\procedure_rhochi\rhochi.py", line 73, in
rhochi_rietveld_refinement
    dict_out = rhochi_rietveld_refinement_with_parameters(
  File "C:\Users\cr9\AppData\Local\anaconda3\lib\site-
packages\crspsy\procedure_rhochi\rhochi.py", line 95, in
rhochi_rietveld_refinement_with_parameters
    chi_sq, parameter_name, dict_in_out, res =
rhochi_rietveld_refinement_by_dictionary(
  File "C:\Users\cr9\AppData\Local\anaconda3\lib\site-
packages\crspsy\procedure_rhochi\rhochi_by_dictionary.py", line 126, in
rhochi_rietveld_refinement_by_dictionary
    print(f"Started chi_sq per number of points is {chi_sq/n_point:.2f}.
")
ZeroDivisionError: division by zero
```

Potential bug in this version of CrysPy (March 2026)

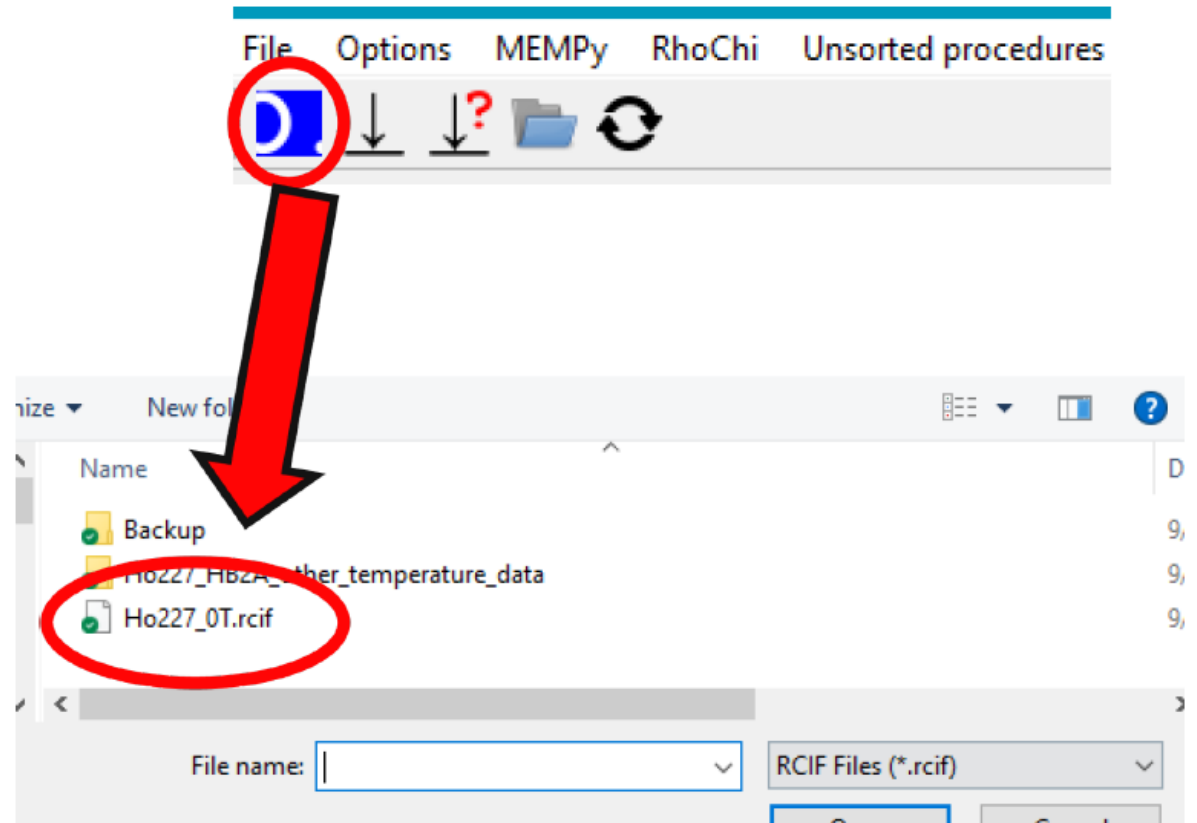
- If the displayed filename ends in “.cif” then you need to open the saved .rcif file
- Now the top left of the program should say:



- *NOTE: It is often easier to edit a known working file when starting a new project, rather than setting everything up from the beginning.*

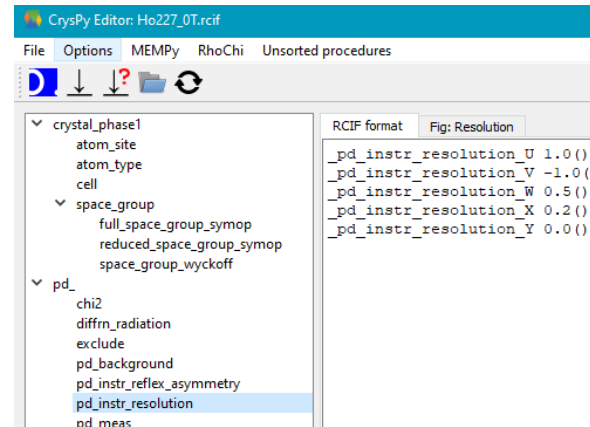
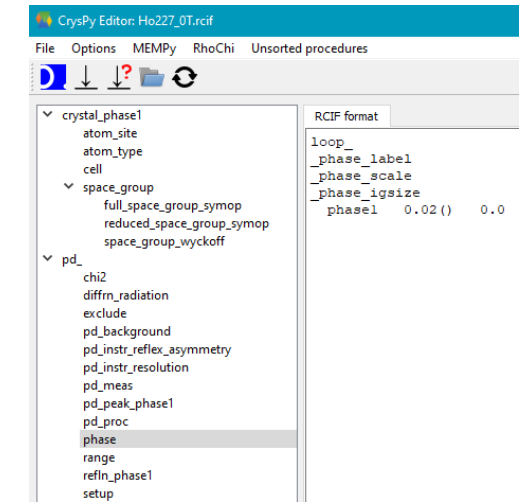
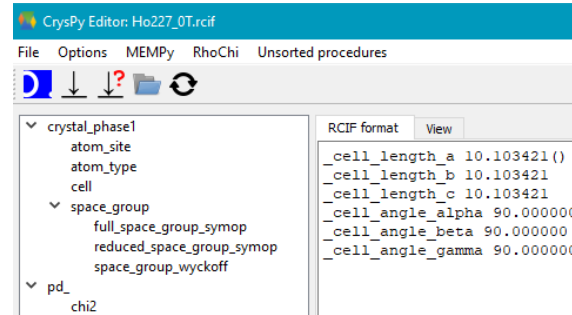


- “Save as” just saves it to the folder, so need to open it.



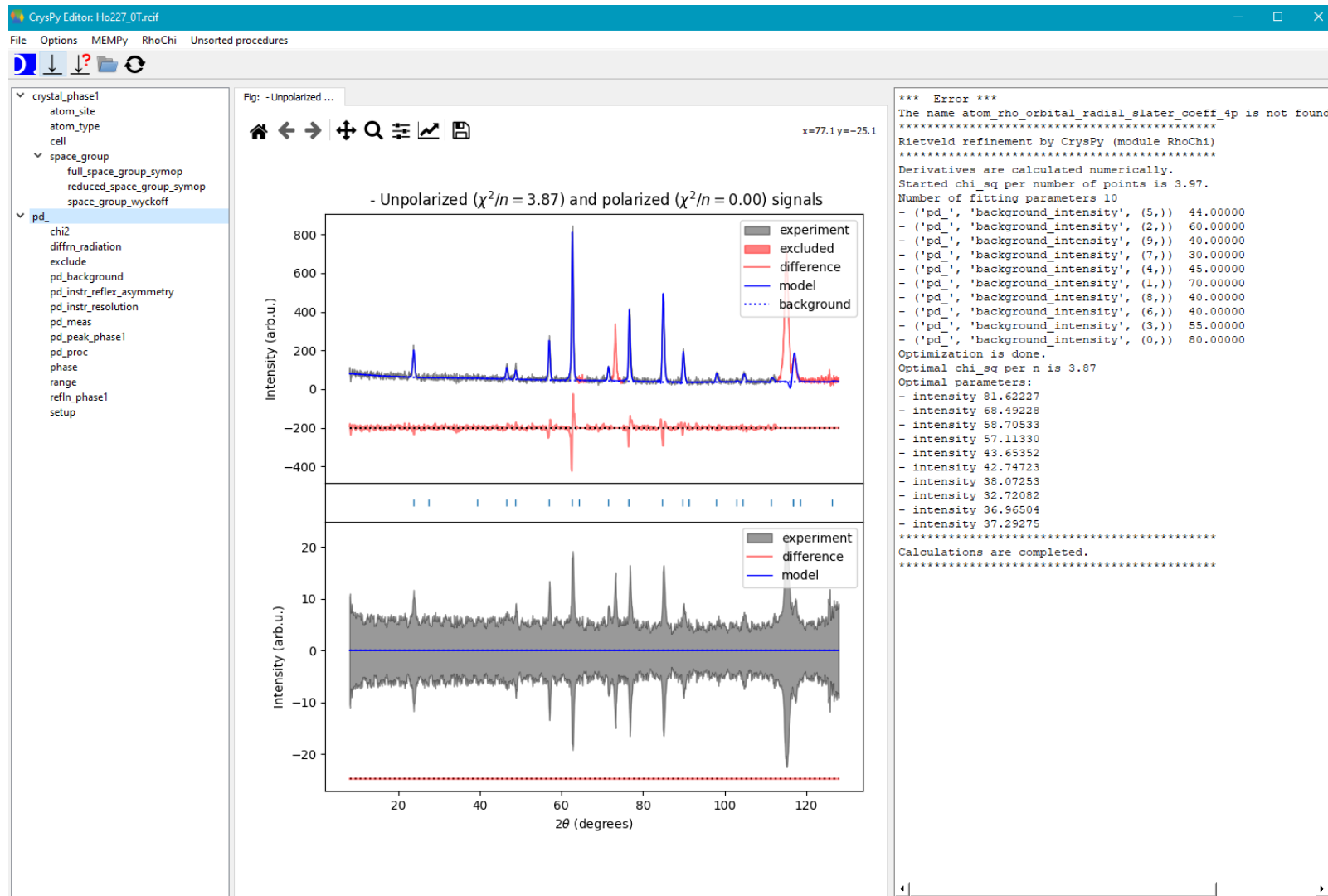
Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- Now refine more parameters to improve the model:
 - Cell (lattice constants)
 - Phase (scale), only refine the first number. Keep second 0.
 - `pd_instr_resolution` (peak shape)
 - Background



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- Run the refinement
 - “RhoChi” → Rietveld Refinement (autorun)
 - The right-hand side should go yellow and show the variables refining.
- Once complete, check the data and model pattern by clicking on the “pd_” category.
- The model and data are very close
- NOTE: The bottom panel (difference) is zero. This is unpolarized data and so is correct!



Step 2: Refine the unpolarized, non-magnetic crystal structure using crysPy

- The fit is very good (by eye and for χ^2).
- Now we can fix (i.e. no longer refine) the instrument parameters (phase, U,V,W,X, etc) when refining the polarized data.
- If this STEP 2 of refining unpolarized zero field data is not done then it can be hard to interpret the polarized difference data.
- This becomes more important the weaker the difference signal or the more change to scattering with an applied magnetic field.

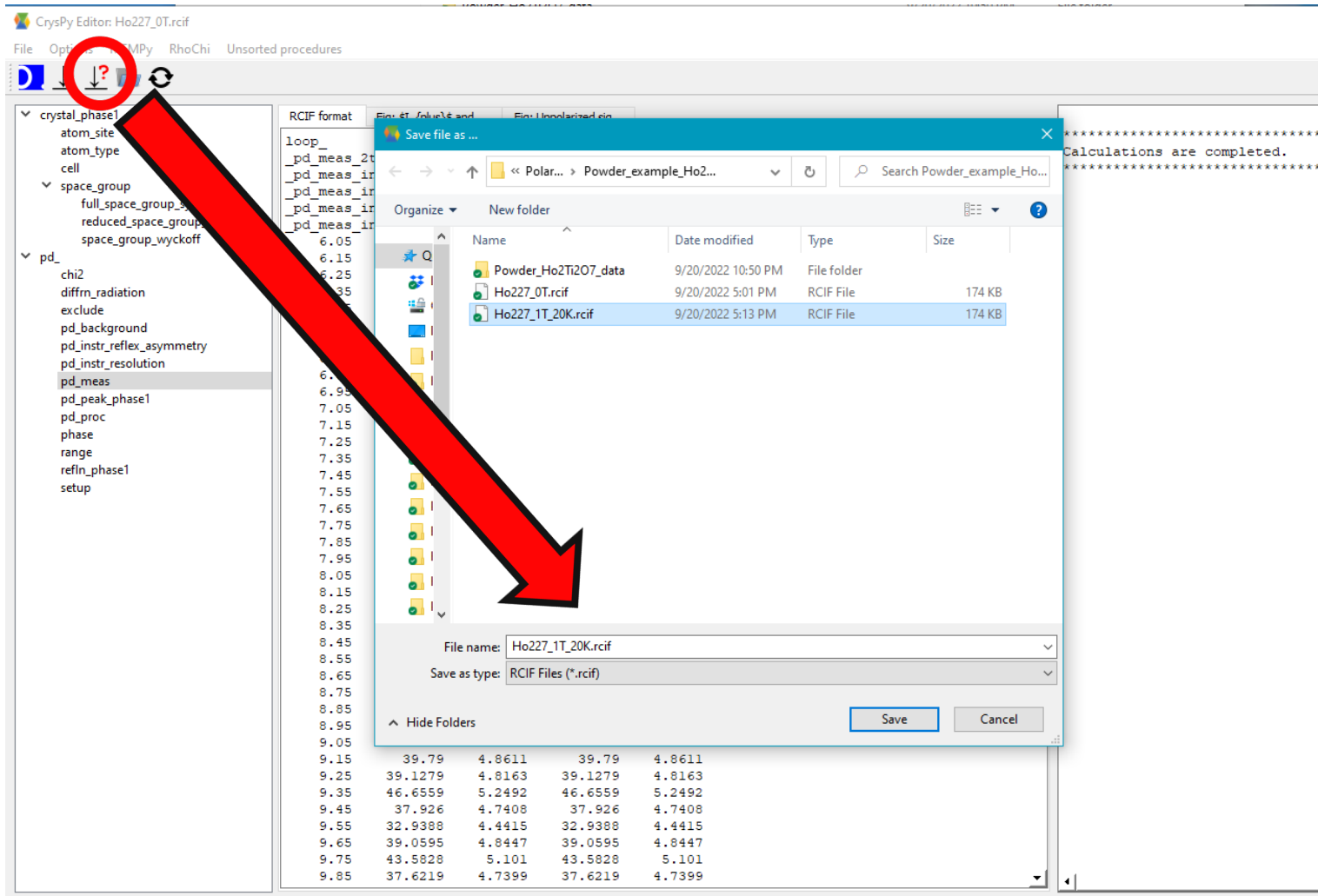


Ho₂Ti₂O₇ Powder Example

- This example will use CrysPy to determine the local site susceptibility in the following steps:
 - Step 1: Installing and running CrysPy
 - Step 2: Refine unpolarized, non-magnetic Ho₂Ti₂O₇ crystal structure using crysPy
 - **Step 3: Refine the polarized Ho₂Ti₂O₇ data using CrysPy**
 - Step 4: Interpreting the results

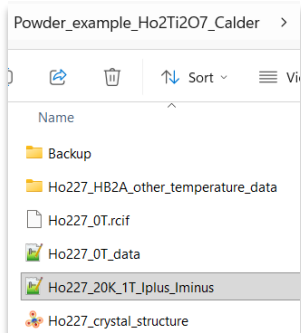
Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- We will start with the .rcif file from STEP 2. Make sure all the refinement label “()” are removed so nothing is refining.
 - This can be done quickly by right clicking on “crystal_phase1” and select “Fix all variables”. Do the same for “pd_”, right click, select “Fix all variables”
- Rename the file to something else. We will call it “Ho227_1T_20K.rcif”
- **NOTE: If the displayed name doesn't change, then open the file (see slide 27 for similar bug)**

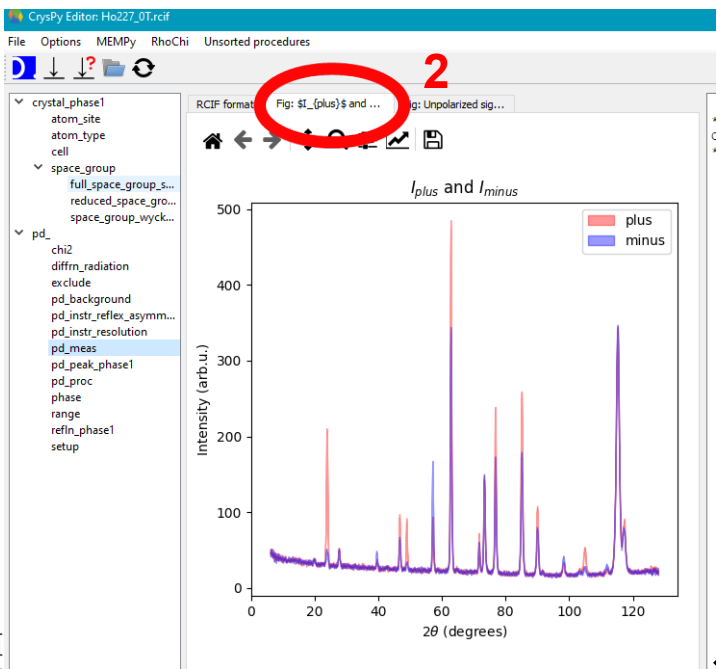


Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- **Add the polarized data**



- **1.** Paste the data in the file “Ho227_20K_1T_Iplus_Iminus.dat” into the **pd_meas** category.
- **2.** You can look at the data in the “Fig:” tabs



CrysPy Editor: Ho227_0T.rcif

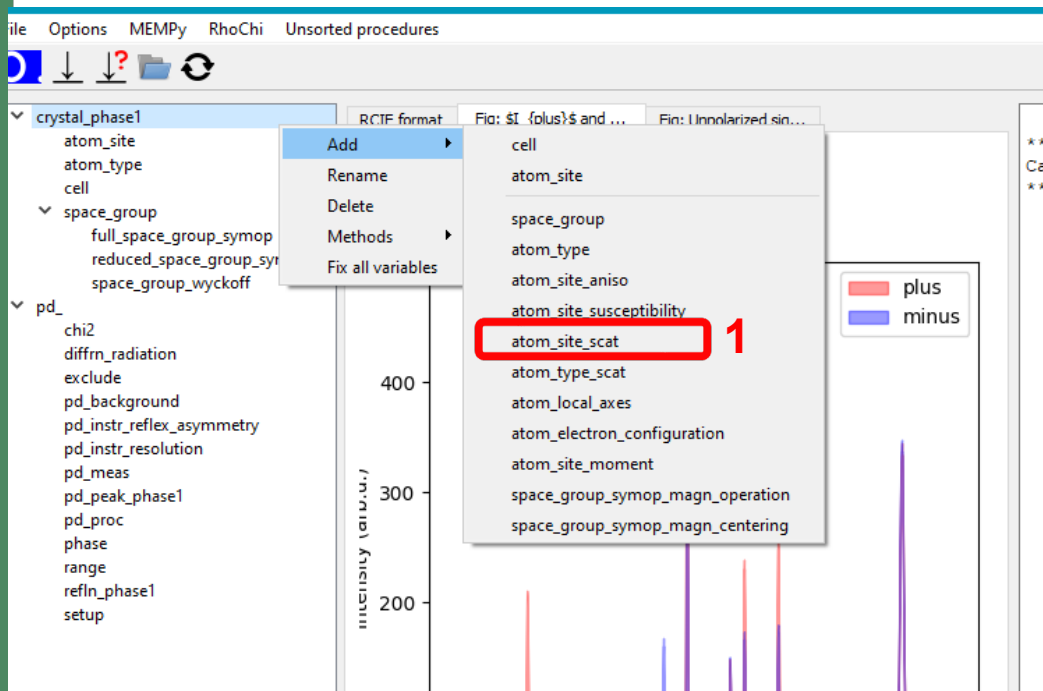
File Options MEMPy RhoChi Unsorted procedures

RCIF format Fig: \$I_{(plus)}\$ and ... Fig: Unpolarized sig...

2θ (degrees)	I _{plus}	I _{minus}	I _{plus} - I _{minus}	I _{plus} + I _{minus}
6.05	48.9707	2.539	45.3246	2.4509
6.15	46.8531	2.4797	43.8996	2.4021
6.25	49.3232	2.5436	46.1803	2.472
6.45	49.3795	2.5499	47.1803	2.4901
6.55	44.8117	2.4338	49.0888	2.5417
6.65	43.4909	2.3941	44.5956	2.4293
6.75	44.0562	2.4143	41.8436	2.3465
6.85	45.8143	2.4594	43.6894	2.4014
6.95	42.5383	2.3669	42.9672	2.3834
7.05	48.292	2.5243	42.5359	2.3668
7.15	45.1823	2.4432	45.44	2.45
7.25	42.7318	2.374	36.8752	2.2077
7.35	41.3968	2.3324	44.8561	2.4327
7.45	38.0564	2.2386	42.3935	2.3662
7.55	44.0705	2.4114	43.5382	2.3967
7.65	39.2861	2.272	42.5519	2.364
7.75	40.7073	2.3158	39.8251	2.2955
7.85	40.1095	2.3004	35.3249	2.1578
7.95	40.2942	2.311	43.6849	2.4011
8.05	38.8663	2.2629	42.9174	2.3806
8.15	41.7386	2.3443	39.1077	2.2693
8.25	40.2918	2.3033	39.1841	2.2737
8.35	43.4978	2.3981	39.1899	2.274
8.45	43.1442	2.3822	39.0625	2.2666
8.55	37.8347	2.2372	38.8147	2.2599

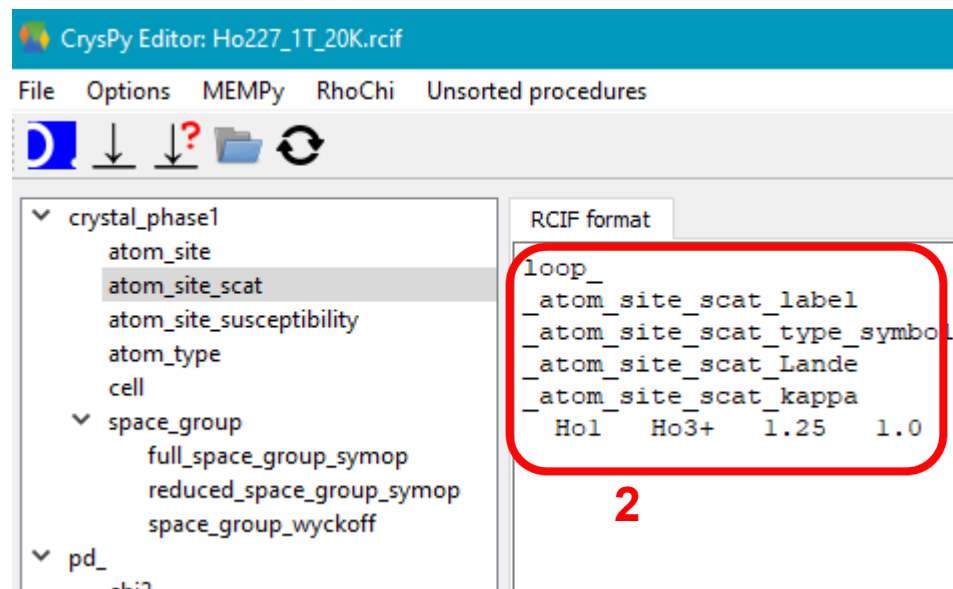
Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- Add the magnetic ion**



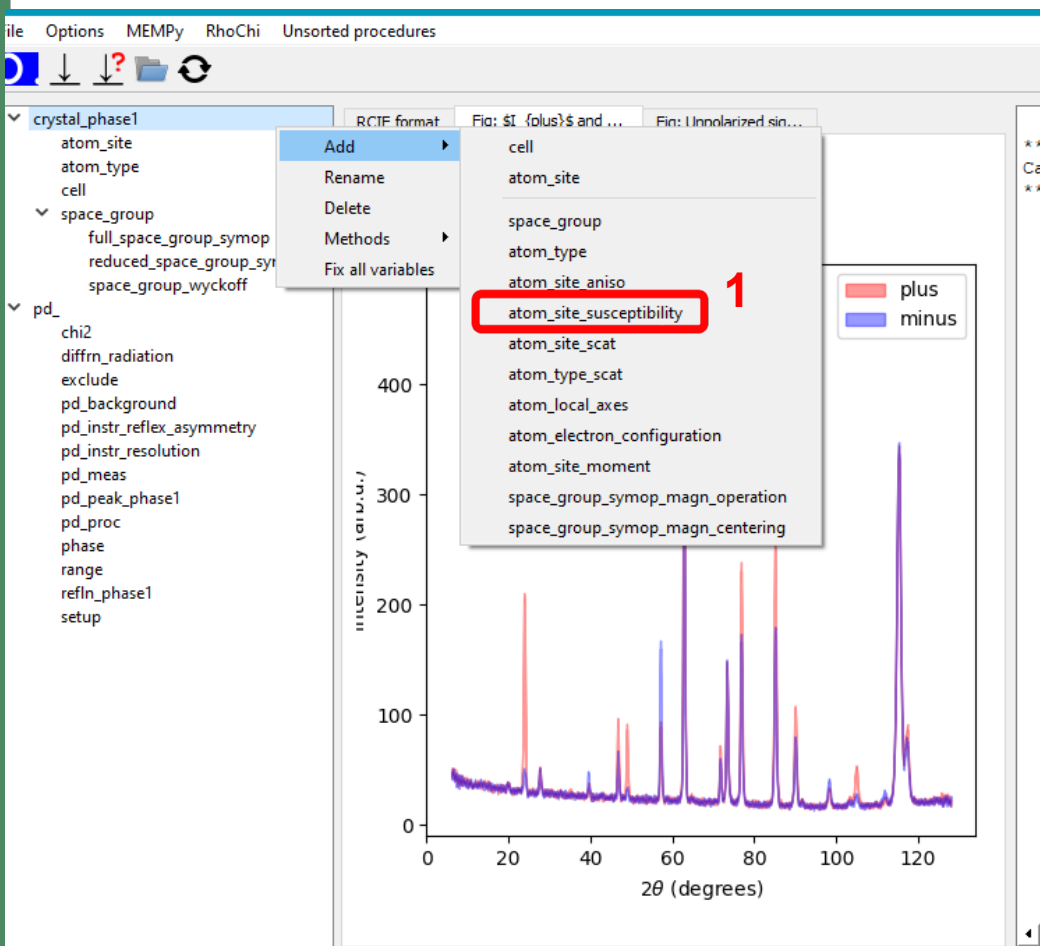
- 1. Right-click on “crystal_phase1” → Add → atom_site_scat
- 2. Put in the Ho ion.
 - The lande-g needs to be looked up. For Ho³⁺ it is 5/4

Ion	4F ⁿ	L	L	J	Ground state	g _J	p th	p ^{exp}
La ³⁺	0	0	0	0	¹ S ₀	0	0	0
Ce ³⁺	1	1/2	3	5/2	² F _{5/2}	6/7	2.535	2.4-2.7
Pr ³⁺	2	1	5	4	³ H ₄	4/5	3.578	3.4-3.6
Nd ³⁺	3	3/2	6	9/2	⁴ I _{9/2}	8/11	3.618	3.4-3.7
Pm ³⁺	4	2	6	4	⁵ I ₄	3/5	2.683	-
Sm ³⁺	5	5/2	5	5/2	⁶ H _{5/2}	2/7	0.845	1.3-1.6
Eu ³⁺	6	3	3	0	⁷ F ₀	0	0	3.2-3.4
Gd ³⁺	7	7/2	0	7/2	⁸ S _{7/2}	2	7.937	7.9-8.0
Tb ³⁺	8	3	3	6	⁷ F ₆	3/2	9.721	9.4-9.8
Dy ³⁺	9	5/2	5	15/2	⁶ H _{15/2}	4/3	10.646	10.5-10.7
Ho ³⁺	10	2	6	8	⁵ I ₈	5/4	10.607	10.3-10.6
Er ³⁺	11	3/2	6	15/2	⁴ I _{15/2}	6/5	9.581	9.4-9.6
Tm ³⁺	12	1	5	6	³ H ₆	7/6	7.561	7.3-7.6
Yb ³⁺	13	1/2	3	7/2	² F _{7/2}	8/7	4.536	4.4-4.6
Lu ³⁺	14	0	0	0	¹ S ₀	0	0	0

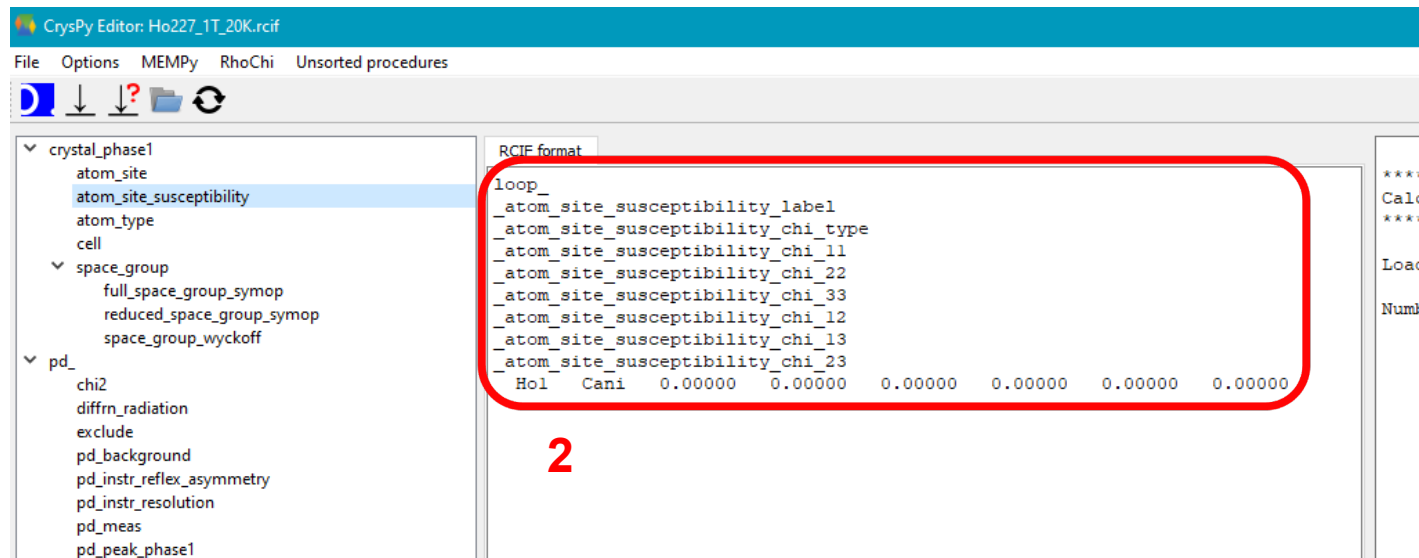


Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- Set-up the site susceptibility refinement for the magnetic ions**



- 1. Right-click on “crystal_phase1” → Add → atom_site_susceptibility
- 2. Put in the Ho ion.
 - C_{ani} will allow for an anisotropic susceptibility tensor. Crispy will only refine symmetry allowed values. C_{iso} is isotropic (only first 3 terms)

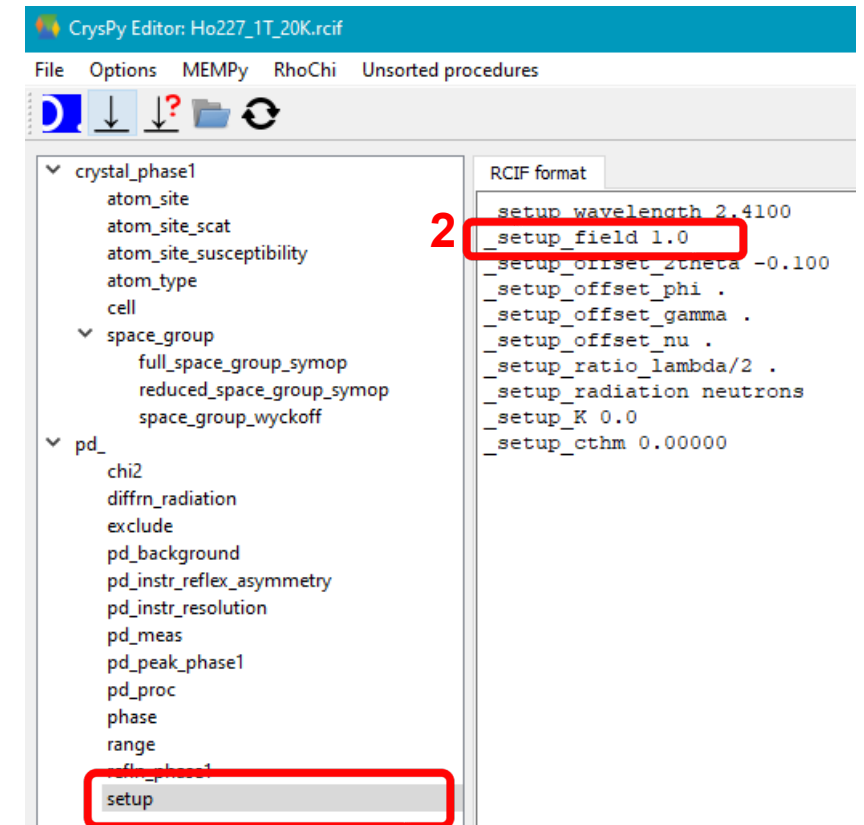
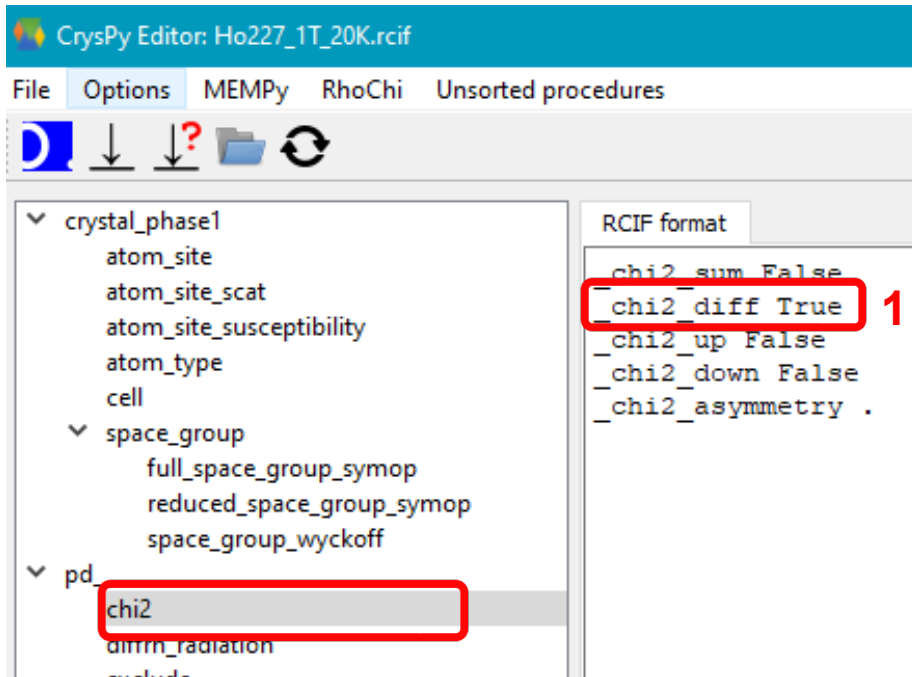


$$\chi = \frac{m}{H} = \begin{pmatrix} \chi_{11} & \chi_{12} & \chi_{13} \\ \chi_{12} & \chi_{22} & \chi_{23} \\ \chi_{13} & \chi_{23} & \chi_{33} \end{pmatrix}$$

Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- **1** In chi2 set “_chi2_diff_” to True, since we want to refine the difference of spin up and spin down.
 - Later we can refine the sum as well.

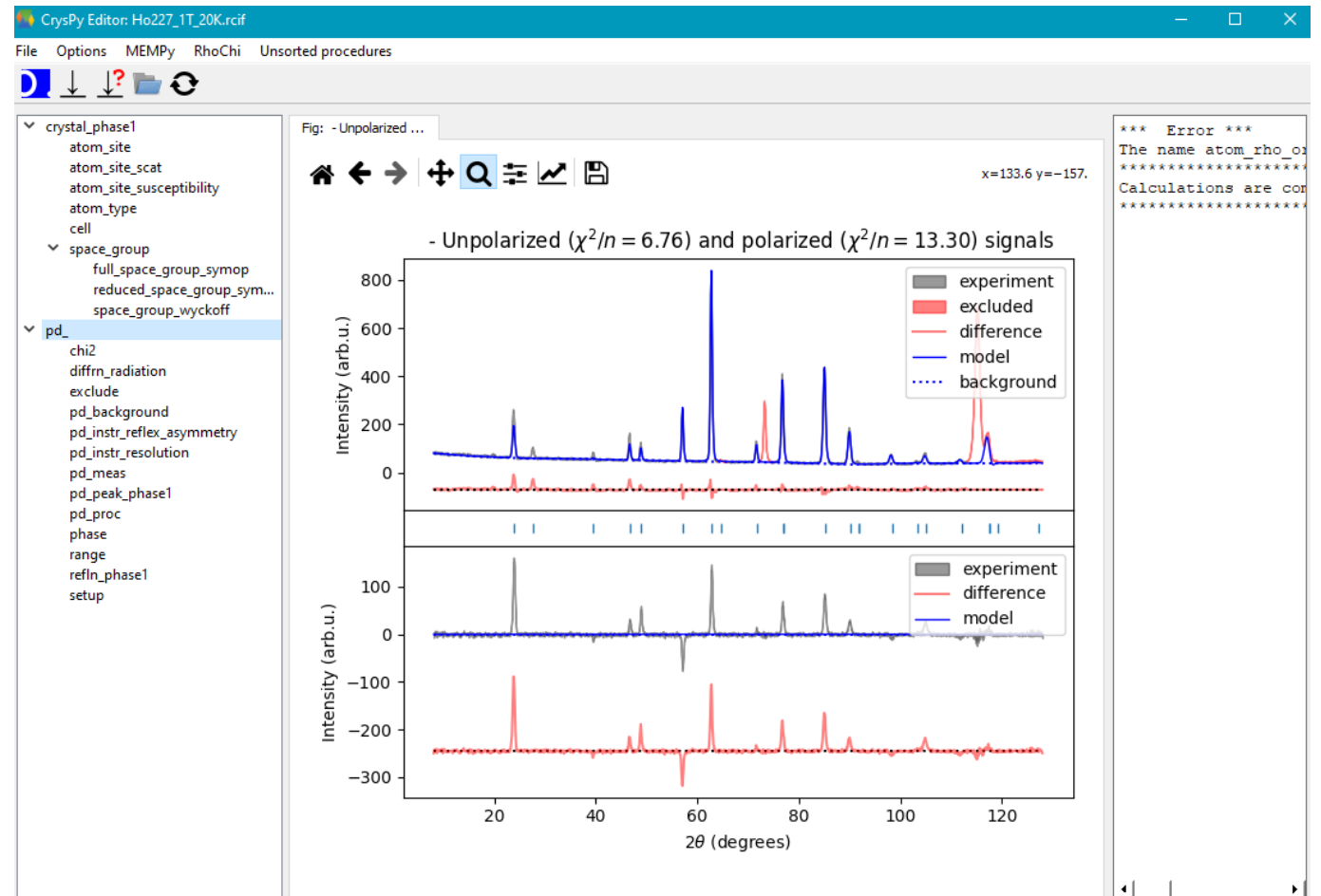
- **2.** Put in the field value used for the measurement in pd_ → setup
- A value of 1 Tesla was used



Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- Check everything is set-up correctly by running: RhoChi → No refinement (autorun) and look at the data against model
- The site susceptibility is zero, so the model for the difference is still a flat line.

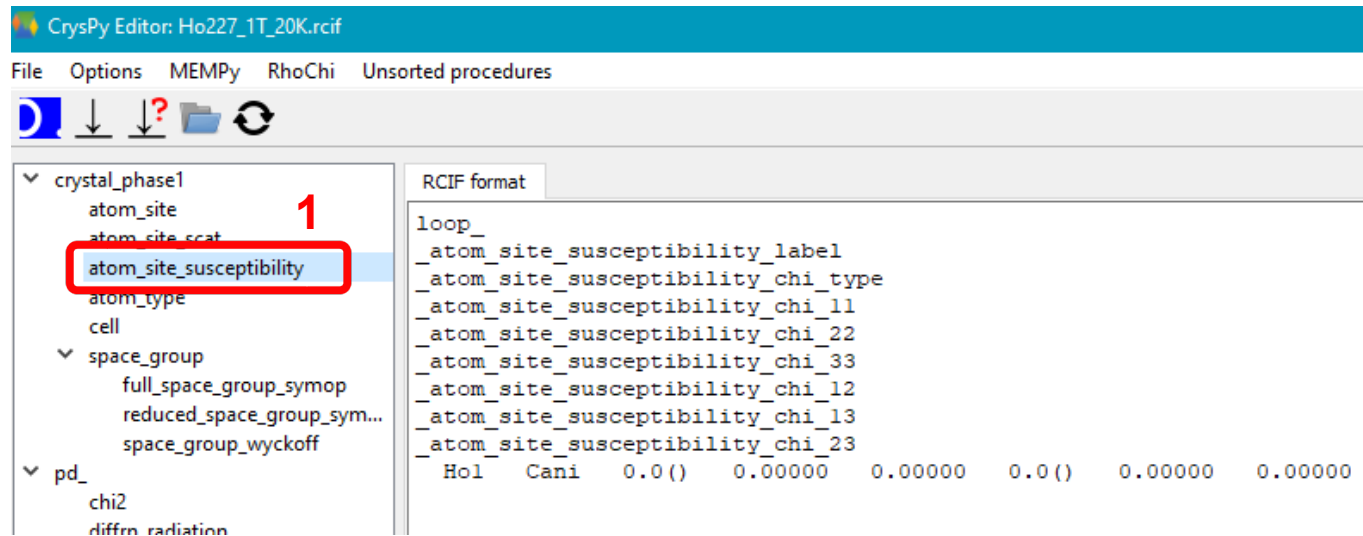
- We're now ready to refine the local site susceptibility



Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- **Refine the local site susceptibility tensor for $\text{Ho}_2\text{Ti}_2\text{O}_7$**

- **1.** Right-click on “atom_site_susceptibility” → Refine all variables
- The symmetry constraints in crysPy determine which matrix elements to refine.
 - Diagonal elements are constrained together
 - Off-diagonal elements are constrained together



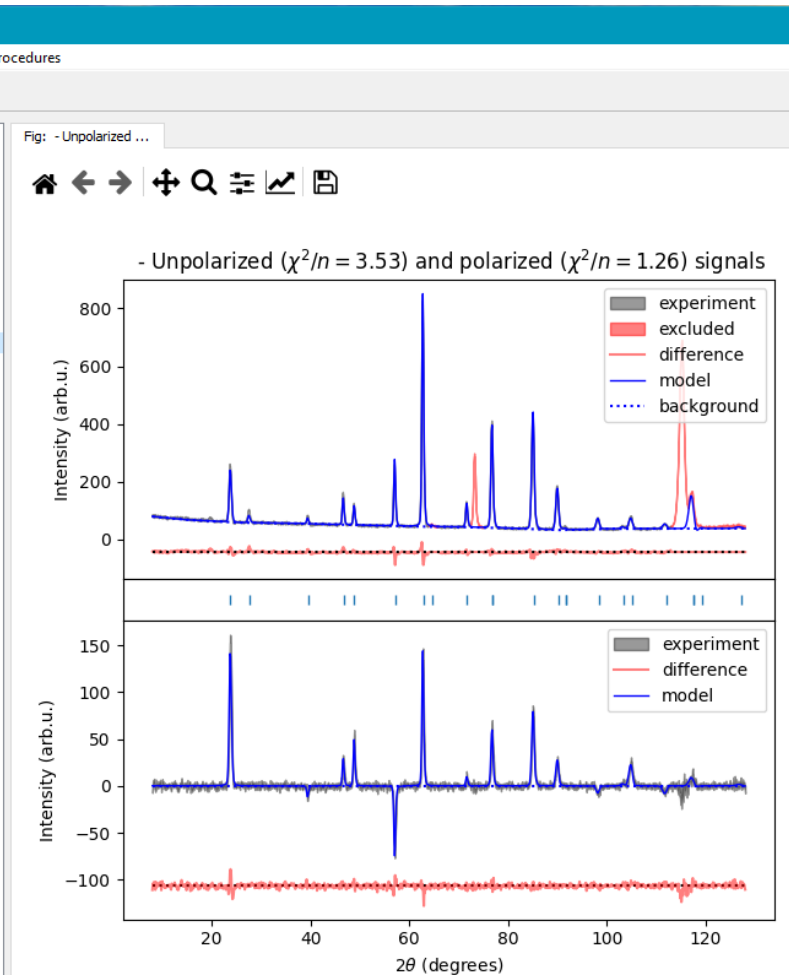
Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- **Refine the local site susceptibility tensor for $\text{Ho}_2\text{Ti}_2\text{O}_7$**

The screenshot shows the CrysPy Editor interface for the file 'Ho227_1T_20K.rcif'. The 'RCIF format' panel is active, displaying a list of parameters for refinement. A terminal window in the foreground shows the following output:

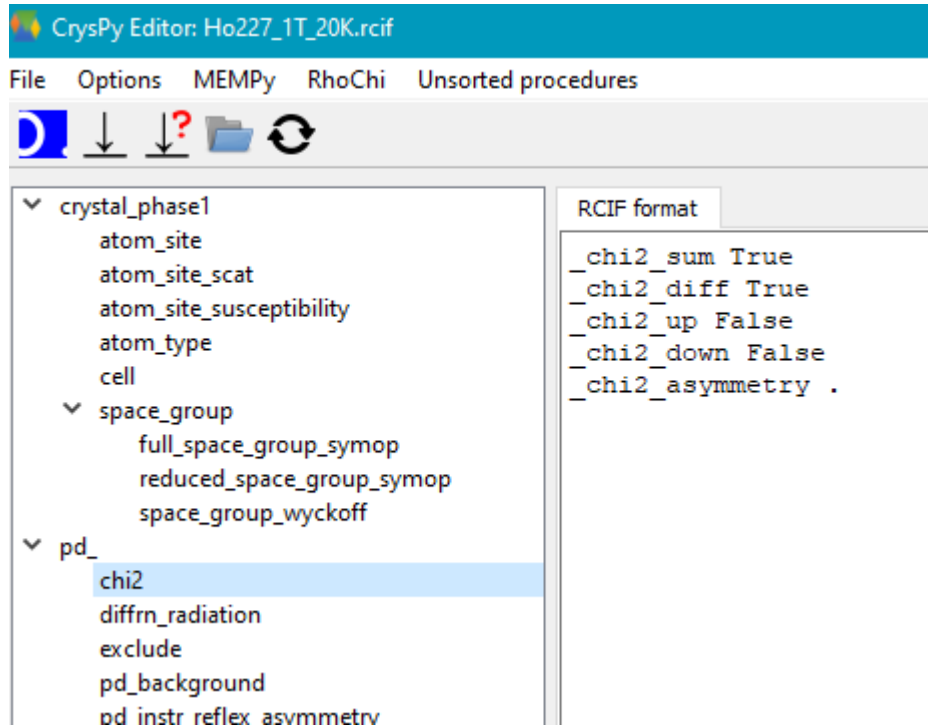
```
*****  
Welcome to CrysPy Editor.  
*****  
Number of variables is 2  
- chi_l1 1.00041  
- chi_l2 0.93718
```

- The fit to the data looks very good!
- In the top panel the red peaks are the excluded Al sample holder peaks.



Step 3: Refine the polarized flipper on and flipper off data using using crysPy

- **Refine the local site susceptibility tensor for $\text{Ho}_2\text{Ti}_2\text{O}_7$**



- Now refine both the difference and sum by turning “_chi2_sum” to True

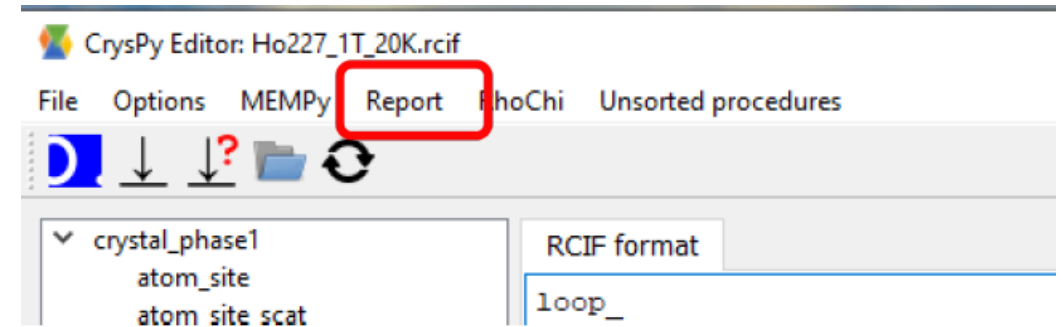
- Try refining further parameters to see how it influences the model.
 - Lattice constants, background,
- Also try “_diffn_radiation_polarization”
 - This can be sensitive to depolarization or if the sample is no longer in the linear M/H regime (check the 1.5 K data as an exercise!)

Ho₂Ti₂O₇ Powder Example

- This example will use crsPy to determine the local site susceptibility in the following steps:
 - Step 1: Installing and running crsPy
 - Step 2: Refine unpolarized, non-magnetic Ho₂Ti₂O₇ crystal structure using crsPy
 - Step 3: Refine the polarized Ho₂Ti₂O₇ data using crsPy
 - **Step 4: Interpreting the results**

Step 4: Interpreting the results

- In the “Report” drop down menu run
 - Powder experiment (autorun)
- This gives the moment. This value should agree with bulk M/H data for the sample at the same field and temperature.
- Note: Care should be taken if there is more than one magnetic ion to make sure you are comparing the same numbers!



```
*****
Calculations are completed.
*****
Result of function is
In experiment ''

For polycrystalline sample at field 1.00T:
-----
Phase      Density      wF  mass M      vF      M  Moment
          g/cm^3                emu/g      emu/cm^3  mu_B/ion
-----
phase1      7.001  100.0%      22  100.0%      155  1.067
-----

wF is weight-fraction of given phase in a mixture;
mass M is mass magnetization of powder sample;
vF is volume-fraction of given phase in a mixture;
M is volume magnetization of powder sample.

Mass magnetization of a mixture is 22.16 [emu/g];
Volume magnetization of a mixture is 155.11 [emu/cm^3].

-----
Phase      Mass susceptibility tensor x 10 000
-----
phase1      22.16  22.16  22.16  -0.00  0.00  0.00
-----

The tensor is defined in Cartesian coordinate system
such as axis Z || c, axis X || a* for each phase in
[emu/(Oe g)].
```

Step 4: Interpreting the results

- Click on “crystal_phase1” to see the Magnetization ellipsoid details.
- These can be visualized using VESTA by populating the thermal parameters.
- The magnetization ellipsoids (black) are anisotropic and drive the all-in, all-out spin ice behavior of $\text{Ho}_2\text{Ti}_2\text{O}_7$

Magnetization ellipsoids

For 'Ho1' the susceptibility is:

Susceptibility (μ_B/T) Orientation: X along inv.a Y is [inv.a, c] Z along c

-0.08553	along:	0.75352	-0.64908	-0.10443
-0.08553	along:	0.31445	0.49534	-0.80979
3.44779	along:	0.57735	0.57735	0.57735

Ellipsoid factor 1.02481.

Use thermal parameters U_{ij} to plot magn. ellipsoid.

U_11 U_22 U_33 U_12 U_13 U_23
3.967 3.967 3.967 3.960 3.960 3.960



VESTA
Visualization for Electronic and Structural Analysis

Ho227_ICSD_structure.cif

Phase: 1 252236-ICSD

Phase Unit cell Structure parameters Volumetric data Crystal shape

Atomic displacement parameter Anisotropic: U Isotropic: B

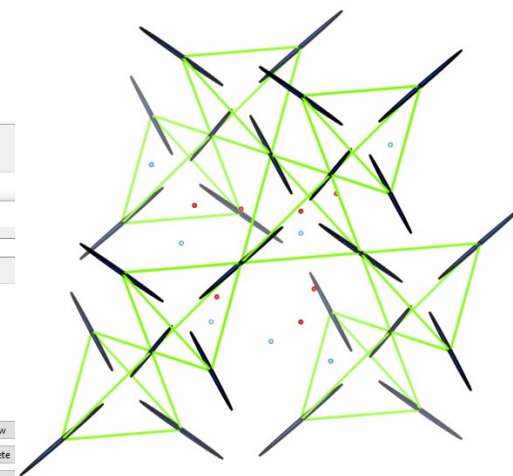
No.: 1/4 Symbol: Ho Label: Ho1 Charge: 3

x: 0.500000 y: 0.500000 z: 0.500000 Occ.: 1

U11: 3.967000 U22: 3.967000 U33: 3.967000

U12: 3.960000 U13: 3.960000 U23: 3.960000

No.	Atom	Label	x	y	z	Occ.	B
1	Ho	Ho1	0.500000	0.500000	0.500000	1	1
2	Ti	Ti1	0.000000	0.000000	0.000000	1	1
3	O	O1	0.375000	0.375000	0.375000	1	1
4	O	O2	0.328360	0.125000	0.125000	1	1



$$\chi = \frac{m}{H} = \begin{pmatrix} \chi_{11} & \chi_{12} & \chi_{13} \\ \chi_{12} & \chi_{22} & \chi_{23} \\ \chi_{13} & \chi_{23} & \chi_{33} \end{pmatrix}$$

Step 4: Interpreting the results

DOI: [10.1103/PhysRevResearch.1.033100](https://doi.org/10.1103/PhysRevResearch.1.033100)

PHYSICAL REVIEW RESEARCH **1**, 033100 (2019)

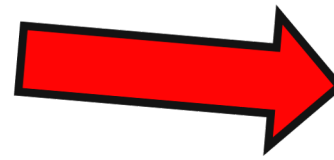
Local magnetic anisotropy by polarized neutron powder diffraction: Application of magnetically induced preferred crystallite orientation

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For the space group $Fd3m$, the symmetry constraints imply that the local susceptibility tensor has only two independent matrix elements χ_{11} and χ_{12} and the principal axes of Ho magnetization ellipsoids are oriented along the four local $\langle 111 \rangle$ axes. Their lengths, given by $\chi_{\parallel} = \chi_{11} + 2\chi_{12}$ and $\chi_{\perp} = \chi_{11} - \chi_{12}$, were determined at each temperature. The thermal evolution of χ_{\parallel} and χ_{\perp} obtained by 2D Rietveld refinement on a polycrystalline sample is shown in Fig. 6 by solid symbols. Open symbols in the figure show the results of a previous study performed using polarized neutron diffraction on a single crystal [6]. One can see that the results of Rietveld refinement are in good agreement with the single-crystal ones and offer the same precision of the susceptibility parameters.



DOI: [10.1103/PhysRevResearch.1.033100](https://doi.org/10.1103/PhysRevResearch.1.033100)

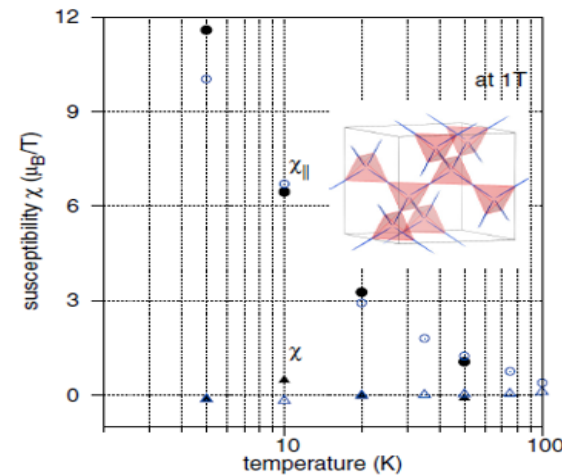


FIG. 6. Temperature dependence of the susceptibility components χ_{\parallel} (circles) and χ_{\perp} (triangles) for single (open symbols, taken from Ref. [6]) and powder (solid symbols) $\text{Ho}_2\text{Ti}_2\text{O}_7$ at 1 T. The inset shows Ho magnetization ellipsoids at 50 K.

- The HB-2A results can be compared to other single crystal and powder measurements on $\text{Ho}_2\text{Ti}_2\text{O}_7$.
- Use CrysPy to fit the 1.5K, 50K and 100K data and follow the symmetry arguments in the paper to reproduce the temperature dependence of the susceptibility.

HB-2A polarized analysis

