



## Data processing with APEX4

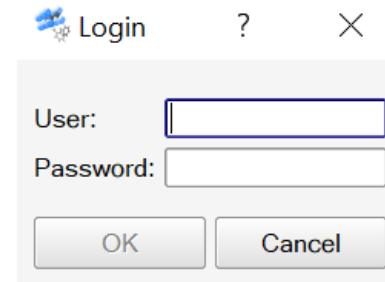
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# APEX4 – New sample

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



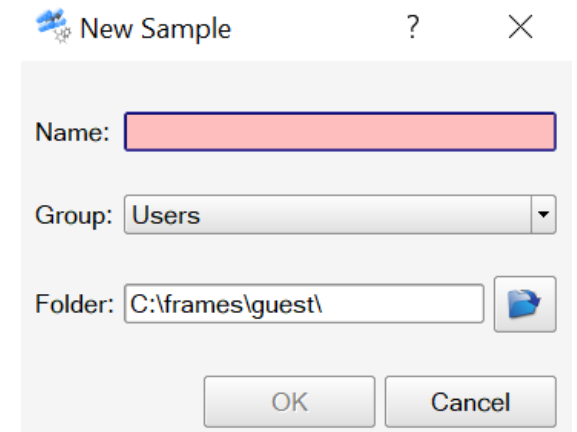
Login ? X

User:

Password:

OK Cancel


- **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



New Sample ? X

Name:


Group: Users

Folder: C:\frames\guest\ 

OK Cancel

# APEX4 – New sample

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Describe Sample 

Set Up

Name:

Compound:

Formula:

Appearance	Intensity	Primary Color	Secondary Color
Crystal Color: <input type="text" value="translucent"/>	<input type="text" value="intense"/>	<input type="text" value="yellow"/>	<input type="text" value="n/a"/>

Crystal Dimensions:  x  x  [mm]

Crystal Shape:

Describe Sample

Center Crystal

Screen Crystal

Structure Now

- **Set Up** menu

Mandatory fields: Formula and Crystal dimensions

# APEX4 – Search Unit Cell

## Evaluate menu

APEX4 v2021.10.0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Unit Cell

Ylid\_Photon17\_MoImo\_matrix\_01\_0001.sfrm

Set Up Evaluate

Determine Unit Cell

Transform Unit Cell

Compare Unit Cells

View Reciprocal Lattice

View Images

Collect Reduce Data Examine Data Find Structure Report

Cursor  
Position [mm] 27.87 34.64  
Position [pixels] 590 511  
Intensity [counts] 11  
HKL index -3.58 -10.15 -0.29  
Resolution [Å] 0.93  
2Theta [°] 45.01

Image Header Tool Editor Cursor Position

Automatic Mode  
Start at: Collect Data  
Stop after: Search  
Run

Manual Mode  
Collect Data  
Harvest Spots  
Index  
Domains  
Bravais  
Refine  
Search

Unit cells:  
Edit...  
Delete  
Delete All

Reflections:  
Edit...  
Delete  
Delete All

Expected Resolution Estimate Exposure Time  
Target Resolution [Å] 0.80  
I/Sigma at Selected Resolution 10  
Recommended Exposure Time: NA  
Crystal Mosaicity [°]: 0.40

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

## Evaluate menu

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Unit Cell

Ylid\_Photon17\_Molmo\_matrix\_01\_0001.sfrm

Set Up Evaluate

Determine Unit Cell

Transform Unit Cell

Compare Unit Cells

View Reciprocal Lattice

View Images

Collect

Reduce Data

Examine Data

Find Structure

Report

Automatic Mode

Start at: Collect Data

Stop after: Search

Run

Manual Mode

Collect Data

Harvest Spots

Index

Domains

Bravais

Refine

Search

Unit cells:

Edit...

Delete

Delete All

Reflections:

Edit...

Delete

Delete All

Expected Resolution Estimate Exposure Time

Target Resolution [Å] 0.80

I/Sigma at Selected Resolution 10

Recommended Exposure Time: NA

Crystal Mosaicity [°]: 0.40

Cursor

Position [mm] 27.87 34.64

Position [pixels] 590 511

Intensity [counts] 11

HKL index -3.58 -10.15 -0.29

Resolution [Å] 0.93

Image Header Tool Editor Cursor Position

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

# APEX4 - Search Unit Cell

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Unit Cell

Ylid\_Photon17\_Mo/mo\_matrix\_01\_0001.sfrm

Set Up Evaluate

Determine Unit Cell

Transform Unit Cell

Compare Unit Cells

View Reciprocal Lattice

View Images

Collect

Reduce Data

Examine Data

Find Structure

Report

Cursor

Position [mm]	51.01	6.49
Position [pixels]	761	303
Intensity [counts]	19	
HKL index	-3.55	-7.22 -7.87
Resolution [Å]	0.89	
$2\theta$ [deg]	47.46	

1600

1400

1200

1000

800

600

400

200

First Image: C:\Users\...Ylid\_Photon17\_Mo/mo\_matrix\_01\_0001.sfrm

Number of Runs: 2 Images per Run: 12

Go to Image: C:\Users\...mo\_matrix\_01\_0001.sfrm

Min. I./sigma(I): 10.00

More Spots Fewer Spots

Smooth images

From [A] To [A]

Add...

Edit...

Delete

Delete All

Excluded Shells:

Store: empty

Save only reflections that span images

Finish Harvest Cancel

## Harvest Spots

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

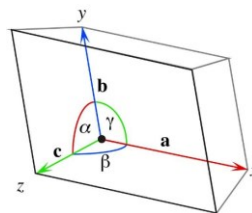
# APEX4 - Search Unit Cell

## Indexing: calculating the unit cell

↪ smallest repeating unit of the crystal lattice

### Unit cell parameters:

axis –  $a, b, c$   
angles –  $\alpha, \beta, \gamma$



Automatic Mode: Start at: Collect Data, Stop after: Search

Manual Mode:  Collect Data,  Harvest Spots

Buttons: Run, **Index**, Domains, Bravais, Refine, Search

Unit cells: [Empty list] Edit..., Delete, Delete All

Reflections: Group 0: 411 reflections Edit..., Delete, Delete All

Expected Resolution: 0.80 Å, I/Sigma at Selected Resolution: 10, Recommended Exposure Time: 7 sec/degree, Crystal Mosaicity [°]: 0.67

Reflections: Group 0: 411 reflections

Go to Image: C:\Users\...\Ylid\_Photon17\_Molmo\_matrix\_01\_0001.sfrm

Min. I/sigma(I): 10.00, Resolution [Å]: 9999.00 - 0.70

Reflections must be isolated,  Reflections must span images,  Reflections must be whole

411 Reflections selected for Indexing

Store: Empty

Corrections:  From store,  From last harvest,  Manual

Distance [mm]: 0.00, Pitch [°]: 0.00, X Beam Center [mm]: -1.60, Roll [°]: 0.00, Y Beam Center [mm]: 0.41, Yaw [°]: 0.00

Methods:  Difference Vectors,  Fast Fourier Transform,  Least Squares

Buttons: Finish, **Index...**, Cancel

Reduced Unit Cells found:

Method	Score	HKL histogram
Method: Difference Vectors	Score: 0.38	0.1: 42.3% (174/411) 0.2: 80.0% (329/411) 0.3: 92.9% (382/411)
Method: Fast Fourier Transform	Score: 0.46	0.1: 46.7% (192/411) 0.2: 75.4% (310/411) 0.3: 89.5% (368/411)

Parameters for the selected method (Fast Fourier Transform):  
a = 5.86 Å, α = 90.18°, V = 955 Å³  
b = 10.67 Å, β = 90.34°  
c = 18.28 Å, γ = 123.39°

Buttons: Finish, **Accept...**, Cancel

Unit Cell: a = 5.86 Å, α = 90.18°, V = 955 Å³, b = 10.67 Å, β = 90.34°, c = 18.28 Å, γ = 123.39°

Parameter	Value
a [Å]	5.945 ± 0.011
b [Å]	10.772 ± 0.016
c [Å]	18.40 ± 0.03
α [°]	90.31 ± 0.04
β [°]	90.23 ± 0.07
γ [°]	123.57 ± 0.05
V [Å³]	982 ± 4

Parameters: Domain translation (x, y, z in mm) and Domain orientation (ω, χ in degrees)

Reflections: Group 0: 411 reflections

Go to Image: C:\Users\...\Ylid\_Photon17\_Molmo\_matrix\_01\_0001.sfrm

Tolerance: 0.35, 214 Reflections selected for Refinement

Show selected Reflections,  Show predicted Reflections

RMS XY [mm]: 0.060, RMS angle [°]: 0.212

Tools: **Refine**, Histograms..., Transformations...

Buttons: Finish, **Accept**, Cancel

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

**Automatic Mode**

Start at:

Stop after:

**Manual Mode**

Collect Data

Harvest Spots

Index

Domains

Bravais

Refine

Search

**Unit cells:**

a= 5.86Å, $\alpha$ = 90.18°, V=955Å <sup>3</sup> b=10.67Å, $\beta$ = 90.34° c=18.28Å, $\gamma$ =123.39°	<input type="button" value="Edit..."/>  <input type="button" value="Delete"/>  <input type="button" value="Delete All"/>
a= 5.95Å, $\alpha$ = 90.31°, V=982Å <sup>3</sup> b=10.77Å, $\beta$ = 90.23° c=18.40Å, $\gamma$ =123.57°	<input type="button" value="Delete All"/>

**Reflections:**

Group 0: 411 reflections	<input type="button" value="Edit..."/>  <input type="button" value="Delete"/>  <input type="button" value="Delete All"/>
--------------------------	--

Expected Resolution    Estimate    Exposure Time

Target Resolution [Å]   

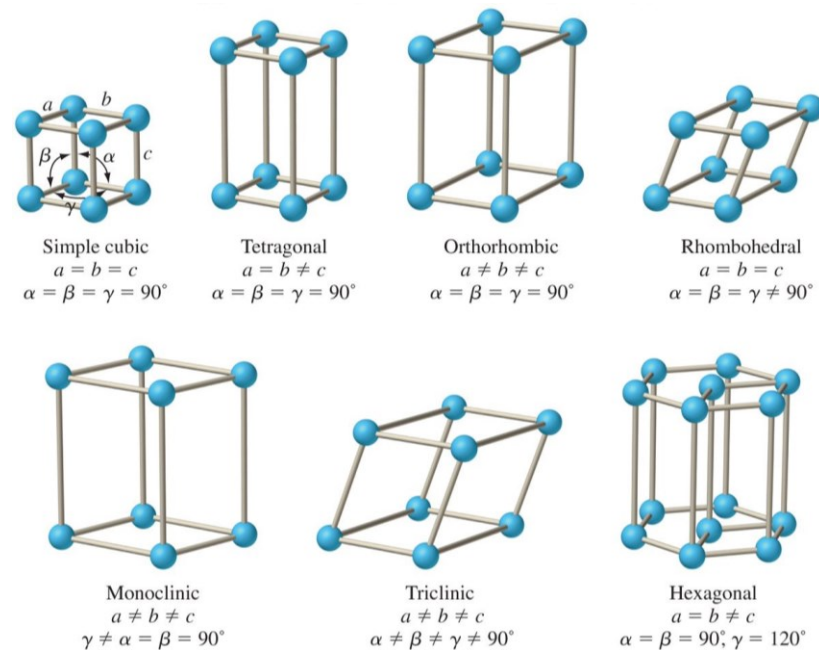
I/Sigma at Selected Resolution   

Recommended Exposure Time: 7 sec/degree

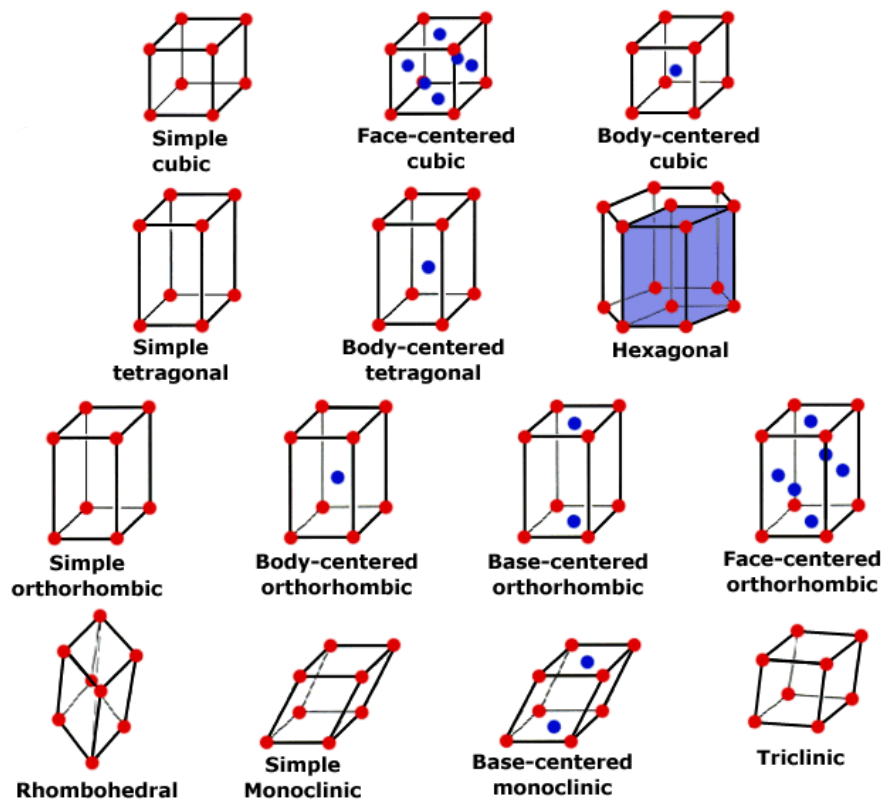
Crystal Mosaicity [°]: 0.67

## Bravais

### 7 Crystal Systems



## 14 Bravais Lattices



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

Initial Unit Cell:

a= 5.95Å,  $\alpha$ = 90.31°, V=982Å<sup>3</sup>  
 b=10.77Å,  $\beta$ = 90.23°  
 c=18.40Å,  $\gamma$ =123.57°

Part:

Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	$\alpha$ [°]	$\beta$ [°]	$\gamma$ [°]
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

Navigation: Finish, Accept, Cancel

- 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 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Unit Cell

Ylid\_Photon17\_Mo\mo\_matrix\_01\_0001.sfrm

Set Up Evaluate

Determine Unit Cell Transform Unit Cell Compare Unit Cells View Reciprocal Lattice View Images

Unit Cell:  $a = 5.95 \text{ \AA}$ ,  $\alpha = 90.00^\circ$ ,  $V = 982 \text{ \AA}^3$   
 $b = 8.98 \text{ \AA}$ ,  $\beta = 90.00^\circ$ , Orthorhombic P  
 $c = 18.40 \text{ \AA}$ ,  $\gamma = 90.00^\circ$

Parameters:

Unit cell	
a [Å]	5.977 ± 0.007
b [Å]	9.013 ± 0.011
c [Å]	18.49 ± 0.02
α [°]	90.00
β [°]	90.00
γ [°]	90.00
V [Å <sup>3</sup> ]	996 ± 3

Domain translation

x [mm]	0.00
y [mm]	0.00
z [mm]	0.00

Domain orientation

ω [°]	-161.46 ± 0.03
χ [°]	80.77 ± 0.03

Reflections: Group 0: 411 reflections

Go to Image: C:\Users\... \Ylid\_Photon17\_Mo\mo\_matrix\_01\_0001.sfrm

Tolerance: 0.18 More Reflections Fewer Reflections

138 Reflections selected for Refinement

Show selected Reflections  
 Show predicted Reflections

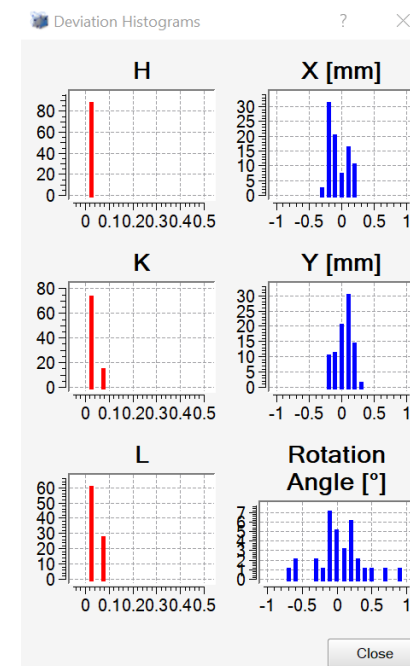
RMS XY [mm]: 0.056    RMS angle [°]: 0.157

Tools: **Refine** Histograms... Transformations...

**Accept** Cancel

Cursor  
 Position [mm] 35.99 -8.93  
 Position [pixels] 650 189  
 Intensity [counts] 5  
 HKL index 0.09 8.28 1.56  
 Resolution [Å] 1.07  
 2θ [°] 32.00

Image Header Tool Editor Cursor Position



## Search

Unit Cell: Part:

a= 5.98Å, α=90.00°, V=1004Å<sup>3</sup>  
 b= 9.04Å, β=90.00°, Orthorhombic P  
 c=18.59Å, γ=90.00°

Formula	a [Å]	b [Å]	c [Å]	α [°]	β [°]
C11 H8 Cl N3	6.11	9.09	18.68	90.00	91.4
(C13 H16 Cd1 Cl2 O9 P2)n,n(H2 O1)	5.84	9.23	18.69	90.00	91.1
C10 H7 Cl2 N O	5.88	8.77	18.67	90.00	90.0
C20 H20 Br2 N2 O2	5.91	18.86	9.01	90.00	91.8
C21 H24 Br2 N2 O2	5.96	18.55	9.05	90.00	90.0
C10 H15 N O4	6.17	9.27	18.63	90.00	90.1
C8 H8 Cl3 N O3	6.00	9.27	18.81	90.00	90.0
C21 H24 Br2 N2 O2	5.96	18.55	9.05	90.00	90.0
C23 H30 N2 O2	18.54	6.06	8.94	90.00	90.0

Elements Filter:

Only These Elements

Databases

<input checked="" type="checkbox"/> APEX DB	<input checked="" type="checkbox"/> 29 CSD	<input checked="" type="checkbox"/> 2 COD	<input type="checkbox"/> local CIF/RES
created: 2021-10-08	created: 2021-10-20	created: 2022-07-09	

Unit Cell: Part:

a= 5.98Å, α=90.00°, V=1004Å<sup>3</sup>  
 b= 9.04Å, β=90.00°, Orthorhombic P  
 c=18.59Å, γ=90.00°

Formula	a [Å]	b [Å]	c [Å]	α [°]	β [°]
C11 H10 O2 S	5.96	9.04	18.40	90.00	90.0
C11 H10 O2 S	5.96	9.04	18.40	90.00	90.0
C14 H10 Br2 Ge2 O4,C4 H8 O2	9.12	5.82	18.78	90.00	91.8
C11 H8 Cl N3	6.11	9.09	18.68	90.00	91.4
(C13 H16 Cd1 Cl2 O9 P2)n,n(H2 O1)	5.84	9.23	18.69	90.00	91.1
C10 H7 Cl2 N O	5.88	8.77	18.67	90.00	90.0
C20 H20 Br2 N2 O2	5.91	18.86	9.01	90.00	91.8
C21 H24 Br2 N2 O2	5.96	18.55	9.05	90.00	90.0
C10 H15 N O4	6.17	9.27	18.63	90.00	90.1

Elements Filter:

Only These Elements

Databases

<input checked="" type="checkbox"/> APEX DB	<input checked="" type="checkbox"/> 29 CSD	<input checked="" type="checkbox"/> 2 COD	<input type="checkbox"/> local CIF/RES
created: 2021-10-08	created: 2021-10-20	created: 2022-07-09	

CCDC FIZ Karlsruhe LEIBNIZ INSTITUTE FOR INFORMATION INFRASTRUCTURE CSD Entry: MSULIN09 Sign In

Simple Search Structure Search Unit Cell Search Formula Search

Your query was: Identifier(s): MSULIN09 and the search returned 1 record.

Results

Database Identifier	Deposition Number
<input checked="" type="checkbox"/> MSULIN09	1433819

MSULIN09 : 2-(dimethylsulfanylidene)-1H-indene-1,3(2H)-dione  
 Space Group: P 2<sub>1</sub> 2<sub>1</sub> (19), Cell: a 5.96330(10)Å b 9.0417(2)Å c 18.4007(4)Å, α 90° β 90° γ 90°

3D viewer

Ball and Stick

Chemical diagram

Additional details

Deposition Number	1433819
Data Citation	L. Krause, R. Herbst-Irmer, D. Stalke CCDC 1433819: Experimental Crystal Structure Determination, 2015, DOI: 10.5517/cc1k407y
Additional Database Identifiers	RUTXOV
Deposited on	28/10/2015

- 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.

## Collect: Calculate strategy

**Calculate Strategy**

**Run Experiment**

**Orion Crystal**

**Reduce Data**

**Examine Data**

**Find Structure**

**Report**

**Prepare** **Edit**

Step 1. Which unit cell should the strategy be based on

Data set with 1993 reflections  
Two theta limit 52.0 degrees  
H from 0 to 7  
K from 0 to 11  
L from -22 to 22  
Applied symmetry:  
Point group 222: Ousdrant

$a = 5.97\text{\AA}$ ,  $\alpha = 90.00^\circ$ ,  $V = 995\text{\AA}^3$   
 $b = 9.07\text{\AA}$ ,  $\beta = 90.00^\circ$ , Orthorhombic P  
 $c = 18.39\text{\AA}$ ,  $\gamma = 90.00^\circ$

Restart

Estimated resolution

Anode: Mo Resolution: 0.81 Å

Detector Masking: Positional

Symmetry: Chiral (222)

Apply

Already measured:

Measure from file:

Step 2. Which runs should be collected

1\_P 12 min ( 0.2h +0 Tue 08:57) 4778 refls, 1977 unique; P90mult 1.7; Av. mult 2.4; Complete 99.20 %  
2\_O 8 min ( 0.3h +0 Tue 09:05) 3793 refls, 15 unique; P90mult 3.1; Av. mult 4.3; Complete 99.95 %

P90 multiplicity: 3.1 Missing reflections: 1 (beamstop reflections: 1)  
Average multiplicity: 4.3 Completeness: 99.95 %

Multiplicity percentiles:  
1% of all reflections have a multiplicity greater than 7.3  
10% of all reflections have a multiplicity greater than 6.3  
50% of all reflections have a multiplicity greater than 4.9  
90% of all reflections have a multiplicity greater than 3.1  
99% of all reflections have a multiplicity greater than 2.0

Cumulative multiplicity:  
Reflections with multiplicity 1 and higher: 99.9 %  
Reflections with multiplicity 2 and higher: 99.4 %  
Reflections with multiplicity 3 and higher: 92.3 %  
Reflections with multiplicity 4 and higher: 75.8 %  
Reflections with multiplicity 5 and higher: 47.5 %  
Reflections with multiplicity 6 and higher: 14.4 %

Determine strategy... Extend strategy... Show reciprocal lattice view

Step 3. How should they be collected

Measurement with 2.00 degree frame angle: 10.0 seconds per frame ( 5.00 [sec/deg], shutterless )  
Total time: 21 [minutes]  
Expected end time: (2021, Tue, Mar 23, 09:05)

Select scan parameters...

Completeness: 99.95  
Average Multiplicity: 4.3

**IMPORTANT**

$$\text{Resolution (d)} = \sin \theta / \lambda$$

## Collect: Run experiment

APEX3 v2019.1.0 - User: (guest) - Sample: Ylid\_Photon2 - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Run Experiment

Set Up Experiment Monitor Experiment

Image location: D:\frames\guest\Ylid\_Photon2

Filename or prefix: YlidData

Number for first run: 1

Scan options:

Shuttered operations

Exposures: Auto Correlate

Default time: 10.000 [sec/image]

Default width: 0.500 [degrees]

Detector format: 1024x1024

Deicing: off

Generate new dark images

Retake if topped

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										

Append Strategy Append Matrix Strategy Load Table... Save Table... Validate Resume Execute

[03/23/2021 08:42:37] New client program connected to BIS

## Reduce data: Integrate images

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Chart Windows Help Integrate Images

Set Up Evaluate Collect Reduce Data Integrate Images Index Crystal Faces Scale Integrate Debye Rings Unwrap and Convert Images Examine Data Find Structure Report

Starting Image Filename	Images	Resolution Limit [Å]	Output Filename
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			

Resolution Limit [Å]: 0.545

Unit Cells:  
a= 5.98Å, α=90.00°, V=1004Å<sup>3</sup>  
b= 9.04Å, β=90.00°, Orthorhombic  
c=18.59Å, γ=90.00°

Edit Domains...

Refinement Options...  
Integration Options...  
Find Runs...  
Import Runs from Experiment  
Start Integration...

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Chart Windows Help Integrate Images

Set Up Evaluate Collect Reduce Data Integrate Images Index Crystal Faces Scale Integrate Debye Rings Unwrap and Convert Images Examine Data Find Structure Report

Starting Image Filename	Images	Resolution Limit [Å]	Output Filename
1 C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Mo\mo_Ylid_01_0001.sfrm	240		C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Molwork\mo_Ylid_01.raw
2 C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Mo\mo_Ylid_02_0001.sfrm	240		C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Molwork\mo_Ylid_02.raw
3 C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Mo\mo_Ylid_03_0001.sfrm	240		C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Molwork\mo_Ylid_03.raw
4 C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Mo\mo_Ylid_04_0001.sfrm	240		C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Molwork\mo_Ylid_04.raw
5 C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Mo\mo_Ylid_05_0001.sfrm	180		C:\Users\user\Desktop\ESC7Ylid_PhotonI7_Molwork\mo_Ylid_05.raw
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			

Resolution Limit [Å]: 0.545

Unit Cells:  
a= 5.98Å, α=90.00°, V=1004Å<sup>3</sup>  
b= 9.04Å, β=90.00°, Orthorhombic  
c=18.59Å, γ=90.00°

Edit Domains...

Refinement Options...  
Integration Options...  
Find Runs...  
Import Runs from Experiment  
Start Integration...

Select Runs

Look in: C:\Users\user\Desktop\ESC7Ylid\_PhotonI7\_Mo

Name	Size	Type	Date Modified
Mo_Ylid.xml	493 KB	xml File	09/07/2022 20:13:48
mo_Matri_013.sfrm	391 KB	sfrm File	07/07/2022 22:52:39
mo_Matri_014.sfrm	391 KB	sfrm File	07/07/2022 22:52:37
mo_Matri_015.sfrm	392 KB	sfrm File	07/07/2022 22:52:37
mo_Matri_016.sfrm	391 KB	sfrm File	07/07/2022 22:52:37
mo_Matri_017.sfrm	391 KB	sfrm File	07/07/2022 22:52:38
mo_Matri_018.sfrm	391 KB	sfrm File	07/07/2022 22:52:37
mo_Matri_019.sfrm	391 KB	sfrm File	07/07/2022 22:52:38
mo_Matri_020.sfrm	391 KB	sfrm File	07/07/2022 22:52:36
mo_Matri_013.sfrm	392 KB	sfrm File	07/07/2022 22:52:36
mo_Matri_014.sfrm	392 KB	sfrm File	07/07/2022 22:52:37
mo_Matri_015.sfrm	391 KB	sfrm File	07/07/2022 22:52:36
mo_Matri_016.sfrm	392 KB	sfrm File	07/07/2022 22:52:36

Run

- mo\_Matrix\_01\_#### (13 - 20)
- mo\_Matrix\_02\_#### (13 - 20)
- mo\_Ylid\_01\_#### (1 - 240)
- mo\_Ylid\_02\_#### (1 - 240)
- mo\_Ylid\_03\_#### (1 - 240)
- mo\_Ylid\_04\_#### (1 - 240)
- mo\_Ylid\_05\_#### (1 - 180)
- mo\_matrix\_01\_#### (1 - 12)
- mo\_matrix\_02\_#### (1 - 12)

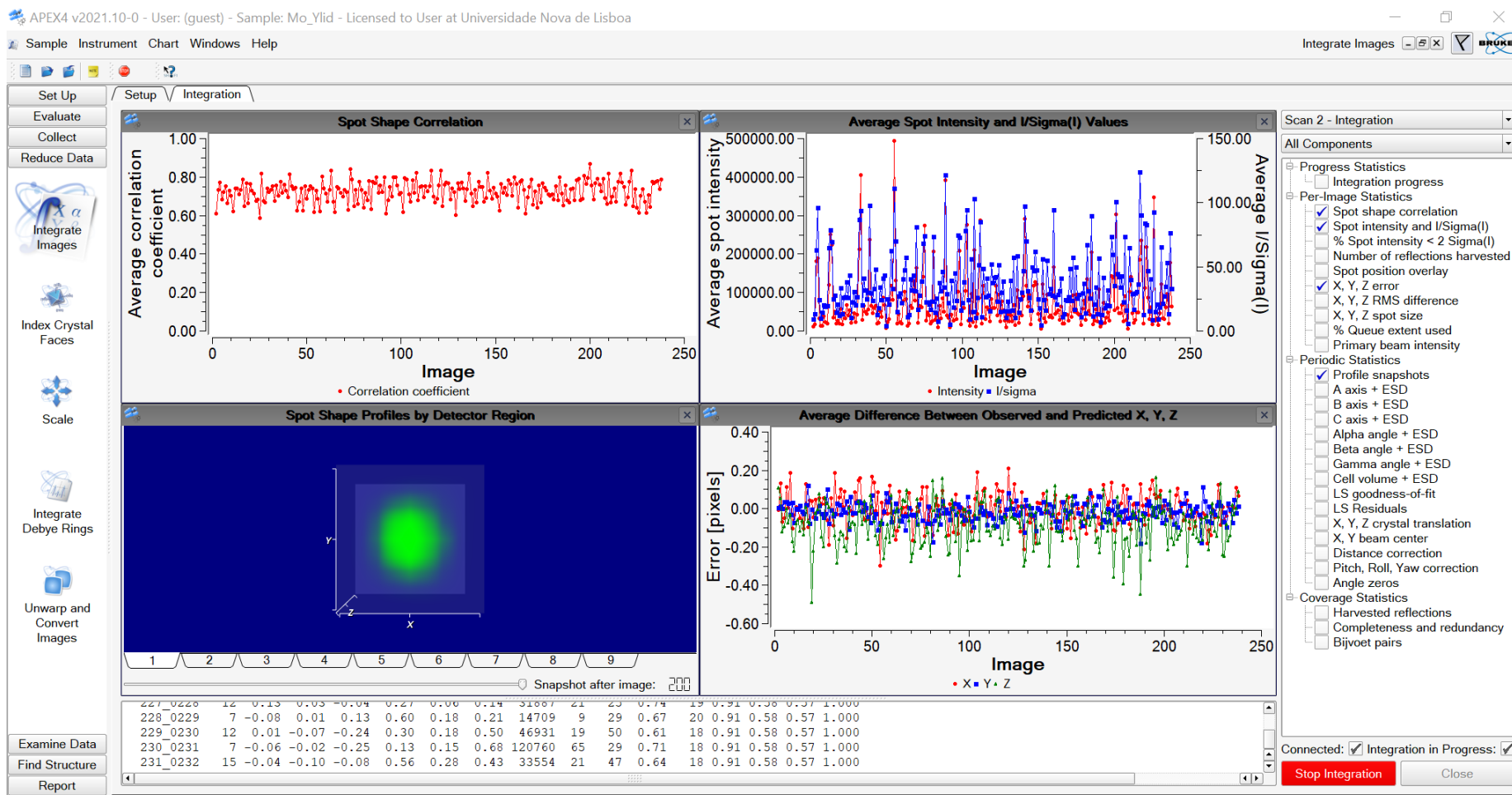
Select all Deselect all

Directory:  Choose

Files of type: Directories Cancel

## 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)



## Reduce data: Scale

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help

Setup Absorption Correction Parameter Refinement Error Model Finalize Diagnostics

Set Up Evaluate Collect Reduce Data

Integrate Images

Index Crystal Faces

Scale

Integrate Debye Rings

Unwrap and Convert Images

Examine Data Find Structure Report

**Setup**

Input Folder:

Input File(s)

- mo\_Mo\_Ylid\_0m.raw

Select All Deselect All

Laue Group:

Point Group:

Additional Spherical Absorption Correction  
Mu\*r of Equivalent Sphere:

Absorption Correction

- Multi-Scan
- Numerical Mu Calculated
- Numerical Mu From Formula

Advanced Setup

Start Over **Start** Finish

Scale

Initial Reflections

Total:

Unique:

Outlier Rejection

High Resolution Limit:

|I-<I> /su ratio for rejection:

g-value:

Reflections after Outlier Rejection

Total:

Percent Rejected [%]:

Unique:

Percent Rejected [%]:

Error Model g-value

Fixed g-value:

Scan	Fast Scan	Domain	2-Theta	R(int)	Incid. Factors	Diffr. Factors	K	g	I/s(lim)	Total	>2sig(I)
<input checked="" type="checkbox"/> 1	<input type="checkbox"/>	1	-28.0	0.0326	0.620 - 1.121	0.975 - 1.086	1.117	0.0461	21.7	2995	1968
<input checked="" type="checkbox"/> 2	<input type="checkbox"/>	1	-28.0	0.0321	0.556 - 0.609	0.966 - 1.047	1.032	0.0461	21.7	2975	2076
<input checked="" type="checkbox"/> 3	<input type="checkbox"/>	1	28.0	0.0309	0.567 - 0.609	0.955 - 1.050	0.958	0.0461	21.7	3025	2217
<input checked="" type="checkbox"/> 4	<input type="checkbox"/>	1	28.0	0.0354	0.537 - 0.606	0.964 - 1.050	0.996	0.0461	21.7	3039	2248
<input checked="" type="checkbox"/> 5f	<input checked="" type="checkbox"/>	1	0.0	0.1049	7.185 - 8.306	0.955 - 1.085	1.375	0.0461	21.7	1029	726

Start Over Error Model Repeat Parameter Refinement Finish

## Examine data: Determine space group

### Required files:

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

APEX4 v2021.10-0 - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Space Group

Set Up Evaluate Collect Reduce Data Examine Data

Setup Bravais Type Space Group Statistics Finalize

**I/sigma Statistics**

Mean I/sigma

Resolution

**Input**

Unit Cell  
 $a = 5.97\text{\AA}$ ,  $\alpha = 90.00^\circ$ ,  $V = 993\text{\AA}^3$   
 $b = 9.04\text{\AA}$ ,  $\beta = 90.00^\circ$   
 $c = 18.40\text{\AA}$ ,  $\gamma = 90.00^\circ$

Force Chiral

Anode Mo Wavelength [Å] 0.71073

HKL File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.hkl

P4P File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.p4p

H																				He	
Li	Be											B	C	N	O	F				Ne	
Na	Mg											Al	Si	P	S	Cl				Ar	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br				Kr	
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I				Xe	
Cs	Ba		Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At				Rn	
Fr	Ra																				
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu							
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr							

Formula: C<sub>11</sub>H<sub>10</sub>O<sub>2</sub>S

**Output**

INS File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.ins

PRP File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.prp

HKL File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.hkl

Create SCA File C:\Users\user\Desktop\ESC7\Ylid\_Photon17\_Mo\work\mo\_Mo\_Ylid\_0m.sca

Transform HKLF5 File

Restart Next Finish

**HKL Analysis (P1)**

H Min/Max	-9 -- 3	Total Reflections	12038	Av. Intensity	11.20
K Min/Max	-14 -- 14	Unique Reflections	8578	Av. I/sigma	9.72
L Min/Max	-29 -- 28	Redundancy	0.68	R(int)	0.0312
		Max. Resolution [Å]	0.62	R(sigma)	0.0642

Find Structure Report

## Examine data: Determine space group

Setup Bravais Type Space Group Statistics Finalize

	P	A	B	C	I	F	Obv	Rev	All
N (total)	0	6040	6002	6008	6009	9025	7985	8020	12038
N (int>3sigma)	0	3840	3778	3928	3843	5773	5093	5131	7696
Mean intensity	0.0	13.0	12.4	12.2	13.3	12.5	13.0	13.0	13.2
Mean int/sigma	0.0	8.8	8.6	9.0	8.8	8.8	8.8	8.8	8.9

Figure Of Demerit

Bravais Lattice Selection

- a= 5.97Å, α=90.00°, V=993Å<sup>3</sup>  
b= 9.04Å, β=90.00°, Orthorhombic P  
c=18.40Å, γ=90.00°, R(sym)=0.060
- Retain Original Cell

Options

Default (12038 data points)

Lattice centering: P

Max. dev. in higher UC search: 1.00

Threshold to terminate cell search: 0.05

R(int) max. for terminating cell search: 0.12

Start Over **Next** Finish

	b--	c--	n--	21--	-c-	-a-	-n-	-21-	--a	--b	--n	--21
Total	561	567	572	13	415	419	412	14	173	180	173	38
N(I > 3s)	419	385	390	0	251	270	225	0	125	127	112	0
I	17.7	17.4	20.6	0.1	23.3	24.7	11.5	0.0	20.5	23.2	17.5	0.1
<I/s>	11.5	10.5	10.7	0.5	10.1	10.3	8.3	0.3	11.3	11.5	10.6	0.9

Figure Of Demerit

Space Group Selection

Option	Space Group	No.	Type	Axes	CSD	R(sym)	N(eq)	Syst. Abs.	CFOM	
<input checked="" type="radio"/>	A	P2(1)2(1)2(1)	# 19	chiral	1	5917	0.060	5053	0.9 / 8.3	1.54

Force Space Group: P2(1)2(1)2(1)

Options

Default (12038 data points)

Max. R(int) gap for SG determination: 0.300

Min. # of data in group for sys. abs. test: 5

Max. <I/s(I)> for sys. abs.: 2.38

Min. I/s(I) gap between abs. and other: 1.76

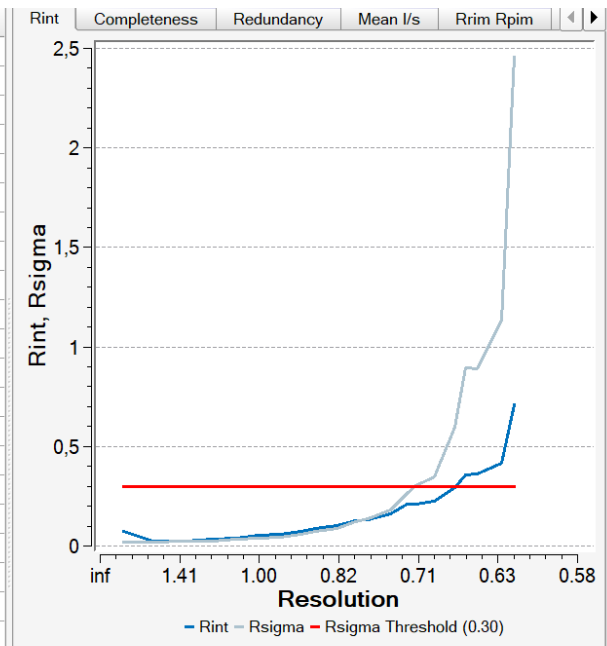
Repeat

Start Over Back **Next** Finish

- 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

## 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.  
 Green = good  
 Orange = medium  
 Red = bad

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

Res.(max) 0.62 Redundancy 2.72 R(int) 0.0531 R(rim) 0.0616

Completeness 93.7 Mean I/s 13.02 R(sigma) 0.0521 R(pim) 0.0237

Start Over Back Next **Finish**

## Examine data: Determine space group

APEX4 v2021.10-U - User: (guest) - Sample: Mo\_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Determine Space Group

Set Up Evaluate Collect Reduce Data Examine Data Determine Space Group Analyze Data Synthesize Precession Images Find a Reflection Find Structure Report

Setup Bravais Type Space Group Statistics Finalize

```
TITL mo_Mo_Ylid_0m_in_P2(1)2(1)2(1)
CELL 0.71073 5.96610 9.04430 18.39690 90.0000 90.0000 90.0000
ZERR 4.00 0.00010 0.00020 0.00030 0.0000 0.0000 0.0000
LATT -1
SYMM 0.5-X, -Y, 0.5+Z
SYMM -X, 0.5+Y, 0.5-Z
SYMM 0.5+X, 0.5-Y, -Z
SFAC C H O S
UNIT 44 40 8 4
TEMP 23.000
SIZE 0.20 0.20 0.20
TREF
HKL 4
```

Unit Cell Information

Unit Cell Crystal System  Laue Group  Space Group  Transformation From Original Cell

Direct Space	1	0	0	Reciprocal Space	1	0	0
	0	1	0		0	1	0
	0	0	1		0	0	1

Warnings

Poor data completeness.  
Low data redundancy.

Res (max) [Å] 0.62  
Completeness [%] 93.7  
Redundancy 2.72  
Mean I/s 13.02  
R(int) 0.0531  
R(sigma) 0.0521

General Information

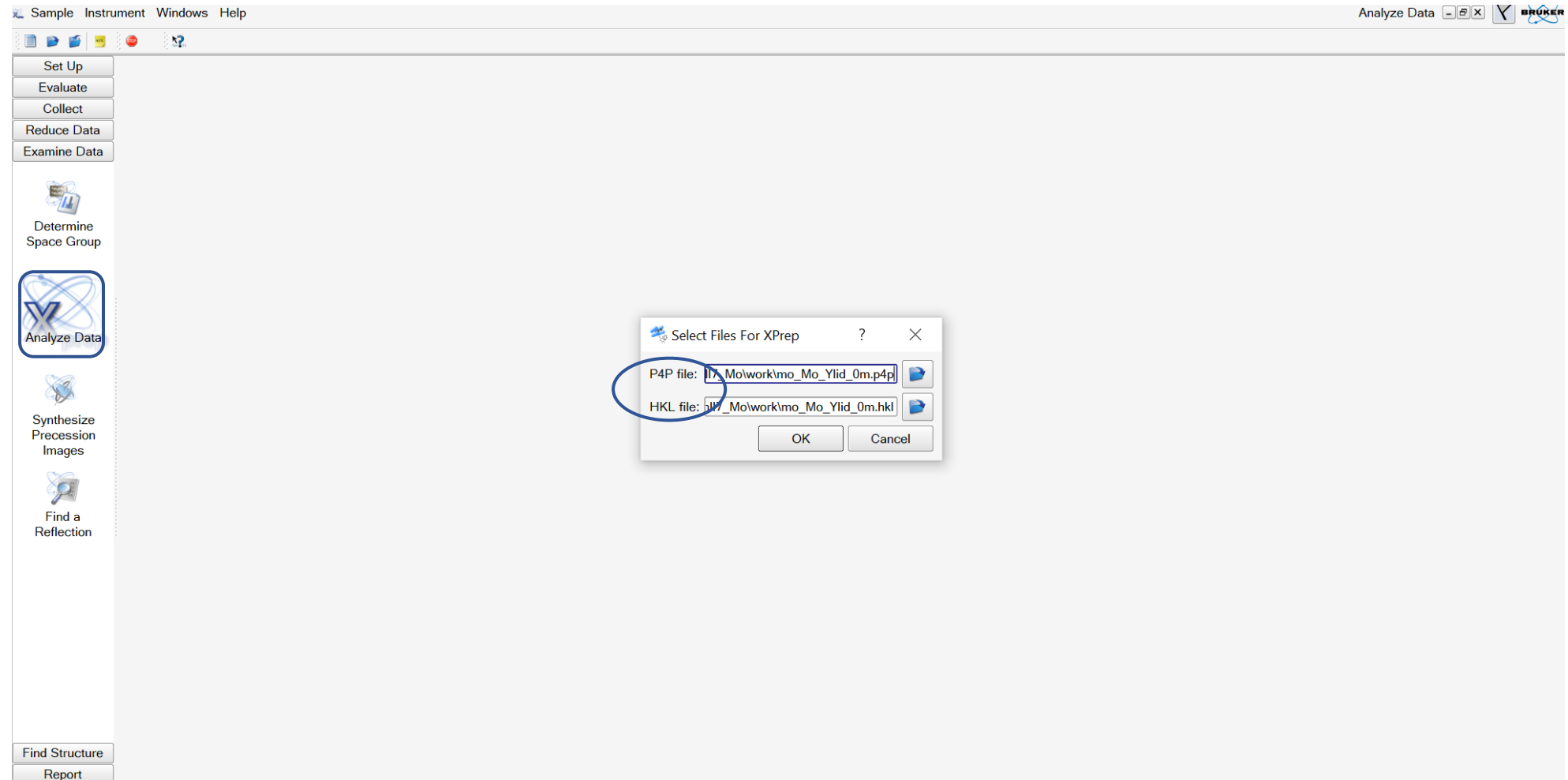
Wavelength [Å] 0.71073  
Formula C11H10O2S  
Z 4.00  
Non-H Atomic Volume 17.7  
Formula Weight [g/mol] 206.26  
Density [g/cm<sup>3</sup>] 1.380  
Mu [mm<sup>-1</sup>] 0.29  
F(000) 432.00

Rho [g/cm<sup>3</sup>] 1.380  
Non-H atomic volume [Å<sup>3</sup>] 17.7  
Formula C11H10O2S  
Tentative Z (units/cell) 4.00 Update

Start Over Back **Exit**

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**.

## Examine data: Xprep – analyze data (alternative to “determine space group”)



The screenshot displays the APEX4 software interface. The main window has a menu bar with 'Sample', 'Instrument', 'Windows', and 'Help'. The right side of the window shows 'Analyze Data' with window control icons and the Bruker logo. On the left, a vertical toolbar contains buttons for 'Set Up', 'Evaluate', 'Collect', 'Reduce Data', 'Examine Data', 'Determine Space Group', 'Analyze Data' (highlighted with a blue border), 'Synthesize Precession Images', 'Find a Reflection', 'Find Structure', and 'Report'. A dialog box titled 'Select Files For XPrep' is open in the center, containing two input fields: 'P4P file: [D:\\_Molwork\mo\_Mo\_Ylid\_0m.p4p]' and 'HKL file: [D:\\_Molwork\mo\_Mo\_Ylid\_0m.hkl]'. Both input fields are circled in blue. The dialog box also features 'OK' and 'Cancel' buttons at the bottom.

## Examine data: Xprep – analyze data

```

XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014
Current dataset: mo_YlidData_Om.hkl      Wavelength: 0.71073 Chiral: ?
-----
Original cell:  5.966  9.045 18.407  90.00  90.00  90.00  Vol  993.3
                Esds:  0.000  0.000  0.000  0.00  0.00  0.00  Lattice: P
-----
Current cell:  5.966  9.045 18.407  90.00  90.00  90.00  Vol  993.3
-----
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-----
Crystal system: Orthorhombic      Lattice: P
-----
[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu or [Q] QUIT program

Select option [S]:

[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal,
[H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT

Select option [O]:

Lattice exceptions: P      A      B      C      I      F      Obv      Rev      All
N (total) =              0  7951  7970  7983  7950 11952 10606 10602 15901
N (int>3sigma) =         0  6555  6434  6715  6523  9852  8677  8640 13022
Mean intensity =        0.0 251.7 240.6 242.0 243.4 244.7 246.4 240.5 239.2
Mean int/sigma =         0.0 15.6 14.9 15.3 15.2 15.3 15.4 15.2 15.2

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:

Mean |E*-1| = 0.685 [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

      b--  c--  n--  21--  -c-  -a-  -n-  -21-  --a  --b  --n  --21
N       819  824  819   20   580  602  572   24  284  281  277   51
N I>3s  683  645  630    2   429  449  370    0  216  229  211    3
<I>    270.9 349.3 348.8  1.8 477.0 449.7 235.4  0.5 333.6 369.4 334.3  2.0
<I/s>   20.0  22.6  21.4  1.0  21.6  20.5  12.0  0.6  19.0  21.6  19.4  1.3

Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym)  N(eq)  Syst. Abs.  CFOM
[A] P2(1)2(1)2(1)  # 19  chiral  1  5917  0.025  3433  1.3 / 12.0  1.20

Select option [A]:

```

```

XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014
-----
Resolution  #Data #Theory %Complete Redundancy Mean I Mean I/s Rmerge Rsigma
Inf - 2.86      37   39      94.9      7.49  149.98  51.45  0.0799  0.0172
2.86 - 1.83     85   85     100.0     8.74   53.34  56.39  0.0316  0.0170
1.83 - 1.43    122  122     100.0     7.82   30.48  49.73  0.0282  0.0184
1.43 - 1.23    126  126     100.0     7.02   14.86  43.60  0.0359  0.0203
1.23 - 1.11    124  125     99.2     5.46   13.87  34.52  0.0393  0.0248
1.11 - 1.02    122  122     100.0     5.86    7.44  28.74  0.0567  0.0283
1.02 - 0.96    122  122     100.0     5.56    6.69  26.12  0.0621  0.0332
0.96 - 0.90    142  142     100.0     5.70    4.71  21.64  0.0772  0.0402
0.90 - 0.86    119  119     100.0     5.18    3.20  15.41  0.0961  0.0568
0.86 - 0.83    102  102     100.0     5.00    2.78  13.29  0.1082  0.0689
0.83 - 0.80    127  127     100.0     4.70    2.66  11.11  0.1327  0.0814
0.80 - 0.77    137  138     99.3     4.51    2.08   9.07  0.1418  0.1032
0.77 - 0.75     96   96     100.0     4.23    1.92   7.93  0.1655  0.1236
0.75 - 0.73    127  127     100.0     4.29    1.33   5.56  0.2147  0.1779
0.73 - 0.71    128  129     99.2     4.24    1.24   4.78  0.2314  0.2083
0.71 - 0.69    143  147     97.3     3.95    1.07   3.85  0.2354  0.2629
0.69 - 0.67    154  156     98.7     3.51    0.75   2.33  0.3177  0.4654
0.67 - 0.66     90   90     100.0     3.17    0.60   1.58  0.4048  0.6683
0.66 - 0.65     97   97     100.0     3.41    0.53   1.55  0.3962  0.7290
0.65 - 0.63    191  214     89.3     2.94    0.49   1.26  0.4618  0.9040
0.63 - 0.62     51  124     41.1     0.52    0.35   0.44  0.4715  2.3871
-----
0.72 - 0.62    789   891     88.6     3.02    0.71   2.19  0.3203  0.5576
Inf - 0.62   2442  2549     95.8     4.72    9.04  17.07  0.0565  0.0399

Merged [A], lowest resolution = 9.04 Angstroms

Graphical output: 1=<I/s>, 2=Rmerge, 3=Rsigma, <Enter>=none: █

```