Data processing with APEX4

Fátima Minas-Piedade (CQE, FC-ULisboa)

Isabel Cordeiro Santos (C2TN, IST-ULisboa)

Clara Gomes (LAQV-REQUIMTE, FCT-NOVA)

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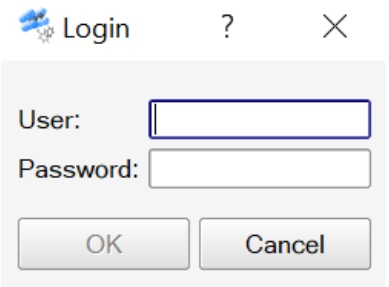
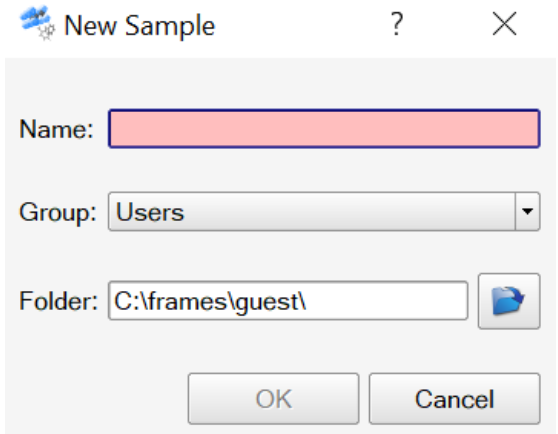
APEX4 – New sample

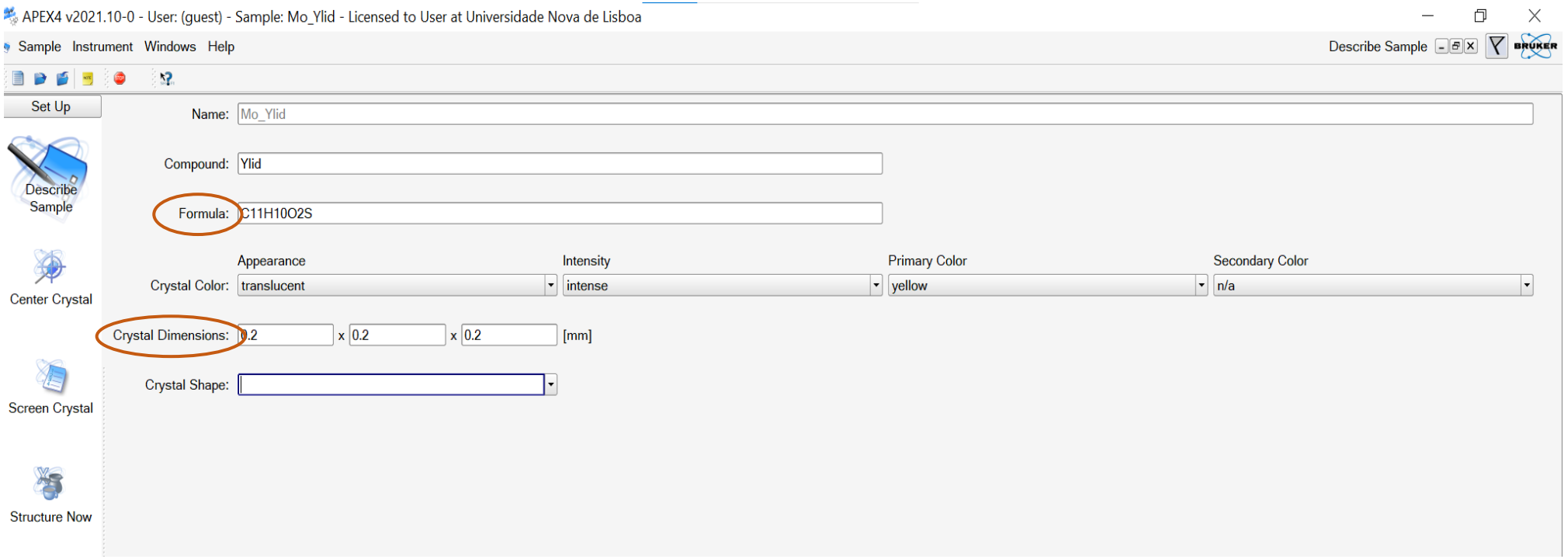
- **Login**
- Sample → Login
- Enter in Username (group name) and Password



- **Create New Sample**
- Sample → New
- Enter in sample name, be sure to check white board or cards to establish next number
- Directory under sample's name will be created in the folder C:\frames\guest\SampleName



APEX4 – New sample

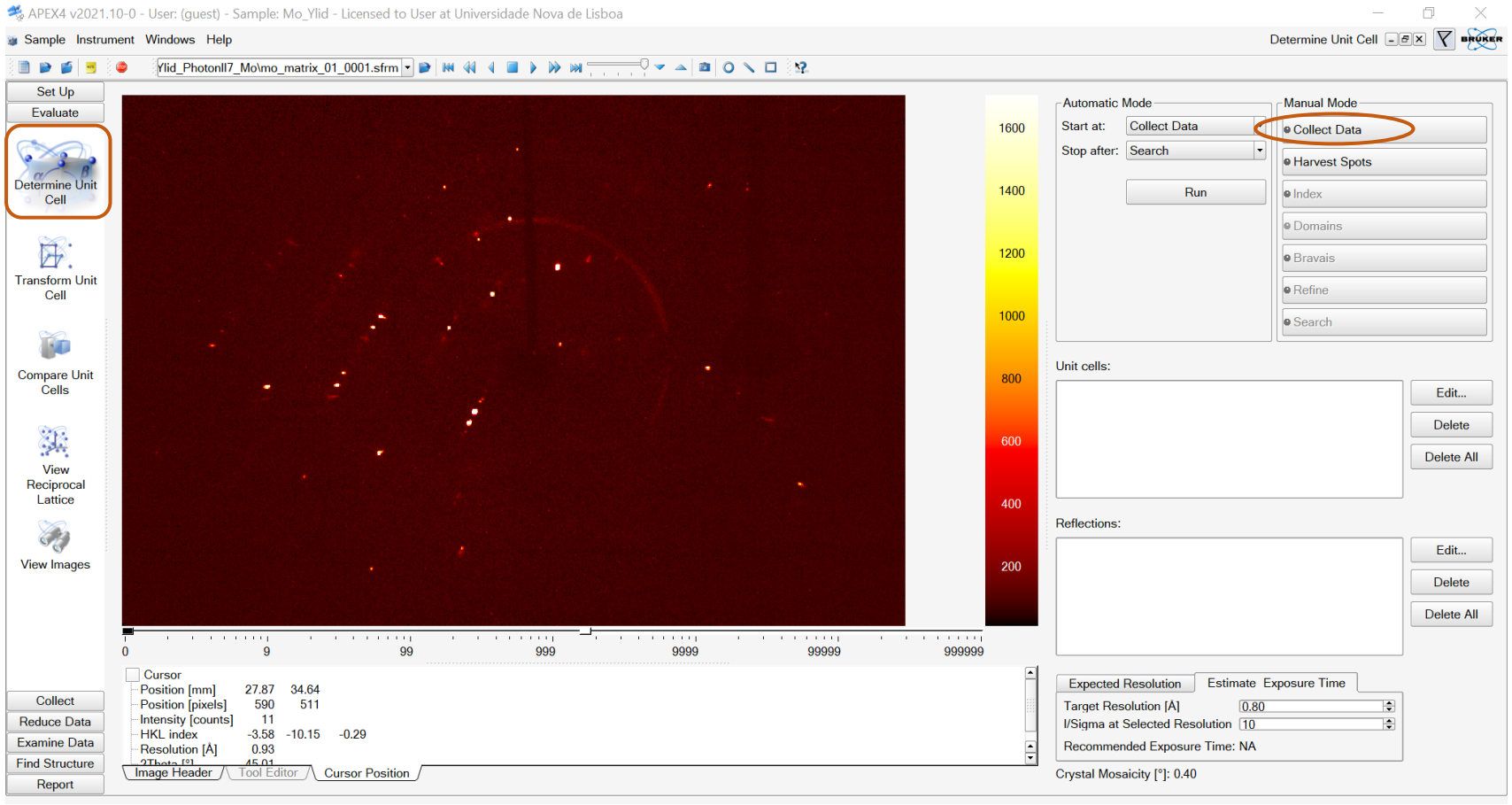


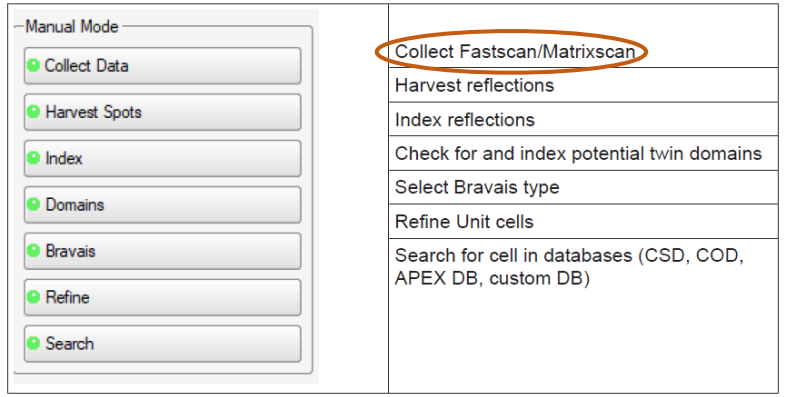
- **Set Up** menu

  Mandatory fields: Formula and Crystal dimensions

APEX4 – Search Unit Cell

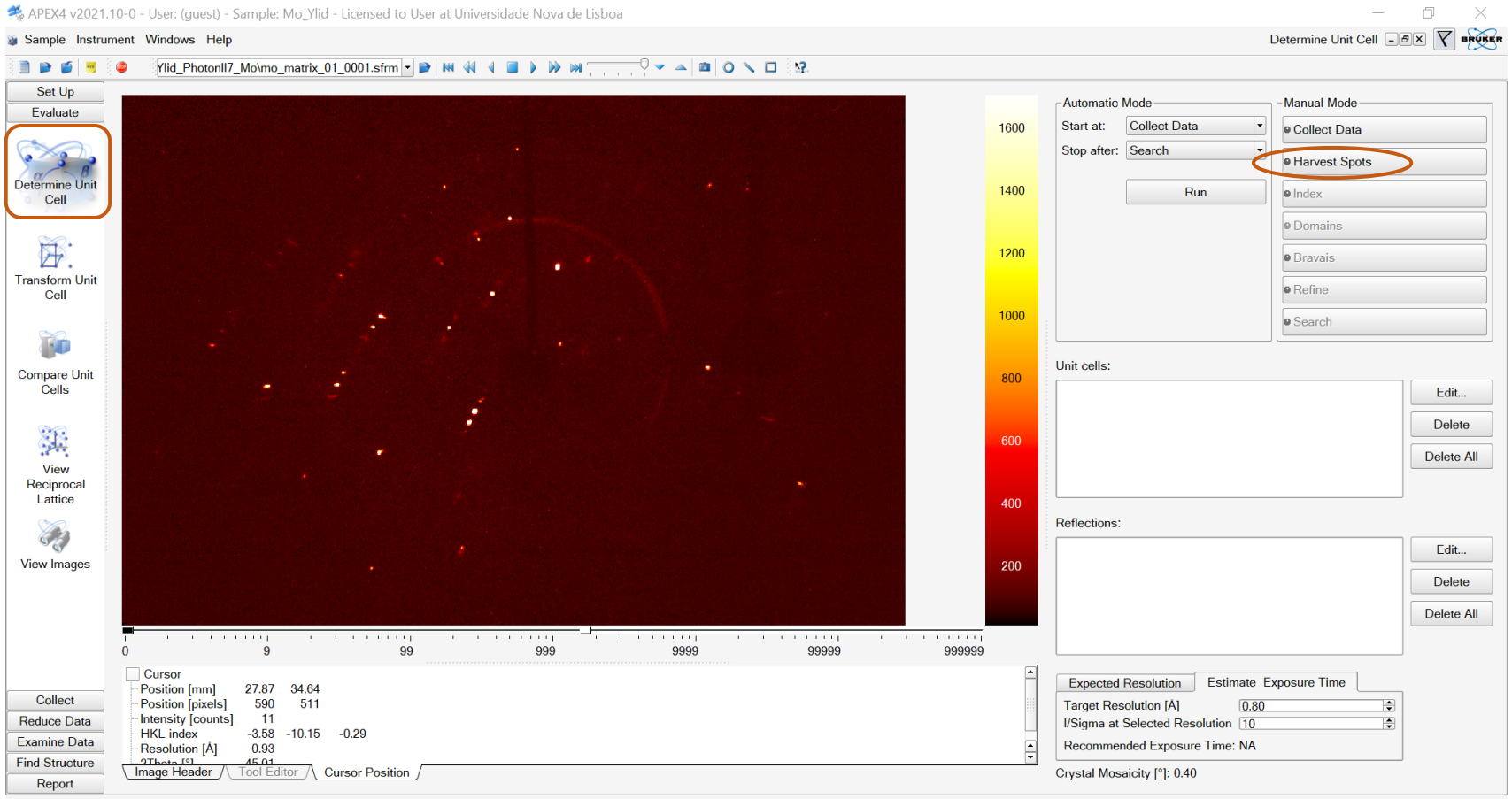
Evaluate menu

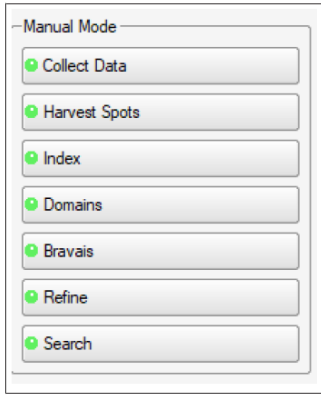




APEX4 – Search Unit Cell

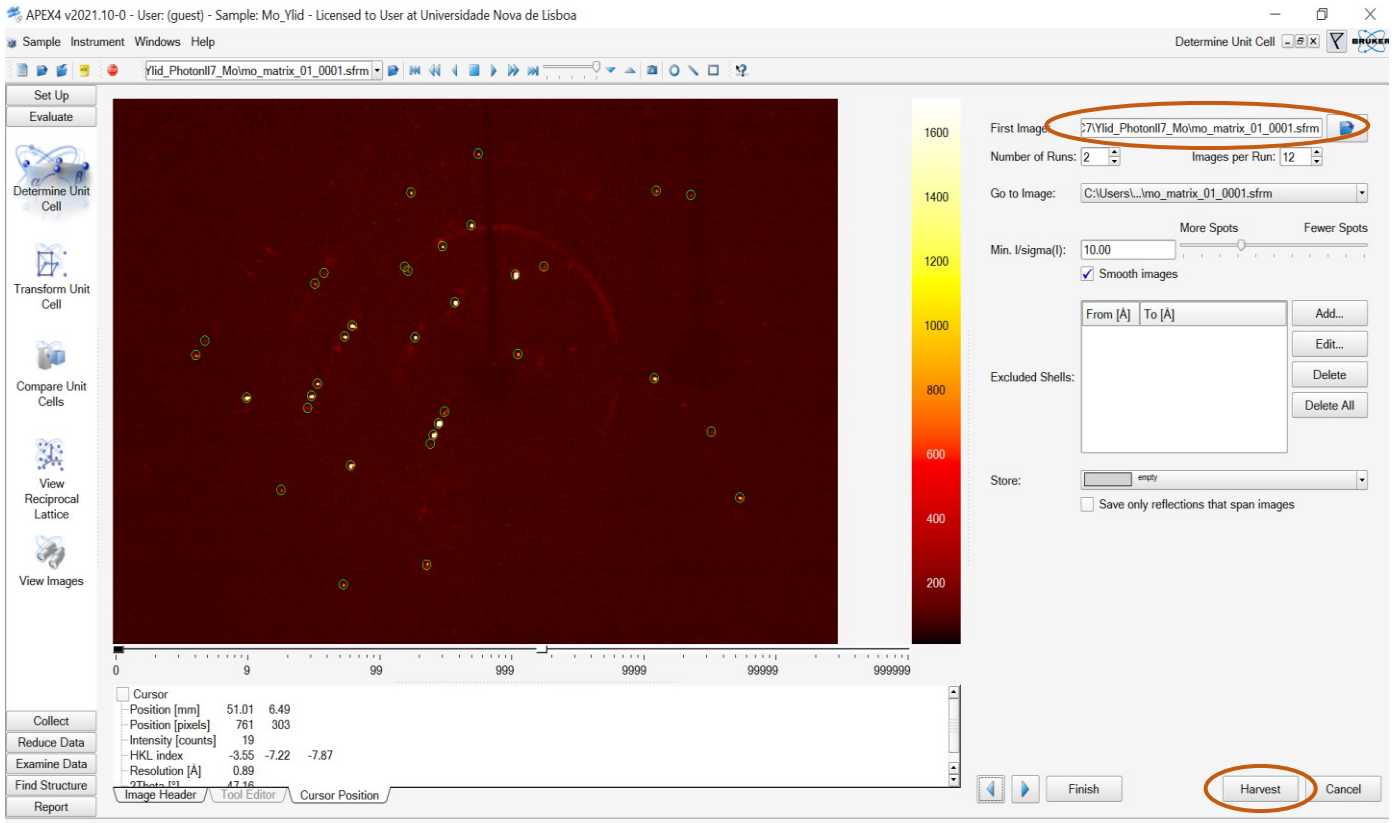
Evaluate menu





Collect Fastscan/Matrixscan
Harvest reflections
Index reflections
Check for and index potential twin domains
Select Bravais type
Refine Unit cells
Search for cell in databases (CSD, COD, APEX DB, custom DB)

APEX4 - Search Unit Cell



Harvest Spots

A. Select Harvest Spots in list on the right side of the screen
B. Select matrix frames from data collection, change Number of Runs and Images per Run from to reflect the number of matrix files
C. Adjust the Min I./sigma(I)
    i. Values can be adjusted by manually entering number in the box or by moving the slider
D. Click Harvest button at the bottom of the page

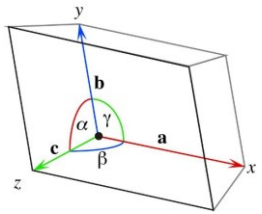
APEX4 - Search Unit Cell

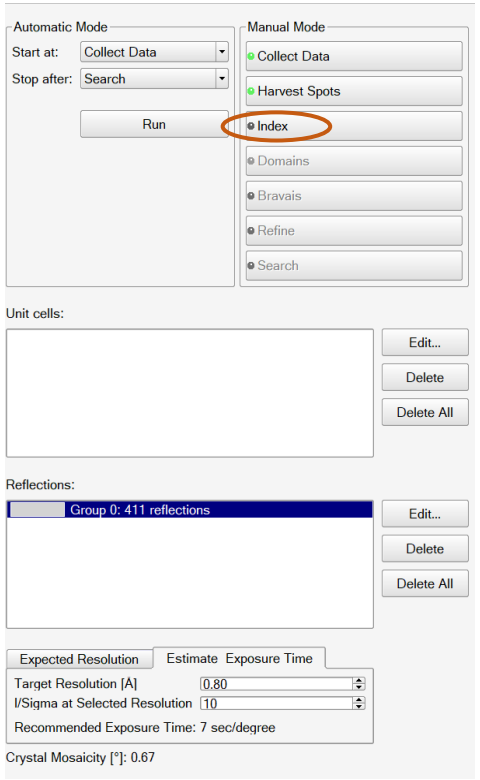
Indexing: calculating the unit cell

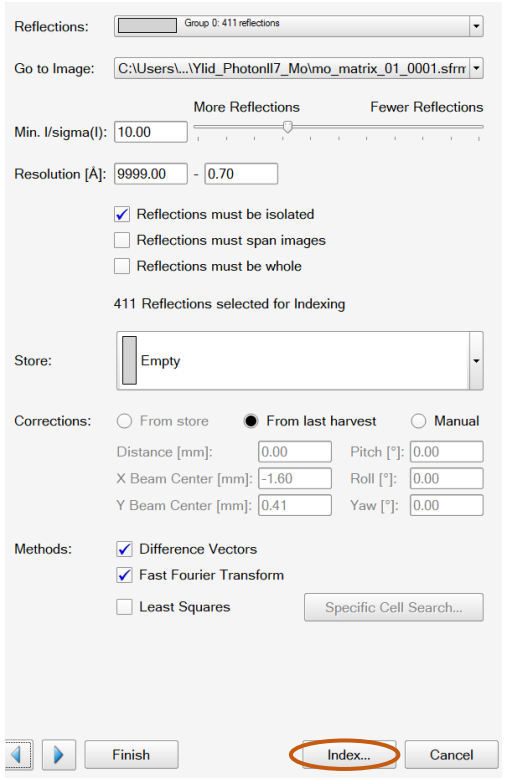
smallest repeating unit of the crystal lattice

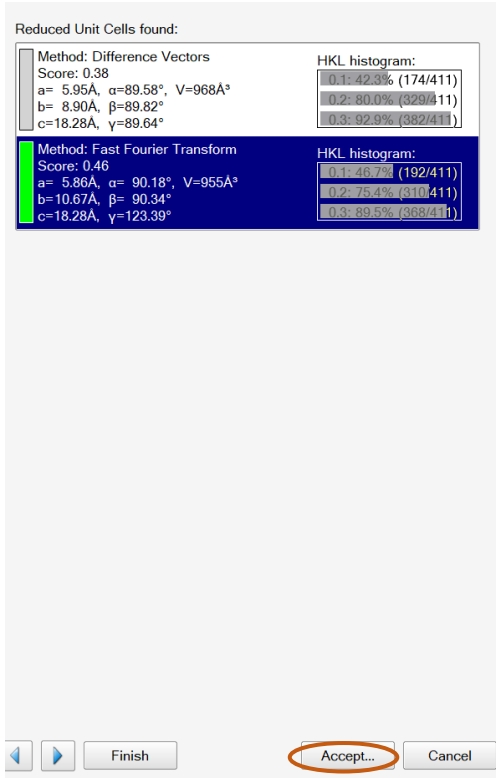
Unit cell parameters:

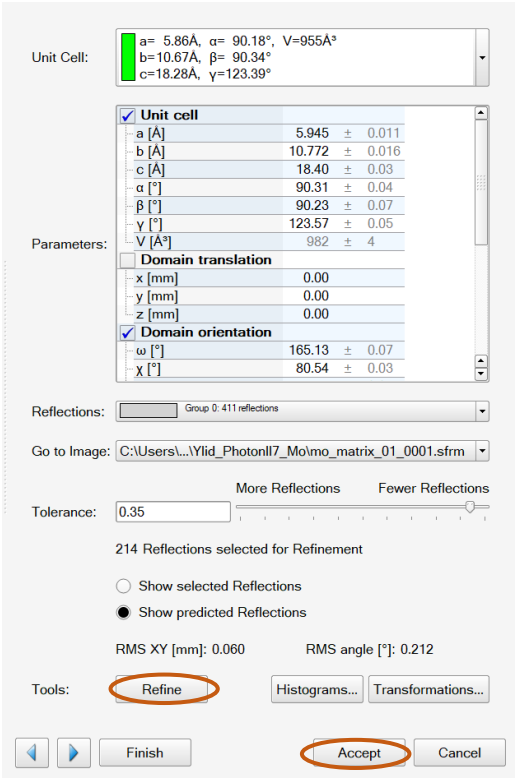
axis – ***a***, ***b***, ***c***
angles - α, β, γ





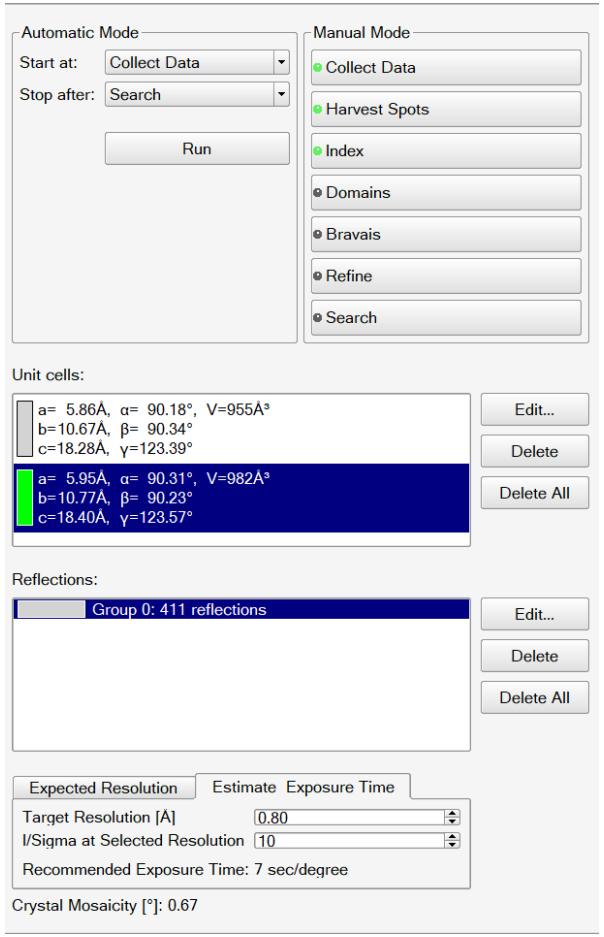






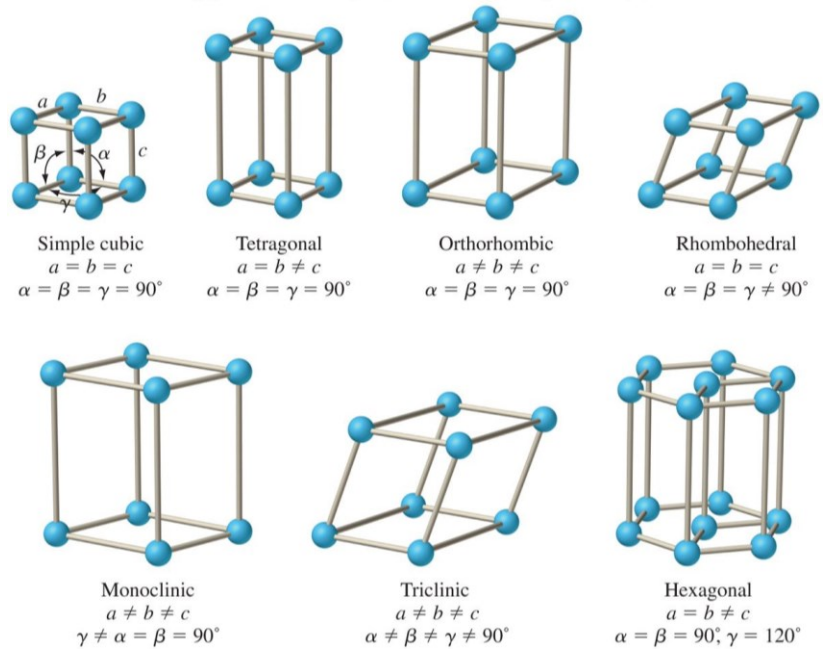
APEX4 - Search Unit Cell

Domains: Allows automated search for non-merohedral twin domains.



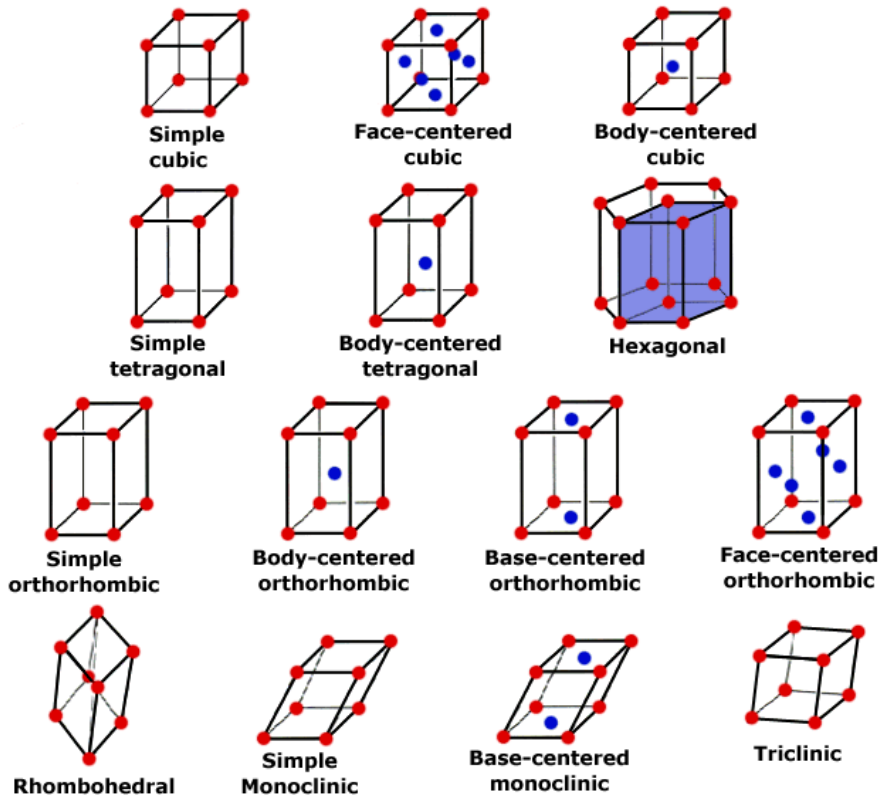
Bravais

7 Crystal Systems

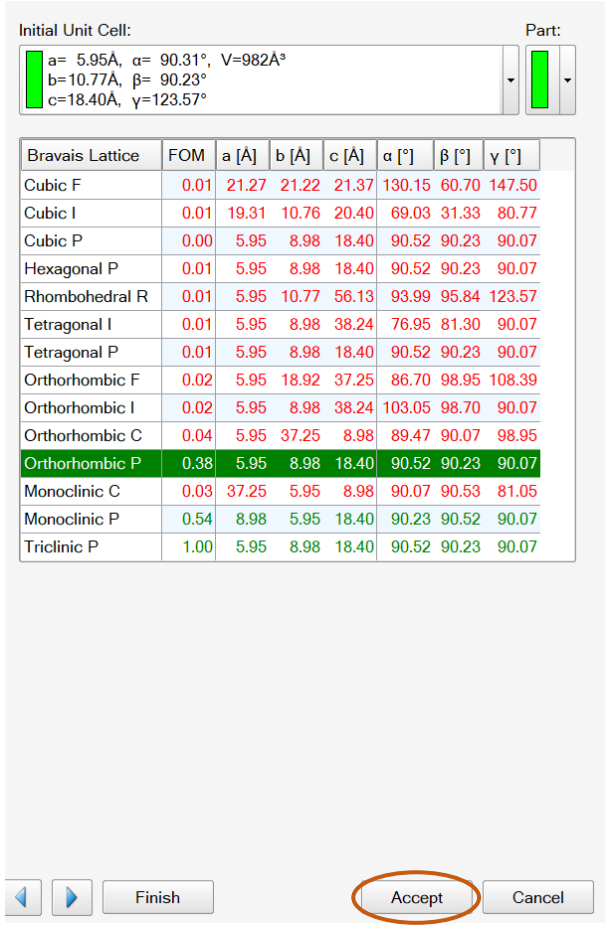


APEX4 - Search Unit Cell

14 Bravais Lattices



Bravais: Allows automated search for non-merohedral twin domains.

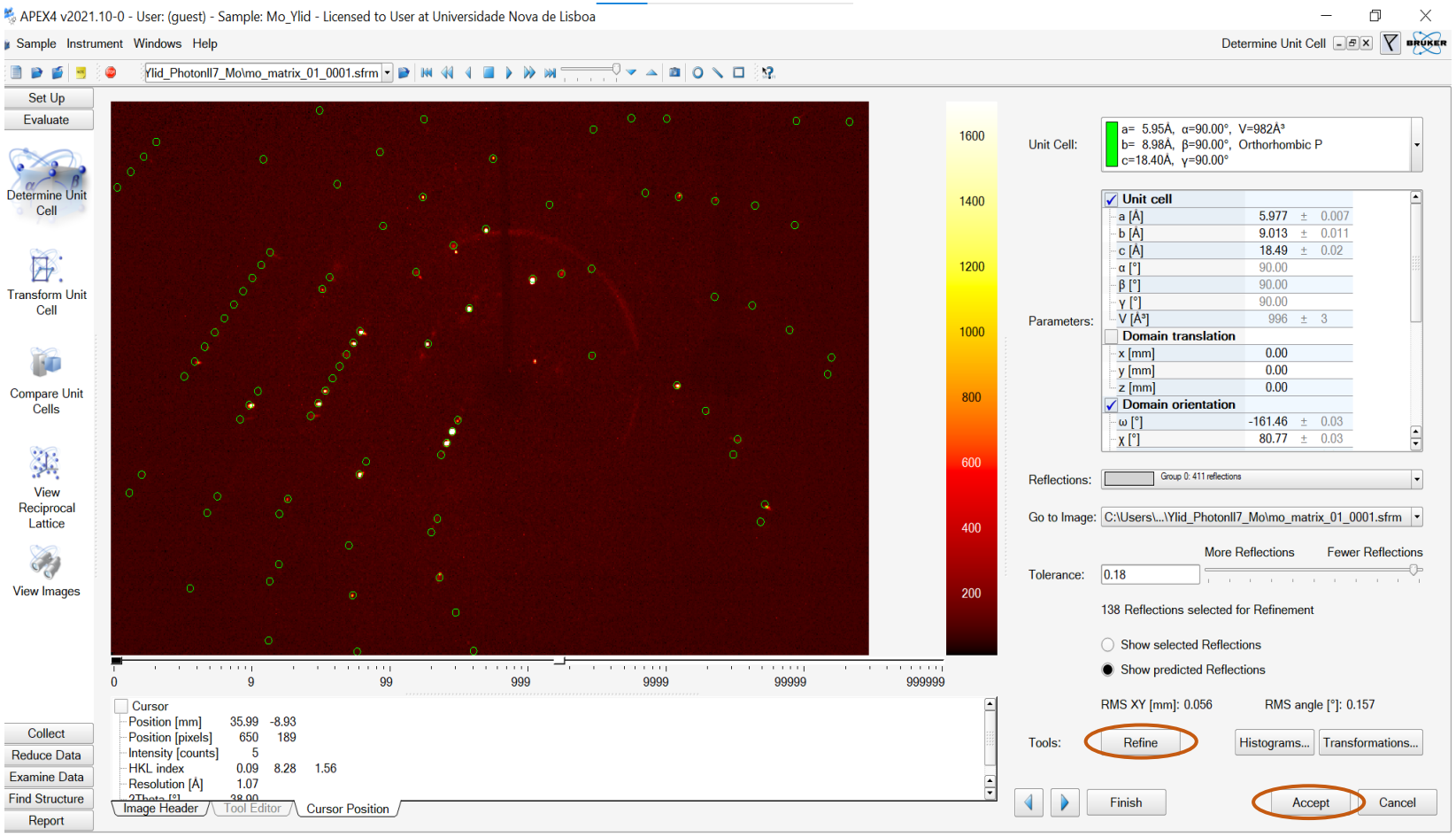


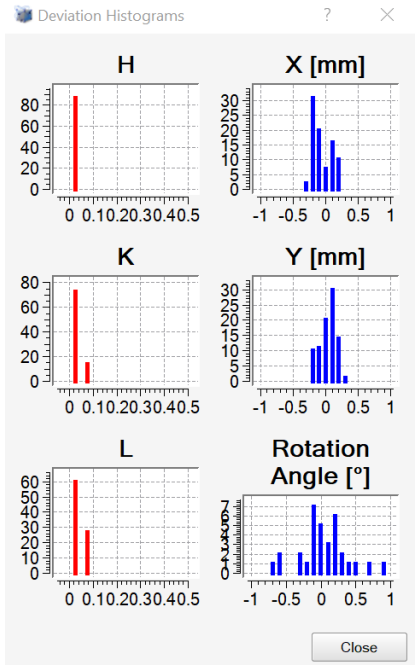
Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
Cubic F	0.01	21.27	21.22	21.37	130.15	60.70	147.50
Cubic I	0.01	19.31	10.76	20.40	69.03	31.33	80.77
Cubic P	0.00	5.95	8.98	18.40	90.52	90.23	90.07
Hexagonal P	0.01	5.95	8.98	18.40	90.52	90.23	90.07
Rhombohedral R	0.01	5.95	10.77	56.13	93.99	95.84	123.57
Tetragonal I	0.01	5.95	8.98	38.24	76.95	81.30	90.07
Tetragonal P	0.01	5.95	8.98	18.40	90.52	90.23	90.07
Orthorhombic F	0.02	5.95	18.92	37.25	86.70	98.95	108.39
Orthorhombic I	0.02	5.95	8.98	38.24	103.05	98.70	90.07
Orthorhombic C	0.04	5.95	37.25	8.98	89.47	90.07	98.95
Orthorhombic P	0.38	5.95	8.98	18.40	90.52	90.23	90.07
Monoclinic C	0.03	37.25	5.95	8.98	90.07	90.53	81.05
Monoclinic P	0.54	8.98	5.95	18.40	90.23	90.52	90.07
Triclinic P	1.00	5.95	8.98	18.40	90.52	90.23	90.07

- Bravais lattices **in agreement with the unit cell** are displayed in **green**. Those that are not are displayed in red.
- They are chosen automatically, but you can override the program's decision.

APEX4 - Search Unit Cell

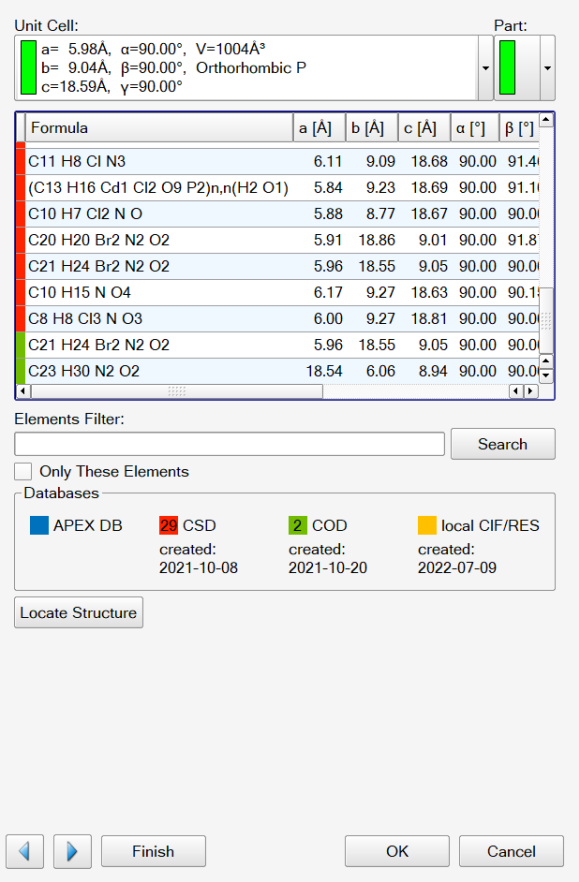
Refine

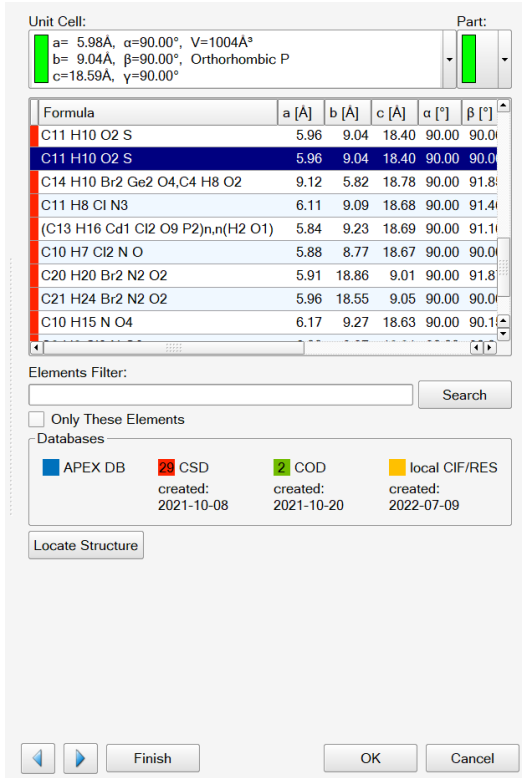


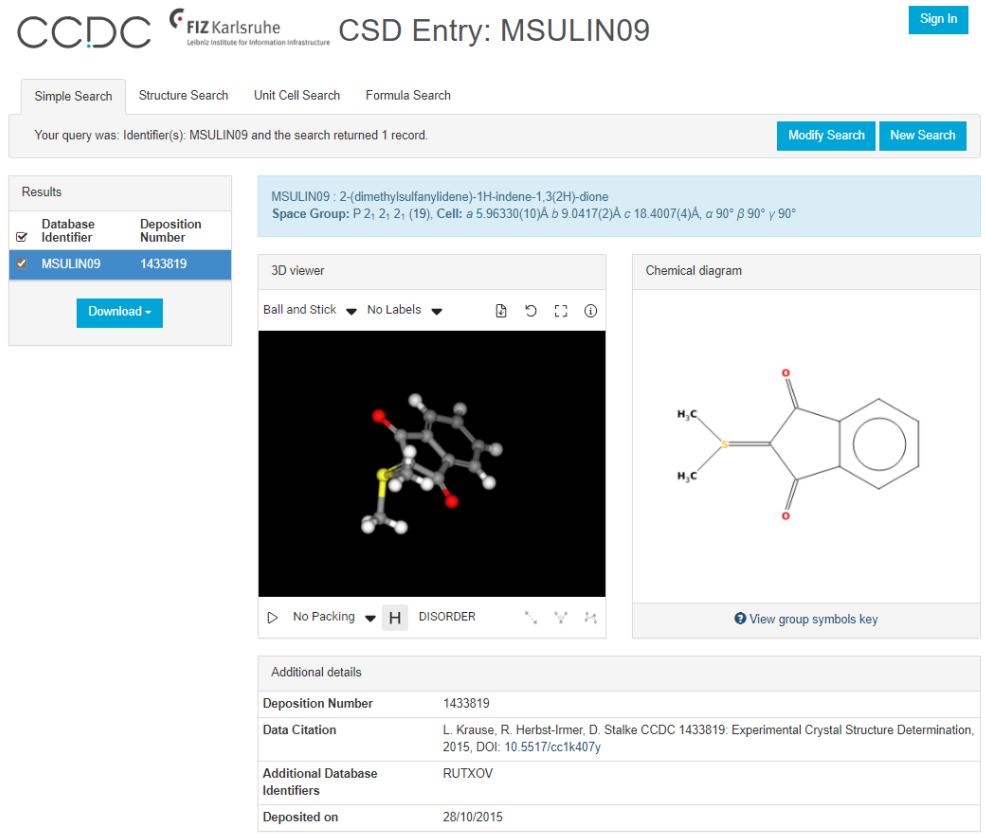


APEX4 - Search Unit Cell

Search



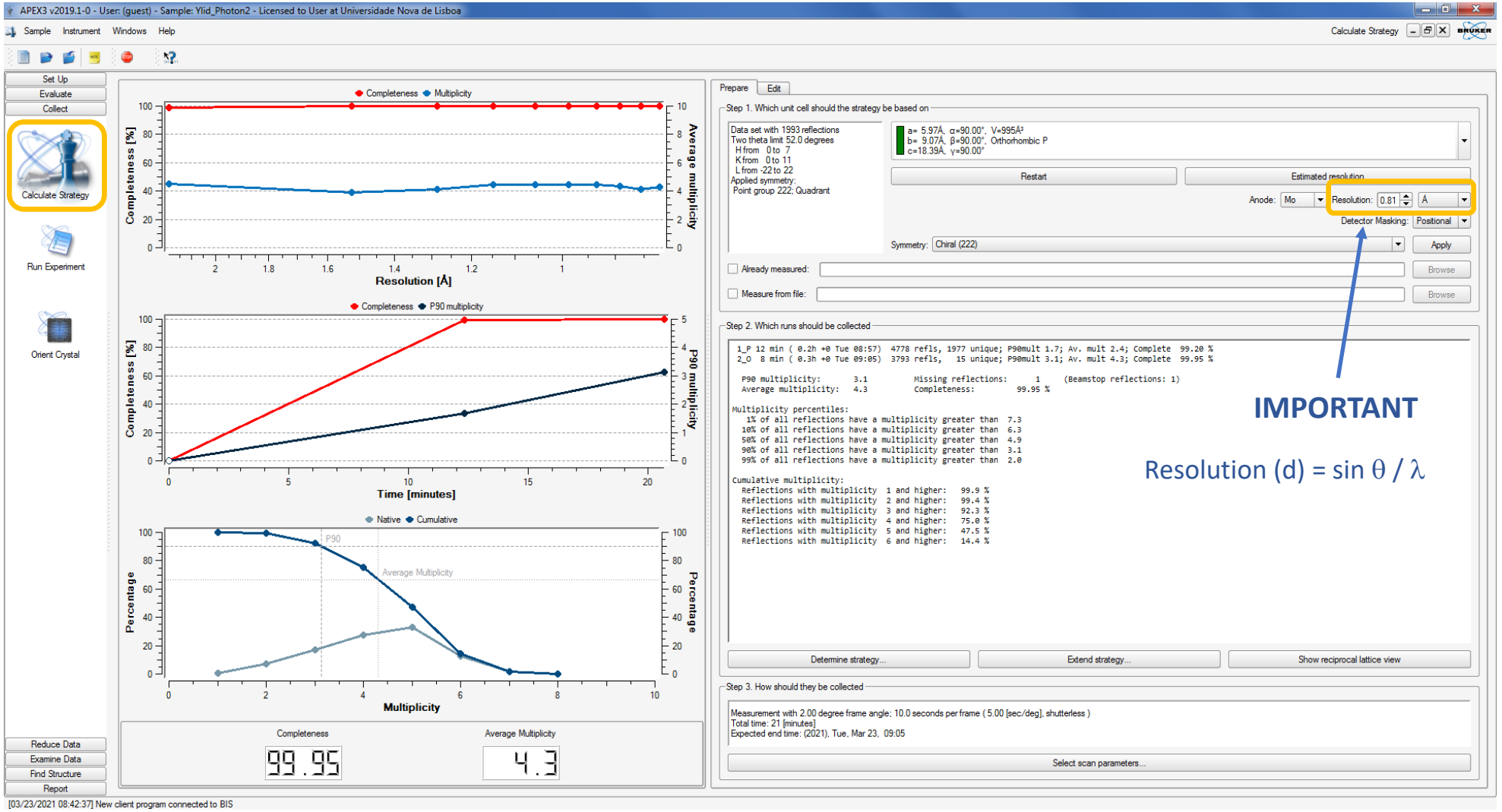




- Available databases: APEX DB, CSD, COD, local CIF/RES. CSD and COD require internet connection.
- Double click on a structure, opens the corresponding DB and shows the molecular structure, unit cell parameters and space group.

APEX4 - Data collection

Collect: Calculate strategy

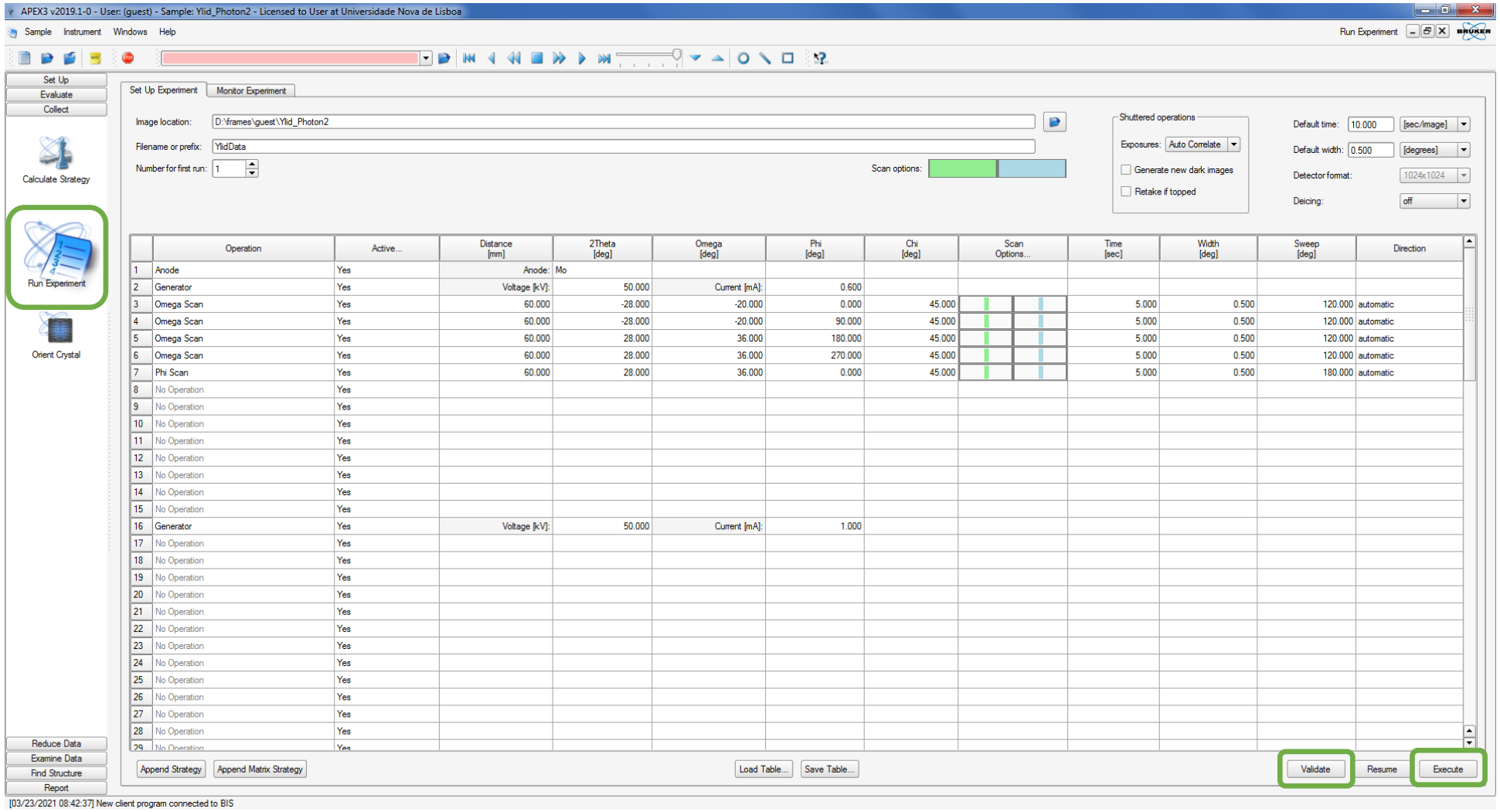


IMPORTANT

Resolution (d) = sin θ / λ

APEX4 - Data collection

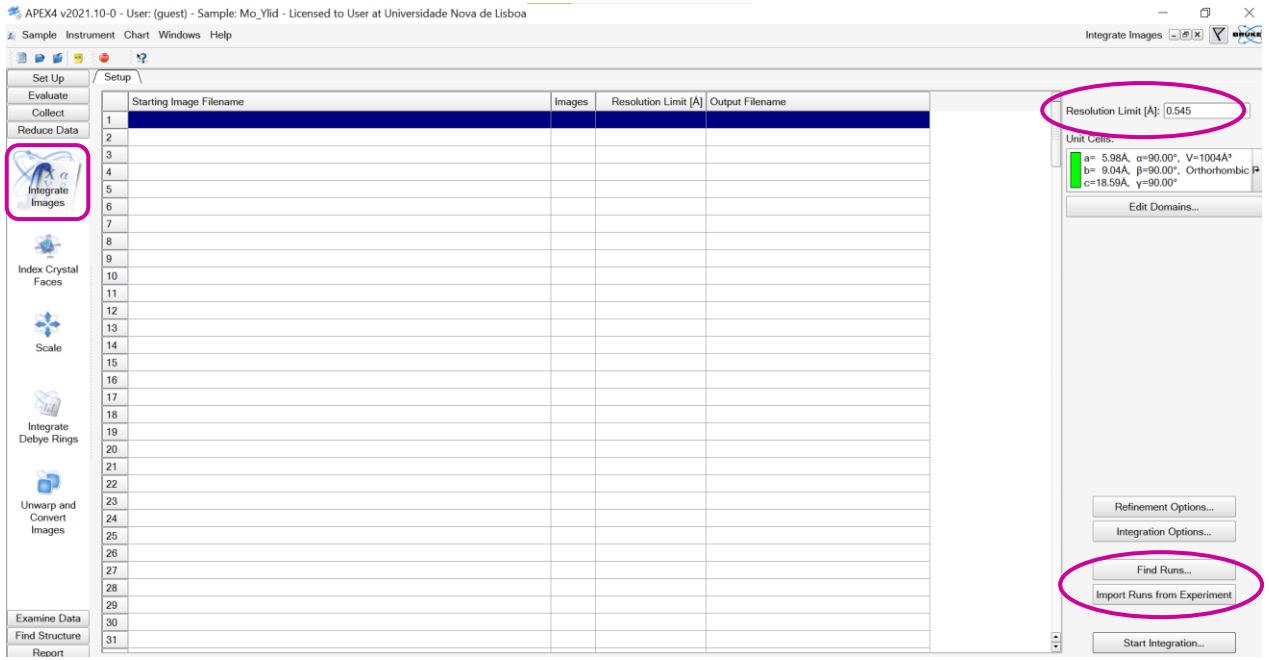
Collect: Run experiment

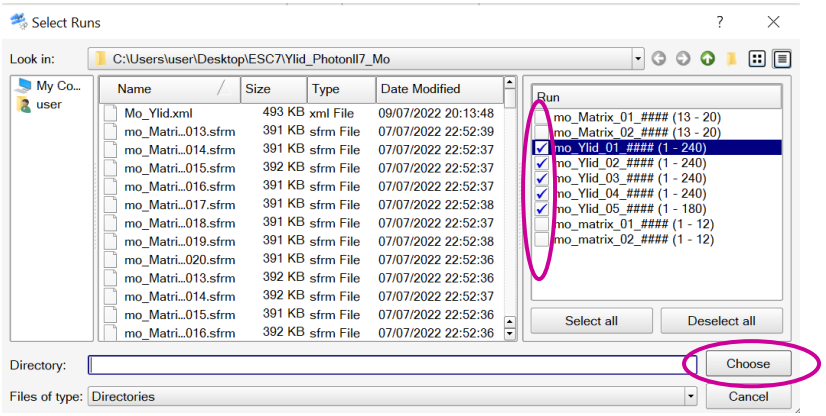


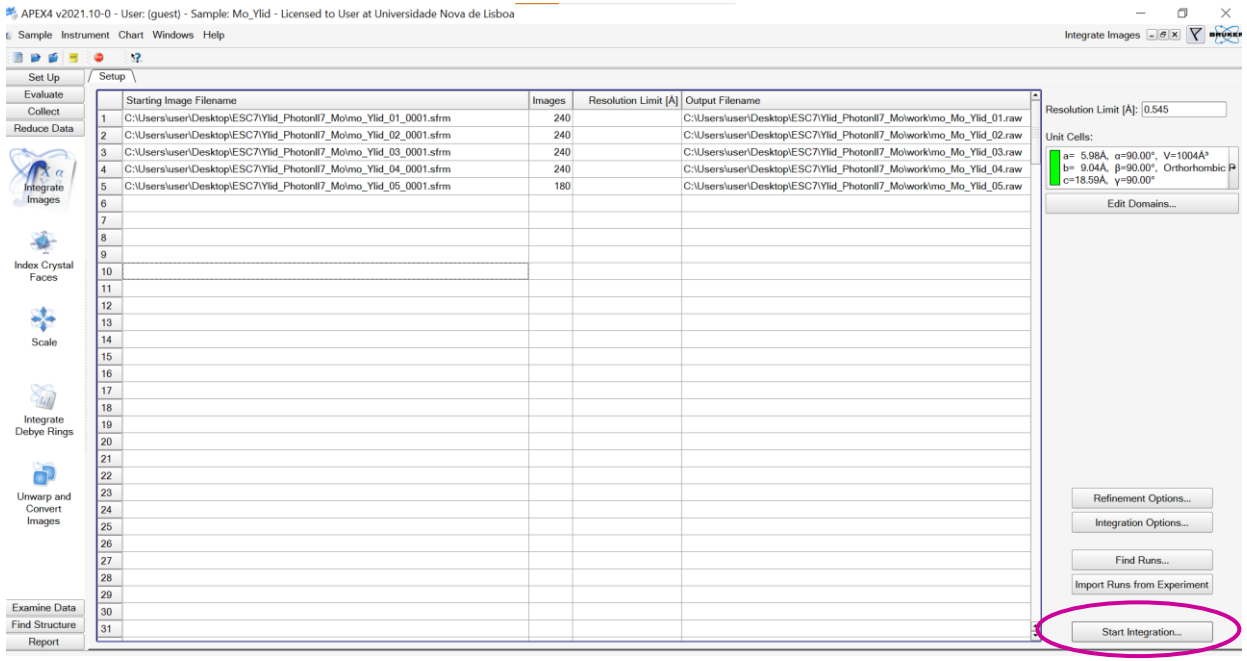
	Operation	Active...	Distance [mm]	2Theta [deg]	Omega [deg]	Phi [deg]	Chi [deg]	Scan Options...	Time [sec]	Width [deg]	Sweep [deg]	Direction
1	Anode	Yes	Anode:	Mo								
2	Generator	Yes	Voltage [kV]:	50.000	Current [mA]:	0.600						
3	Omega Scan	Yes	60.000	-28.000	-20.000	0.000	45.000		5.000	0.500	120.000	automatic
4	Omega Scan	Yes	60.000	-28.000	-20.000	90.000	45.000		5.000	0.500	120.000	automatic
5	Omega Scan	Yes	60.000	28.000	36.000	180.000	45.000		5.000	0.500	120.000	automatic
6	Omega Scan	Yes	60.000	28.000	36.000	270.000	45.000		5.000	0.500	120.000	automatic
7	Phi Scan	Yes	60.000	28.000	36.000	0.000	45.000		5.000	0.500	180.000	automatic
8	No Operation	Yes										
9	No Operation	Yes										
10	No Operation	Yes										
11	No Operation	Yes										
12	No Operation	Yes										
13	No Operation	Yes										
14	No Operation	Yes										
15	No Operation	Yes										
16	Generator	Yes	Voltage [kV]:	50.000	Current [mA]:	1.000						
17	No Operation	Yes										
18	No Operation	Yes										
19	No Operation	Yes										
20	No Operation	Yes										
21	No Operation	Yes										
22	No Operation	Yes										
23	No Operation	Yes										
24	No Operation	Yes										
25	No Operation	Yes										
26	No Operation	Yes										
27	No Operation	Yes										
28	No Operation	Yes										
29	No Operation	Yes										

APEX4 - Data processing

Reduce data: Integrate images



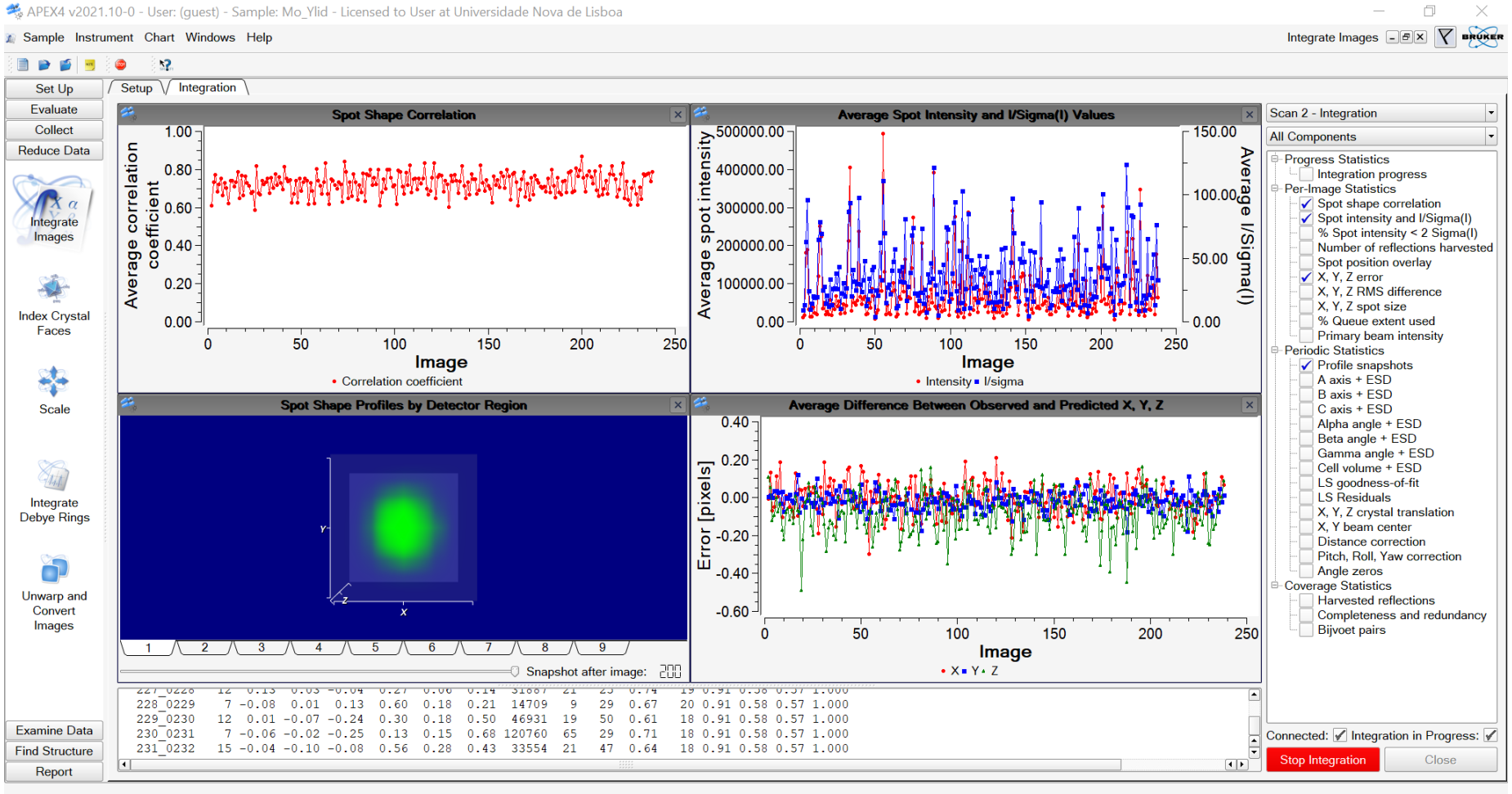




APEX4 - Data processing

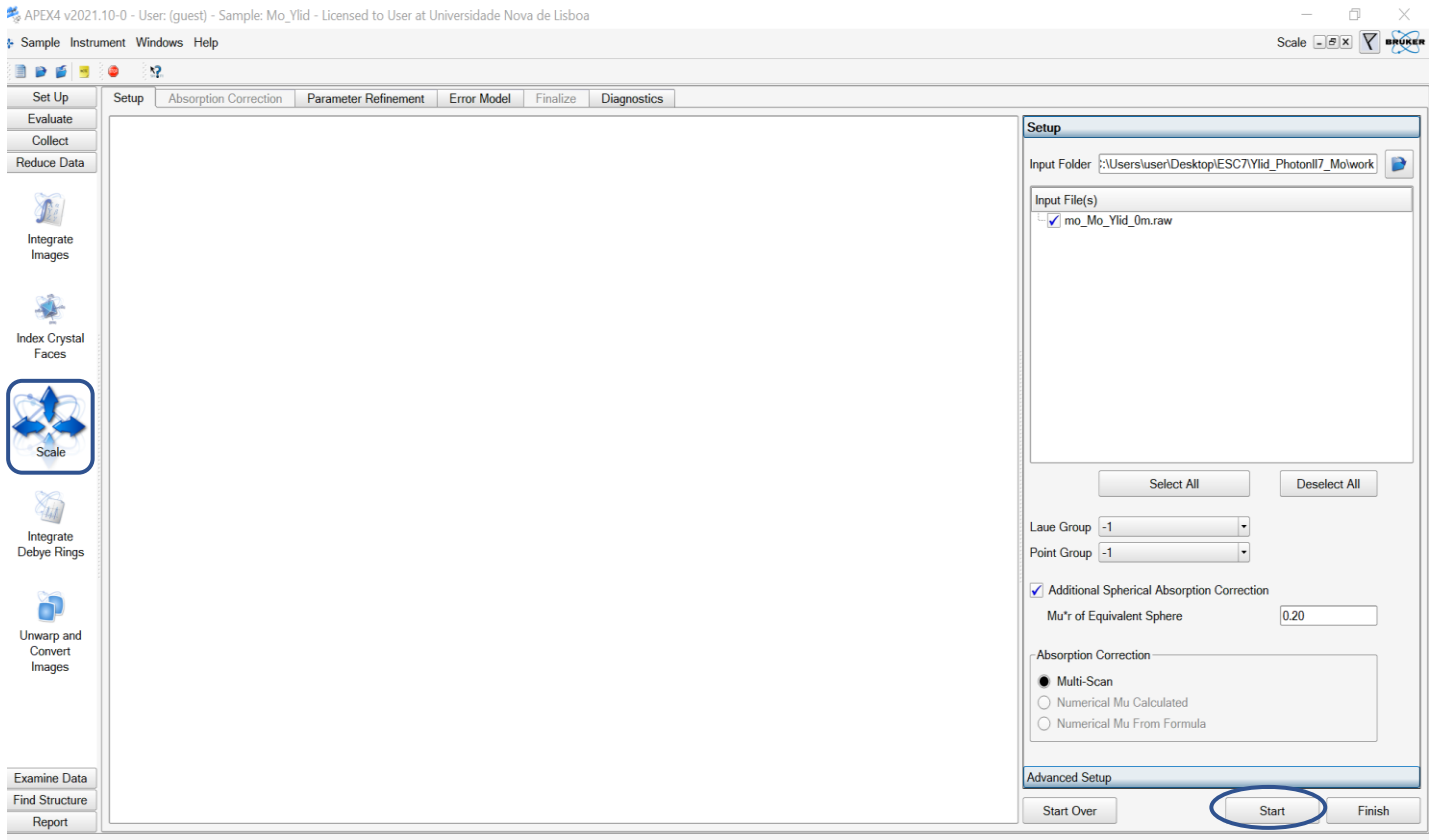
Reduce data: Integrate images

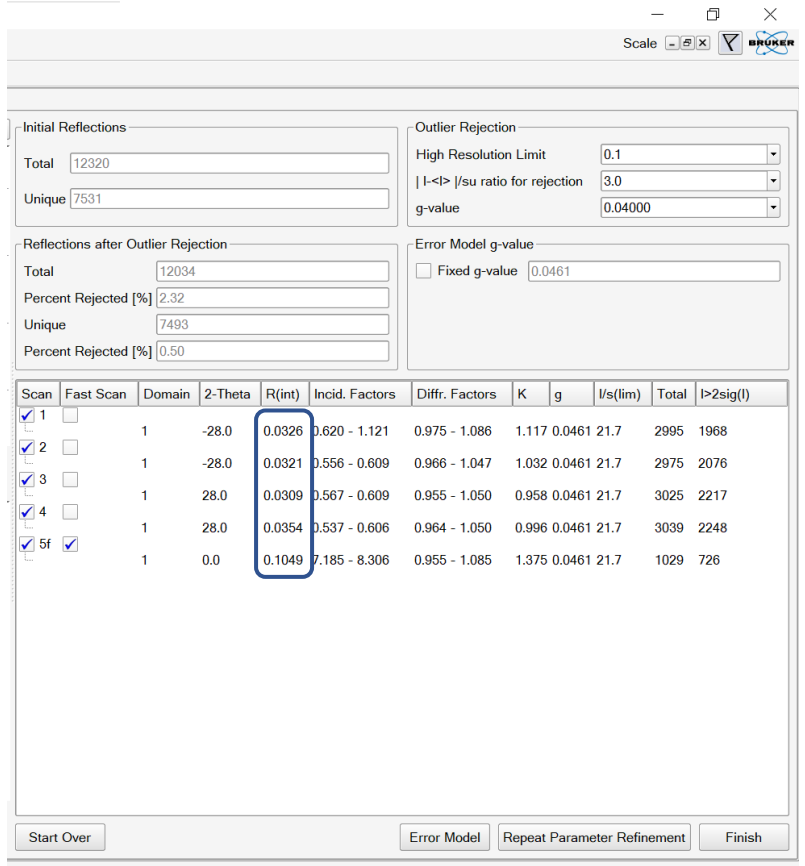
Integration (images in a single file - list of intensities), **Merge** (peaks present in more than one image) and **Scale** (relative intensities)



APEX4 - Data processing

Reduce data: Scale



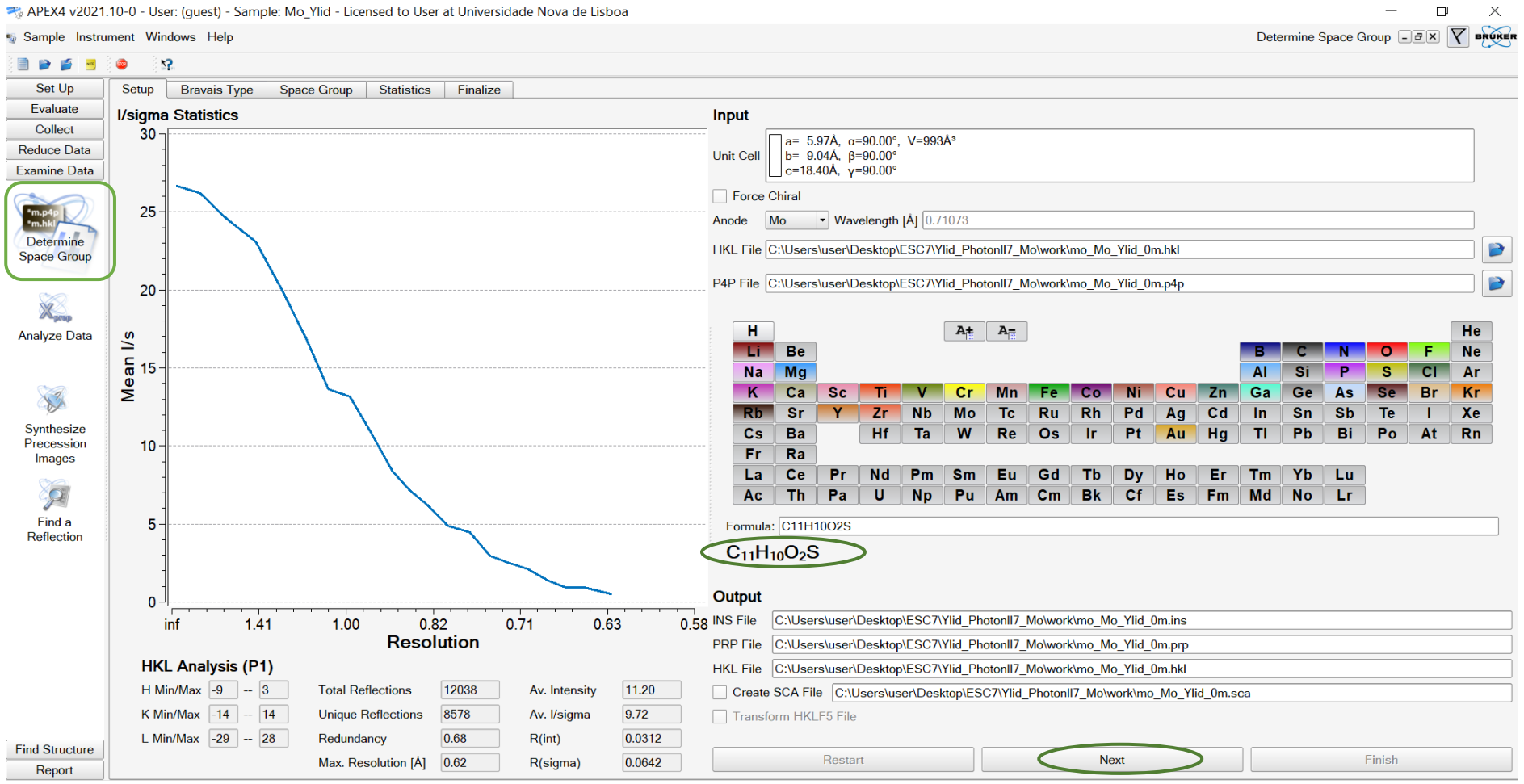


APEX4 - Data processing

Examine data: Determine space group

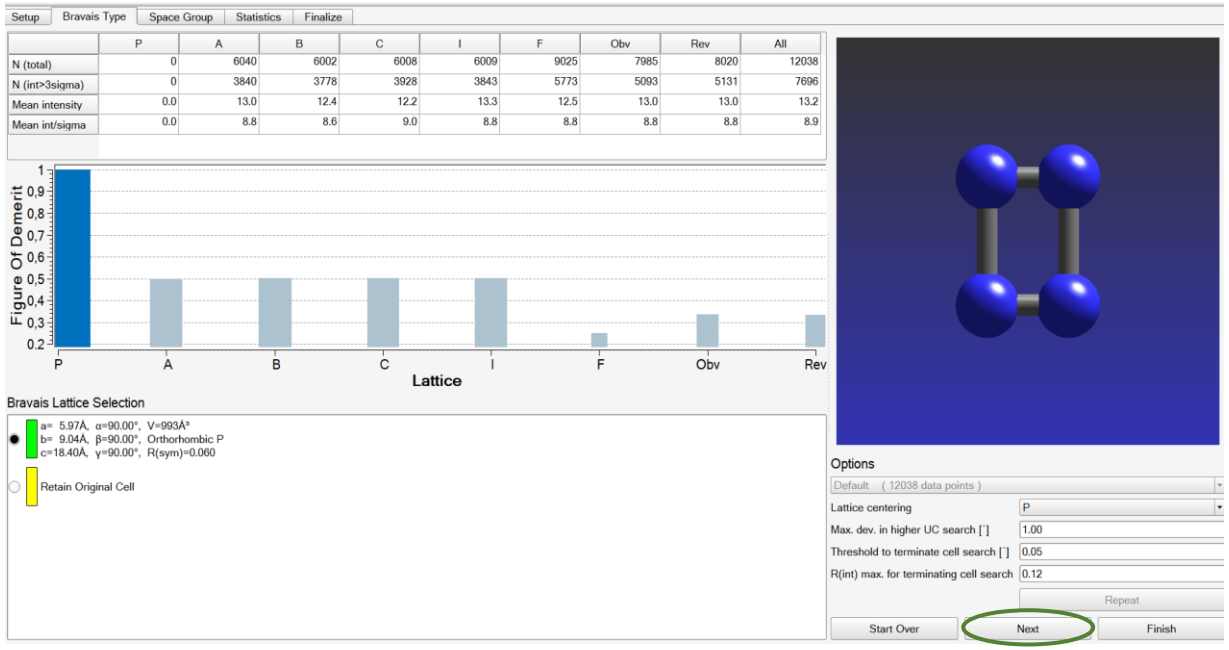
Required files:

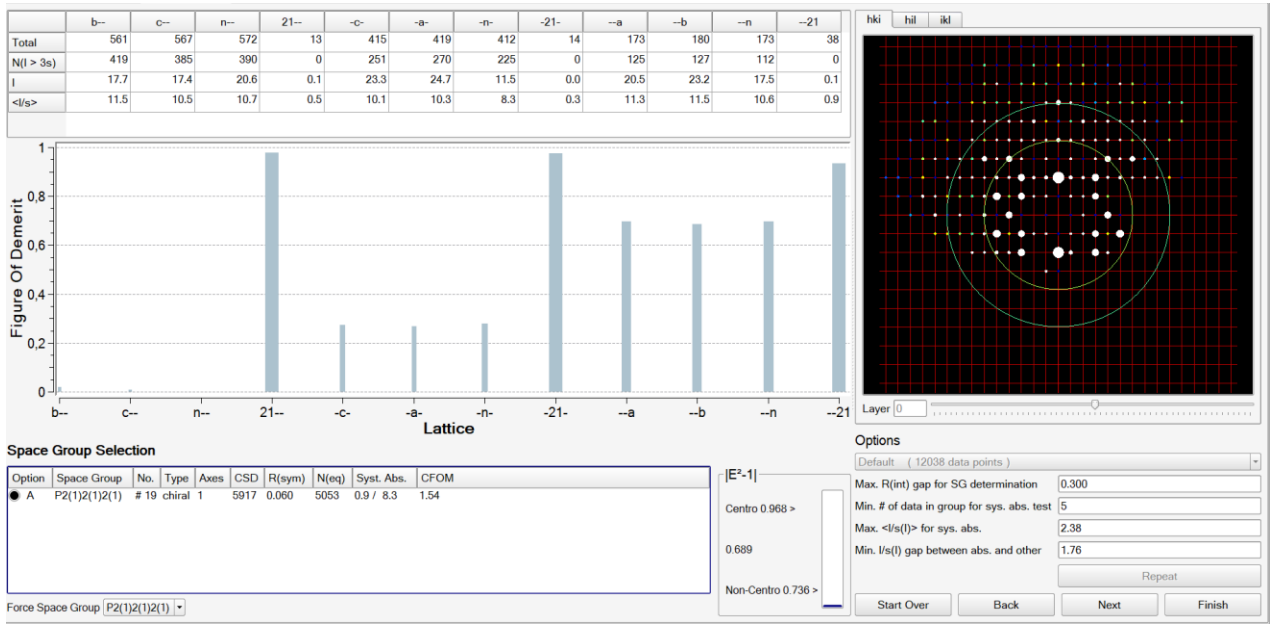
Name.p4p (contains final unit cell parameters from integration) and ***Name.hkl*** (contains corrected intensities)



APEX4 - Data processing

Examine data: Determine space group



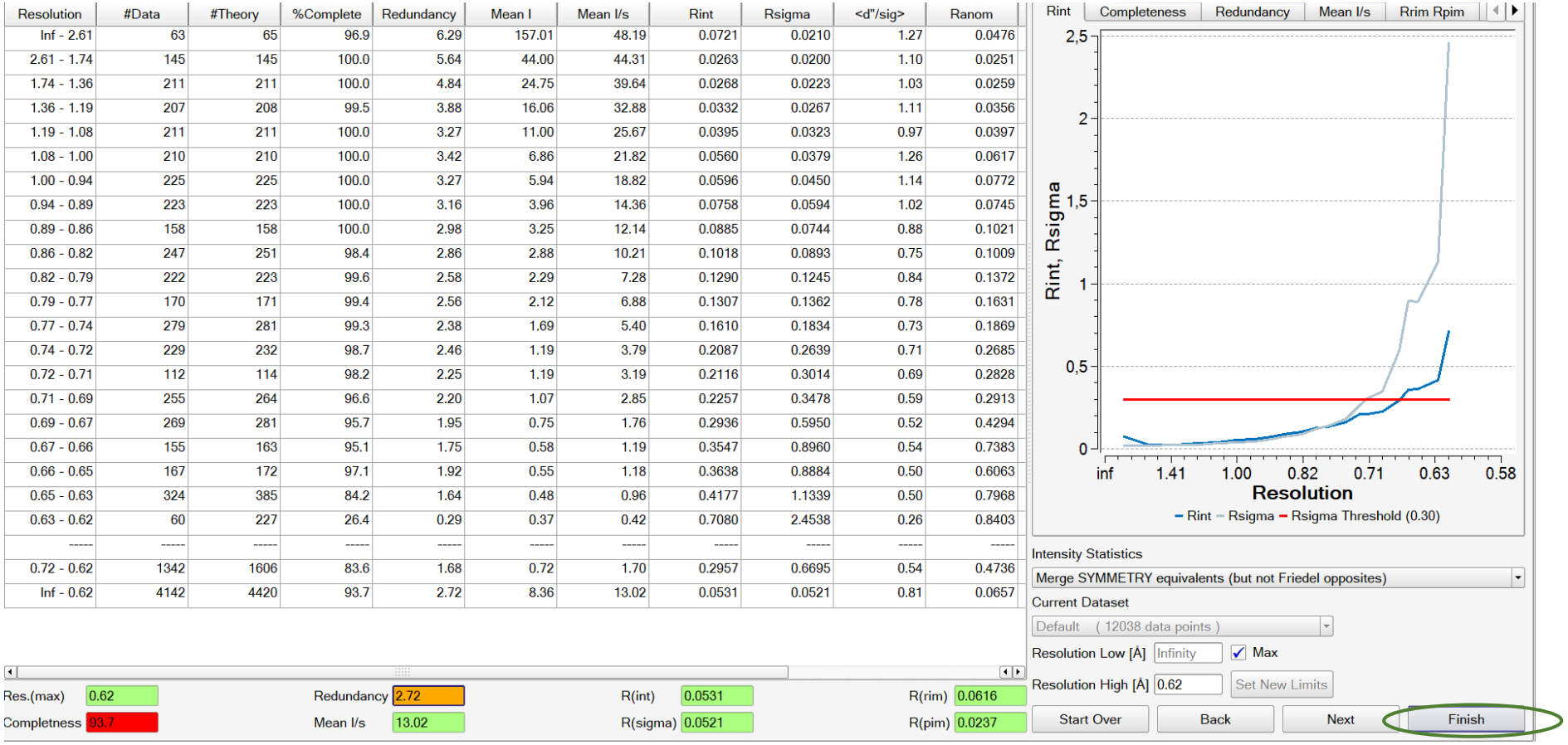


- Suggestion of space group based on the previously chosen Bravais lattice type.
- The higher and wider a peak, the more likely the correct space group

APEX4 - Data processing

Examine data: Determine space group

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma	<d"/sig>	Ranom
Inf - 2.61	63	65	96.9	6.29	157.01	48.19	0.0721	0.0210	1.27	0.0476
2.61 - 1.74	145	145	100.0	5.64	44.00	44.31	0.0263	0.0200	1.10	0.0251
1.74 - 1.36	211	211	100.0	4.84	24.75	39.64	0.0268	0.0223	1.03	0.0259
1.36 - 1.19	207	208	99.5	3.88	16.06	32.88	0.0332	0.0267	1.11	0.0356
1.19 - 1.08	211	211	100.0	3.27	11.00	25.67	0.0395	0.0323	0.97	0.0397
1.08 - 1.00	210	210	100.0	3.42	6.86	21.82	0.0560	0.0379	1.26	0.0617
1.00 - 0.94	225	225	100.0	3.27	5.94	18.82	0.0596	0.0450	1.14	0.0772
0.94 - 0.89	223	223	100.0	3.16	3.96	14.36	0.0758	0.0594	1.02	0.0745
0.89 - 0.86	158	158	100.0	2.98	3.25	12.14	0.0885	0.0744	0.88	0.1021
0.86 - 0.82	247	251	98.4	2.86	2.88	10.21	0.1018	0.0893	0.75	0.1009
0.82 - 0.79	222	223	99.6	2.58	2.29	7.28	0.1290	0.1245	0.84	0.1372
0.79 - 0.77	170	171	99.4	2.56	2.12	6.88	0.1307	0.1362	0.78	0.1631
0.77 - 0.74	279	281	99.3	2.38	1.69	5.40	0.1610	0.1834	0.73	0.1869
0.74 - 0.72	229	232	98.7	2.46	1.19	3.79	0.2087	0.2639	0.71	0.2685
0.72 - 0.71	112	114	98.2	2.25	1.19	3.19	0.2116	0.3014	0.69	0.2828
0.71 - 0.69	255	264	96.6	2.20	1.07	2.85	0.2257	0.3478	0.59	0.2913
0.69 - 0.67	269	281	95.7	1.95	0.75	1.76	0.2936	0.5950	0.52	0.4294
0.67 - 0.66	155	163	95.1	1.75	0.58	1.19	0.3547	0.8960	0.54	0.7383
0.66 - 0.65	167	172	97.1	1.92	0.55	1.18	0.3638	0.8884	0.50	0.6063
0.65 - 0.63	324	385	84.2	1.64	0.48	0.96	0.4177	1.1339	0.50	0.7968
0.63 - 0.62	60	227	26.4	0.29	0.37	0.42	0.7080	2.4538	0.26	0.8403
-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
0.72 - 0.62	1342	1606	83.6	1.68	0.72	1.70	0.2957	0.6695	0.54	0.4736
Inf - 0.62	4142	4420	93.7	2.72	8.36	13.02	0.0531	0.0521	0.81	0.0657

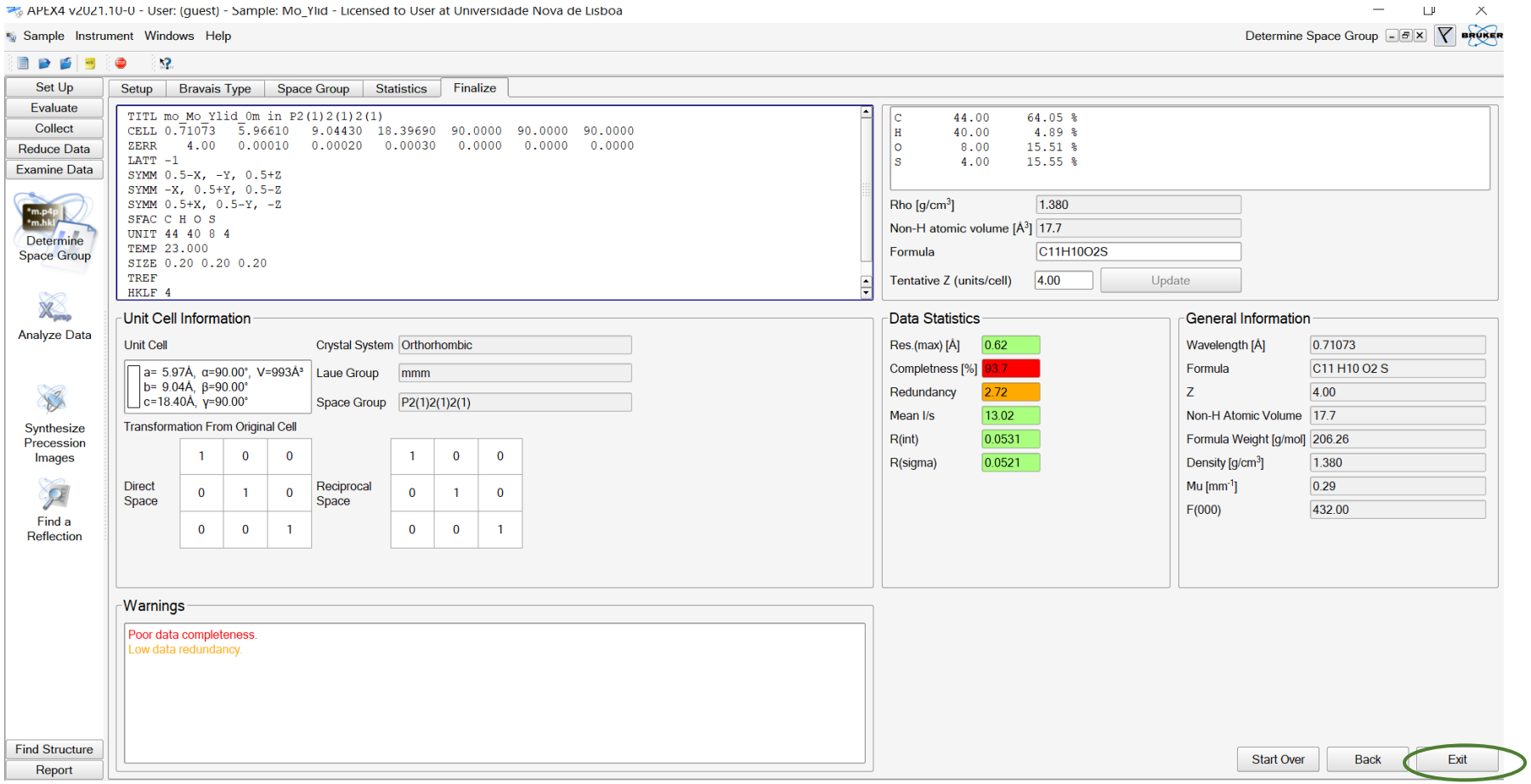


The **quality of the dataset** are listed in correlation to a given resolution.
Gree = good
Orange = medium
Red = bad

Examine **completeness**, **Redundancies**, $\mathbf{R_{int}}$ and $\mathbf{R_{sigma}}$.

APEX4 - Data processing

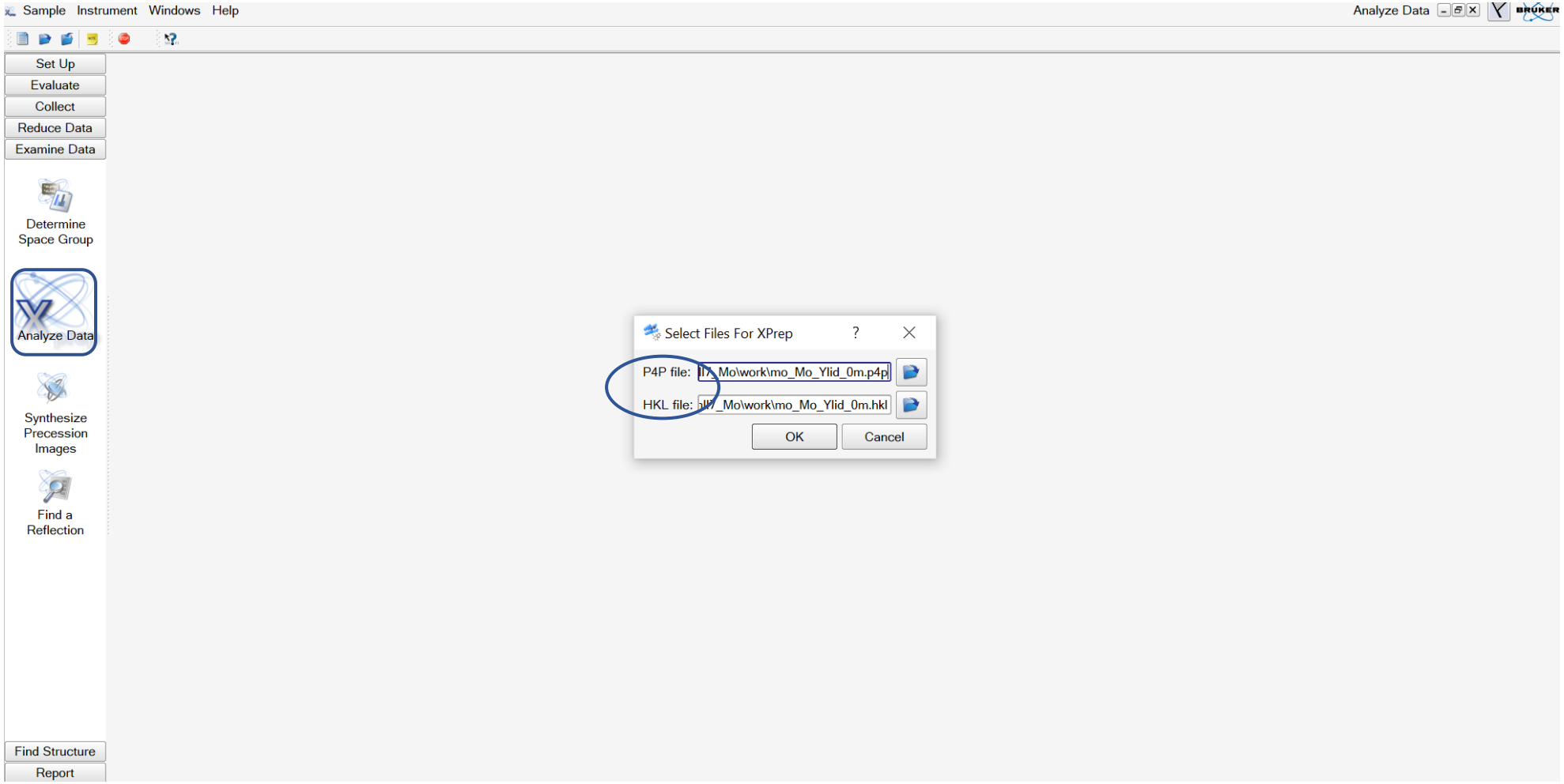
Examine data: Determine space group



Check **Rho/density** (1.1 to 1.4 for organic molecules, higher for inorganic compounds), **Z**, **Non-H atomic volume** (around 16 to 20), **Crystal system**, **Laue Group** and **Space Group**.

APEX4 - Data processing

Examine data: Xprep – analyze data
(alternative to "determine space group")



APEX4 - Data processing

Examine data: Xprep – analyze data