

From Crystal to Diffraction Pattern

- Judging the Quality of a Crystal.
- Determining the Best Exposure Time.
- Data Collection Strategy.

Judging the Quality of the X-tal

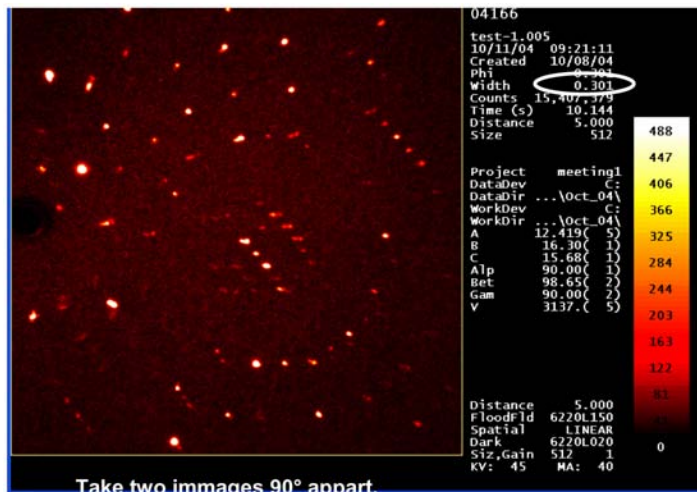


Figure by MIT OCW.

Judging the Quality of the X-tal

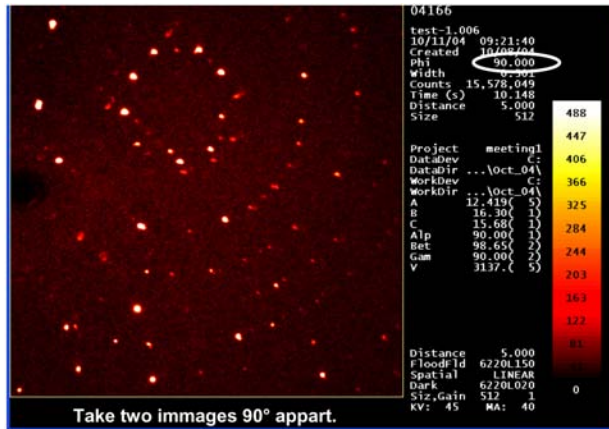


Figure by MIT OCW.

Judging the Quality of the X-tal

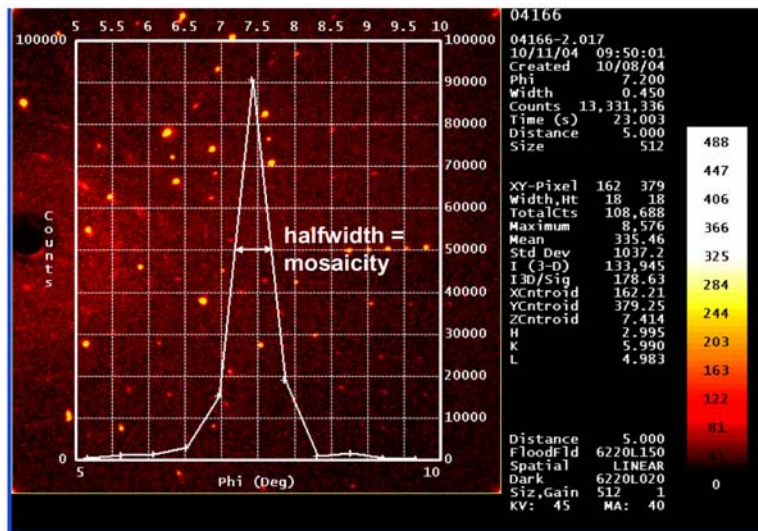


Figure by MIT OCW.

Determining the Exposure Time

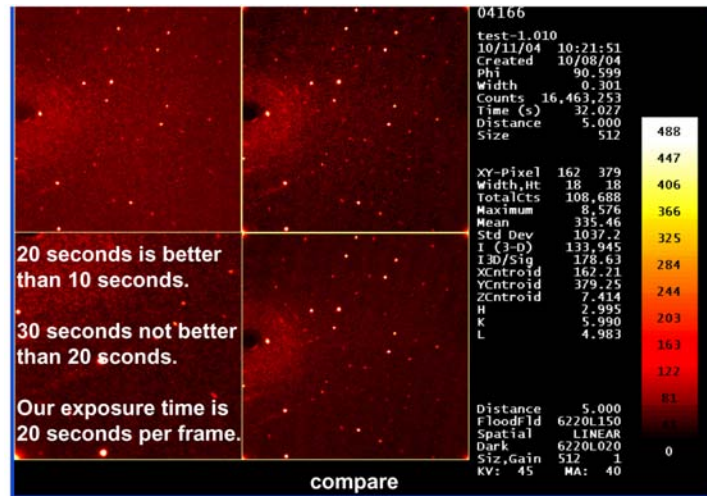


Figure by MIT OCW.

Collect Dark Current

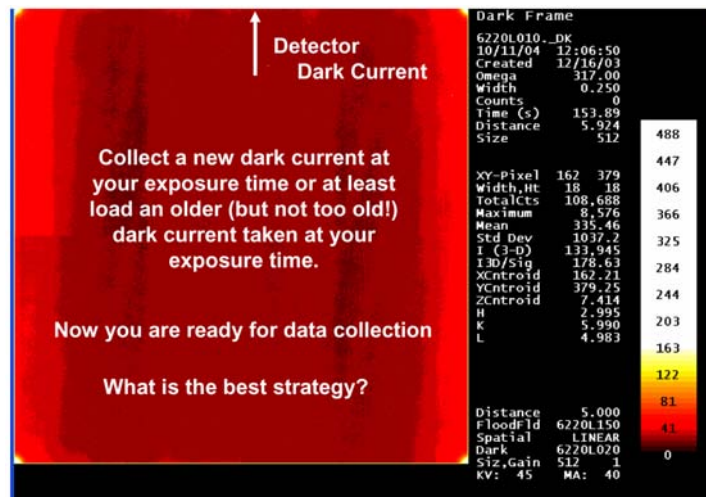


Figure by MIT OCW.

ACA 2005 in Orlando FL: Session 09:01
Data Collection Strategies

MoO is No Schmu –
Why Less is Not Always More.

Peter Müller

MIT

Roland Boese distinguishes between unavoidable errors (*aka* artifacts), avoidable errors, and really avoidable errors.

Artifacts

- Libration.
- C-C and C-N triple bonds determined too short.
- Inaccurately determined hydrogen positions.
- Fourier truncation effects.

Avoidable Errors

- Wrong unit-cell.
- Twin refined as disorder.
- Wrong atom type assignment.
- Incorrect space group.
- Fourier truncation peaks mistaken for hydrogen atoms.

Roland Boese distinguishes between unavoidable errors (*aka* artifacts), avoidable errors, and really avoidable errors.

Really Avoidable Errors

- Typos in unit-cell parameters.
- Misadjustment of the diffractometer (zero points, *etc.*).
- No absorption correction.
- Data collection at room temperature.
- **Wrong data collection strategy.**

The **Standard Data Collection Strategy** for the Platform geometry as recommended by Bruker for many years is still used in many labs:

Three 180° ω -scans, $\phi = 0^\circ, 90^\circ$ and 180° ;
Scan width: 0.3°

Is this good enough?

In most cases probably yes.

But can we do better?

In any case!

What is the optimal Scan Width?

The narrow frame algorithm in SAINT allows for 3D profile fitting, which is a good thing.

For this to work, the scan width needs to be smaller than the mosaicity, but not *much* smaller. 0.3° is fine, but 0.5° suffices in almost any case.

Going from a scan width of 0.3° to 0.5° saves memory and time. These gains can be used to collect more frames, which will increase the quality of the dataset.

Z. Dauter (1999), *Acta Cryst.* **D55**, 1703-1717.

What is MoO?

Redundancy: **a**: the quality or state of being redundant, **b**: the use of redundant components, **c**: dismissal from a job by layoff.

Also: number of times the average reflection has been observed, regardless of the Ψ -angle.

Multiplicity: Number of symmetry equivalents in a certain Laue group. *E.g.* The multiplicity is 4 in 2/m (monoclinic).

True Redundancy: Number of times the average reflection has been observed independently at different Ψ -angles. Not such a nice term.

What is MoO?

Redundancy: **a**: the quality or state of being redundant, **b**: the use of redundant components, **c**: dismissal from a job by layoff.

Also: number of times the average reflection has been observed, regardless of the Ψ -angle.

Multiplicity: Number of symmetry equivalents in a certain Laue group. *E.g.* The multiplicity is 4 in 2/m (monoclinic).

MoO: Multiplicity of Observations. Same as “true redundancy” but a nicer and clearer term.

What is MoO?

P. Müller *et al.* (2005), *Acta Cryst.* **D61**, 309-315:

“MoO: Multiplicity of Observations. This term was defined at the SHELX workshop in Göttingen in September 2003 to distinguish the MoO from redundancy, or multiplicity, with which the MoO has frequently been confused in the past. In contrast to redundancy, which is repeated recording of the same reflection obtained from the same crystal orientation (performing scans that rotate the crystal by more than 360°), MoO, sometimes also referred to as “true redundancy”, describes multiple measurements of the same reflection obtained from different crystal orientations (*i.e.* measured at different Ψ -angles).”

What is MoO?

With a one-circle diffractometer the maximum value for the MoO is a direct function of the Laue group.

With a three-circle, four-circle or a Kappa goniometer, the MoO_{max} is a function mostly of beamtime, radiation damage and personal patience.

For most small molecule structures a MoO of > 7 or 8 can be assumed “good”. A MoO of 4 to 6 should be considered the minimum (in my private opinion, that is), and everything in the double-digits is “way cool”.

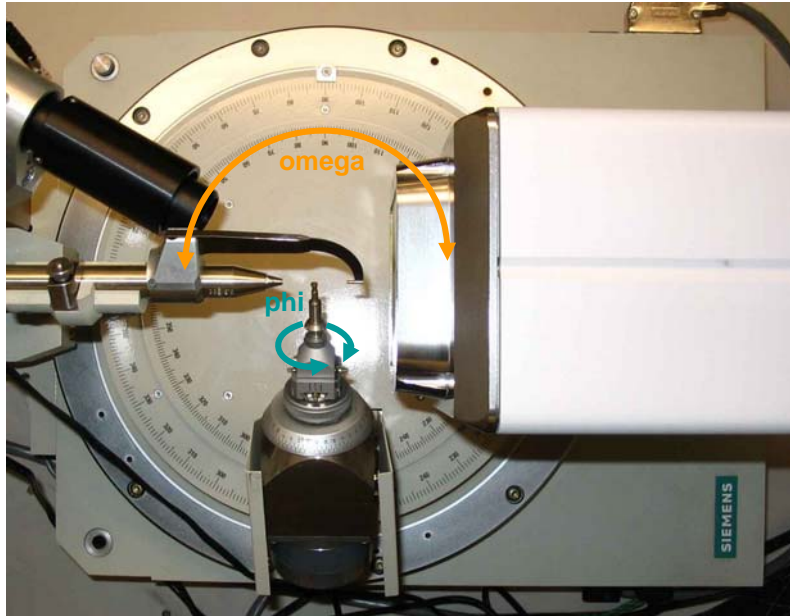
MoO is no Schmu?

Whenever possible try to collect as many frames as you can.

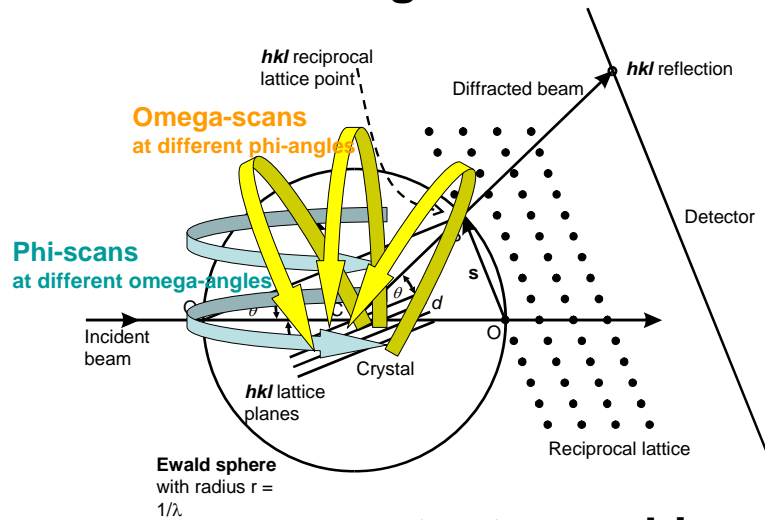
Try to distribute the frames all over the reciprocal space, collecting the same reflections from different Ψ -angles. This will give you the same reflections together on the same frame with different other reflections, which helps with semi-empirical absorption correction.

How to do that?

The Platform Geometry



phi- and omega-scans are orthogonal



...try to combine them!

Practical Test

Three structures – the famous “Ylid” and two monoclinic real

SCAN MultiRun List (50 lines) MIT Standard									
Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
1	001	-30.00	-20.00	0.00	54.74	2	-0.500	400	20.00
2	001	-30.00	-30.00	0.00	54.74	3	0.450	800	20.00
3	001	-30.00	-20.00	90.00	54.74	2	-0.500	400	20.00
4	001	-30.00	-20.00	180.00	54.74	2	-0.500	400	20.00
5	001	-30.00	210.00	0.00	54.74	3	0.450	800	20.00

3. Three 200° ω -scans plus two full 360° ϕ -scans:

MIT Standard.

A full scan takes ca. 9 hours (10 second exposures), frequently one can interrupt right before or during the fifth run. This allows for one to two data collections per day.

Ylid three 180° ω -scans

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
Inf - 2.13	84	84	100.0	7.15	1302.5	87.38	0.0160	0.0088
2.13 - 1.66	85	85	100.0	8.59	519.7	75.21	0.0186	0.0100
1.66 - 1.42	91	91	100.0	8.95	377.3	69.92	0.0233	0.0114
1.42 - 1.27	87	87	100.0	8.63	200.9	54.92	0.0342	0.0152
1.27 - 1.17	87	87	100.0	8.37	217.5	51.48	0.0373	0.0159
1.17 - 1.10	85	85	100.0	7.81	130.7	36.59	0.0436	0.0209
1.10 - 1.03	99	99	100.0	7.65	86.5	29.40	0.0563	0.0269
1.03 - 0.98	95	95	100.0	6.99	92.2	27.35	0.0619	0.0303
0.98 - 0.94	93	93	100.0	6.99	60.8	21.83	0.0769	0.0383
0.94 - 0.90	104	104	100.0	6.66	56.1	18.55	0.0815	0.0431
0.90 - 0.87	87	87	100.0	6.47	43.2	15.85	0.0917	0.0527
0.87 - 0.84	103	103	100.0	6.08	35.4	13.38	0.1074	0.0639
0.84 - 0.82	88	88	100.0	5.74	37.4	12.49	0.1009	0.0648
0.82 - 0.79	130	130	100.0	5.82	28.5	10.65	0.1193	0.0786
0.79 - 0.77	92	92	100.0	5.50	25.7	9.59	0.1359	0.0896
0.77 - 0.75	116	116	100.0	5.27	24.7	8.60	0.1375	0.0997
0.75 - 0.72	127	127	100.0	5.00	17.4	6.75	0.1920	0.1292

0.82 - 0.72	512	512	100.0	5.42	25.3	9.14	0.1349	0.0918
Inf - 0.72	1653	1653	100.0	6.81	173.1	30.19	0.0313	0.0185

Merged [A], lowest resolution = 18.39 Angstroms, 368 outliers downweighted

Ylid two 180° ω -scans, one 180° ϕ -scan

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
Inf - 2.14	83	83	100.0	7.01	1310.4	90.29	0.0168	0.0087
2.14 - 1.67	83	83	100.0	8.30	529.8	77.82	0.0179	0.0097
1.67 - 1.43	84	84	100.0	8.58	401.5	73.22	0.0225	0.0113
1.43 - 1.29	84	84	100.0	8.25	203.5	57.47	0.0337	0.0146
1.29 - 1.18	88	88	100.0	7.27	204.2	49.07	0.0346	0.0164
1.18 - 1.11	83	83	100.0	6.73	140.4	36.81	0.0403	0.0216
1.11 - 1.05	85	85	100.0	6.92	104.8	32.28	0.0454	0.0236
1.05 - 0.99	95	95	100.0	6.05	95.4	27.31	0.0566	0.0309
0.99 - 0.95	95	95	100.0	6.05	71.5	23.16	0.0684	0.0364
0.95 - 0.91	99	99	100.0	5.79	58.8	19.53	0.0768	0.0442
0.91 - 0.88	87	87	100.0	5.53	43.4	14.90	0.0826	0.0559
0.88 - 0.85	95	95	100.0	5.40	35.1	13.81	0.0987	0.0641
0.85 - 0.82	126	126	100.0	5.17	38.2	13.08	0.1014	0.0666
0.82 - 0.79	126	127	99.2	4.89	29.7	10.63	0.1078	0.0833
0.79 - 0.77	90	91	98.9	4.86	25.4	9.89	0.1254	0.0887
0.77 - 0.75	115	116	99.1	4.41	25.5	8.59	0.1356	0.1068
0.75 - 0.72	120	123	97.6	4.33	17.8	7.07	0.1637	0.1298

0.82 - 0.72	501	507	98.8	4.64	25.9	9.28	0.1250	0.0951
Inf - 0.72	1638	1644	99.6	6.05	175.1	30.59	0.0293	0.0188
Merged [A], lowest resolution = 18.37 Angstroms, 314 outliers downweighted								

Ylid three 200° ω -scans, two 360° ϕ -scans

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
Inf - 2.14	83	83	100.0	16.82	1309.8	113.83	0.0170	0.0070
2.14 - 1.67	83	83	100.0	20.17	531.6	104.63	0.0229	0.0075
1.67 - 1.43	84	84	100.0	20.31	404.7	100.57	0.0270	0.0084
1.43 - 1.29	84	84	100.0	18.54	203.6	80.06	0.0365	0.0107
1.29 - 1.18	89	89	100.0	16.30	202.9	69.65	0.0376	0.0116
1.18 - 1.10	95	95	100.0	15.26	148.4	55.40	0.0424	0.0141
1.10 - 1.04	83	83	100.0	15.04	83.5	42.66	0.0540	0.0177
1.04 - 0.99	86	86	100.0	13.15	100.9	42.70	0.0582	0.0204
0.99 - 0.95	93	93	100.0	13.16	68.9	35.22	0.0704	0.0235
0.95 - 0.91	100	100	100.0	12.80	58.0	30.01	0.0798	0.0277
0.91 - 0.88	87	87	100.0	12.01	42.6	23.13	0.0940	0.0344
0.88 - 0.85	95	95	100.0	11.92	35.1	22.05	0.1032	0.0390
0.85 - 0.82	125	125	100.0	10.93	37.1	20.34	0.1017	0.0411
0.82 - 0.79	128	128	100.0	10.89	28.8	17.18	0.1171	0.0492
0.79 - 0.77	91	91	100.0	10.47	25.4	15.55	0.1280	0.0555
0.77 - 0.75	116	116	100.0	9.62	24.7	13.71	0.1344	0.0616
0.75 - 0.72	126	126	100.0	9.60	17.4	11.15	0.1777	0.0785

0.82 - 0.72	509	509	100.0	10.18	25.2	14.74	0.1310	0.0570
Inf - 0.72	1648	1648	100.0	13.55	173.9	43.40	0.0310	0.0128
Merged [A], lowest resolution = 18.38 Angstroms, 883 outliers downweighted								

Ylid three 180° ω -scans,

Refinement Statistics:

R1 = 0.0403 for 2571 $F_o > 4\text{sig}(F_o)$

wR2 = 0.1015 for all 2783 data

Goof = 1.068 for all 2783 data

Flack x parameter = -0.0169 with esd 0.0743

Average s.u. on all C-C bonds: 0.0026 Å

Ylid two 180° ω -scans, one 180° ϕ -scan

Refinement Statistics:

R1 = 0.0402 for 2559 $F_o > 4\text{sig}(F_o)$

wR2 = 0.1010 for all 2759 data

Goof = 1.065 for all 2759 data

Flack x parameter = 0.0131 with esd 0.0749

Average s.u. on all C-C bonds: 0.0026 Å

Ylid three 200° ω -scans, two 360° ϕ -scans

Refinement Statistics:

R1 = 0.0359 for 2658 $F_o > 4\text{sig}(F_o)$

wR2 = 0.0917 for all 2773 data

Goof = 1.094 for all 2773 data

Flack x parameter = -0.0133 with esd 0.0660

Average s.u. on all C-C bonds: 0.0023 Å

C₁₁H₁₀O₂S: Three Strategies, data statistics

Bruker Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.82 - 0.72	512	512	100.0	5.42	25.3	9.14	0.1349	0.0918
Inf - 0.72	1653	1653	100.0	6.81	173.1	30.19	0.0313	0.0185

Small Combo

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.82 - 0.72	501	507	98.8	4.64	25.9	9.28	0.1250	0.0951
Inf - 0.72	1638	1644	99.6	6.05	175.1	30.59	0.0293	0.0188

MIT Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.82 - 0.72	509	509	100.0	10.18	25.2	14.74	0.1310	0.0570
Inf - 0.72	1648	1648	100.0	13.55	173.9	43.40	0.0310	0.0128

C₁₁ H₁₀ O₂ S: Three Strategies, refinement statistics

Bruker Standard

R1	wR2	Goof	s.u.
0.0403	0.1015	1.068	0.0026 Å

Small Combo

R1	wR2	Goof	s.u.
0.0402	0.1010	1.065	0.0026 Å

MIT Standard

R1	wR2	Goof	s.u.
0.0359	0.0917	1.093	0.0023 Å

C₁₁₄ H₁₅₉ Cl Cr N₄ in Cc: Three Strategies, Data

Bruker Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	4672	4689	99.6	2.99	19.9	5.95	0.1515	0.1487
Inf - 0.72	15819	15838	99.9	3.95	75.7	15.97	0.0489	0.0425

Small Combo

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	4491	4700	95.6	2.54	20.3	5.64	0.1510	0.1676
Inf - 0.72	15643	15887	98.5	3.47	77.6	15.75	0.0446	0.0459

MIT Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	4694	4700	99.9	5.13	19.5	8.20	0.1624	0.1061
Inf - 0.72	15872	15880	99.9	7.15	75.8	22.21	0.0484	0.0302

C₁₁₄H₁₅₉ClCrN₄ in Cc: Three Strategies, Refinement

Bruker Standard

R1	wR2	Goof	s.u.
0.0502	0.1224	1.016	0.0029 Å

Small Combo

R1	wR2	Goof	s.u.
0.0493	0.1215	1.024	0.0030 Å

MIT Standard

R1	wR2	Goof	s.u.
0.0451	0.1128	1.026	0.0025 Å

C₃₄H₄₆F₁₂N₂O₄W₂ in P2₁/c: Three Strategies, Data

Bruker Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	3858	3862	99.9	2.92	84.6	4.45	0.1674	0.1899
Inf - 0.72	13089	13104	99.9	3.84	178.6	9.66	0.0822	0.0790

Small Combo

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	3840	3840	100.0	2.49	93.2	4.33	0.1475	0.2038
Inf - 0.72	13043	13053	99.9	3.38	196.1	9.84	0.0720	0.0819

MIT Standard

Resolution	#Data	#Theory	%Complete	MoO	Mean I	Mean I/s	R(int)	Rsigma
0.81 - 0.72	3859	3859	100.0	5.41	86.1	6.79	0.1700	0.1223
Inf - 0.72	13085	13093	99.9	7.52	185.1	14.76	0.0790	0.0507

C₃₄H₄₆F₁₂N₂O₄W₂ in *P*2₁/*c*: Three Strategies, Refinement

Bruker Standard

R1	wR2	Goof	s.u.	Peak / hole
0.0499	0.1312	1.056	0.0103 Å	5.59 / -1.74

Small Combo

R1	wR2	Goof	s.u.	Peak / hole
0.0502	0.1305	1.050	0.0104 Å	5.00 / -2.36

MIT Standard

R1	wR2	Goof	s.u.	Peak / hole
0.0455	0.1269	1.138	0.0098 Å	3.94 / -3.03

Overview

	Compl.	MoO	R_{sigma}	R1	wR2	s. u.	Strat.
Ylid	100	6.8	0.0185	0.0403	0.1068	0.0026	3 ω
Ylid	100	13.6	0.0128	0.0359	0.0917	0.0023	MIT
CrCl	99.9	4.0	0.0425	0.0502	0.1224	0.0029	3 ω
CrCl	99.9	7.2	0.0302	0.0451	0.1128	0.0025	MIT
W ₂	99.9	4.8	0.0790	0.0499	0.1312	0.0103	3 ω
W ₂	99.9	7.5	0.0507	0.0455	0.1269	0.0098	MIT

MoO is no Schmu

The take home message of this talk is:

Collect as many frames as you can.

Combine ω - and ϕ -scans.

High MoO is good for you.

MIT Standard Strategy

Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
0	001	0.00	0.00	0.00	54.74	3	0.500	1	20.00
1	001	-30.00	-20.00	0.00	54.74	2	-0.500	400	20.00
0	002	0.00	0.00	0.00	54.74	3	0.500	1	20.00
2	001	-30.00	-30.00	0.00	54.74	3	0.450	800	20.00
0	003	0.00	0.00	0.00	54.74	3	0.500	1	20.00
3	001	-30.00	-20.00	90.00	54.74	2	-0.500	400	20.00
0	004	0.00	0.00	0.00	54.74	3	0.500	1	20.00
4	001	-30.00	-20.00	180.00	54.74	2	-0.500	400	20.00
0	005	0.00	0.00	0.00	54.74	3	0.500	1	20.00
5	001	-30.00	210.00	0.00	54.74	3	0.450	800	20.00
0	006	0.00	0.00	0.00	54.74	3	0.500	1	20.00

Three 200° omega-scans and two 360° phi-scans that's plenty for any situation.

Two 200° omega-scans and one 360° phi-scan is enough for *mmm* and higher.

Figure by MIT OCW.

The Zero-Frames

Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
0	001	0.00	0.00	0.00	54.74	3	0.500	1	20.00
1	001	-30.00	-20.00	0.00	54.74	2	-0.500	400	20.00
0	002	0.00	0.00	0.00	54.74	3	0.500	1	20.00
2	001	-30.00	-30.00	0.00	54.74	3	0.450	800	20.00
0	003	0.00	0.00	0.00	54.74	3	0.500	1	20.00
3	001	-30.00	-20.00	90.00	54.74	2	-0.500	400	20.00
0	004	0.00	0.00	0.00	54.74	3	0.500	1	20.00
4	001	-30.00	-20.00	180.00	54.74	2	-0.500	400	20.00
0	005	0.00	0.00	0.00	54.74	3	0.500	1	20.00
5	001	-30.00	210.00	0.00	54.74	3	0.450	800	20.00
0	006	0.00	0.00	0.00	54.74	3	0.500	1	20.00

Figure by MIT OCW.

The Zero-Frames

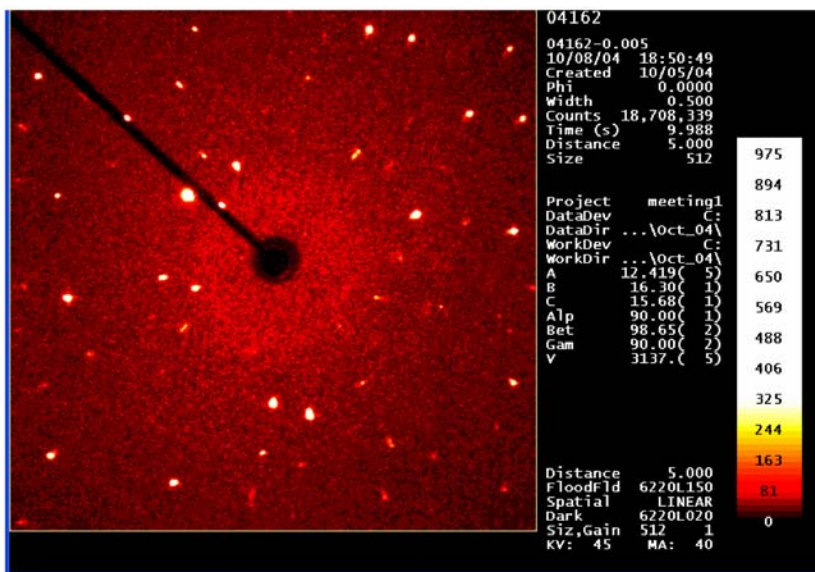


Figure by MIT OCW.

Data Reduction

- Using SAINT
- Using SADABS
- File Name Confusion

SAINT Now and Then

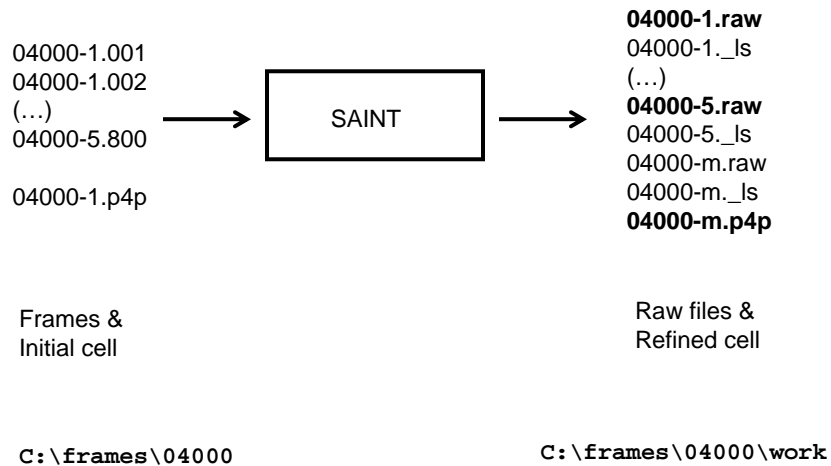
There was a time before GUIs where SAINT did not run on PCs. One had to call SAINT from the command line. Like so:

```
saint /l1:1023 /l2:768 /k1:1 /k2:3 /bg:2 /h:2
```

This translates to: run SAINT, treat everything as constant during integration, allow everything but crystal translations and goniometer zeros to be refined in post-refinement, assume triclinic Laue symmetry during integration, assume monoclinic Laue symmetry in post-refinement, assume low background noise level, assume small allocated memory.

There were more qualifiers for special cases (like very weak diffraction, etc.).

SAINT: Data Flow



SADABS: Default Magic (The Wizzard of Göttingen)

Absorption (and other) corrections from equivalents

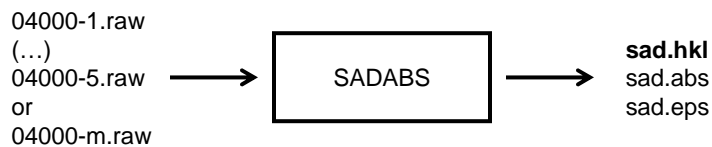
- Always use SADABS
- Generally SADABS comes right after SAINT
- You can use all defaults except for one: the LAUE symmetry
- Look at the SADABS output (sad.eps)
- Use the corrected data (sad.hkl) to go into XPREP, not the 04000-m.raw

SADABS: The Laue Symmetry

The answer to the third question in SADABS is critical. You must give the same Laue symmetry and setting you used in SAINT, even if you happen to know that this is not the true Laue symmetry.

What you should do in such a case is go back to SMART find an orientation matrix in the correct Laue symmetry, repeat the integration in SAINT with this matrix and then run SADABS.

SADABS: Data Flow

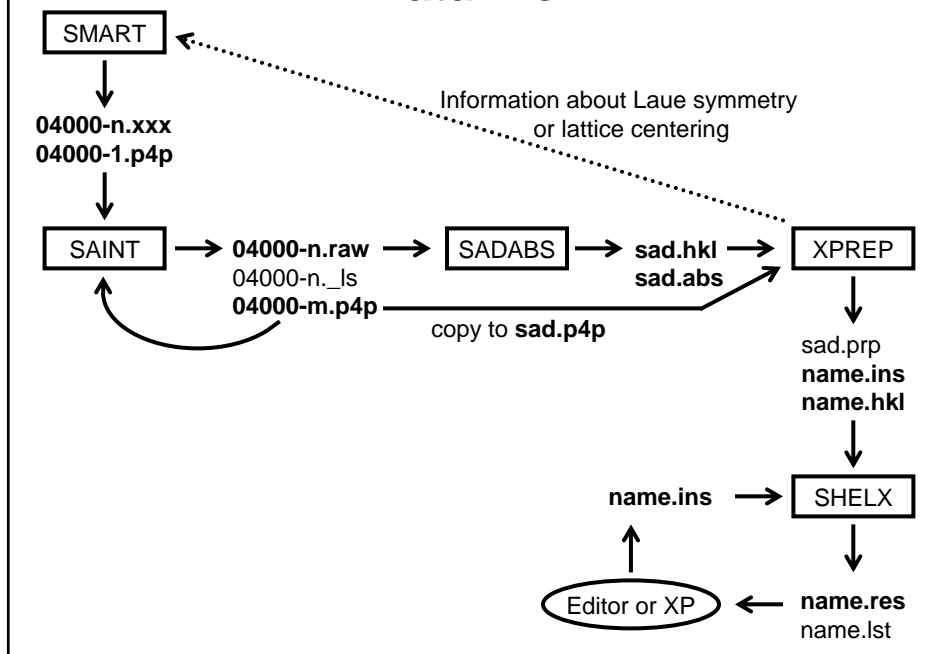


SADABS: Not Very Graphical

Open SADABS either from the SAINTPLUS interface by clicking on SADABS (will open a DOS window) or directly from a DOS-prompt.

And then just follow the Yellow Brick Road.

Data Flow



Playing with the Data

Things to do in XPREP

- Higher metric symmetry search
- Space group determination
- Judging the quality of the data
- High resolution cutoff
- Anomalous scattering
- Merohedral twinning

Between SADABS and SHELXS

Search for higher metric symmetry

The metric symmetry is the symmetry of the crystal lattice without taking into account the reflex intensities

XPREP searches for twofold axes in real space (LePage algorithm). For each metric symmetry there is a typical pattern of twofold axes.

E.g. 3 twofolds perpendicular to one another: orthorhombic

14 Bravais lattices (taking into account possible lattice centering).

The 14 Bravais Lattices

Triclinic:	$a \neq b \neq c, \alpha \neq \beta \neq \gamma \neq 90^\circ$	Lattice: P
Monoclinic:	$a \neq b \neq c, \alpha = \gamma = 90^\circ \neq \beta$	Lattice: P, C
Orthorhombic:	$a \neq b \neq c, \alpha = \beta = \gamma = 90^\circ$	Lattice: P, C, I, F
Tetragonal:	$a = b \neq c, \alpha = \beta = \gamma = 90^\circ$	Lattice: P, I
Trigonal/Hexagonal:	$a = b \neq c, \alpha = 90^\circ, \gamma = 120^\circ$	Lattice: P, R
Cubic:	$a = b = c, \alpha = \beta = \gamma = 90^\circ$	Lattice: P, I, F

Laue Symmetry

Laue symmetry is the symmetry in reciprocal space (taking into account the reflex intensities). Friedel's law is assumed to be true.

The Laue symmetry can be lower than the highest metric symmetry, but never higher.

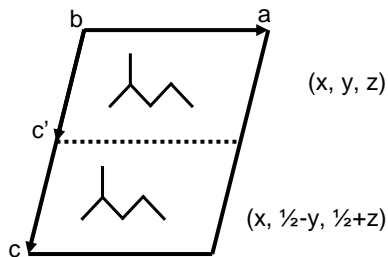
Twinning can simulate both too high or too low Laue symmetry.

Laue Symmetry

Crystal System	Laue Group	Point Group
Triclinic	-1	1, -1
Monoclinic	2/m	2, m, 2/m
Orthorhombic	mmm	222, mm2, mmm
Tetragonal	4/m	4, -4, 4/m
	4/mmm	422, 4mm, -42m, 4/mmm
Trigonal/ Rhombohedral	-3	3, -3
	-3/m	32, 3m, -3m
Hexagonal	6/m	6, -6, 6/m
	6/mmm	622, 6mm, -6m2, 6/mmm
Cubic	m3	23, m3
	m3m	432, -43m, m3m

Systematic Absences

Monoclinic cell, projection along b with c glide plane (e.g. Pc).



In this two 2D projection the structure is repeated at $c/2$. Thus, the unit cell seems to half the size: $c' = c/2$. This doubles the reciprocal cell accordingly: $c'^* = 2c^*$. Therefore, the reflections corresponding to this projection ($h, 0, l$) will be according to the larger reciprocal cell.

That means ($h, 0, l$) reflections with $l \neq 2n$ are not observed.

E²-1 Statistics

We measure intensities I

$I \longrightarrow F^2$ F: structure factors

Normalized structure factors E:

$E^2 = F^2 / \langle F^2 \rangle$ $\langle F^2 \rangle$: mean value for reflections at same resolution

$\langle E^2 \rangle = 1$

$\langle |E^2 - 1| \rangle = 0.736$ for non-centrosymmetric structures

0.968 for centrosymmetric structures

Heavy atoms on special positions and twinning tend to lower this value.

Pseudo translational symmetry tend to increase this value.

Maximum Resolution

While it is very important to use all reflections even the very weak ones, it does not make any sense to refine against noise.

If *all* reflections at a certain resolution and higher are very very weak, then it is appropriate to truncate the dataset accordingly.

What is very very weak?

$I/\sigma \leq 2$

$R_{\text{sym}} \geq 0.4$ and/or $R_{\text{int}} \geq 0.4$

It is very difficult to give hard numbers. Depends much on the individual case. SADABS output can help (plot of R_{int} v/s resolution).

Merge the Data in XPREP

1. Centrosymmetric space groups:
Merge all
2. Non-centrosymmetric space groups with no atom heavier than fluorine:
Merge all
3. Non-centrosymmetric space groups with some atoms heavier than silicon:
Merge symmetry equivalents but not Friedel opposites
4. Non-centrosymmetric space groups with some atoms heavier than fluorine but no atom heavier than Al:
Examine anomalous signal then decide whether 2. or 3.

With the latest version of XPREP: Do not merge XPPREP!

Merge the Data in XPREP

When you merge the data in XPREP, make sure that you do not lose the sad.prp file, as this is the only place where you can find the total number of observed reflections (before merging), which is needed to calculate the MoO, and the R_{int} for your dataset.

You will need these two numbers later for the .cif file.