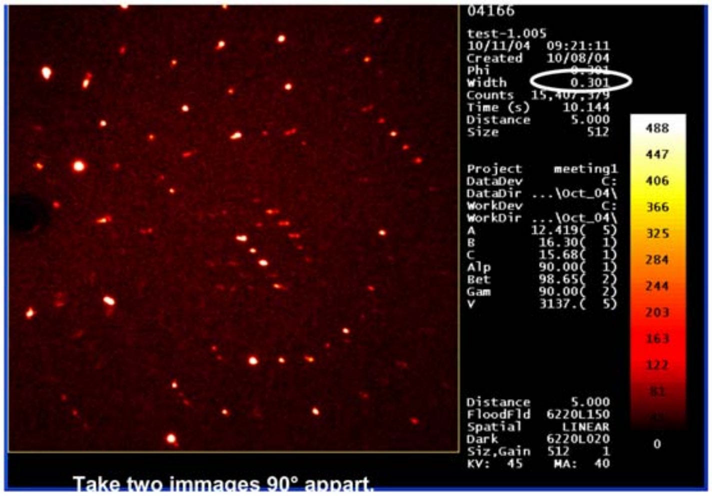
From Crystal to Diffraction Pattern

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

Judging the Quality of the X-tal

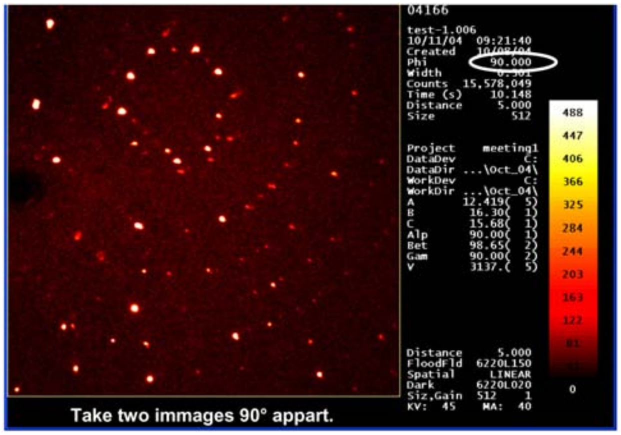


```
04166

test-1.005
10/11/04  09:21:11
Created   10/08/04
Phi          0.301
Width        0.301
Counts  15,407,379
Time (s)    10.144
Distance     5.000
Size           512

Project    meeting1
DataDev          C:
DataDir ...\Oct_04\
WorkDev          C:
WorkDir ...\Oct_04\
A       12.419(  5)
B        16.30(  1)
C        15.68(  1)
Alp      90.00(  1)
Bet      98.65(  2)
Gam      90.00(  2)
V        3137.(  5)

Distance     5.000
FloodFld  6220L150
Spatial     LINEAR
Dark      6220L020
Siz,Gain  512    1
KV:  45    MA:  40
```

Take two immages 90° appart.

Figure by MIT OpenCourseWare.

Judging the Quality of the X-tal

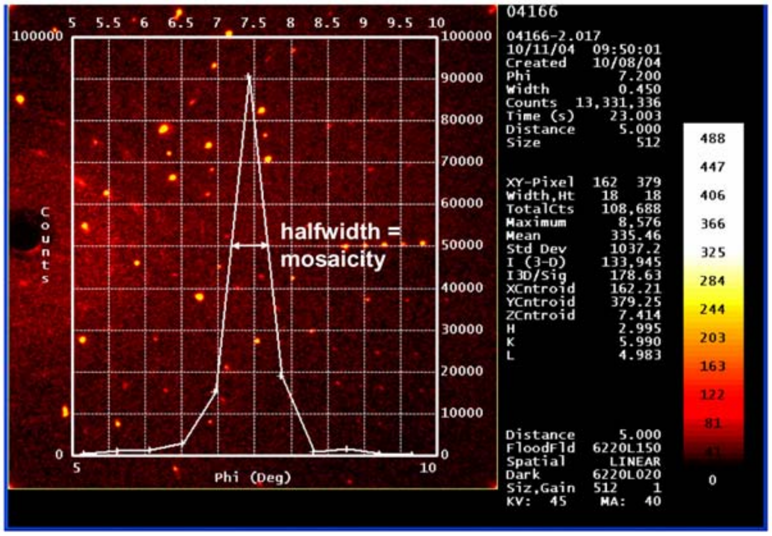


```
04166
test-1.006
10/11/04  09:21:40
Created    10/08/04
Phi          90.000
Width         0.301
Counts   15,578,049
Time (s)     10.148
Distance      5.000
Size            512

Project    meeting1
DataDev          C:
DataDir ...\Oct_04\
WorkDev          C:
WorkDir ...\Oct_04\
A         12.419(  5)
B          16.30(  1)
C          15.68(  1)
Alp        90.00(  1)
Bet        98.65(  2)
Gam        90.00(  2)
V          3137.(  5)

Distance      5.000
FloodFld   6220L150
Spatial      LINEAR
Dark       6220L020
Siz,Gain   512    1
KV:  45    MA:   40
```

Take two immages 90° appart.

Figure by MIT OpenCourseWare.

Judging the Quality of the X-tal

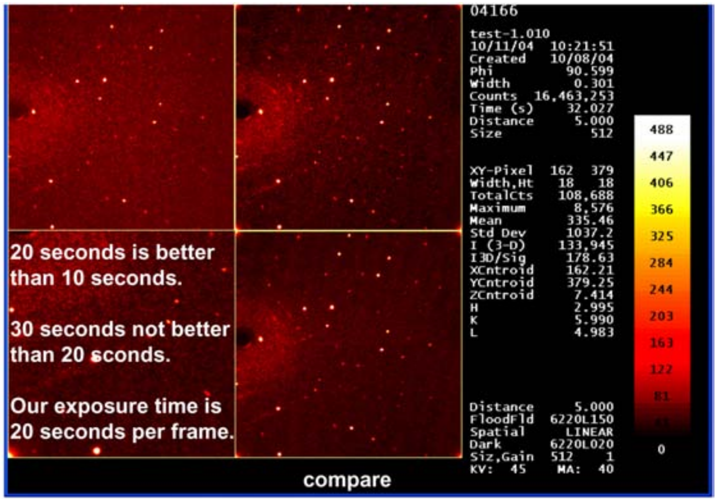


halfwidth = mosaicity

```
04166
04166-2.017
10/11/04  09:50:01
Created   10/08/04
Phi          7.200
Width        0.450
Counts  13,331,336
Time (s)    23.003
Distance     5.000
Size           512

XY-Pixel  162  379
Width,Ht   18   18
TotalCts   108,688
Maximum      8,576
Mean        335.46
Std Dev     1037.2
I (3-D)    133,945
I3D/Sig     178.63
XCntroid    162.21
YCntroid    379.25
ZCntroid     7.414
H            2.995
K            5.990
L            4.983

Distance     5.000
FloodFld  6220L150
Spatial     LINEAR
Dark      6220L020
Siz,Gain  512    1
KV:  45   MA:   40
```

Figure by MIT OpenCourseWare.

Determining the Exposure Time



20 seconds is better than 10 seconds.

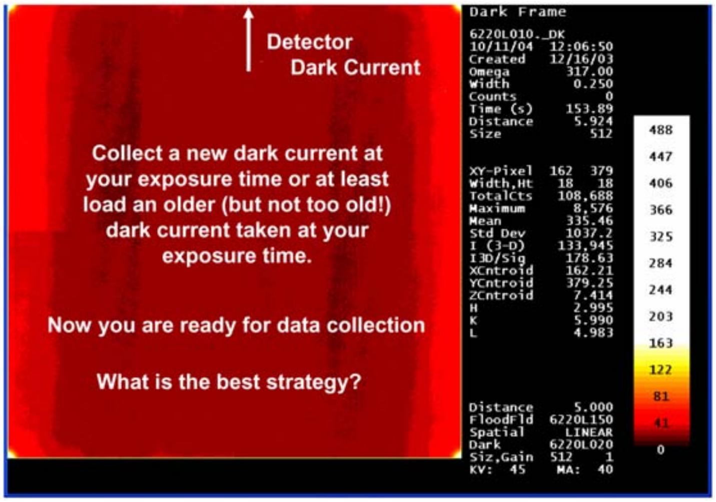
30 seconds not better than 20 sconds.

Our exposure time is 20 seconds per frame.

```
04166

test-1.010
10/11/04   10:21:51
Created    10/08/04
Phi          90.599
Width         0.301
Counts   16,463,253
Time (s)     32.027
Distance      5.000
Size            512


XY-Pixel   162  379
Width,Ht    18   18
TotalCts    108,688
Maximum       8,576
Mean         335.46
Std Dev      1037.2
I (3-D)     133,945
I3D/Sig      178.63
XCntroid     162.21
YCntroid     379.25
ZCntroid      7.414
H             2.995
K             5.990
L             4.983




Distance      5.000
FloodFld   6220L150
Spatial      LINEAR
Dark       6220L020
Siz,Gain   512    1
KV:  45     MA:  40
```

Figure by MIT OpenCourseWare.

Collect Dark Current



Detector Dark Current

Collect a new dark current at your exposure time or at least load an older (but not too old!) dark current taken at your exposure time.

Now you are ready for data collection

What is the best strategy?

```
Dark Frame

6220L010._DK
10/11/04   12:06:50
Created    12/16/03
Omega        317.00
Width         0.250
Counts            0
Time (s)     153.89
Distance      5.924
Size            512


XY-Pixel   162  379
Width,Ht    18   18
TotalCts    108,688
Maximum       8,576
Mean         335.46
Std Dev      1037.2
I (3-D)     133,945
I3D/Sig      178.63
XCntroid     162.21
YCntroid     379.25
ZCntroid      7.414
H             2.995
K             5.990
L             4.983




Distance      5.000
FloodFld   6220L150
Spatial      LINEAR
Dark       6220L020
Siz,Gain   512    1
KV:  45     MA:  40
```

Figure by MIT OpenCourseWare.

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, ϕ = 0°, 90° 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”.

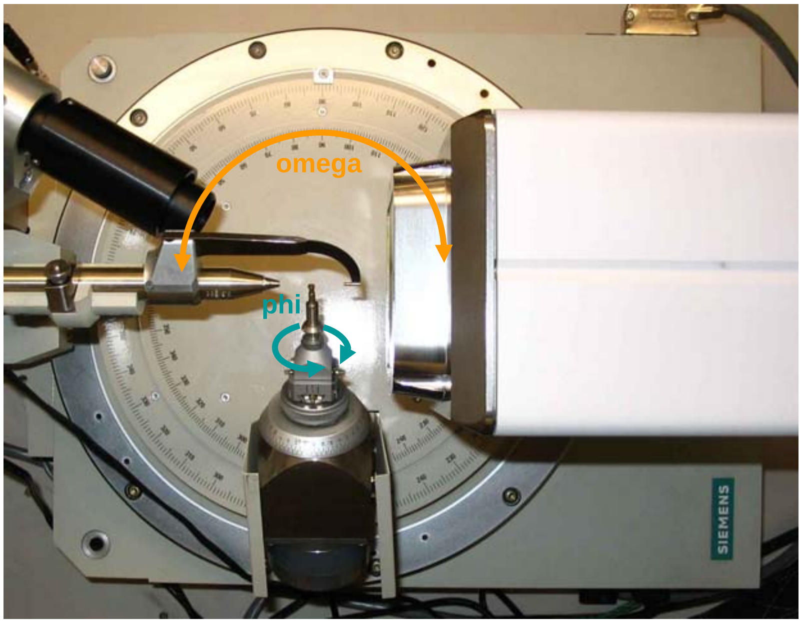
MoO is no Schmu?

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

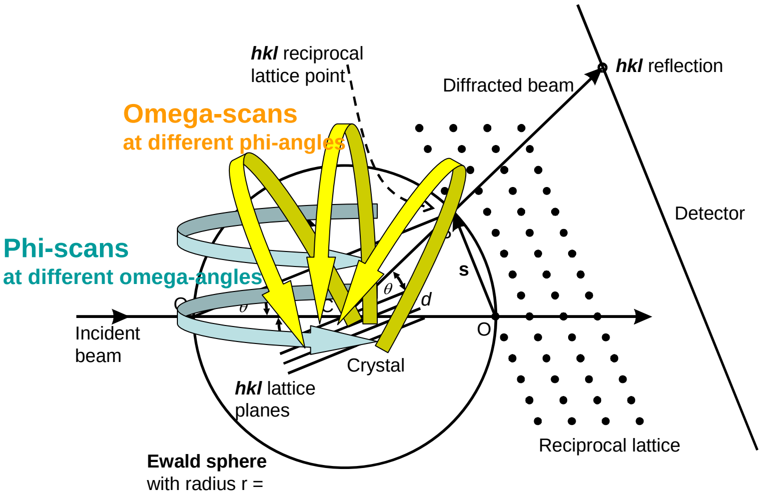
Try to distribute the frames all over the reciprocal space, collecting the same reflections from different Ψ-angles. This will give you the same reflections together on the same frame with different other reflections, which helps with semi-empirical absorption correction.

How to do that?

The Platform Geometry



phi- and omega-scans are orthogonal

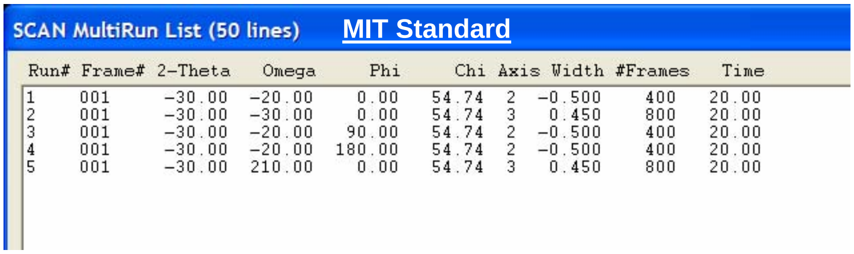

Omega-scans
at different phi-angles

Phi-scans
at different omega-angles

...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 Fo > 4sig(Fo)
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 Fo > 4sig(Fo)
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 Fo > 4sig(Fo)
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
```

C_{11} H_{10} O_2 S: 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
```

$C_{11}H_{10}O_2S$: 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
```

$C_{114}H_{159}ClCrN_4$ 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
```

$C_{114} H_{159}$ Cl Cr N_4 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
```

$C_{34}H_{46}F_{12}N_2O_4W_2$ in $P2_1/c$: 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
```

$C_{34}H_{46}F_{12}N_2O_4W_2$ in $P2_1/c$: 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}	*R*1	*wR*2	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

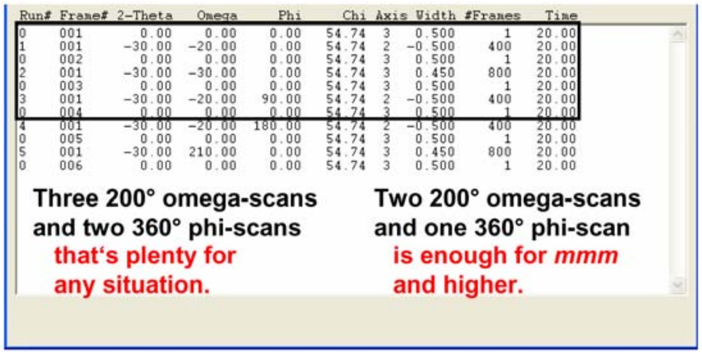
The take home message of this talk is:

Collect as many frames as you can.

Combine ω- and ϕ-scans.

High MoO is good for you.

MIT Standard Strategy



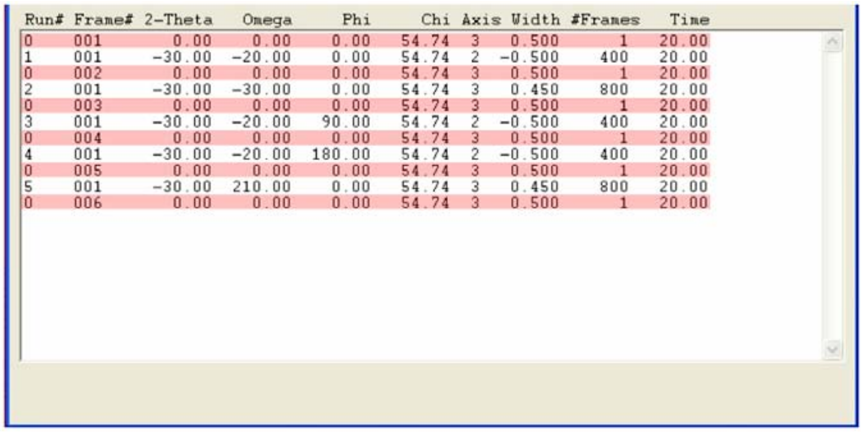
```
Run# Frame# 2-Theta   Omega     Phi     Chi Axis Width #Frames   Time
0    001      0.00     0.00     0.00   54.74  3   0.500      1   20.00
1    001    -30.00   -20.00     0.00   54.74  2  -0.500    400   20.00
0    002      0.00     0.00     0.00   54.74  3   0.500      1   20.00
2    001    -30.00   -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 OpenCourseWare.

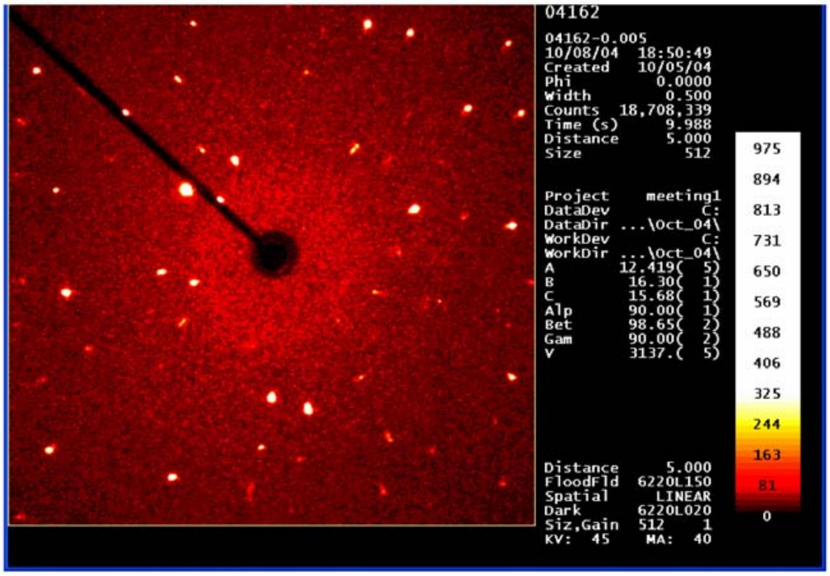
The Zero-Frames



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 OpenCourseWare.

The Zero-Frames



```
04162

04162-0.005
10/08/04   18:50:49
Created    10/05/04
Phi          0.0000
Width         0.500
Counts   18,708,339
Time (s)      9.988
Distance      5.000
Size            512

Project    meeting1
DataDev          C:
DataDir ...\Oct_04\
WorkDev          C:
WorkDir ...\Oct_04\
A        12.419(  5)
B         16.30(  1)
C         15.68(  1)
Alp       90.00(  1)
Bet       98.65(  2)
Gam       90.00(  2)
V         3137.(  5)

Distance      5.000
FloodFld   6220L150
Spatial      LINEAR
Dark       6220L020
Siz,Gain   512    1
KV:   45   MA:   40
```

Figure by MIT OpenCourseWare.

Data Reduction

- Using SAINT
- Using SADABS
- File Name Confusion

SAINT Now and Then

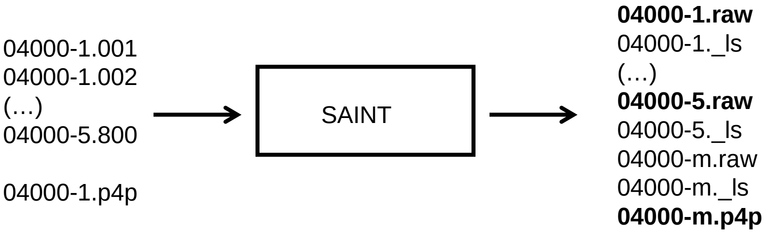
There was a time before GUIs where SAINT did not run on PCs. One had to call SAINT from the command line.
Like so:

```
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 monocinic 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



04000-1.001
04000-1.002
(…)
04000-5.800

04000-1.p4p

SAINT

04000-1.raw
04000-1._ls
(…)
04000-5.raw
04000-5._ls
04000-m.raw
04000-m._ls
04000-m.p4p

Frames &
Initial cell

Raw files &
Refined cell

`C:\frames\04000`

`C:\frames\04000\work`

SADABS: Default Magic

(The Wizzard of Göttingen)

Absorption (and other) corrections from equivalents

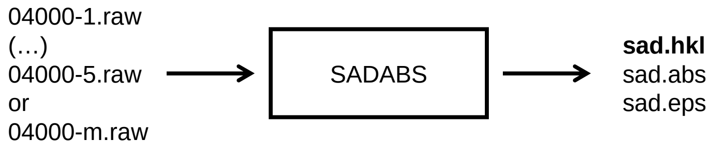
- Always use SADABS
- Generally SADABS comes right after SAINT
- You can use all defaults except for one: the LAUE symmetry
- Look at the SADABS output (sad.eps)
- Use the corrected data (sad.hkl) to go into XPREP, not the 04000-m.raw

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



04000-1.raw
(…)
04000-5.raw
or
04000-m.raw

SADABS

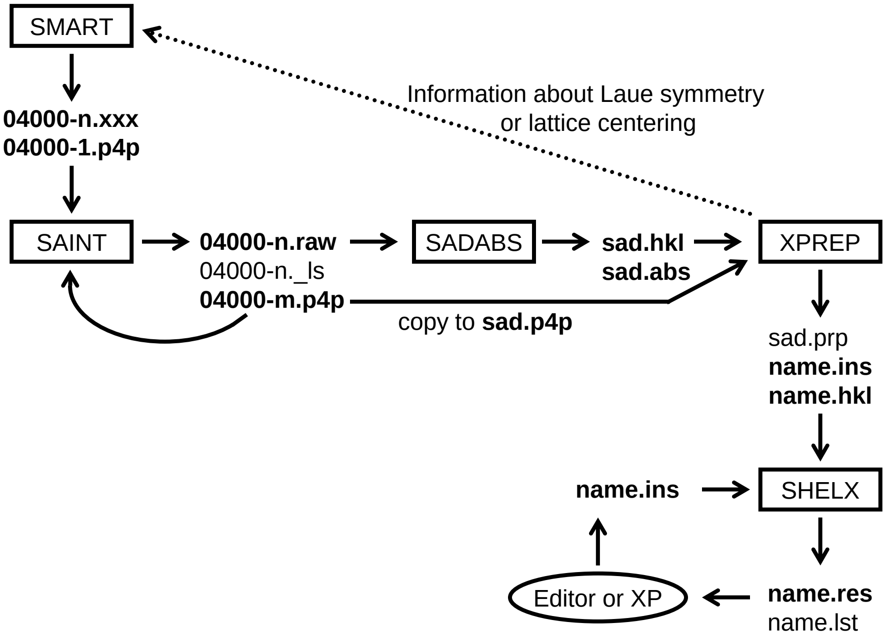
sad.hkl
sad.abs
sad.eps

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


SMART

04000-n.xxx
04000-1.p4p

Information about Laue symmetry
or lattice centering

SAINT

04000-n.raw
04000-n._ls
04000-m.p4p

SADABS

sad.hkl
sad.abs

copy to **sad.p4p**

XPREP

sad.prp
name.ins
name.hkl

name.ins

SHELX

Editor or XP

name.res
name.lst

Playing with the Data

Things to do in XPREP

- Higher metric symmery search
- Space grup 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:	$a \neq b \neq c, \alpha \neq \beta \neq \gamma \neq 90°$	Lattice: P
Monoclinic:	$a \neq b \neq c, \alpha = \gamma = 90° \neq \beta$	Lattice: P, C
Orthorhombic:	$a \neq b \neq c, \alpha = \beta = \gamma = 90°$	Lattice: P, C, I, F
Tetragonal:	$a = b \neq c, \alpha = \beta = \gamma = 90°$	Lattice: P, I
Trigonal/Hexagonal:	$a = b \neq c, \alpha = 90°, \gamma = 120°$	Lattice: P, R
Cubic:	$a = b = c, \alpha = \beta = \gamma = 90°$	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.

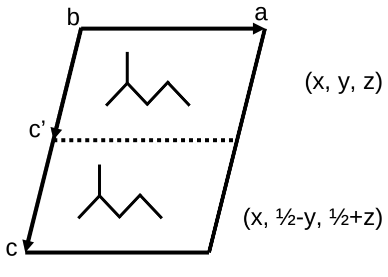
Twinning can simulate both too high or too low Laue symmetry.

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. P*c).



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: $c^{*\prime} = 2c^*$. 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.

E^2-1 Statistics

We measure intensities I
$I \longrightarrow F^2$ F: structure factors

Normalized structure factors E:
$E^2 = F^2/\langle F^2 \rangle$ <F2>: 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 week ones, it does not make any sense to refine against noise.

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

What is very very week?
$I/\sigma \leq 2$
$R_{sym} \geq 0.4$ and/or $R_{int} \geq 0.4$
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.

5.067 Crystal Structure Refinement
Fall 2009