

Factors affecting data quality



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Advanced Light Source
21th April 2015

Scattering Efficiency

- Scattering Efficiency = $\frac{\sum f^2 V_{\text{crystal}}}{V_{\text{cell}}^2}$

- where:-

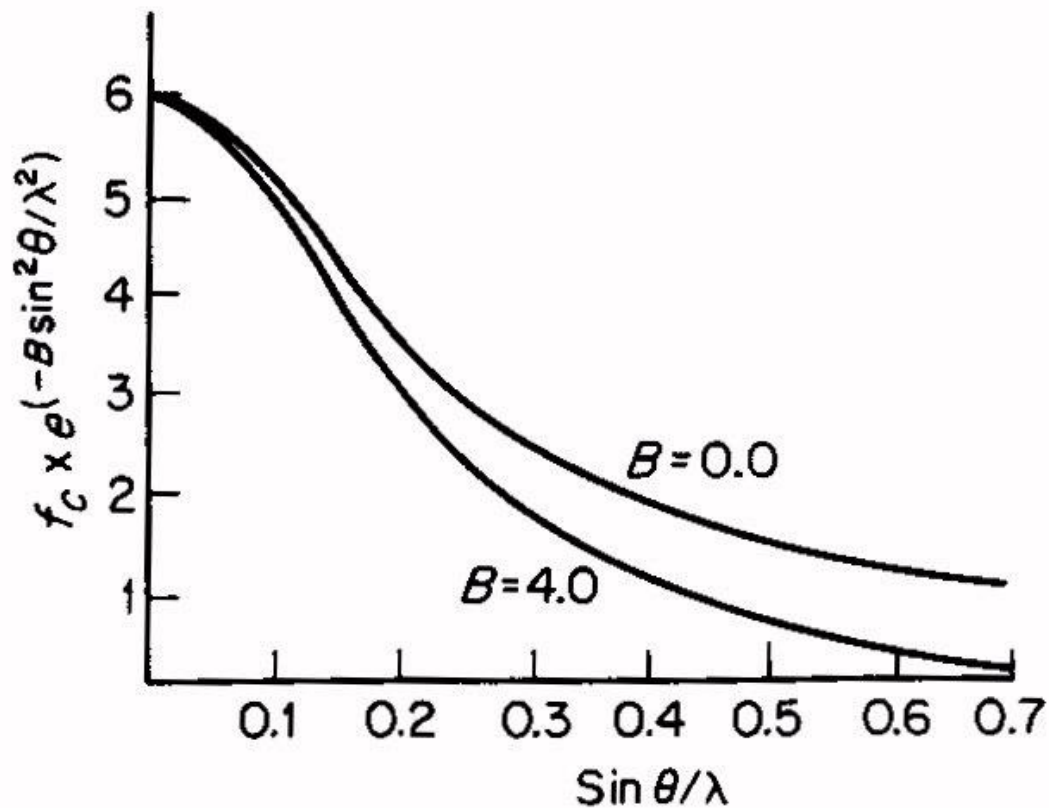
- f = number of electrons per atom

- V_{crystal} = volume of the crystal

- V_{cell} = volume of the unit cell

M.M.Harding *J. Synchrotron Radiation*, 250-259 1996

Effect of disorder



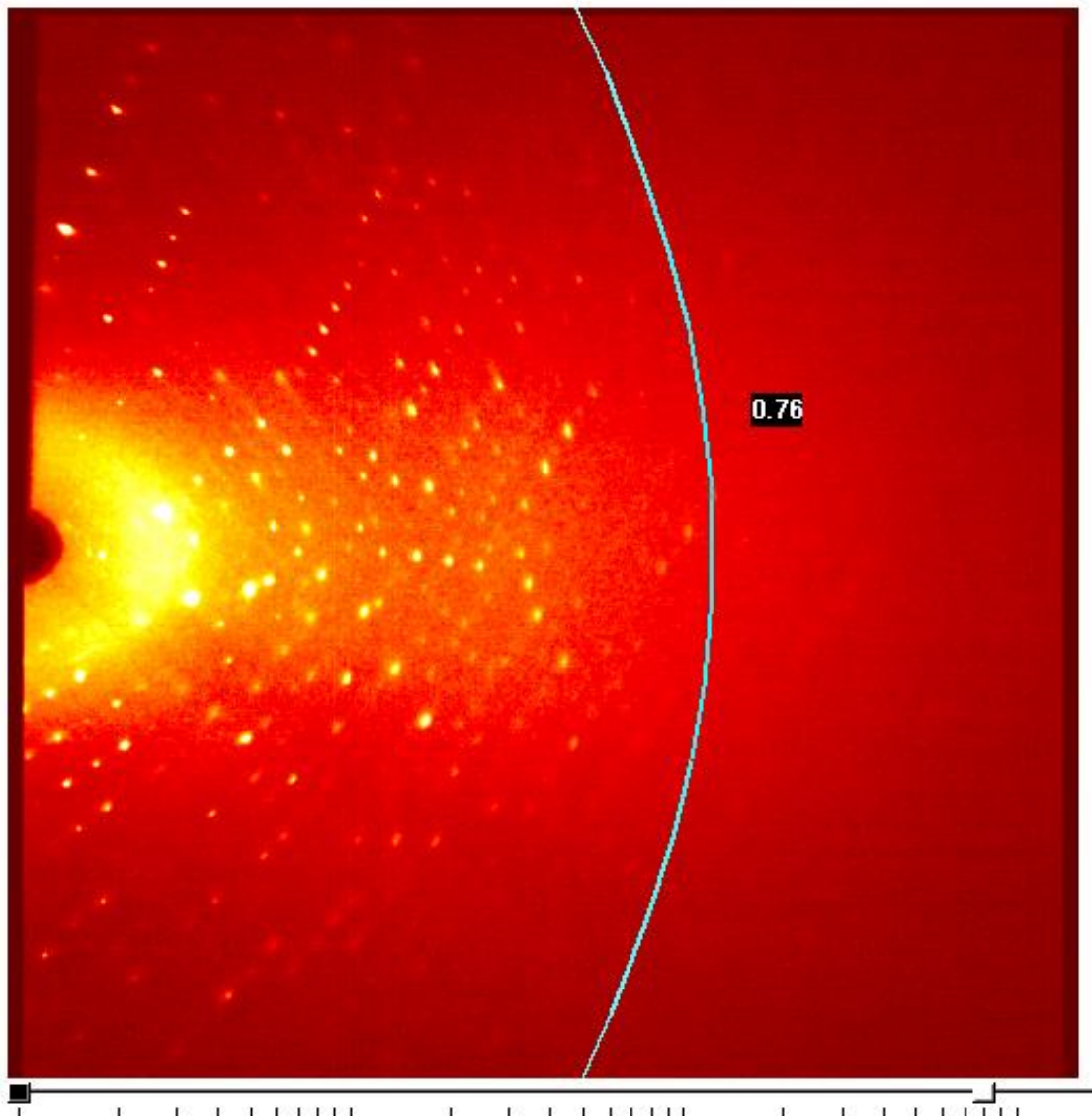
- Wavelength
 - modify strength of interaction
- Rocking width/Mosaicity and size broadening
 - Signal to noise
- Intensity of incident beam

Data: What do you want from it?

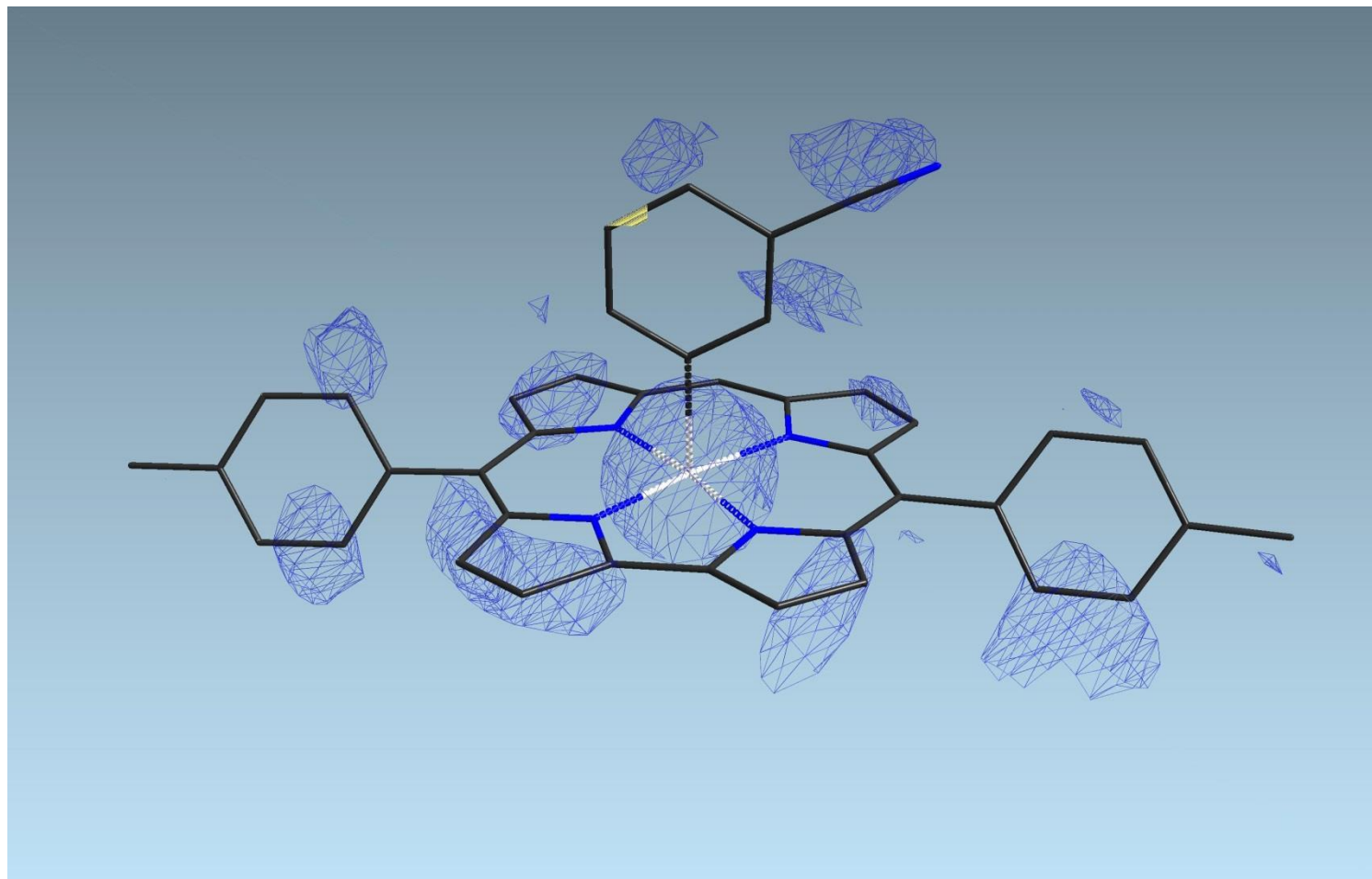
- **Connectivity**
- **Hydrogen position**
- **Accurate bond lengths and angles**
- **Absolute configuration**

Data: Any good?

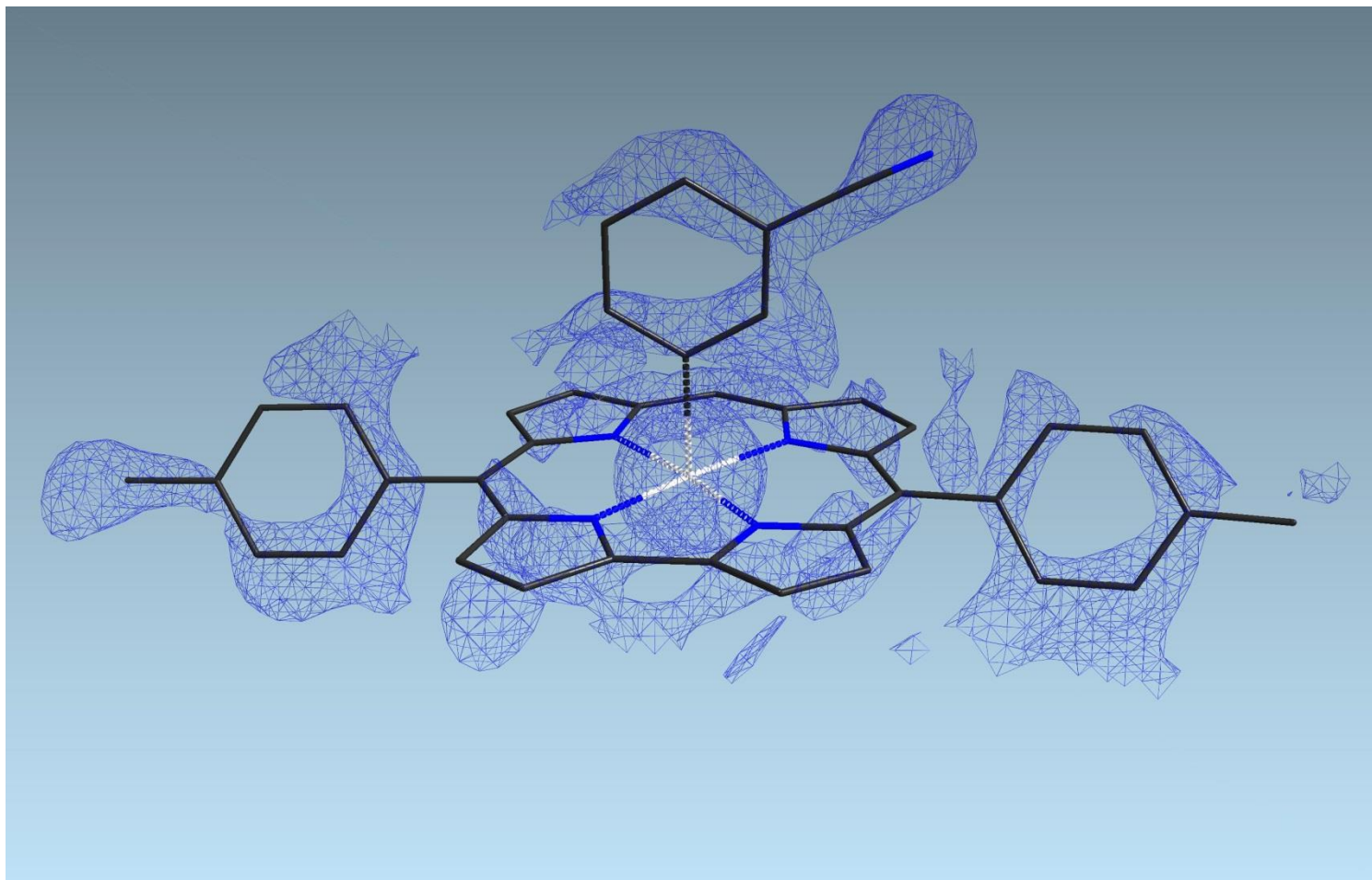
- What does the diffraction look like?
- How well does it index?
- What does the reciprocal lattice look like?
- How well does it integrate?
- Space group determination ease?
- Structure solves easily?
- Structure refines well?



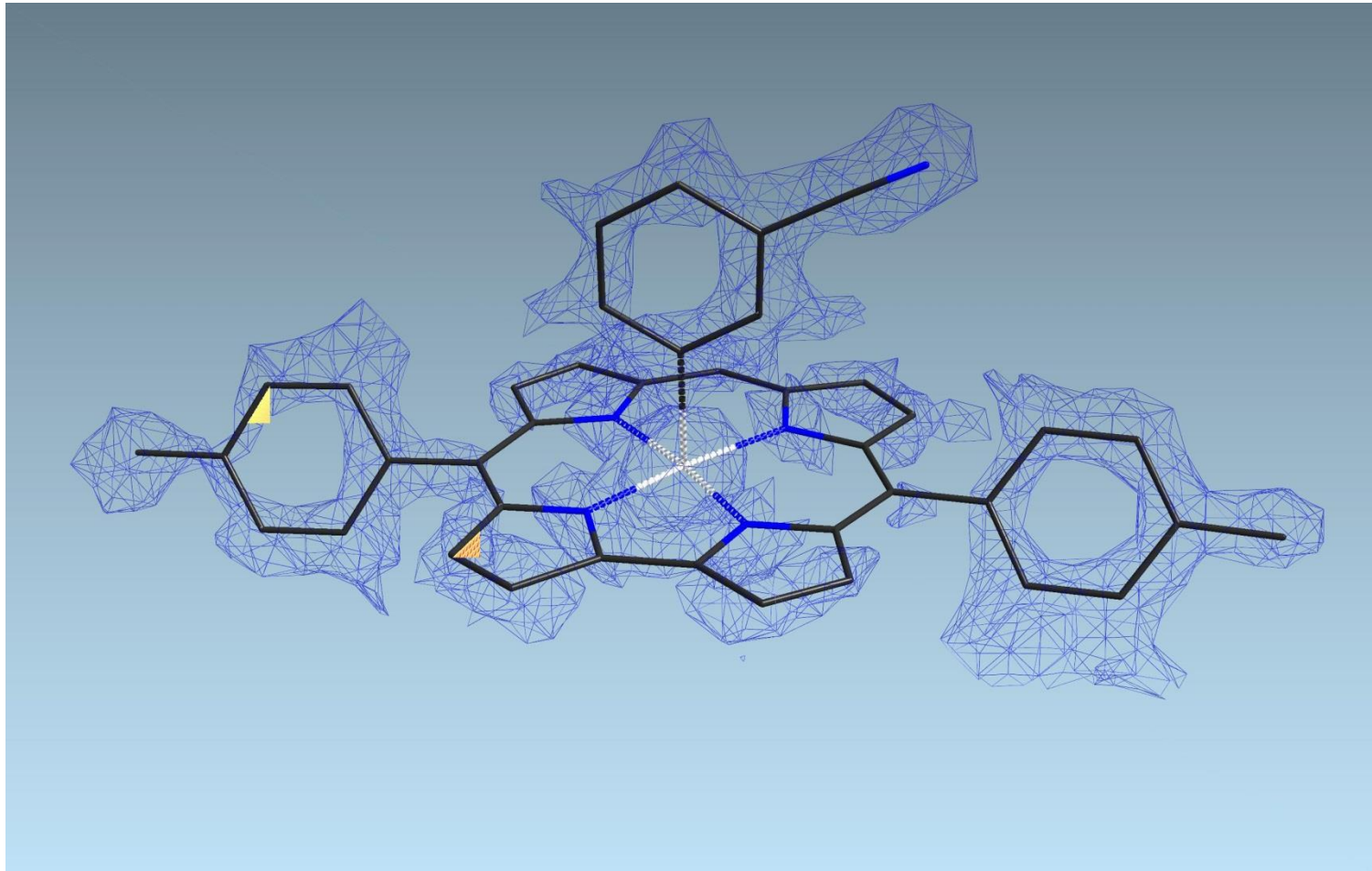
F_{obs} Map at 2.50Å



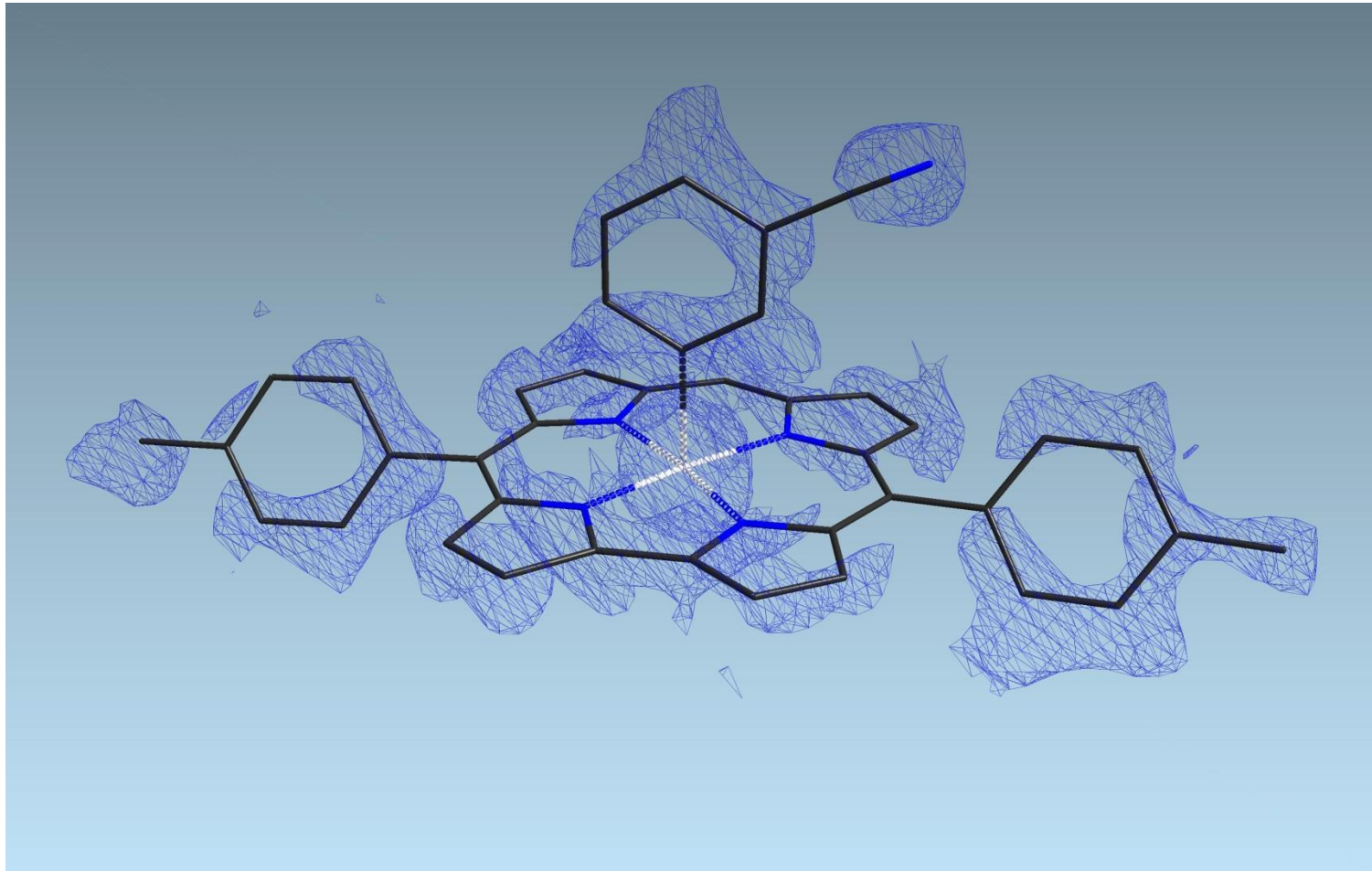
F_{obs} Map at 2.0Å



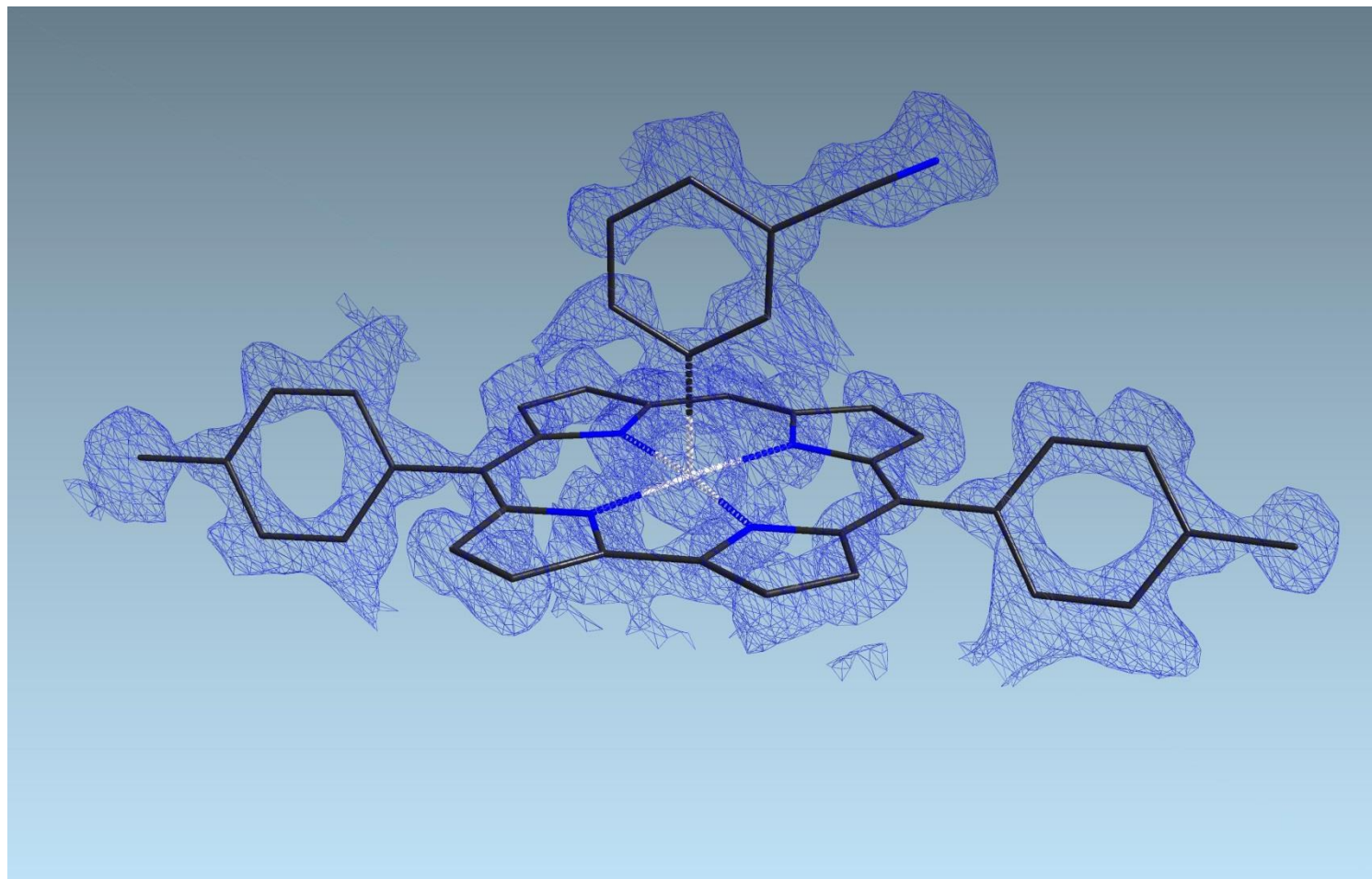
Fobs Map at 1.75Å



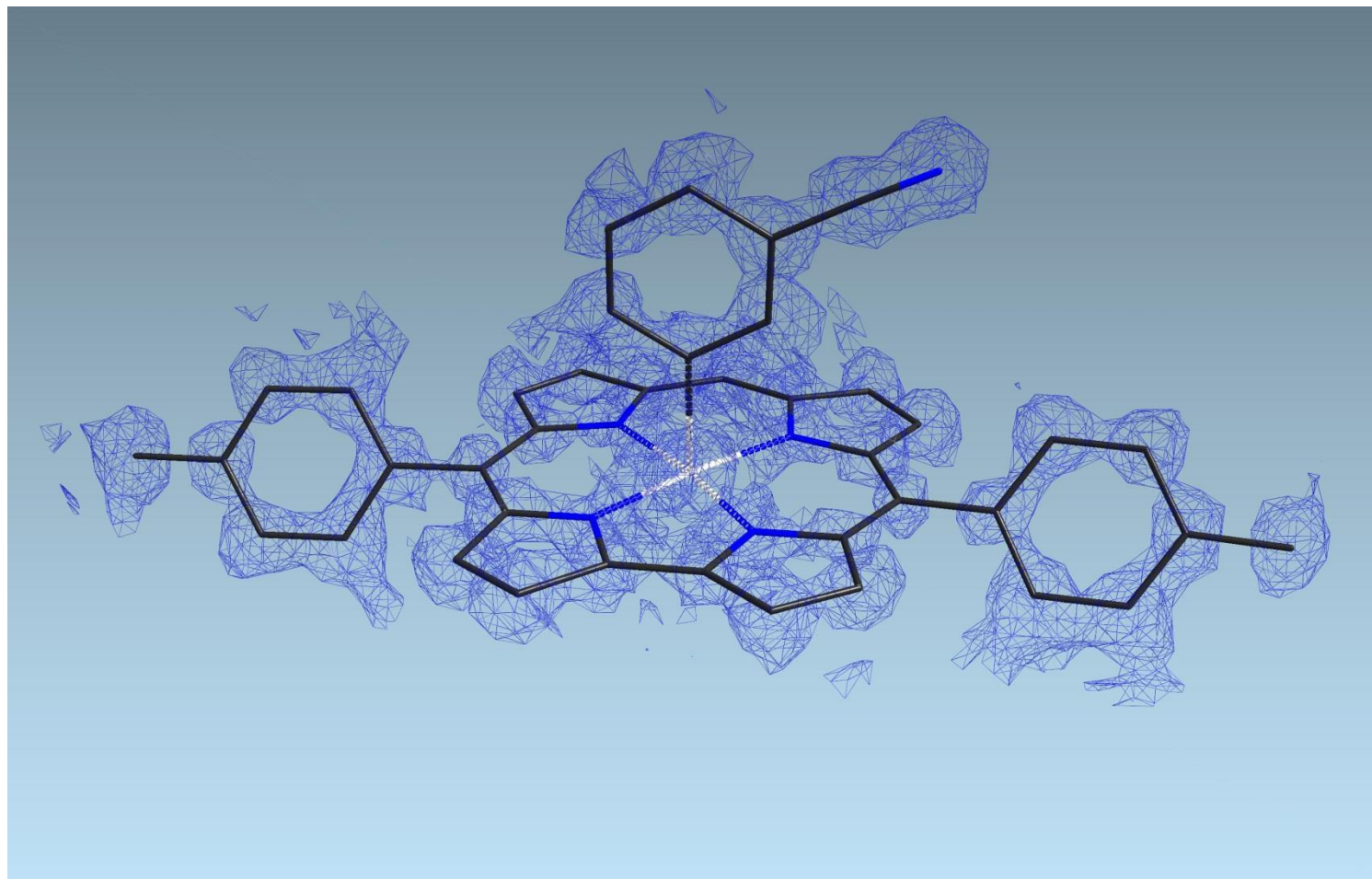
F_{obs} Map at 1.50Å



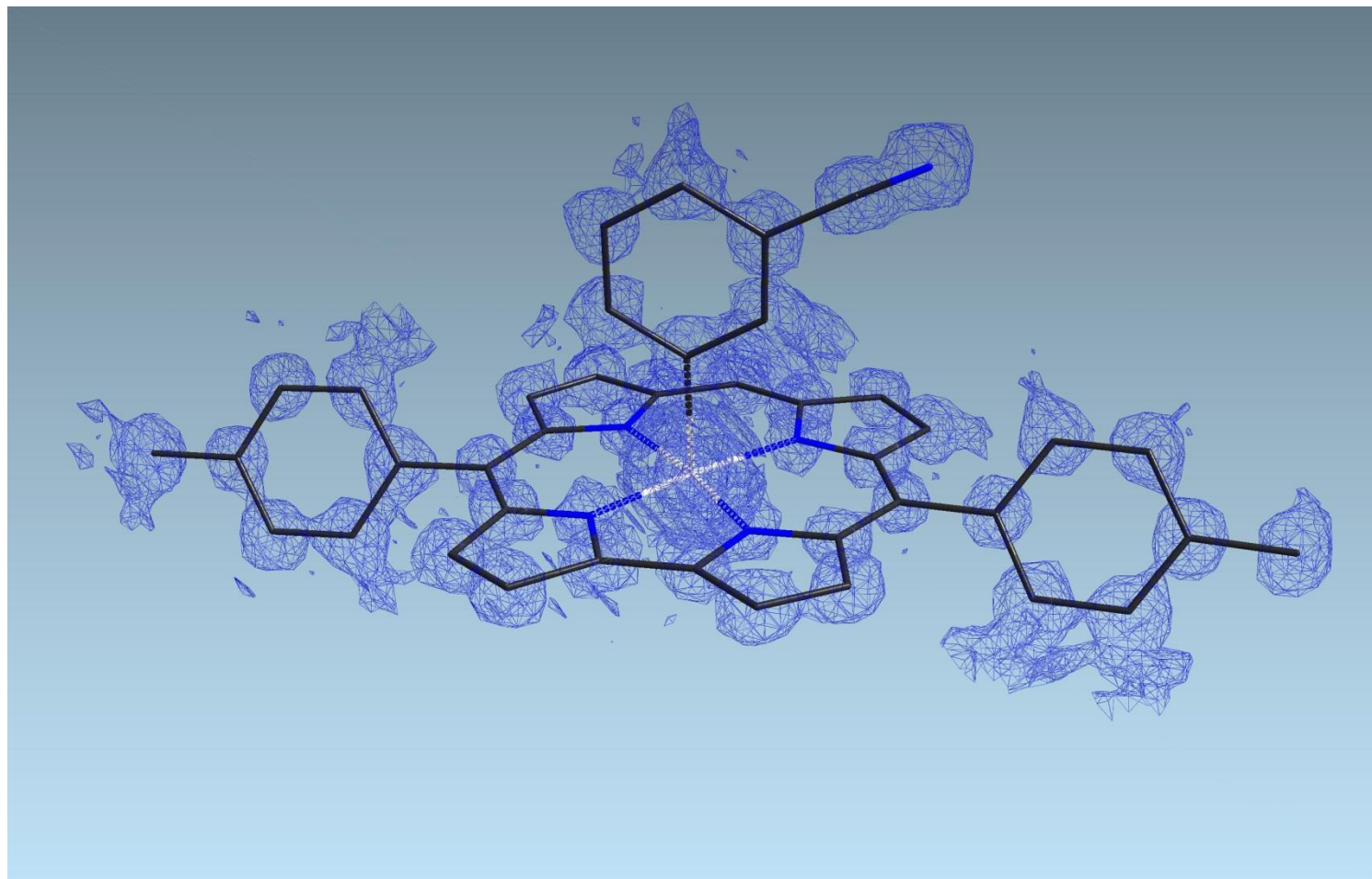
F_{obs} Map at 1.25Å

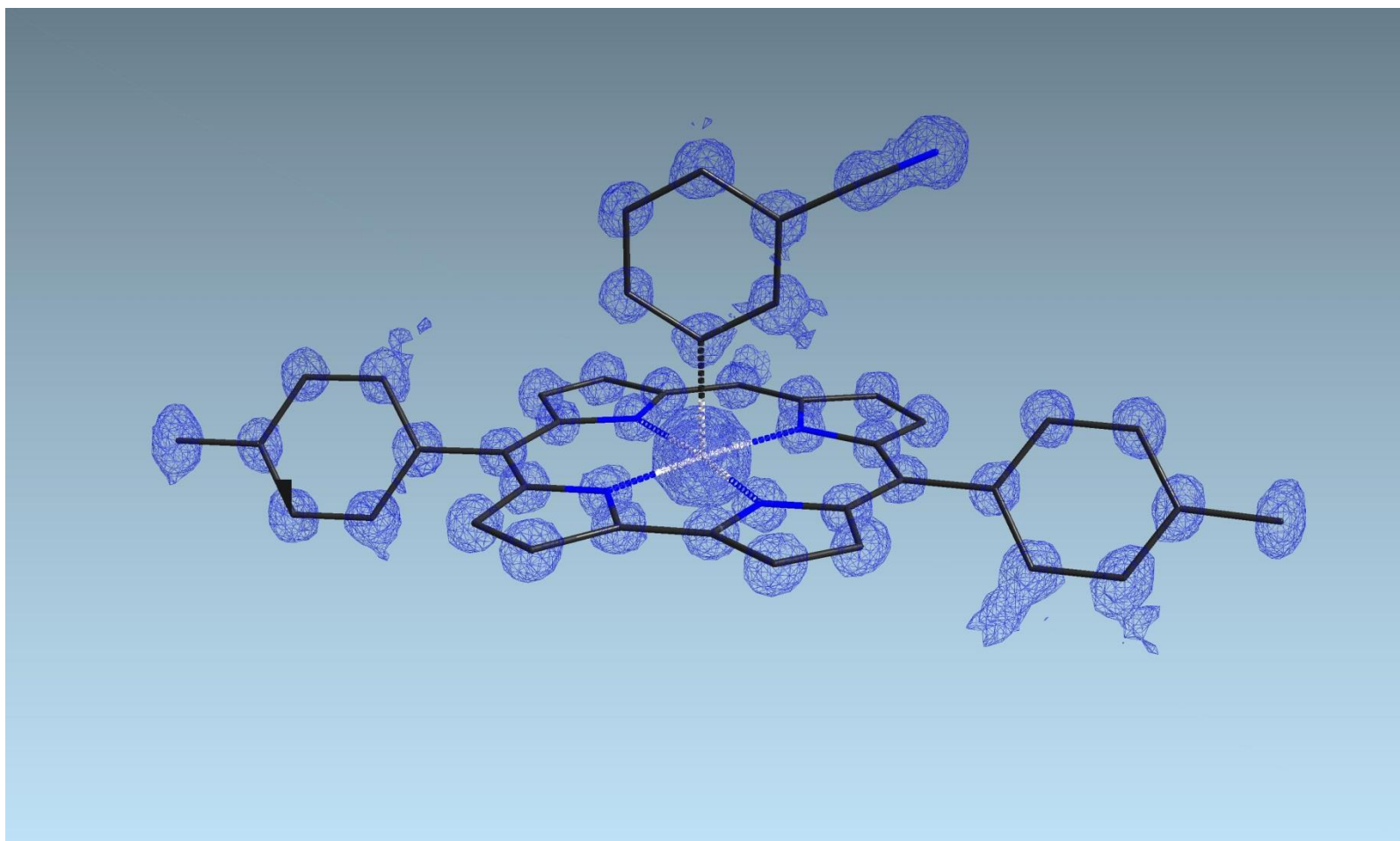


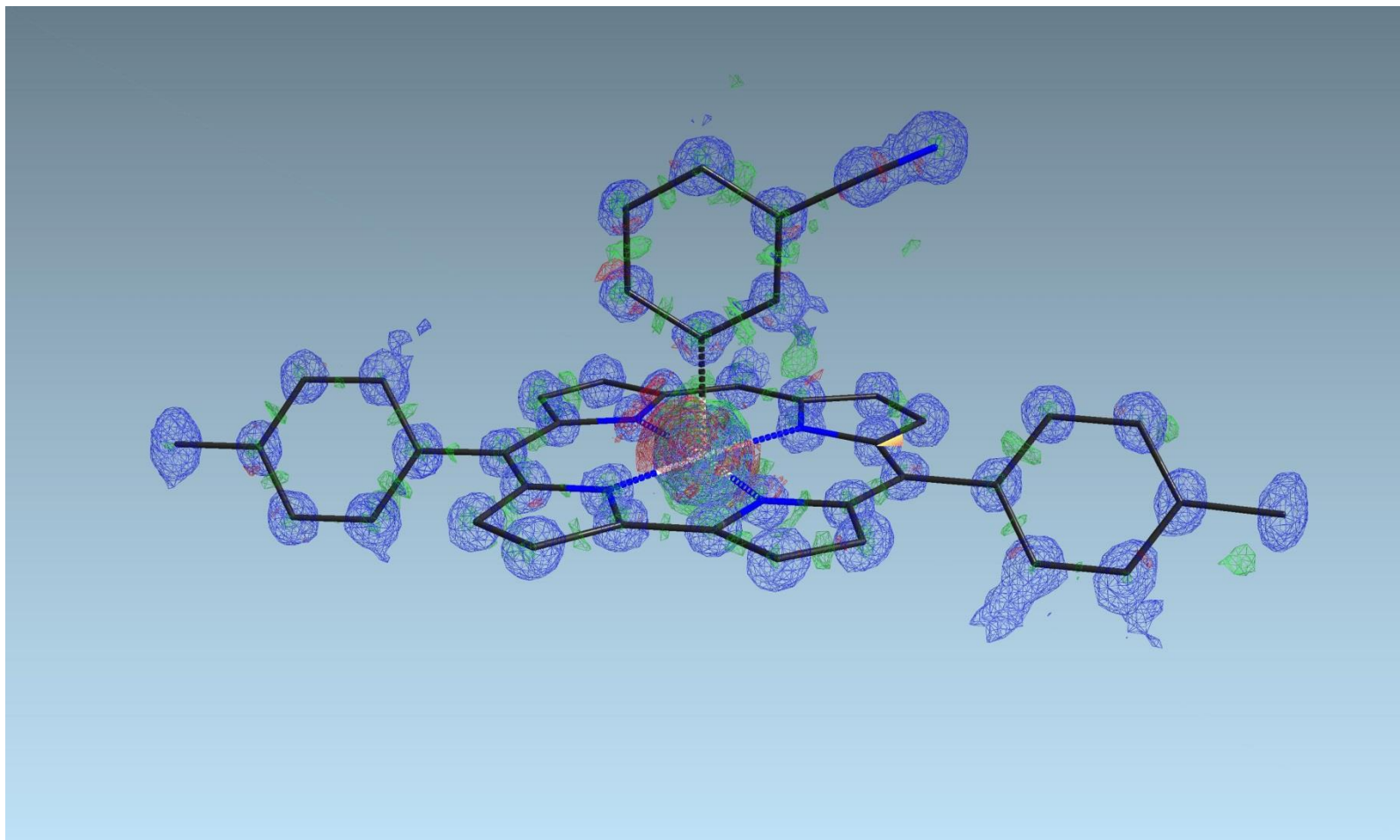
F_{obs} Map at 1.00Å

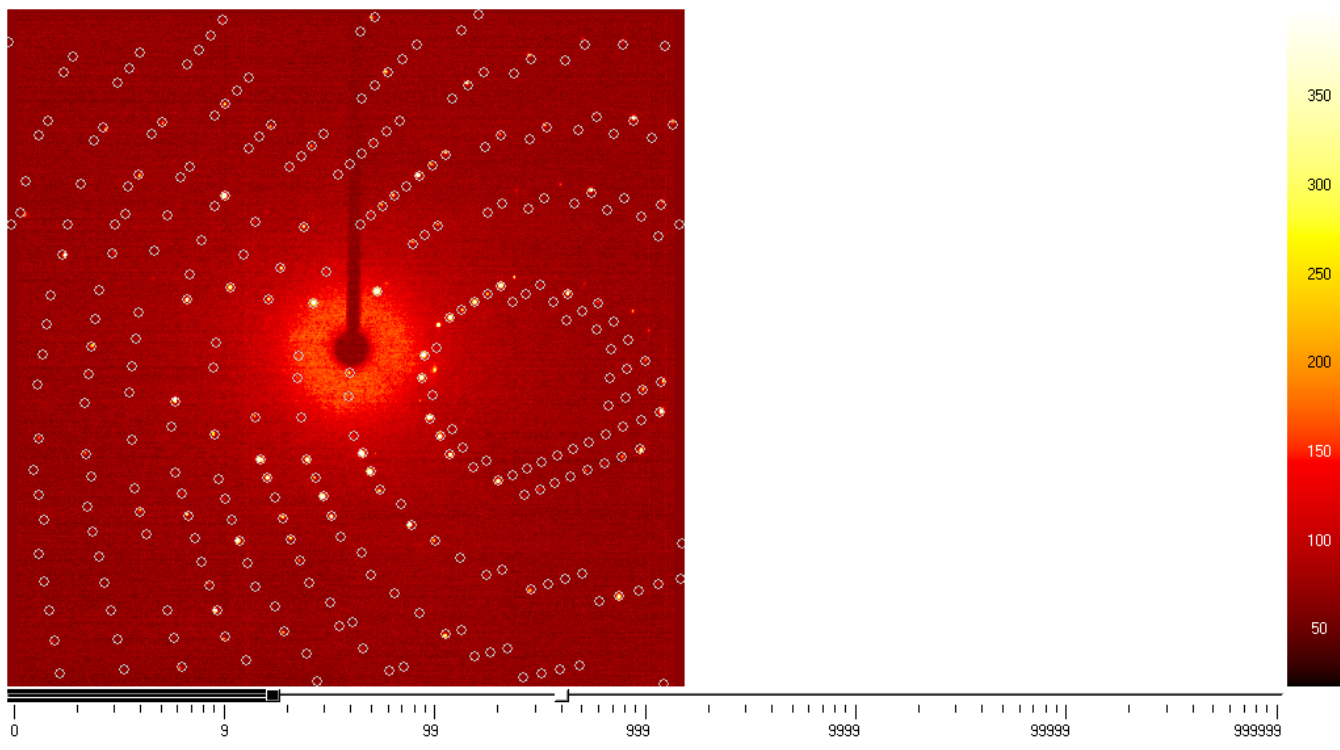


F_{obs} Map at 0.75Å









Reduced Unit Cells found:

Method: Difference Vectors
 Score: 1.73
 $a=10.50\text{\AA}$, $\alpha=90.11^\circ$, $V=3116\text{\AA}^3$
 $b=14.51\text{\AA}$, $\beta=97.67^\circ$
 $c=20.63\text{\AA}$, $\gamma=90.00^\circ$

HKL histogram:

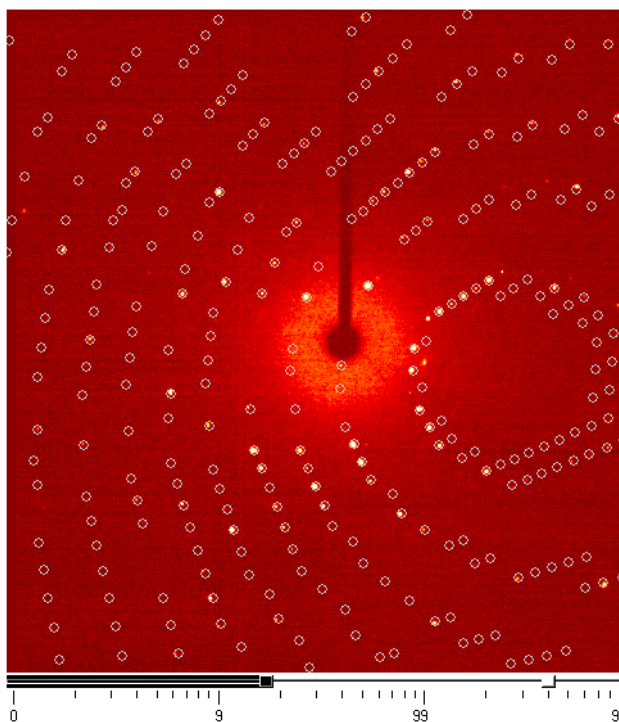
0.1: 95.1% [8317/8745]
 0.2: 95.5% [8353/8745]
 0.3: 95.6% [8363/8745]

Method: Fast Fourier Transform
 Score: 1.73
 $a=10.50\text{\AA}$, $\alpha=90.05^\circ$, $V=3115\text{\AA}^3$
 $b=14.52\text{\AA}$, $\beta=97.63^\circ$
 $c=20.62\text{\AA}$, $\gamma=90.00^\circ$

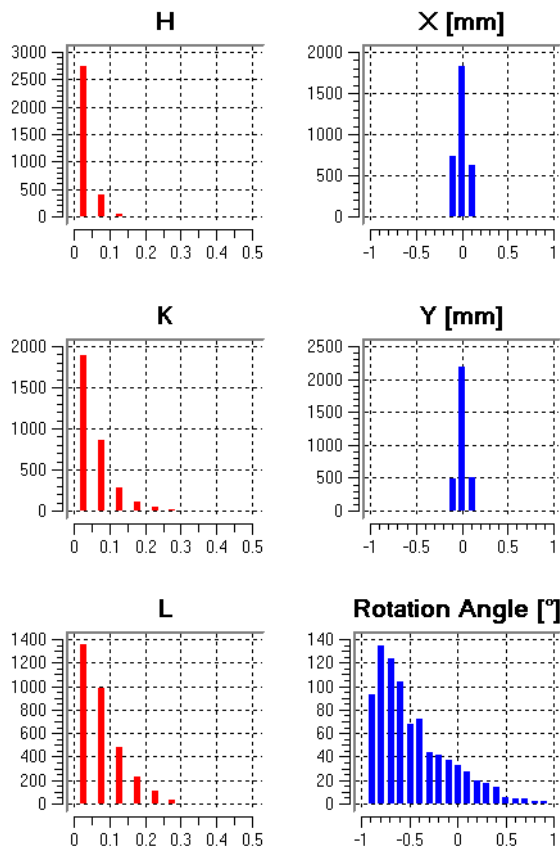
HKL histogram:

0.1: 95.0% [8308/8745]
 0.2: 95.4% [8344/8745]
 0.3: 95.6% [8359/8745]

Cell Refinement



Cursor		
Position [mm]	-34.75	-14.59
Position [pixels]	150	359
Intensity [counts]	46	
HKL index	5.57	-0.10 11.59
Resolution [Å]	1.21	
2Theta [°]	37.50	



Unit Cell: $a=10.50\text{Å}$, $\alpha=90.11^\circ$, $V=3116\text{Å}^3$
 $b=14.51\text{Å}$, $\beta=97.67^\circ$
 $c=20.63\text{Å}$, $\gamma=90.00^\circ$

Parameters:

Unit cell	
a [Å]	10.5036 ± 0.0004
b [Å]	14.5093 ± 0.0007
c [Å]	20.6542 ± 0.0010
α [°]	90.035 ± 0.003
β [°]	97.649 ± 0.003
γ [°]	90.009 ± 0.003
V [Å ³]	3119.7 ± 0.3
Domain translation	
x [mm]	0.00
y [mm]	0.00

Reflections: Group 0: 8745 reflections

Go to Image: D:\frames\guest\LA2_19F\LA2_19F_02_0001.sfm

Tolerance: 0.23

More Reflections Fewer Reflections

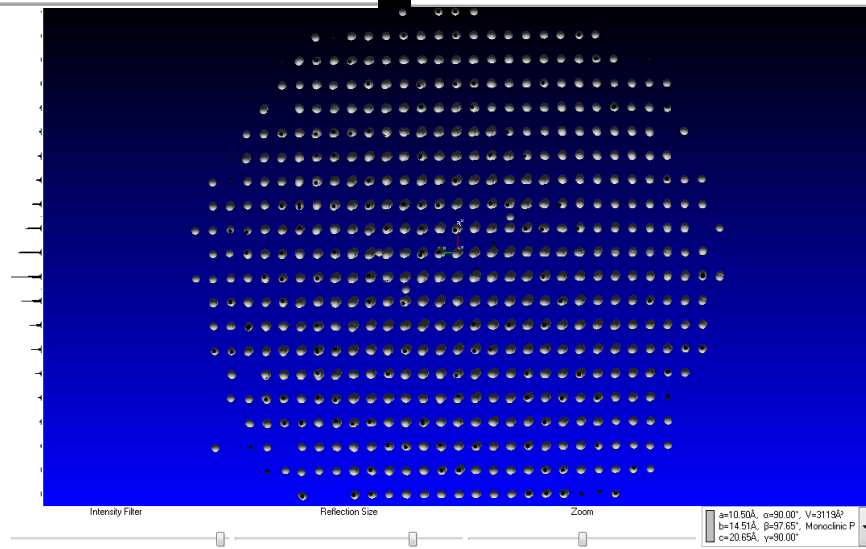
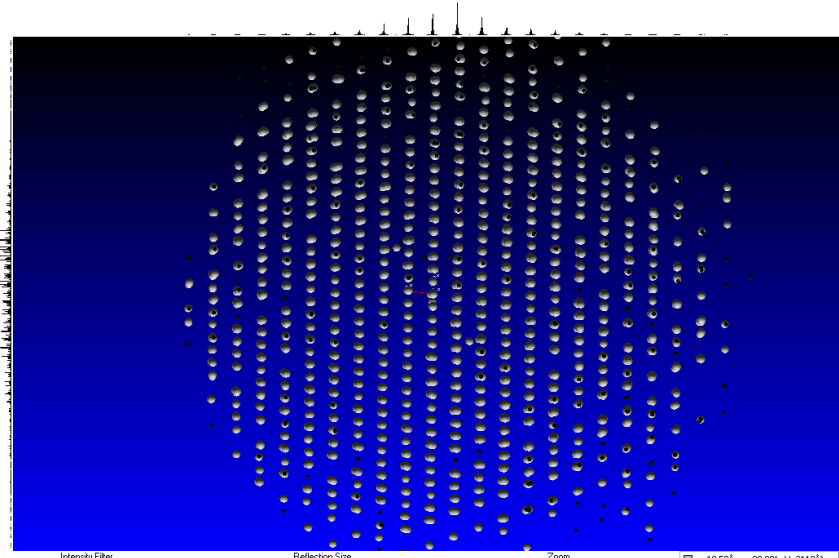
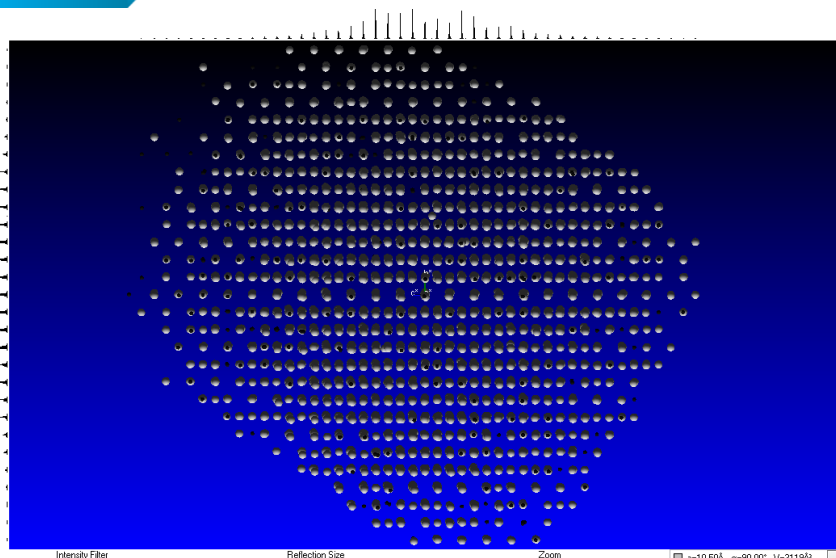
3193 Reflections selected for Refinement

- Show selected Reflections
 Show predicted Reflections

RMS XY [mm]: 0.012 RMS angle [°]: 0.396

Tools:

View the lattice



350

300

250

200

150

Initial Unit Cell:

a=10.50Å, $\alpha=90.04^\circ$, V=3120Å³
 b=14.51Å, $\beta=97.65^\circ$
 c=20.65Å, $\gamma=90.01^\circ$

Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
Cubic F	0.01	28.37	26.25	26.27	112.92	81.85	134.97
Cubic I	0.01	21.89	17.91	25.23	66.33	43.97	78.04
Cubic P	0.01	10.50	14.51	20.65	90.04	97.65	90.01
Hexagonal P	0.01	10.50	14.51	20.65	90.04	97.65	90.01
Rhombohedral R	0.01	17.91	24.39	26.25	119.59	74.09	108.56
Tetragonal I	0.01	10.50	14.51	43.71	70.65	83.43	90.01
Tetragonal P	0.02	10.50	14.51	20.65	90.04	97.65	90.01
Orthorhombic F	0.02	10.50	30.86	41.25	92.40	96.97	70.09
Orthorhombic I	0.02	10.50	14.51	43.71	109.35	96.57	90.01
Orthorhombic C	0.05	10.50	41.25	14.51	89.96	90.01	96.97
Orthorhombic P	0.04	10.50	14.51	20.65	90.04	97.65	90.01
Monoclinic C	0.04	41.25	10.50	14.51	90.01	90.04	83.03
Monoclinic P	0.83	10.50	14.51	20.65	90.04	97.65	90.01
Triclinic P	1.00	10.50	14.51	20.65	90.04	97.65	90.01

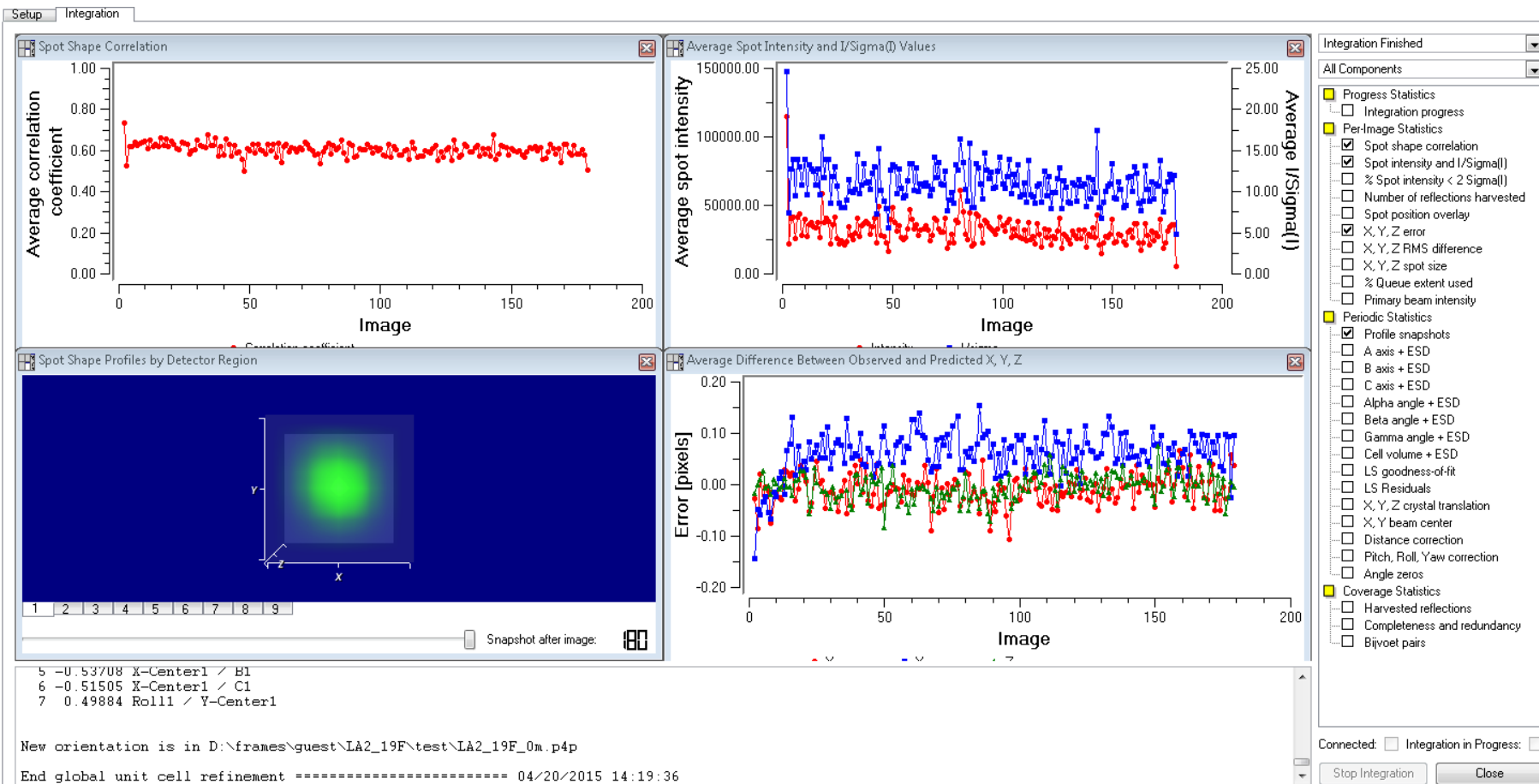
Resolution Limit [Å]: 0.680

Unit Cells:



a=10.50Å, $\alpha=90.00^\circ$, V=3119Å³
 b=14.51Å, $\beta=97.65^\circ$, Monoclinic P
 c=20.65Å, $\gamma=90.00^\circ$

Integration



Laue group numbers:

[1] -1	[8] -3m (rhombohedral axes)
[2] 2/m (Y unique)	[9] -31m (Z unique)
[3] mmm	[10] -3m1 (Z unique)
[4] 4/m (Z unique)	[11] 6/m (Z unique)
[5] 4/mmm (Z unique)	[12] 6/mmm (Z unique)
[6] -3 (rhombohedral axes)	[13] m-3
[7] -3 (Z unique)	[14] m-3m

Run	2theta	R(int)	Incid. factors	Diffr. factors	K	g	I/s(lim)	Total	I>2sig(I)
1	0.0	0.0468	1.823 - 2.083	0.970 - 1.028	0.909	0.0253	39.6	3339	2672
2	0.0	0.0346	0.784 - 1.029	0.967 - 1.028	0.994	0.0253	39.6	12643	10362
3	-45.0	0.0583	0.730 - 1.064	0.967 - 1.028	0.987	0.0253	39.6	14472	9769
4	-45.0	0.0539	0.779 - 1.124	0.968 - 1.022	0.933	0.0253	39.6	14518	9868
5	-45.0	0.0543	0.758 - 1.134	0.967 - 1.028	0.966	0.0253	39.6	14574	10003

original cell in Angstroms and degrees:

10.498 14.526 20.636 90.00 97.61 90.00

59546 Reflections read from file LA2_19f_0m.hkl; mean (I/sigma) = 9.87

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	29768	29796	29818	29732	44691	39723	39679	59546
N (int>3sigma) =	0	19166	19388	19542	19379	29048	25684	25994	38832
Mean intensity =	0.0	10.1	10.2	10.0	10.2	10.1	9.8	10.3	10.2
Mean int/sigma =	0.0	9.9	9.9	10.0	9.9	9.9	9.7	10.0	9.9

Lattice type: P chosen volume: 3119.33

 DETERMINATION OF REDUCED (NIGGLI) CELL

Transformation from original cell (HKLf-matrix):

-1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 -1.0000

unitcell: 10.498 14.526 20.636 90.00 97.61 90.00

Niggli form: a.a = 110.22 b.b = 211.01 c.c = 425.84
 b.c = 0.00 a.c = -28.69 a.b = 0.00

 Search for higher metric symmetry

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

Option A: FOM = 0.000 deg. MONOCLINIC P-lattice R(sym) = 0.032 [9557]

Cell: 10.498 14.526 20.636 90.00 97.61 90.00 Volume: 3119.33

Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000

option A selected

SPACE GROUP DETERMINATION

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	29768	29796	29818	29732	44691	39723	39679	59546
N (int>3sigma) =	0	19166	19388	19542	19379	29048	25684	25994	38832
Mean intensity =	0.0	10.1	10.2	10.0	10.2	10.1	9.8	10.3	10.2
Mean int/sigma =	0.0	9.9	9.9	10.0	9.9	9.9	9.7	10.0	9.9

Crystal system M and Lattice type P selected

Mean |E*-1| = 0.922 [expected .968 centrosym and .736 non-centrosym]

chiral flag NOT set

systematic absence exceptions:

	-21-	-a-	-c-	-n-
N	44	1275	1284	1283
N I>3s	38	525	1	524
<I>	29.2	11.0	0.2	10.9
<I/s>	19.4	8.4	0.5	8.3

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/c	# 13	centro	1	292	0.032	9557	0.5 / 8.3	1.82
[B]	Pc	# 7	non-cen	1	226	0.032	9557	0.5 / 8.3	5.18

option [A] chosen

INTENSITY STATISTICS FOR DATASET # 1 LA2_19f_0m.hkl

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rmerge	Rsigma
Inf - 2.89	155	158	98.1	10.60	46.62	77.36	0.0249	0.0105
2.89 - 1.91	368	368	100.0	12.00	31.71	72.61	0.0273	0.0098
1.91 - 1.50	514	514	100.0	11.60	20.09	56.43	0.0367	0.0121
1.50 - 1.31	510	510	100.0	8.62	12.34	42.87	0.0315	0.0157
1.31 - 1.18	562	562	100.0	8.52	10.59	37.78	0.0356	0.0181
1.18 - 1.10	487	487	100.0	8.38	9.17	32.92	0.0408	0.0209
1.10 - 1.03	559	559	100.0	7.86	7.99	28.29	0.0489	0.0252
1.03 - 0.98	491	492	99.8	6.23	6.35	20.44	0.0629	0.0354
0.98 - 0.93	619	619	100.0	5.47	5.19	16.85	0.0747	0.0453
0.93 - 0.90	433	433	100.0	4.66	4.51	13.70	0.0820	0.0562
0.90 - 0.87	481	481	100.0	4.22	3.60	11.06	0.0959	0.0695
0.87 - 0.84	578	578	100.0	3.96	3.37	9.95	0.1016	0.0788
0.84 - 0.81	637	637	100.0	4.01	3.13	9.08	0.1151	0.0858
0.81 - 0.79	507	507	100.0	3.90	2.97	8.42	0.1270	0.0949
0.79 - 0.77	544	544	100.0	3.88	2.63	7.62	0.1469	0.1073
0.77 - 0.76	300	300	100.0	3.82	2.53	6.96	0.1472	0.1160
0.76 - 0.74	631	631	100.0	3.80	2.39	6.62	0.1653	0.1238
0.74 - 0.72	715	715	100.0	3.65	2.26	6.13	0.1831	0.1365
0.72 - 0.71	380	380	100.0	3.60	2.01	5.19	0.2015	0.1579
0.71 - 0.70	391	391	100.0	3.53	2.04	5.29	0.2035	0.1619
0.70 - 0.69	452	472	95.8	3.20	1.76	4.63	0.2381	0.1900

0.79 - 0.69	3413	3433	99.4	3.65	2.25	6.13	0.1779	0.1369
Inf - 0.69	10314	10338	99.8	5.76	7.13	20.47	0.0464	0.0371

Merged [A], lowest resolution = 10.41 Angstroms

Determination of unit-cell contents

Formula: CdC40H40O10N2

Formula weight = 821.16

Tentative Z (number of formula units/cell) = 4.0 giving rho = 1.749,
non-H atomic volume = 14.7 and following cell contents and analysis:

C	160.00	58.51 %	H	160.00	4.91 %
N	8.00	3.41 %	O	40.00	19.48 %
Cd	4.00	13.69 %			

F(000) = 1688.0 ? -K(alpha) radiation Mu (mm-1) = 0.00

Monoclinic

DETERMINATION OF REDUCED (NIGGLI) CELL

Transformation from original cell (HKLF-matrix):

```
0.0000 -1.0000  0.0000   1.0000  0.0000  0.0000   0.0000  0.0000  1.0000
```

```
Unitcell:      6.812   11.631   12.200   90.34   90.00   90.00
```

```
Niggli form:   a.a =    46.40      b.b =   135.28      c.c =   148.84
                b.c =   -0.85      a.c =    0.00      a.b =    0.00
```

 Search for higher metric symmetry

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

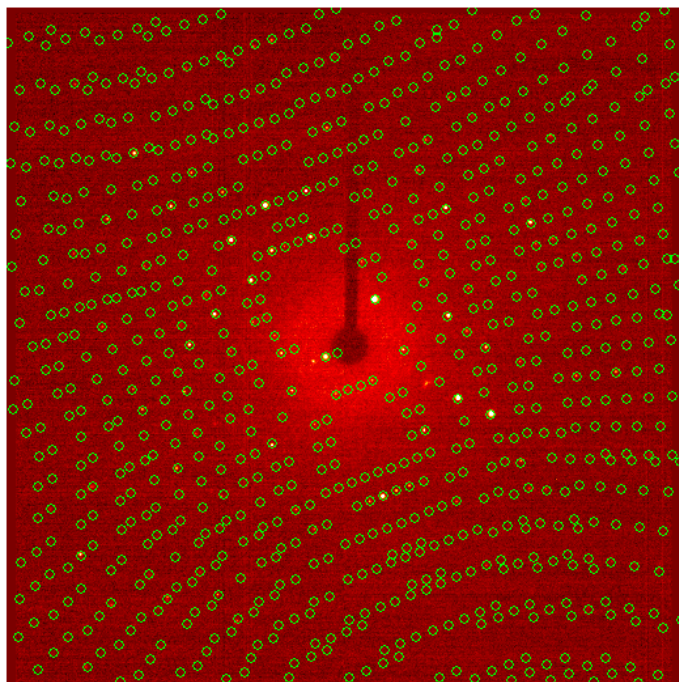
```
-----
Option A: FOM = 0.343 deg.  ORTHORHOMBIC P-lattice  R(sym) = 0.518 [ 6543]
Cell:      6.812  11.631  12.200  90.34  90.00  90.00  Volume: 966.55
Matrix: 0.0000 -1.0000  0.0000  1.0000  0.0000  0.0000  0.0000  0.0000  1.0000
```

```
-----
Option B: FOM = 0.000 deg.  MONOCLINIC P-lattice  R(sym) = 0.016 [ 4214]
Cell:     11.631   6.812  12.200  90.00  90.34  90.00  Volume: 966.55
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
```

```
-----
Option C: FOM = 0.343 deg.  MONOCLINIC P-lattice  R(sym) = 0.544 [ 4319]
Cell:      6.812  11.631  12.200  90.34  90.00  90.00  Volume: 966.55
Matrix: 0.0000 -1.0000  0.0000 -1.0000  0.0000  0.0000  0.0000  0.0000 -1.0000
```

```
-----
Option D: FOM = 0.343 deg.  MONOCLINIC P-lattice  R(sym) = 0.550 [ 4327]
Cell:      6.812  12.200  11.631  89.66  90.00  90.00  Volume: 966.55
Matrix: 0.0000 -1.0000  0.0000  0.0000  0.0000  1.0000 -1.0000  0.0000  0.0000
```

option B selected



Reduced Unit Cells found:

Method: Difference Vectors	HKL histogram:
Score: 1.33 a=14.20Å, $\alpha=69.50^\circ$, V=9000Å ³ b=21.71Å, $\beta=89.92^\circ$ c=31.17Å, $\gamma=89.89^\circ$	0.1: 93.3% (2695/2890) 0.2: 94.0% (2716/2890) 0.3: 94.0% (2716/2890)
Method: Fast Fourier Transform	HKL histogram:
Score: 1.34 a=14.21Å, $\alpha=106.68^\circ$, V=8987Å ³ b=25.95Å, $\beta=90.09^\circ$ c=31.02Å, $\gamma=123.17^\circ$	0.1: 93.9% (2713/2890) 0.2: 94.1% (2720/2890) 0.3: 94.1% (2720/2890)



Cursor			
Position [mm]	-24.96	-36.19	
Position [pixels]	252	134	
Intensity [counts]	75		
HKL index	-6.02	-5.79	-11.74
Resolution [Å]	1.09		
2Theta [°]	41.55		

Image Header | Tool Editor | Cursor Position

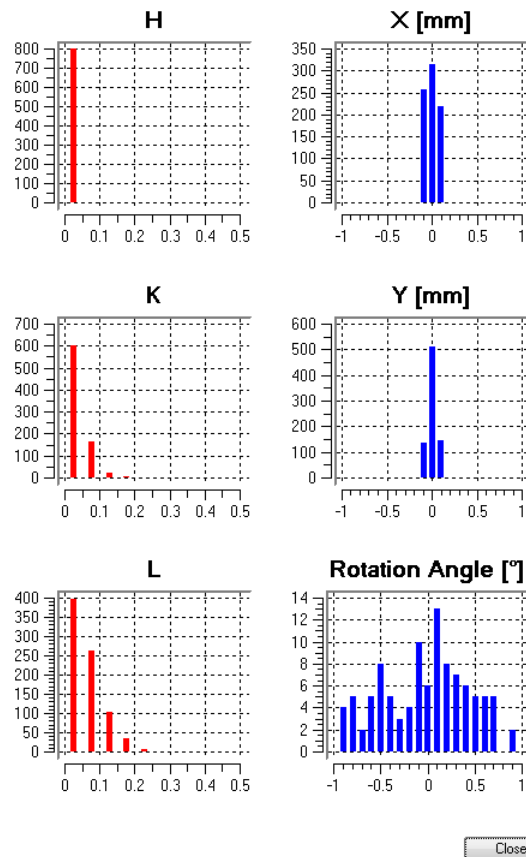
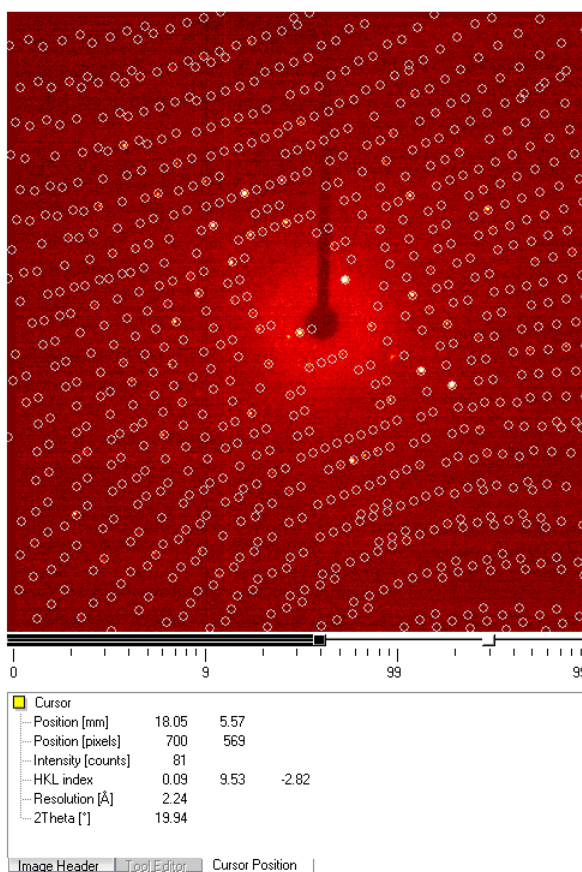


Finish

Accept...

Cancel

Cell Refinement



Unit Cell:

a=14.21Å, α =106.68°, V=8987Å³
 b=25.95Å, β = 90.09°
 c=31.02Å, γ =123.17°

Unit cell

a [Å]	14.246 ± 0.007
b [Å]	26.021 ± 0.014
c [Å]	31.139 ± 0.017
α [°]	106.661 ± 0.013
β [°]	90.070 ± 0.012
γ [°]	123.204 ± 0.010
V [Å ³]	9071 ± 14

Domain translation

x [mm]	0.00
y [mm]	0.00

Reflections: Group 0: 2890 reflections

Go to Image: D:\frames\guest\la2_32b\la2_32b_02_0001.sfrm

More Reflections Fewer Reflections

Tolerance: 0.22

799 Reflections selected for Refinement

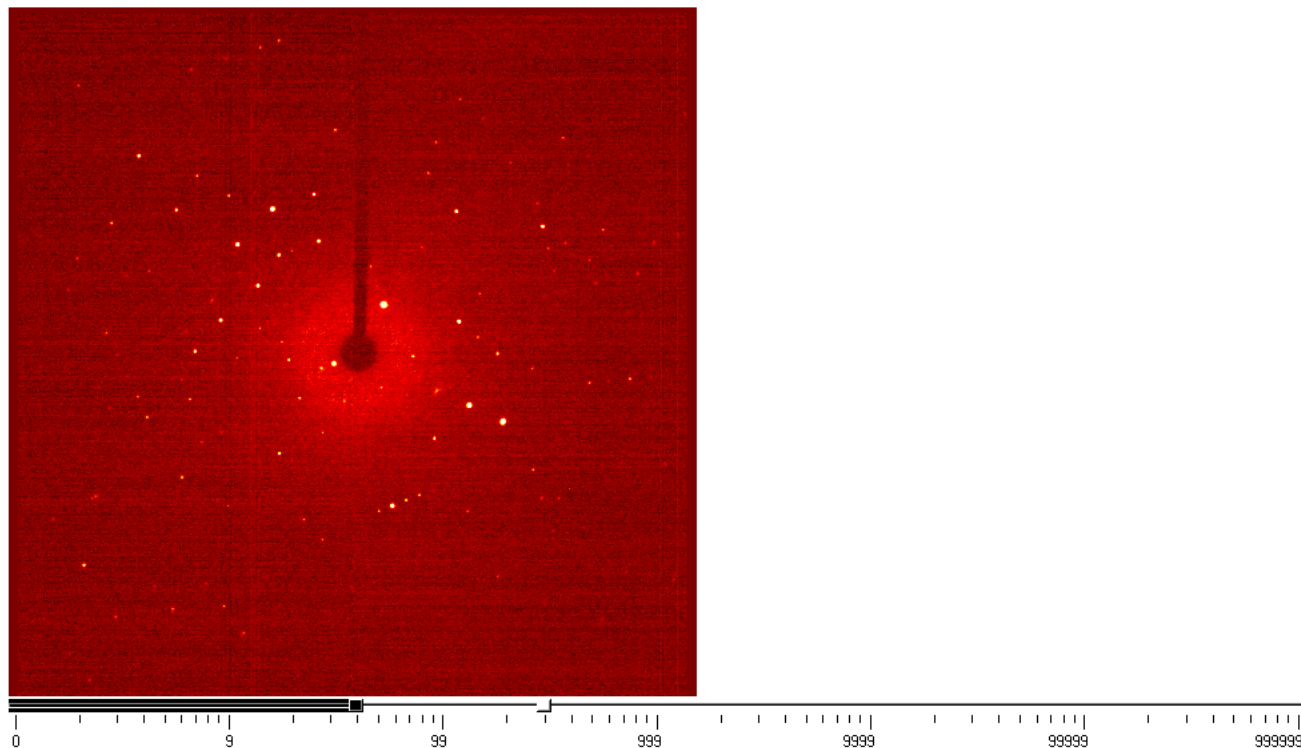
Show selected Reflections
 Show predicted Reflections

RMS XY [mm]: 0.012 RMS angle [°]: 0.661

Tools: Refine Histograms... Transformations...

Finish Accept Cancel

Lattice Choice



280

260

240

220

200

180

160

140

120

100

80

60

Initial Unit Cell:

a=14.25Å, $\alpha=106.66^\circ$, V=9071Å³
 b=26.02Å, $\beta=90.07^\circ$
 c=31.14Å, $\gamma=123.20^\circ$

Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
Cubic F	0.01	34.26	46.25	34.23	100.96	49.17	116.55
Cubic I	0.01	34.23	26.02	31.27	72.80	46.54	91.96
Cubic P	0.01	14.25	21.77	31.14	110.09	90.07	90.01
Hexagonal P	0.02	21.77	31.14	14.25	90.07	90.01	110.09
Rhombohedral R	0.01	14.25	26.02	89.60	78.85	99.07	123.19
Tetragonal I	0.01	14.25	21.77	60.18	89.64	76.39	90.01
Tetragonal P	0.01	14.25	21.77	31.14	110.09	90.07	90.01
Orthorhombic F	0.01	26.02	26.02	84.29	45.12	81.29	66.40
Orthorhombic I	0.02	14.25	21.77	60.18	90.36	103.61	90.01
Orthorhombic C	0.48	21.77	58.49	14.25	89.92	90.01	90.37
Orthorhombic P	0.02	14.25	21.77	31.14	110.09	90.07	90.01
Monoclinic C	0.43	58.49	21.77	14.25	90.01	90.08	89.63
Monoclinic P	0.80	21.77	14.25	31.14	90.07	110.09	90.01
Triclinic P	1.00	14.25	21.77	31.14	110.09	90.07	90.01

Cursor			
Position [mm]	7.58	-35.42	
Position [pixels]	591	142	
Intensity [counts]	69		
HKL index	-9.82	9.92	-11.25
Resolution [Å]	1.27		
2Theta [°]	35.60		

Image Header | Tool Editor | Cursor Position

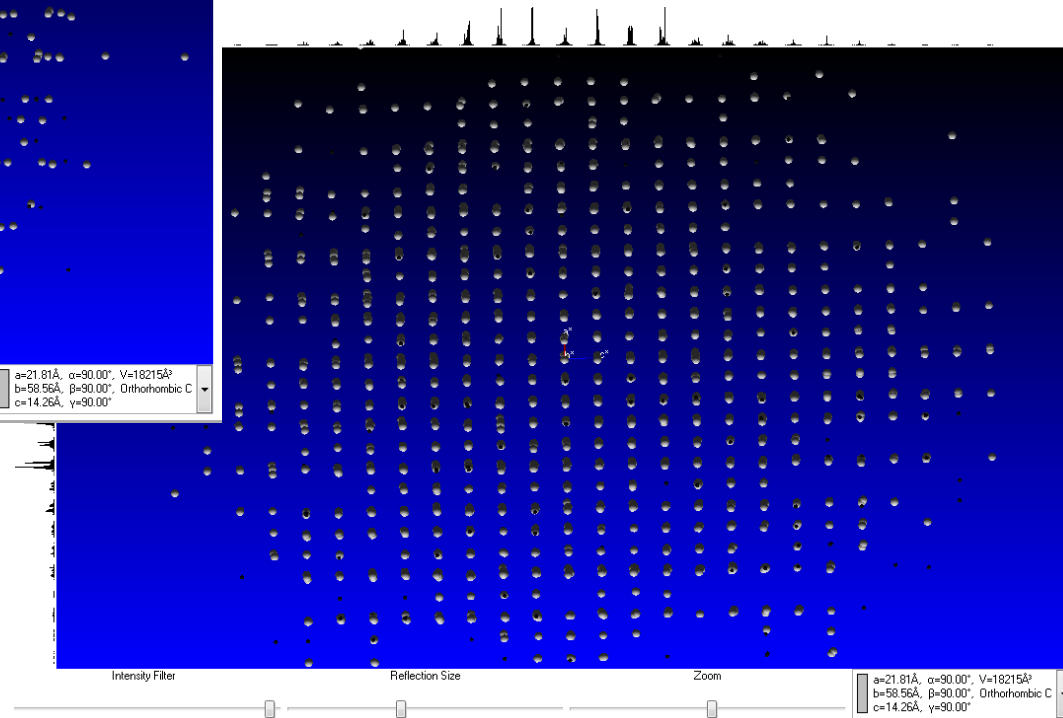
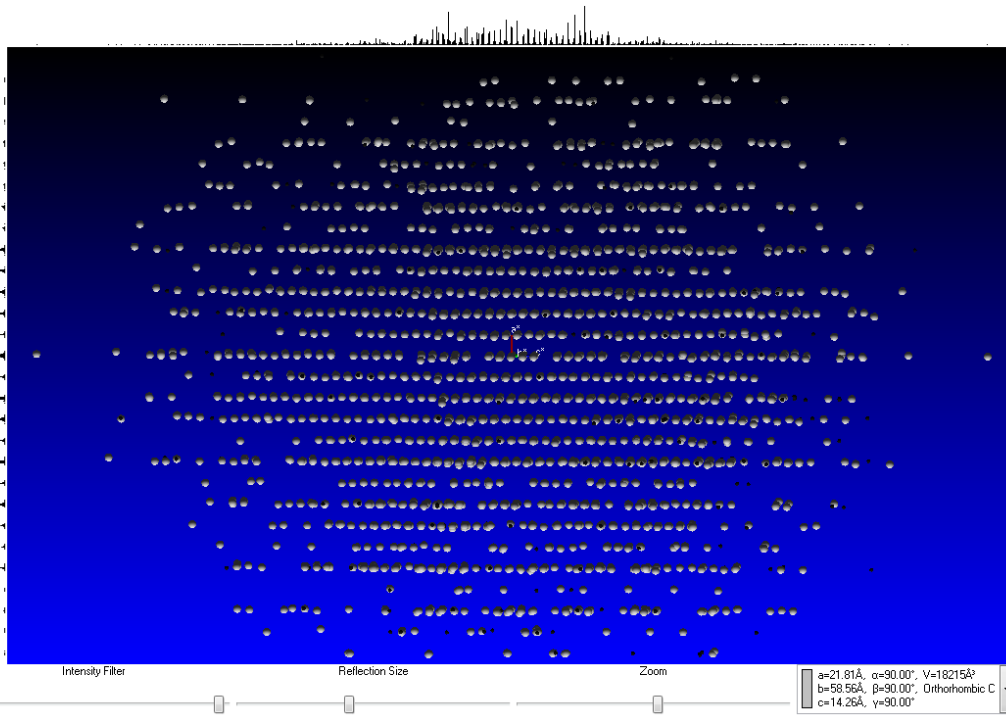


Finish

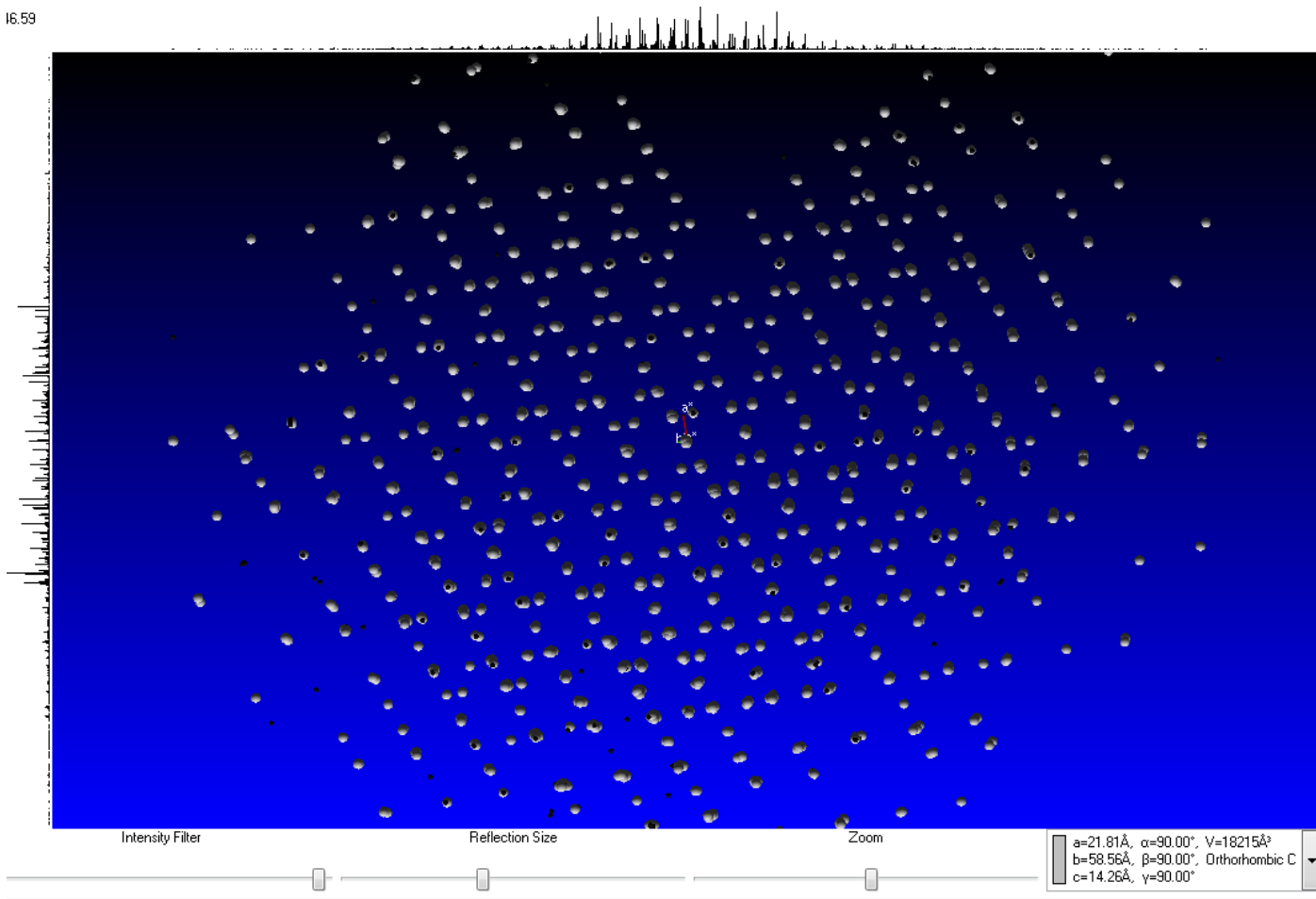
Accept

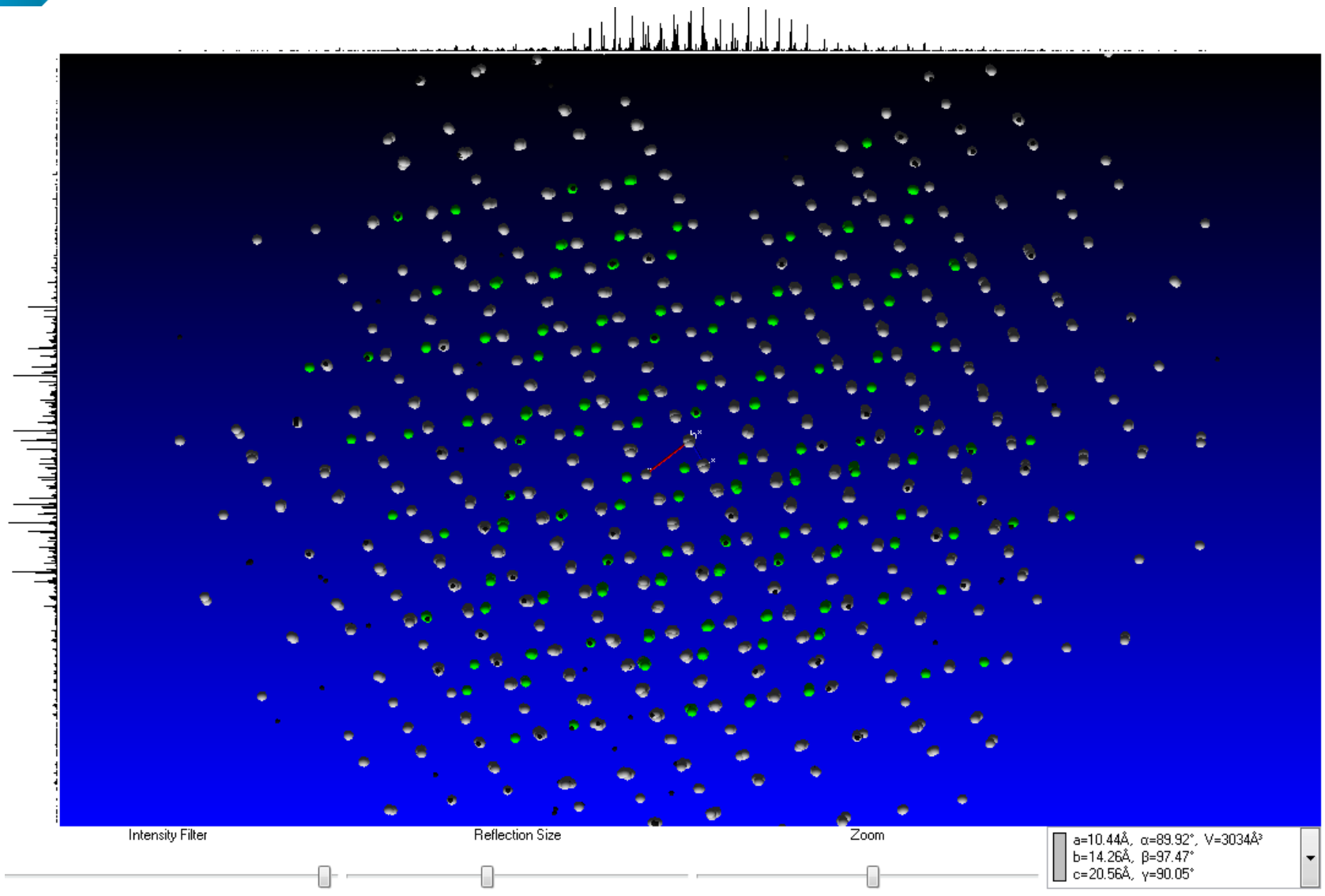
Cancel

View of the Reciprocal Space

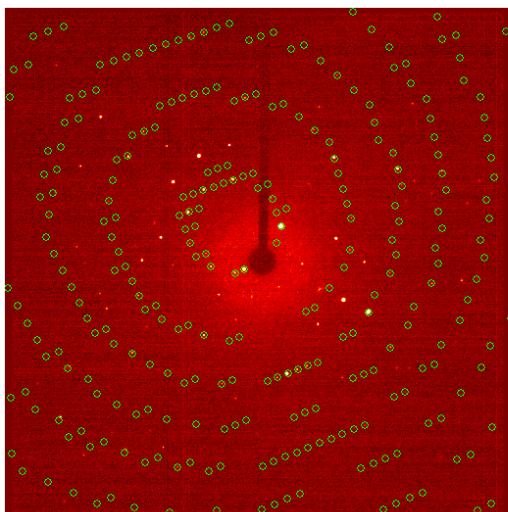
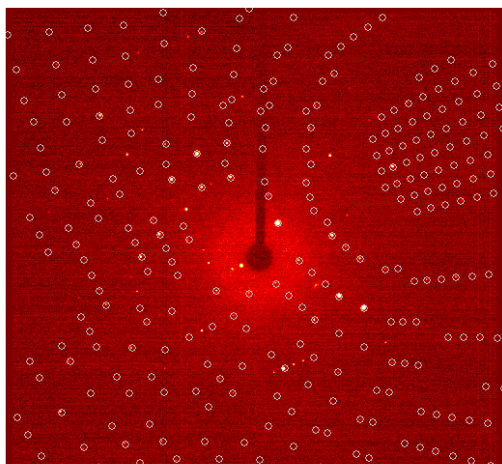


16.59





Both Orientations



Cursor	
Position [mm]	33.70 -48.38
Position [pixels]	863 7
Intensity [counts]	71
HKL index	-6.46 12.22 -2.79
Resolution [Å]	0.93
2Theta [°]	49.42

Image Header | [Data Editor](#) | Cursor Position

280
260
240
220
200
180
160
140
120
100
80
60

Unit Cell: $a=10.44\text{\AA}$, $\alpha=90.00^\circ$, $V=3034\text{\AA}^3$
 $b=14.26\text{\AA}$, $\beta=97.47^\circ$, Monoclinic P
 $c=20.56\text{\AA}$, $\gamma=90.00^\circ$

Unit cell

Parameter	Value
a [Å]	10.425 ± 0.004
b [Å]	14.245 ± 0.003
c [Å]	20.528 ± 0.007
α [°]	90.00
β [°]	97.543 ± 0.013
γ [°]	90.00
V [Å ³]	3022 ± 3

Domain translation

x [nm]	0.00
y [nm]	0.00

Reflections: Group 0: 2389 reflections

Go to Image: D:\frames\guest\la2_32b\la2_32b_02_0001.sfm

More Reflections Fewer Reflections

Tolerance: 0.22

789 Reflections selected for Refinement

280
260
240
220
200
180
160
140
120
100
80
60

Unit Cell: $a=10.44\text{\AA}$, $\alpha=90.00^\circ$, $V=3034\text{\AA}^3$
 $b=14.26\text{\AA}$, $\beta=97.48^\circ$, Monoclinic P
 $c=20.56\text{\AA}$, $\gamma=90.00^\circ$

Unit cell

Parameter	Value
a [Å]	10.440 ± 0.006
b [Å]	14.259 ± 0.006
c [Å]	20.557 ± 0.013
α [°]	90.00
β [°]	97.48 ± 0.03
γ [°]	90.00
V [Å ³]	3034 ± 5

Domain translation

x [nm]	0.00
y [nm]	0.00

Reflections: Group 1: 1414 reflections

Go to Image: D:\frames\guest\la2_32b\la2_32b_02_0001.sfm

More Reflections Fewer Reflections

Tolerance: 0.24

468 Reflections selected for Refinement

Show selected Reflections
 Show predicted Reflections

RMS XY [nm]: n/a RMS angle [°]: n/a

Tools: [Refine](#) [Histograms...](#) [Transformations...](#)

[Finish](#) [Accept](#) [Cancel](#)

The Crystal is a Reticular Twin

- Integrate both components
- TWINABS- HKLF 4 and 5

1	0	0	28.20	1.50
2	0	0	2215.38	30.80
3	0	0	869.51	12.10
4	0	0	28.90	5.00
5	0	0	15.30	6.30
-5	1	0	133.29	10.10
-4	1	0	205.88	7.10
-3	1	0	225.38	7.40
-2	1	0	4917.21	51.19
-1	1	0	1636.54	11.70
0	1	0	2938.01	64.69
1	1	0	9999.00	66.19
2	1	0	189.98	5.50
3	1	0	211.38	6.10
4	1	0	153.08	8.00
5	1	0	34.00	13.50
-5	2	0	60.19	11.40

1	0	0	28.4078	1.61155	1
-2	1	1	2879.34	40.0417	-2
2	0	0	2879.34	40.0417	1
3	0	0	892.362	14.0204	1
-4	2	2	14.4204	12.3454	-2
4	0	0	14.4204	12.3454	1
5	0	0	15.6010	6.59970	1
-5	1	0	130.455	10.8995	1
-4	1	0	206.247	7.58213	1
-3	1	0	233.431	8.34012	1
-2	0	1	6414.16	70.8154	-2
-2	1	0	6414.16	70.8154	1
-1	1	0	1639.80	14.4143	1
0	1	0	3744.00	66.1207	-2
0	1	0	3744.00	66.1207	1
1	1	0	9999.99	106.633	1
-2	2	1	234.163	5.60337	-2

- HKLF 4 data through XPREP
- Potentially go back to TWINABS and XPREP
- Solve on HKLF 4 data
- Refine with both HKLF 4 and 5 data

Types of Twinning

– Merohedral

- The twinning operator is part of the Laue Class not the point group
 - Racemic twinning e.g. looks like $2/m$ but is 2
 - Tetrahedral, Trigonal/hexagonal, Cubic e.g. looks like $4/mmm$ but is $4/m$

– Pseudo-merohedral

- The twinning operator is part of the symmetry of the higher cell e.g. look orthorhombic but is monoclinic.

– Reticular

- e.g. obverse/reverse twinning, false centred cell.

– Non-Merohedral

- The twinning operator transforms between the two orientations can be anything but commonly 180°

Indication of twinning

- Multiple diffraction patterns
- $|E^*E-1|$ smaller than would be expected
- Problems choosing spacegroup
- Problems solving structure
- Large residual peak in non-sensible places
- K-values for weak data
- Disagreement reflects $F_{\text{obs}} \gg F_{\text{calc}}$
- Weighting scheme large
- Poor refinement R1 high than expected

Not Twinned?

DETERMINATION OF REDUCED (NIGGLI) CELL

Transformation from original cell (HKLF-matrix):

```
1.0000  0.0000  0.0000  0.0000  0.0000  1.0000  0.0000 -1.0000  0.0000
```

```
Unitcell:      15.814  24.601  29.023  90.00  90.00  90.25
```

```
Niggli form:   a.a =    250.08      b.b =    605.21      c.c =    842.33
                b.c =      0.00      a.c =      0.00      a.b =    -1.68
```

 Search for higher metric symmetry

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

```
-----
Option A: FOM = 0.247 deg.  ORTHORHOMBIC P-lattice  R(sym) = 0.442 [ 40491]
Cell:   15.814  24.601  29.023  90.00  90.00  90.25  volume:  11290.87
Matrix: 1.0000  0.0000  0.0000  0.0000  0.0000  1.0000  0.0000 -1.0000  0.0000
-----
```

```
Option B: FOM = 0.000 deg.  MONOCLINIC P-lattice  R(sym) = 0.064 [ 27105]
Cell:   15.814  29.023  24.601  90.00  90.25  90.00  volume:  11290.87
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-----
```

```
Option C: FOM = 0.247 deg.  MONOCLINIC P-lattice  R(sym) = 0.458 [ 26936]
Cell:   15.814  24.601  29.023  90.00  90.00  90.25  volume:  11290.87
Matrix: -1.0000  0.0000  0.0000  0.0000  0.0000 -1.0000  0.0000 -1.0000  0.0000
-----
```

```
Option D: FOM = 0.247 deg.  MONOCLINIC P-lattice  R(sym) = 0.471 [ 26611]
Cell:   24.601  15.814  29.023  90.00  90.00  89.75  volume:  11290.87
Matrix: 0.0000  0.0000 -1.0000  1.0000  0.0000  0.0000  0.0000 -1.0000  0.0000
-----
```

Option B selected

SPACE GROUP DETERMINATION

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	830	831	831	833	1246	1099	1104	1657
N (int>3sigma) =	0	687	679	698	688	1032	838	902	1366
Mean intensity =	0.0	38.3	38.4	36.0	38.1	37.6	24.5	37.8	37.9
Mean int/sigma =	0.0	23.4	23.3	23.1	23.2	23.3	18.7	23.4	23.3

Crystal system A and Lattice type P selected

Mean |E*E-1| = 0.783 [expected .968 centrosym and .736 non-centrosym]

Chiral flag NOT set

Systematic absences not required for triclinic

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]	P-1	# 2	centro	1	8646	0.000	0	0.0 / 23.3	3.87
[B]	P1	# 1	chiral	1	700	0.000	0	0.0 / 23.3	0.79

option [A] chosen

LST file indications

Analysis of variance for reflections employed in refinement $K = \text{Mean}[F_o^2] / \text{Mean}[F_c^2]$ for group

Fc/Fc(max)	0.000	0.012	0.017	0.022	0.028	0.034	0.041	0.051	0.065	0.093	1.000
Number in group	6329.	5120.	5450.	6202.	5301.	5139.	5726.	5484.	5515.	5598.	
Goof	0.837	0.860	0.970	1.021	1.090	1.081	1.099	1.050	1.019	1.003	
K	2.857	1.371	1.171	1.069	1.049	1.026	1.049	1.037	1.040	1.094	
Resolution(A)	0.75	0.78	0.81	0.85	0.89	0.95	1.02	1.12	1.28	1.61	inf
Number in group	5644.	5661.	5595.	5446.	5585.	5558.	5692.	5503.	5596.	5584.	
Goof	0.828	0.830	0.856	0.888	0.894	0.917	0.949	0.975	1.087	1.593	
K	1.075	1.043	1.051	1.048	1.046	1.045	1.069	1.074	1.086	1.107	
R1	0.255	0.221	0.187	0.159	0.133	0.111	0.104	0.090	0.080	0.101	

Recommended weighting scheme: WGHT 0.1327 0.0000
 Note that in most cases convergence will be faster if fixed weights (e.g. the default WGHT 0.1) are retained until the refinement is virtually complete, and only then should the above recommended values be used.

Most Disagreeable Reflections (* if suppressed or used for Rfree)

h	k	l	Fo ²	Fc ²	Delta(F ²)/esd	Fc/Fc(max)	Resolution(A)
-1	0	5	3306.90	195.66	9.72	0.015	4.70
1	-6	3	17930.58	317.53	9.17	0.020	4.03
-5	-1	1	16842.12	2148.40	9.15	0.051	3.12
-3	0	11	3588.74	35.68	8.97	0.007	2.06
5	-6	3	3787.50	78.23	8.88	0.010	2.52
1	6	3	20125.86	317.42	8.54	0.020	4.03
3	-7	3	6924.66	913.74	8.38	0.033	3.03
-1	11	3	14265.78	2393.98	7.84	0.054	2.48
3	11	1	6296.08	324.45	7.77	0.020	2.35
3	-11	1	7107.52	326.79	7.73	0.020	2.35
-3	13	1	3836.08	113.46	7.70	0.012	2.05
-1	-11	1	9117.20	1395.21	7.64	0.041	2.59
-10	-1	2	2209.62	66.25	7.53	0.009	1.57
-1	11	1	10622.40	1398.51	7.52	0.041	2.59
-7	-10	3	4039.27	1011.37	7.42	0.035	1.74
3	15	1	4619.09	830.09	7.42	0.032	1.81
-1	-11	3	13777.74	2380.37	7.41	0.054	2.48
-1	13	11	3326.11	57.67	7.32	0.008	1.57
-1	15	1	8343.10	1256.92	7.30	0.039	1.91
-1	-7	3	29590.46	3135.91	7.24	0.061	3.60
3	0	9	3830.77	770.17	7.07	0.016	2.47

INS format for TWIN

```

TITL gac440a in P2(1)
CELL 0.77490 15.8138 29.023 24.6010 90.000 90.247 90.000
ZERR 8.00 0.0012 0.002 0.0019 0.000 0.002 0.000
LATT -1
SYMM -X, 0.5+Y, -Z
SFAC C H N O NI
DISP Ni 0.19064 1.30243 5779.11
DISP O -0.00415 0.00737 40.17
DISP N -0.00306 0.00403 23.92
DISP C -0.00202 0.00197 13.60
DISP H 0.00000 0.00000 0.67
UNIT 512 440 104 48 24

TEMP -123
SIZE 0.50 0.30 0.20

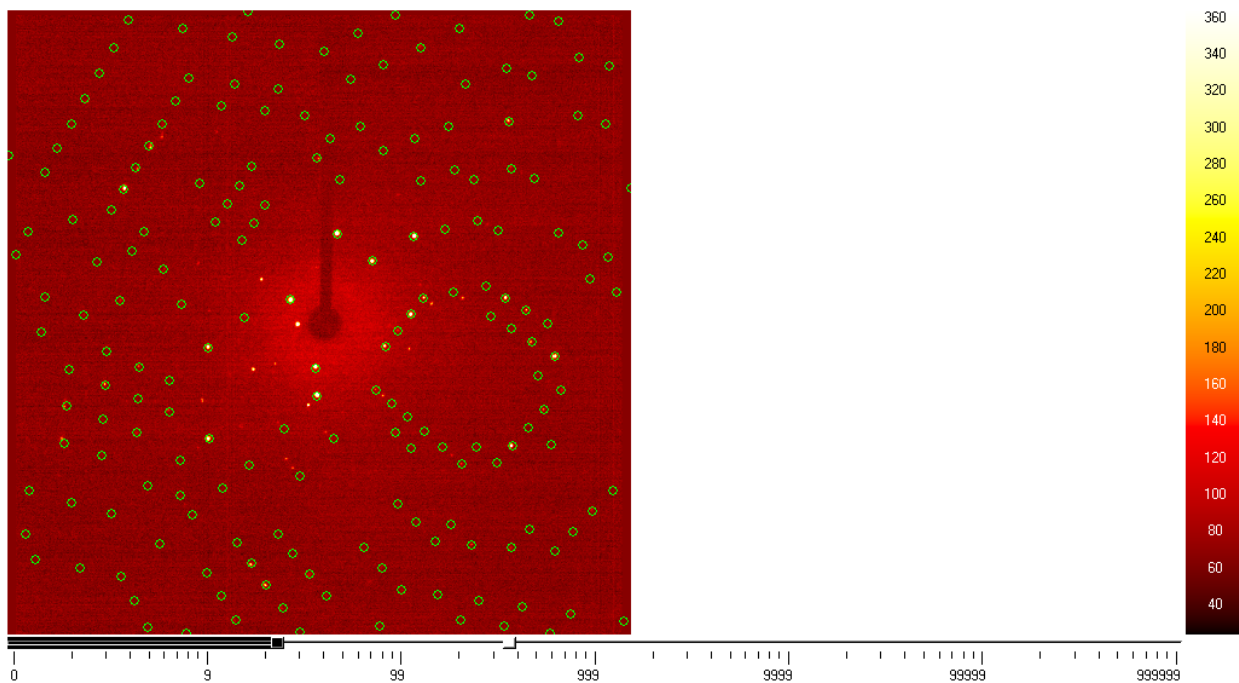
REM blue block
REM data cut at 0.81 angstrom

L.S. 4
BOND $H
ACTA
FMAP 2
PLAN 10

SIMU 0.016 N1S > C20S
DELU 0.004 N1S > C20S
ISOR 0.008 N22 C109 C314
ISOR 0.01 C119 C120 C206 C220 C305 C357
ISOR C32 C33 C204 C219 C228 C246 C251 C141 O36 C304 C311 C327
FLAT N2S > C10S
FLAT N4S > C20S
FLAT N3S > C15S
SADI N4S C16S N4S C20S
SADI C16S C17S C17S C18S C18S C19S C19S C20S

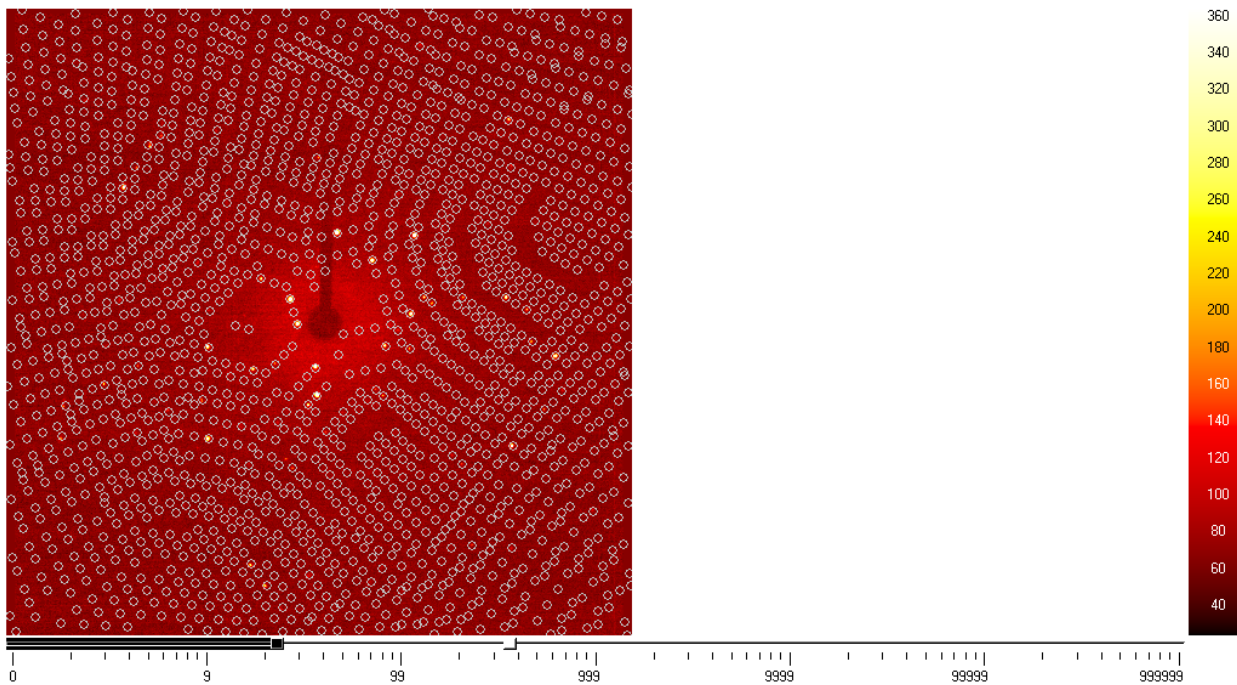
TWIN 1 0 0 0 -1 0 0 0 -1 -4
BASF 0.03796 0.52811 -0.00368

```



Reduced Unit Cells found:

<p>Method: Difference Vectors Score: 1.24 $a=25.93\text{\AA}$, $\alpha=90.50^\circ$, $V=20822\text{\AA}^3$ $b=28.29\text{\AA}$, $\beta=90.01^\circ$ $c=28.38\text{\AA}$, $\gamma=90.17^\circ$</p>	<p>HKL histogram:</p> <table border="1"> <tbody> <tr> <td>0.1:</td> <td>95.1%</td> <td>[2374/2496]</td> </tr> <tr> <td>0.2:</td> <td>95.8%</td> <td>[2390/2496]</td> </tr> <tr> <td>0.3:</td> <td>96.0%</td> <td>[2395/2496]</td> </tr> </tbody> </table>	0.1:	95.1%	[2374/2496]	0.2:	95.8%	[2390/2496]	0.3:	96.0%	[2395/2496]
0.1:	95.1%	[2374/2496]								
0.2:	95.8%	[2390/2496]								
0.3:	96.0%	[2395/2496]								
<p>Method: Fast Fourier Transform Score: 1.54 $a=12.60\text{\AA}$, $\alpha=89.92^\circ$, $V=2078\text{\AA}^3$ $b=12.72\text{\AA}$, $\beta=89.87^\circ$ $c=12.96\text{\AA}$, $\gamma=89.79^\circ$</p>	<p>HKL histogram:</p> <table border="1"> <tbody> <tr> <td>0.1:</td> <td>72.2%</td> <td>[1802/2496]</td> </tr> <tr> <td>0.2:</td> <td>76.3%</td> <td>[1904/2496]</td> </tr> <tr> <td>0.3:</td> <td>80.3%</td> <td>[2005/2496]</td> </tr> </tbody> </table>	0.1:	72.2%	[1802/2496]	0.2:	76.3%	[1904/2496]	0.3:	80.3%	[2005/2496]
0.1:	72.2%	[1802/2496]								
0.2:	76.3%	[1904/2496]								
0.3:	80.3%	[2005/2496]								



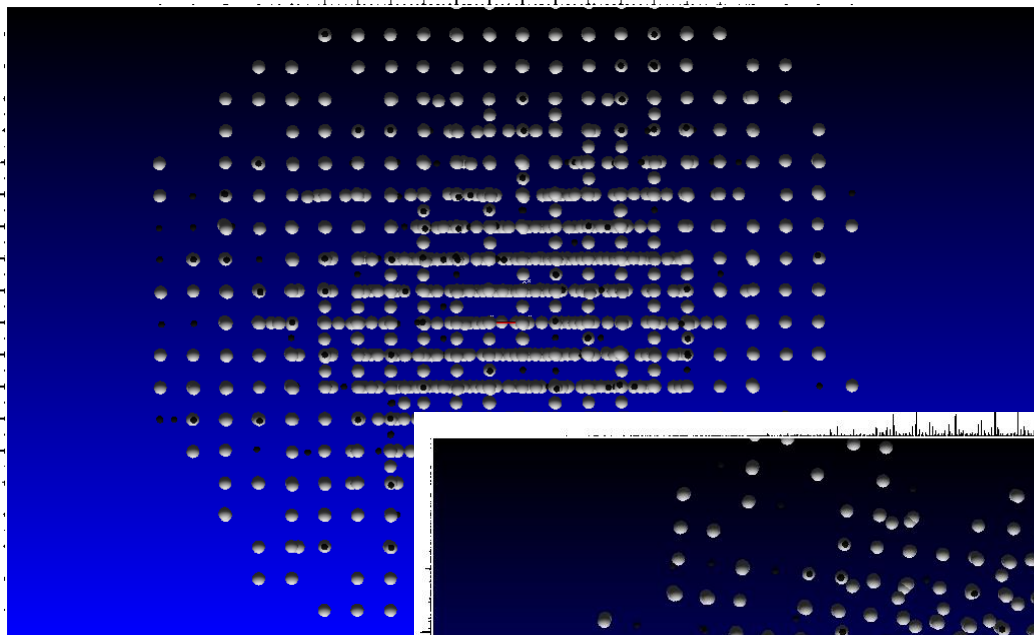
Reduced Unit Cells found:

<p>Method: Difference Vectors Score: 1.24 $a=25.93\text{\AA}$, $\alpha=90.50^\circ$, $V=20822\text{\AA}^3$ $b=28.29\text{\AA}$, $\beta=90.01^\circ$ $c=28.38\text{\AA}$, $\gamma=90.17^\circ$</p>	<p>HKL histogram:</p> <table border="1"> <tbody> <tr> <td>0.1:</td> <td>95.1%</td> <td>(2374/2496)</td> </tr> <tr> <td>0.2:</td> <td>95.8%</td> <td>(2390/2496)</td> </tr> <tr> <td>0.3:</td> <td>96.0%</td> <td>(2395/2496)</td> </tr> </tbody> </table>	0.1:	95.1%	(2374/2496)	0.2:	95.8%	(2390/2496)	0.3:	96.0%	(2395/2496)
0.1:	95.1%	(2374/2496)								
0.2:	95.8%	(2390/2496)								
0.3:	96.0%	(2395/2496)								
<p>Method: Fast Fourier Transform Score: 1.54 $a=12.60\text{\AA}$, $\alpha=89.92^\circ$, $V=2078\text{\AA}^3$ $b=12.72\text{\AA}$, $\beta=89.87^\circ$ $c=12.96\text{\AA}$, $\gamma=89.79^\circ$</p>	<p>HKL histogram:</p> <table border="1"> <tbody> <tr> <td>0.1:</td> <td>72.2%</td> <td>(1802/2496)</td> </tr> <tr> <td>0.2:</td> <td>76.3%</td> <td>(1904/2496)</td> </tr> <tr> <td>0.3:</td> <td>80.3%</td> <td>(2005/2496)</td> </tr> </tbody> </table>	0.1:	72.2%	(1802/2496)	0.2:	76.3%	(1904/2496)	0.3:	80.3%	(2005/2496)
0.1:	72.2%	(1802/2496)								
0.2:	76.3%	(1904/2496)								
0.3:	80.3%	(2005/2496)								

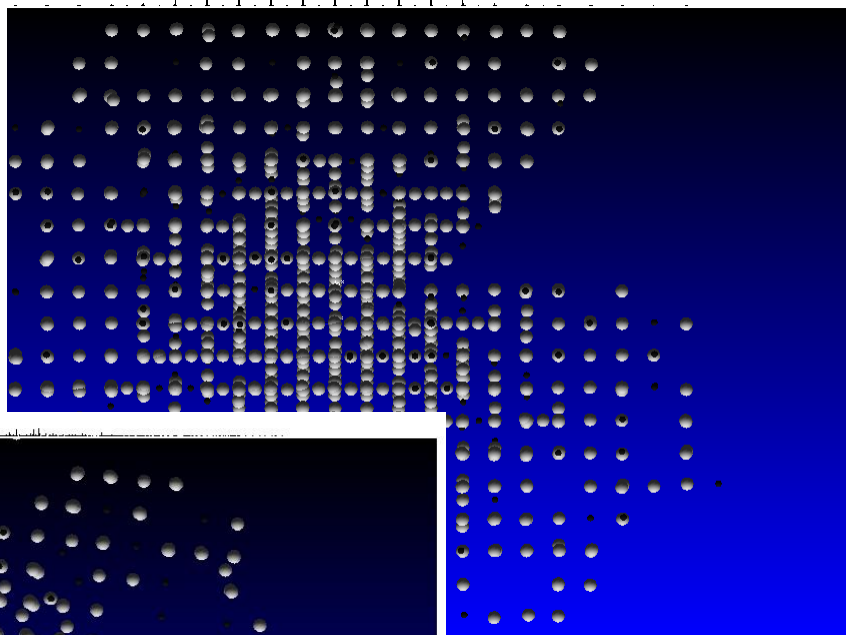
Initial Unit Cell:

a=12.64Å, $\alpha=89.93^\circ$, V=2100Å³
 b=12.78Å, $\beta=89.85^\circ$
 c=13.01Å, $\gamma=89.79^\circ$

Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
Cubic F	0.01	22.17	22.15	22.18	70.39	110.53	71.92
Cubic I	0.01	18.11	17.94	18.25	60.25	59.35	60.73
Cubic P	0.19	12.64	12.78	13.01	89.93	89.85	89.79
Hexagonal P	0.01	12.64	12.78	13.01	90.07	89.85	90.21
Rhombohedral R	0.21	17.94	18.11	22.24	91.31	89.53	119.27
Tetragonal I	0.01	12.64	12.78	31.60	66.12	66.48	89.79
Tetragonal P	0.35	12.64	12.78	13.01	89.93	89.85	89.79
Orthorhombic F	0.01	17.94	18.00	47.82	41.39	111.55	89.37
Orthorhombic I	0.01	12.64	12.78	31.60	113.88	113.52	89.79
Orthorhombic C	0.35	17.94	18.00	13.01	90.15	89.94	89.37
Orthorhombic P	0.56	12.64	12.78	13.01	89.93	89.85	89.79
Monoclinic C	0.31	18.00	17.94	13.01	90.06	90.15	90.63
Monoclinic P	0.63	12.64	13.01	12.78	90.07	90.21	89.85
Triclinic P	1.00	12.64	12.78	13.01	89.93	89.85	89.79



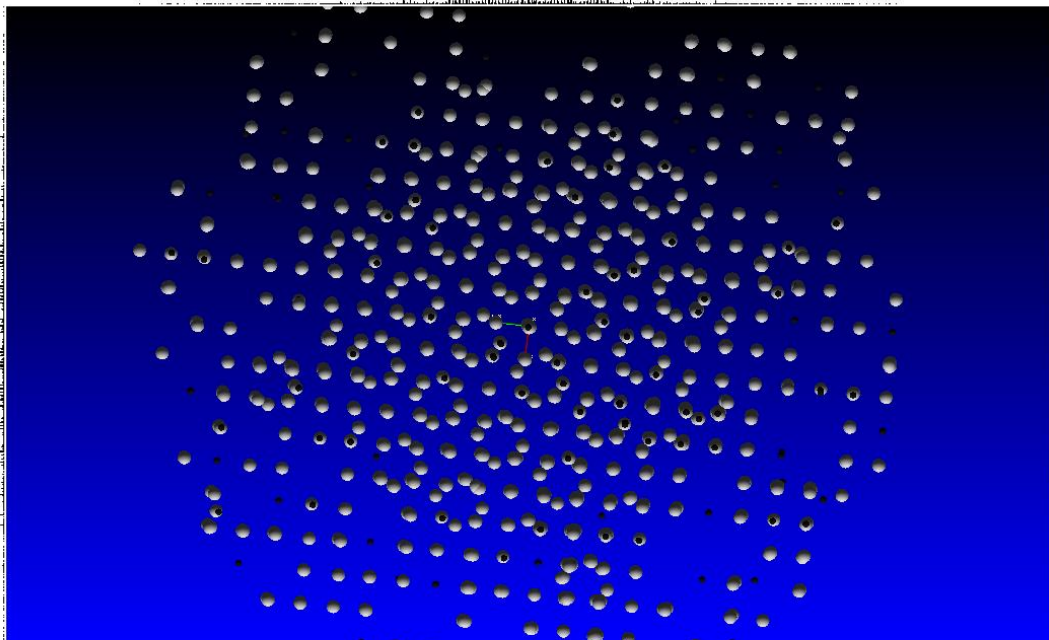
Intensity Filter Reflection Size



Zoom



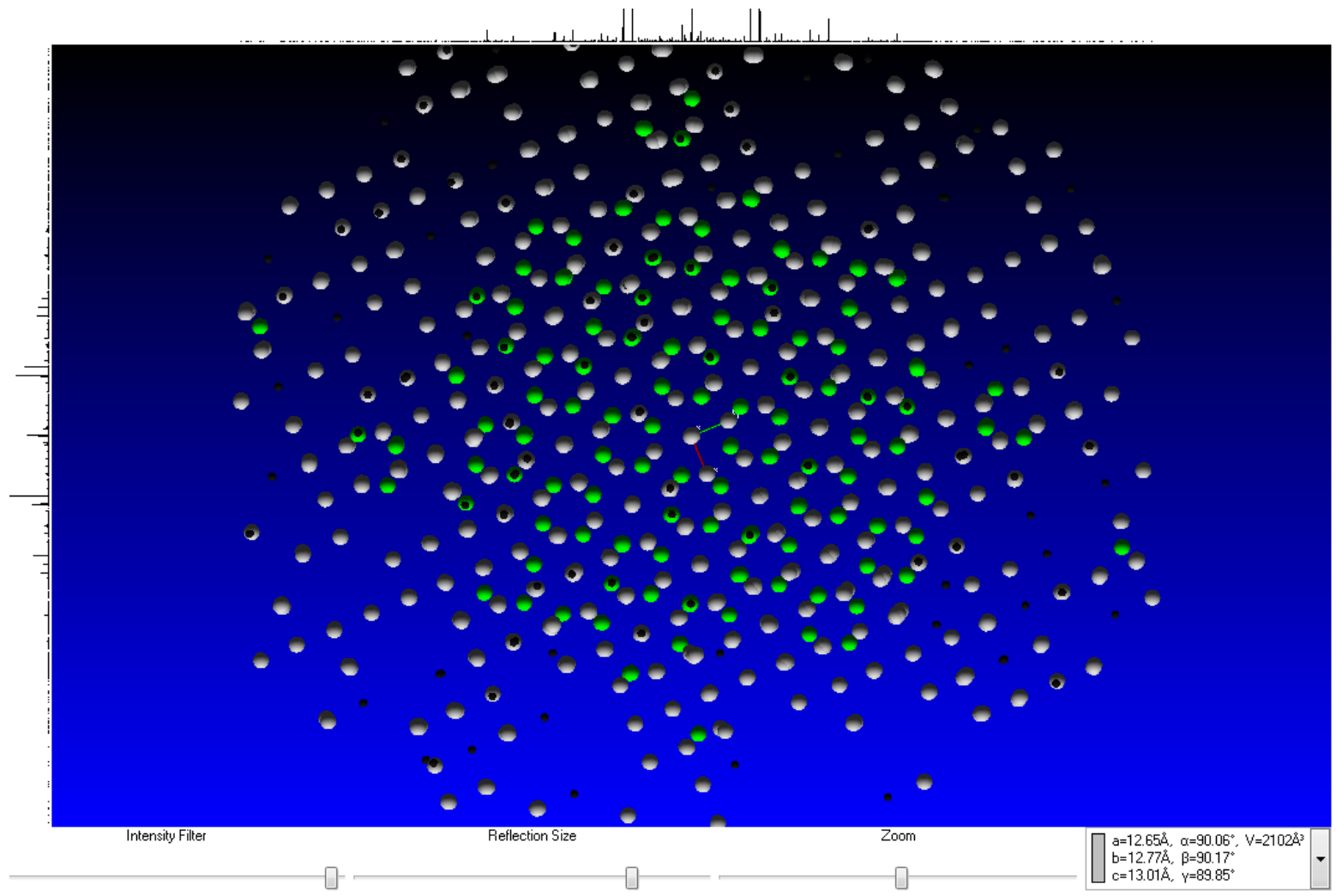
a=12.64Å, α=89.93°, V=2100Å³
 b=12.78Å, β=89.85°
 c=13.01Å, γ=89.79°

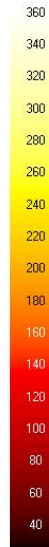
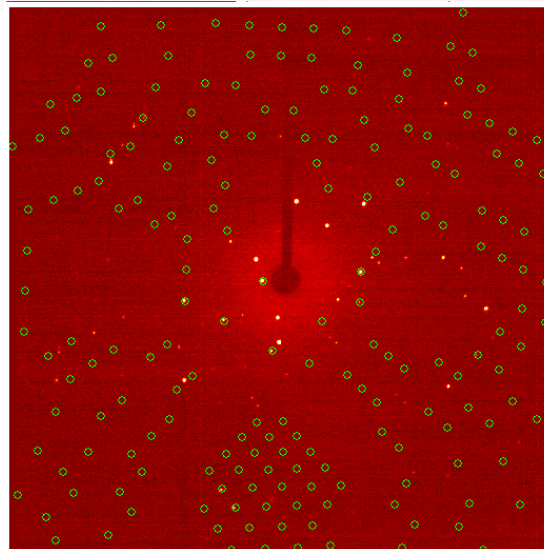
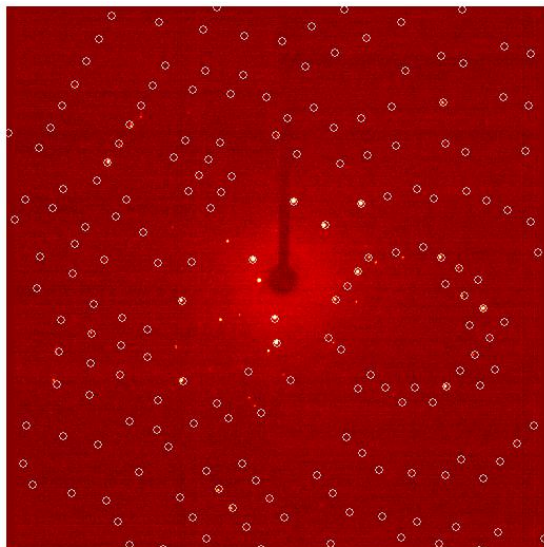


Intensity Filter Reflection Size Zoom



a=12.64Å, α=89.93°, V=2100Å³
 b=12.78Å, β=89.85°
 c=13.01Å, γ=89.79°





Unit Cell: $a=12.65\text{\AA}$, $\alpha=90.00^\circ$, $V=2102\text{\AA}^3$
 $b=12.77\text{\AA}$, $\beta=90.17^\circ$, Monoclinic P
 $c=13.01\text{\AA}$, $\gamma=90.00^\circ$

Unit cell

a [Å]	12.631 ± 0.004
b [Å]	12.765 ± 0.003
c [Å]	13.011 ± 0.003
α [°]	90.00
β [°]	90.098 ± 0.013
γ [°]	90.00
V [Å ³]	2098.0 ± 1.4
<input type="checkbox"/> Domain translation	
x [mm]	0.00
y [mm]	0.00

Reflections: Group 0: 1887 reflections

Go to Image: D:\frames\guest\MCJC_D4\MCJC_D4_01_0001.sfm

Tolerance: 0.26

650 Reflections selected for Refinement

Show selected Reflections

Unit Cell: $a=12.65\text{\AA}$, $\alpha=90.00^\circ$, $V=2102\text{\AA}^3$
 $b=12.77\text{\AA}$, $\beta=90.17^\circ$, Monoclinic P
 $c=13.01\text{\AA}$, $\gamma=90.00^\circ$

Unit cell

a [Å]	12.667 ± 0.008
b [Å]	12.761 ± 0.006
c [Å]	13.006 ± 0.007
α [°]	90.00
β [°]	89.91 ± 0.02
γ [°]	90.00
V [Å ³]	2102 ± 3
<input type="checkbox"/> Domain translation	
x [mm]	0.00
y [mm]	0.00

Reflections: Group 1: 883 reflections

Go to Image: D:\frames\guest\MCJC_D4\MCJC_D4_01_0001.sfm

Tolerance: 0.24

279 Reflections selected for Refinement

Show selected Reflections

Show predicted Reflections

12.646 12.991 12.749 90.00 90.16 90.00

5063 Reflections read from file junk.hkl; mean (I/sigma) = 13.23

Lattice exceptions: P A B C I F Obv Rev All
 N (total) = 0 2532 2522 2532 2537 3793 3366 3367 5063
 N (int>3sigma) = 0 1627 1633 1616 1578 2438 2148 2172 3262
 Mean intensity = 0.0 21.1 20.6 21.1 20.9 20.9 20.6 20.8 20.5
 Mean int/sigma = 0.0 13.2 13.4 13.1 13.3 13.2 13.3 13.3 13.2
 Lattice type: P chosen volume: 2094.55

 DETERMINATION OF REDUCED (NIGGLI) CELL

Transformation from original cell (HKLF-matrix):
 1.0000 0.0000 0.0000 0.0000 0.0000 1.0000 0.0000 -1.0000 0.0000
 unitcell: 12.646 12.749 12.991 90.00 90.00 90.16
 Niggli form: a.a = 159.93 b.b = 162.53 c.c = 168.78
 b.c = 0.00 a.c = 0.00 a.b = -0.46

 Search for higher metric symmetry
 Identical indices and Friedel opposites combined before calculating R(sym)

Option A: FOM = 0.231 deg. TETRAGONAL P-lattice R(sym) = 0.141 [3465]
 Cell: 12.646 12.749 12.991 90.00 90.00 90.16 volume: 2094.55
 Matrix: 1.0000 0.0000 0.0000 0.0000 0.0000 1.0000 0.0000 -1.0000 0.0000

Option B: FOM = 0.540 deg. TETRAGONAL P-lattice R(sym) = 0.801 [3450]
 Cell: 12.749 12.991 12.646 90.00 90.16 90.00 volume: 2094.55
 Matrix: 0.0000 0.0000 1.0000 0.0000 -1.0000 0.0000 1.0000 0.0000 0.0000

Option C: FOM = 0.164 deg. ORTHORHOMBIC P-lattice R(sym) = 0.528 [3309]
 Cell: 12.646 12.749 12.991 90.00 90.00 90.16 volume: 2094.55
 Matrix: 1.0000 0.0000 0.0000 0.0000 0.0000 1.0000 0.0000 -1.0000 0.0000

Option D: FOM = 0.000 deg. MONOCLINIC P-lattice R(sym) = 0.036 [2086]
 Cell: 12.646 12.991 12.749 90.00 90.16 90.00 volume: 2094.55
 Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000

Option E: FOM = 0.164 deg. MONOCLINIC P-lattice R(sym) = 0.555 [2139]
 Cell: 12.646 12.749 12.991 90.00 90.00 90.16 volume: 2094.55
 Matrix: -1.0000 0.0000 0.0000 0.0000 0.0000 -1.0000 0.0000 -1.0000 0.0000

Option F: FOM = 0.164 deg. MONOCLINIC P-lattice R(sym) = 0.555 [2123]
 Cell: 12.749 12.646 12.991 90.00 90.00 89.84 volume: 2094.55
 Matrix: 0.0000 0.0000 -1.0000 1.0000 0.0000 0.0000 0.0000 -1.0000 0.0000

option D selected

$\text{Al}(\text{O}_3\text{PCH}_2\text{CH}_2\text{PO}_3)(\text{OH})$

$(\text{H}_3\text{NCH}_2\text{CH}_2\text{NH}_3)$

Monoclinic $P2_1/m$

$a = 8.052$

$\alpha = 90$

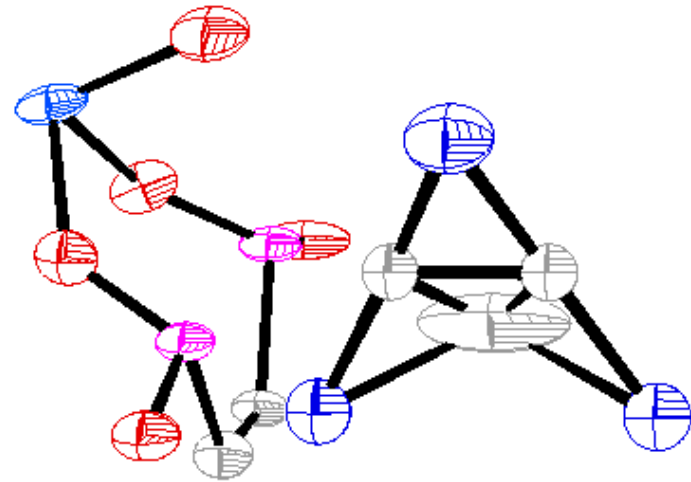
$b = 7.029$

$\beta = 98.441$

$c = 8.977$

$\gamma = 90$

Volume = 503.2



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SPACE GROUP DETERMINATION

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	2532	2522	2532	2537	3793	3366	3367	5063
N (int>3sigma) =	0	1627	1633	1616	1578	2438	2148	2172	3262
Mean intensity =	0.0	21.1	20.6	21.1	20.9	20.9	20.6	20.8	20.5
Mean int/sigma =	0.0	13.2	13.4	13.1	13.3	13.2	13.3	13.3	13.2

Crystal system M and Lattice type P selected

Mean |E*E-1| = 1.014 [expected .968 centrosym and .736 non-centrosym]

chiral flag NOT set

systematic absence exceptions:

	-21-	-a-	-c-	-n-
N	6	144	146	142
N I>3s	5	53	53	0
<I>	213.2	19.3	18.9	0.3
<I/s>	27.6	11.8	11.6	0.6

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/n	# 13	centro	1	292	0.036	2086	0.6 / 11.6	1.46
[B]	Pn	# 7	non-cen	1	226	0.036	2086	0.6 / 11.6	9.09

option [A] chosen

Supercells



Lab Cell

Monoclinic $P2_1/m$

$$a = 8.052$$

$$\alpha = 90$$

$$b = 7.029$$

$$\beta = 98.441$$

$$c = 8.977$$

$$\gamma = 90$$

$$\text{Volume} = 503.2$$

Synchrotron Cell

Monoclinic $P2_1/n$

$$a = 11.142$$

$$\alpha = 90$$

$$b = 7.008$$

$$\beta = 96.24$$

$$c = 12.903$$

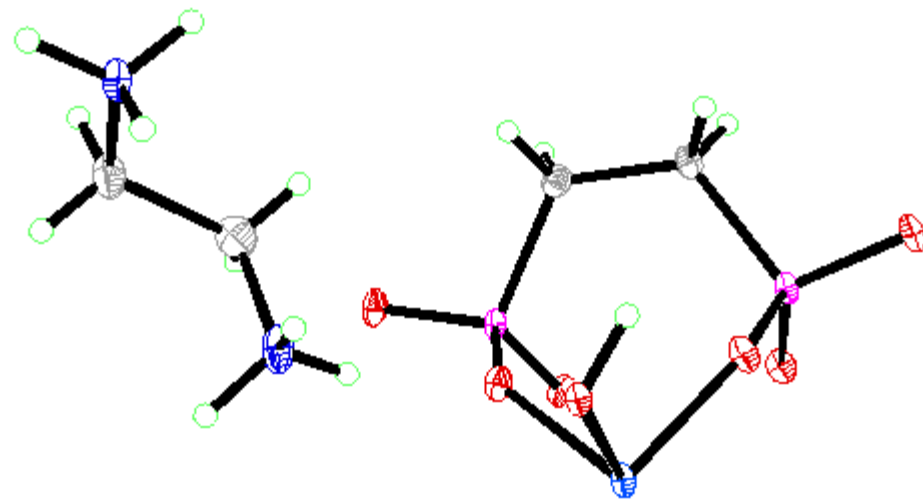
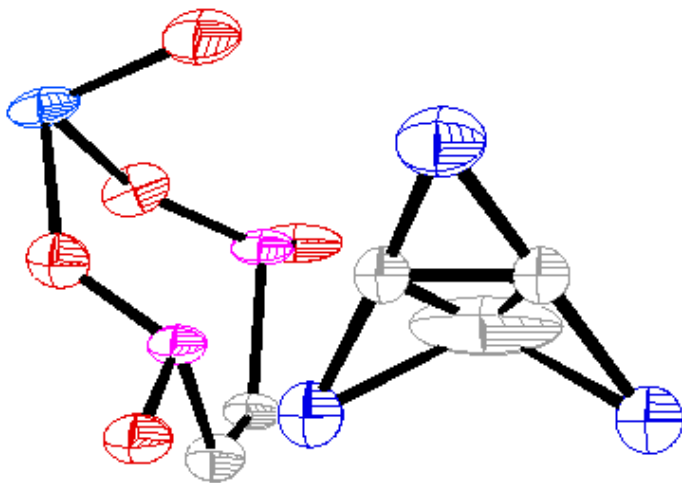
$$\gamma = 90$$

$$\text{Volume} = 1001.50$$

Supercells

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	2987	2979	2982	2987	4474	3965	3964	5956
N (int>3sigma) =	0	1639	1233	1592	1583	2232	2163	2171	3233
Mean intensity =	0.0	22.2	8.3	22.1	19.3	17.5	21.2	20.7	20.9
Mean int/sigma =	0.0	6.0	3.9	5.9	5.8	5.3	6.0	5.9	5.9

Supercells



NiC₂₈H₁₄O₇N₂

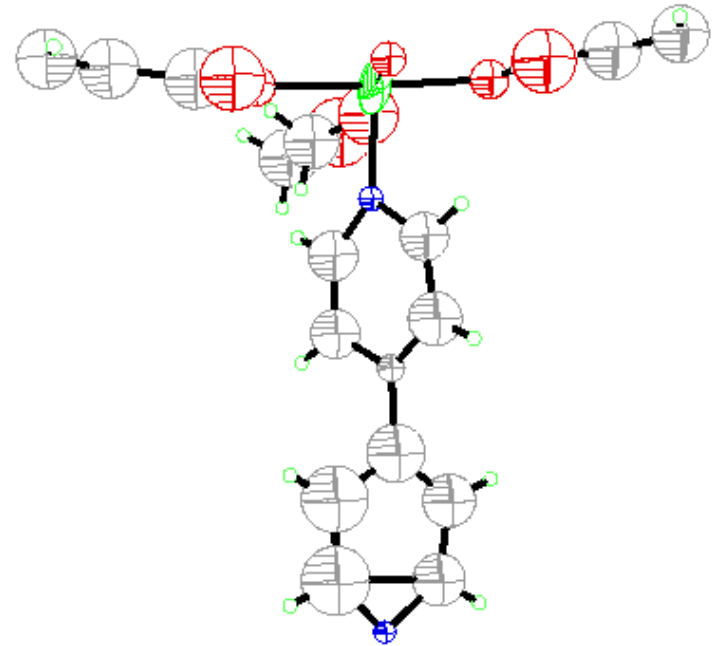
Trigonal P3

a = 19.285 $\alpha = 90$

b = 19.285 $\beta = 90$

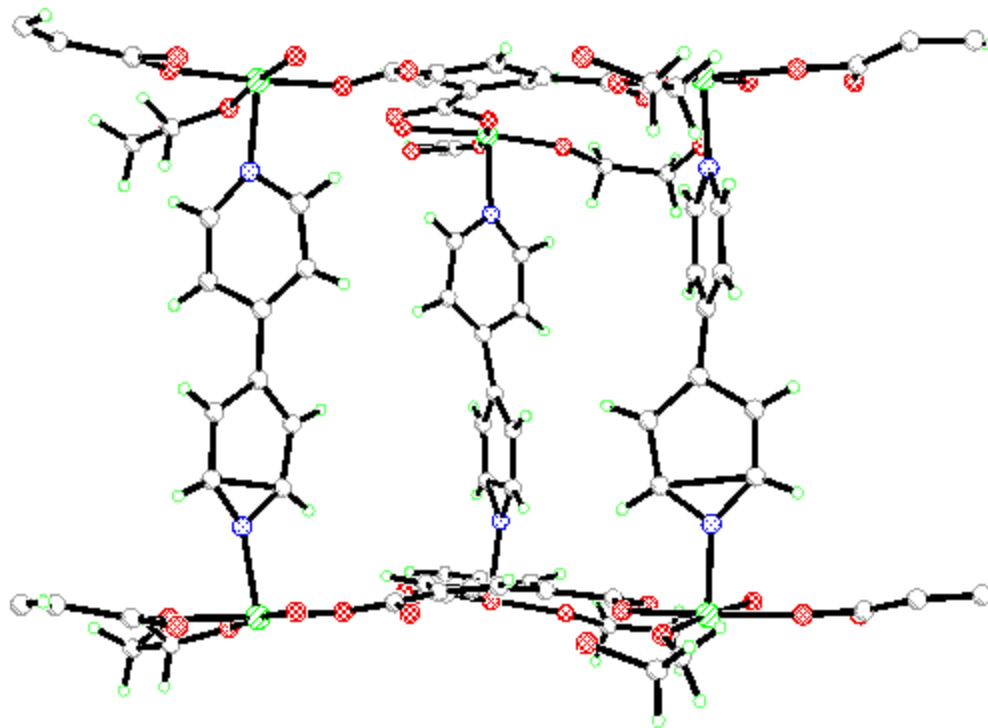
c = 11.270 $\gamma = 120$

Volume = 3665.9

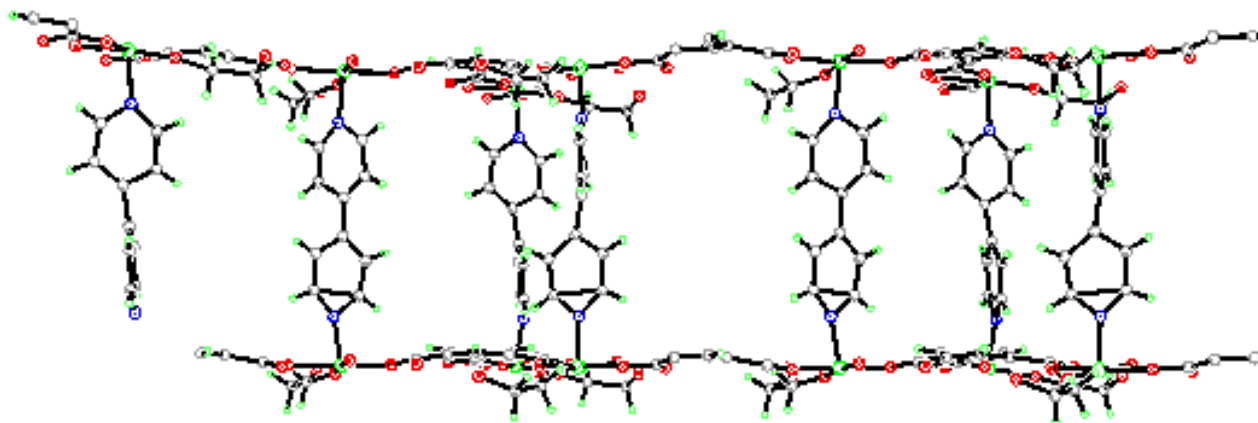


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Supercells



Supercells



NiC₂₈H₁₄O₇N₂

Trigonal P3

$$a = 19.285 \quad \alpha = 90$$

$$b = 19.285 \quad \beta = 90$$

$$c = 11.270 \quad \gamma = 120$$

$$\text{Volume} = 3665.9$$

NiC₂₈H₁₄O₇N₂

Trigonal P3

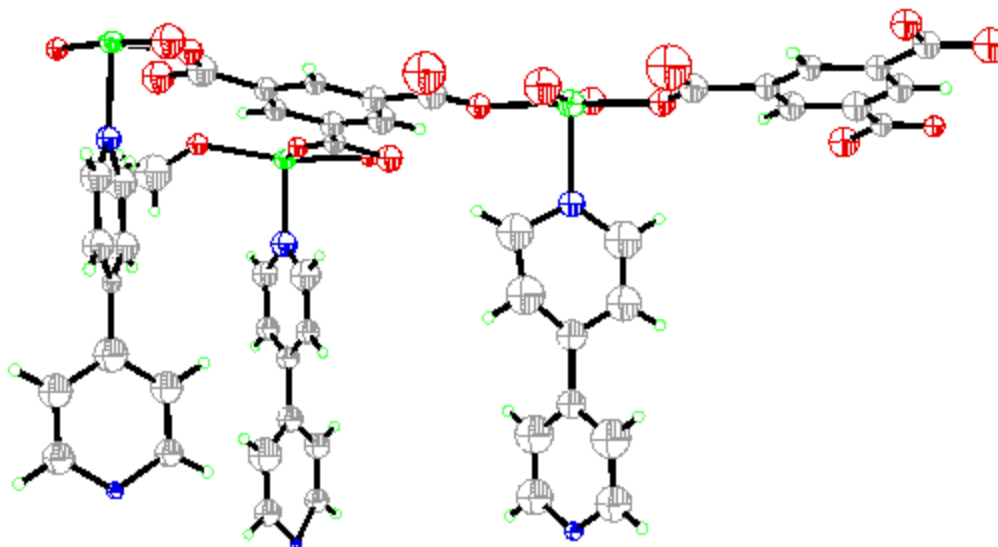
$$a = 33.521 \quad \alpha = 90$$

$$b = 33.521 \quad \beta = 90$$

$$c = 11.302 \quad \gamma = 120$$

$$\text{Volume} = 10998$$

Supercells



Supercells

