# Data Reduction
# SMART

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version 1.0.0

# What to know before you begin

Name :_____Date_____

Office number _____ Telephone _____ Fax _____ e-mail _____

Principle Investigator (Advisor) _____ Dept. _____

Account to be billed _____ to _____ for $60.00

Original Sample Number _____Location _____

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✓ Chemical Formula **$C_{12}H_{22}O_{11}$** Formula weight **342.3 g/mole**

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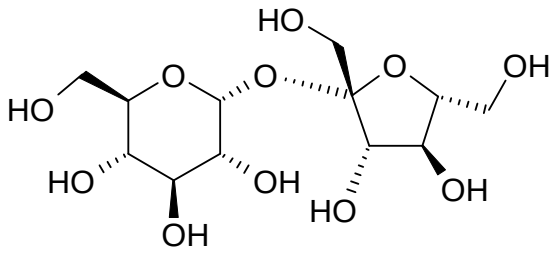
Density _____?_____ Solvents used ____$H_2O$_____ Sensitivity _____

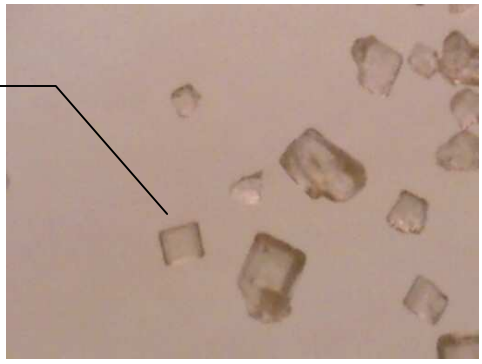
Crystal color _____Colorless_____ Is the sample chiral or racemic ? _***chiral (D)***___

Draw Structure (label all chiral centers).



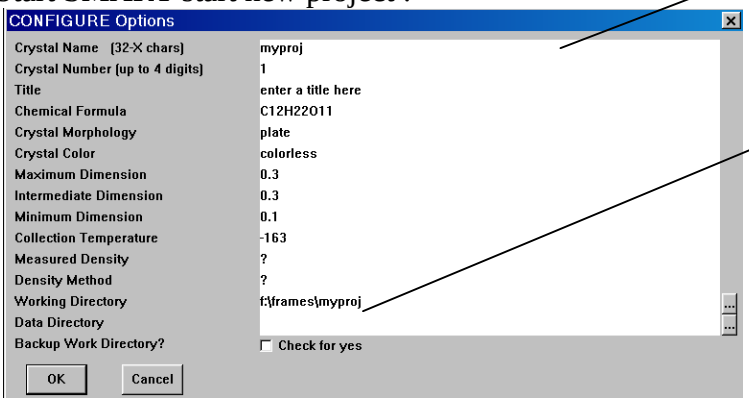

Choosing the crystal

Clear
Sharp edges
Flat faces



# Screening the Crystal

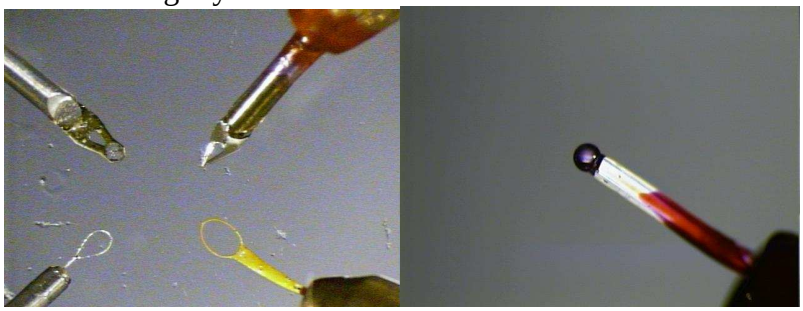
- Start SMART start new project :



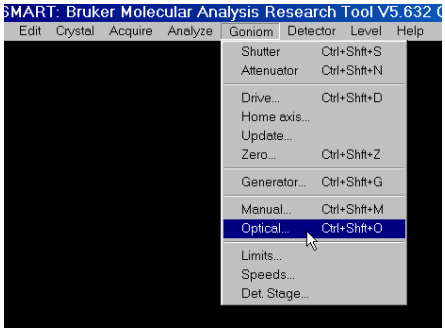

Input the known information

For working directory input f:\frames\myproj change f: if needed use your project name

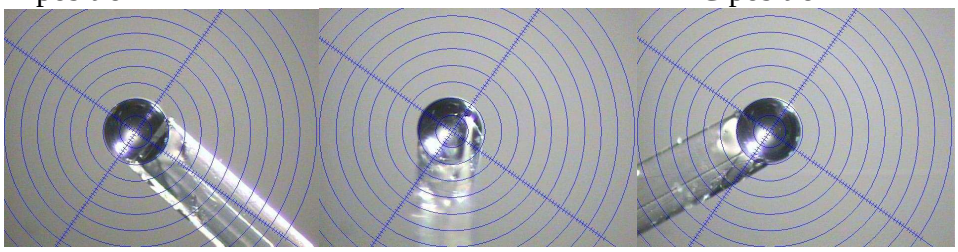
- Select SMALL MOLECULE when asked
- Mounting crystal



Start VIDEO point to File/NEW and GRAB Point to Goniom and Optical. Goto the external control. Select B and push AXIS print. Position the crystal as shown below. Always come from below. Using the "wrench" adjust the height of the crystal so that the crystal is near the crosshairs. Adjust the seldge below the height and adjust the X axis. Punch A and adjust Y axis. Punch C and check the height.



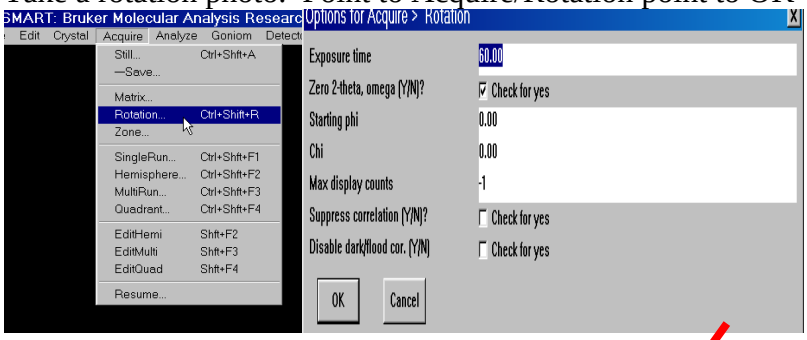

A position

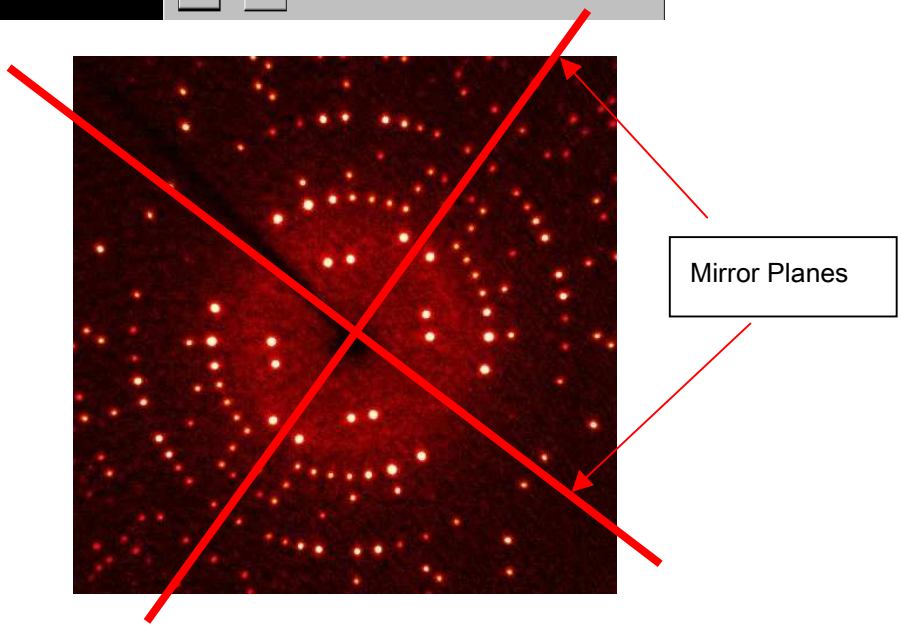


C position

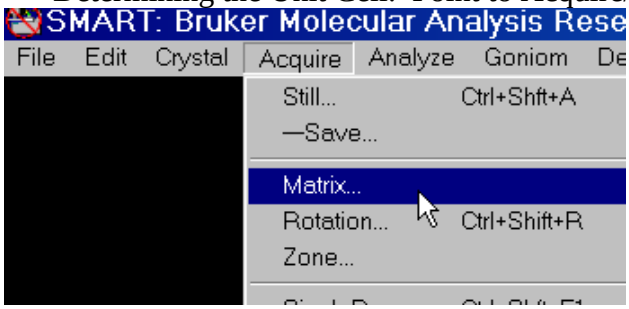
When finished punch the "esc" key on the keyboard.

- Rotation Photo
  Take a rotation photo. Point to Acquire/Rotation point to OK

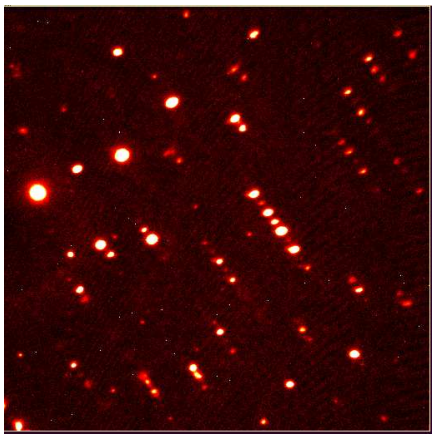


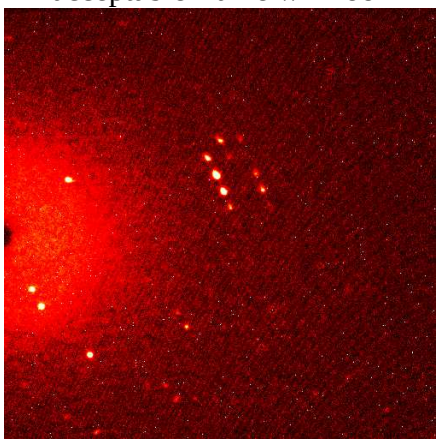

- Determining the Unit Cell. Point to Acquire/Matrix



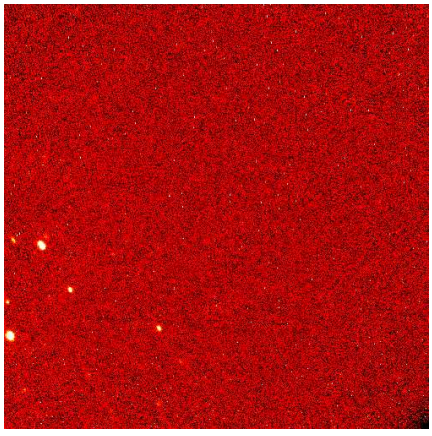
Good frame will look like this



An acceptable frame will look like this



A bad frame will look like this



- Unit cell

**good cell**

Lattice parameters & Standard deviations:

| | | | | | | |
|---|---|---|---|---|---|---|
| 18.0820 | 18.6483 | 18.6871 | 90.050 | 90.054 | 89.958 | 6301.27 |
| 0.0052 | 0.0055 | 0.0082 | 0.029 | 0.034 | 0.023 | 3.94 |

Standard deviations corrected for GOF:

| | | | | | | |
|---|---|---|---|---|---|---|
| 0.0039 | 0.0041 | 0.0061 | 0.021 | 0.025 | 0.017 | 2.94 |

Histograms:

| | | .00 | .05 | .10 | .15 | .20 | .25 | .30 | .35 | .40 | .45 | +Inf |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | 57 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | |
| K | 57 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | |
| L | 57 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | |
| Omega | 55 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | |

**acceptable cell**

Lattice parameters & Standard deviations:

| | | | | | | |
|---|---|---|---|---|---|---|
| 11.2914 | 11.1571 | 15.0720 | 89.760 | 109.387 | 90.316 | 1791.07 |
| 0.0046 | 0.0044 | 0.0055 | 0.010 | 0.011 | 0.010 | 2.04 |

Standard deviations corrected for GOF:

| | | | | | | |
|---|---|---|---|---|---|---|
| 0.0132 | 0.0128 | 0.0159 | 0.029 | 0.033 | 0.030 | 5.89 |

Histograms:

| | | .00 | .05 | .10 | .15 | .20 | .25 | .30 | .35 | .40 | .45 | +Inf |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | 207 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| K | 207 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| L | 201 | 6 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| Omega | 87 | 70 | 28 | 17 | 2 | 1 | 1 | 1 | 0 | 0 | | |

**a bad cell**

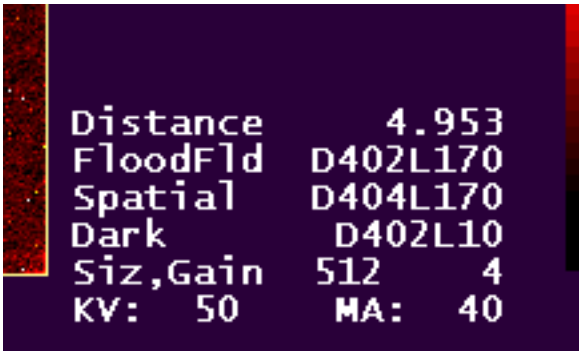
Lattice parameters & Standard deviations:

| | | | | | | |
|---|---|---|---|---|---|---|
| 16.9188 | 16.7568 | 17.0997 | 86.145 | 86.462 | 86.316 | 4818.86 |
| 0.6570 | 0.3144 | 0.2764 | 1.543 | 1.477 | 0.801 | 325.74 |

Standard deviations corrected for GOF:

| | | | | | | |
|---|---|---|---|---|---|---|
| 5.1056 | 2.4433 | 2.1478 | 11.988 | 11.475 | 6.226 | 2531.40 |

- Crystal will not index
  - Bad Crystal?
  - check the distance with the detector distance on the instrument.
    - if there not the same the cell will not index properly

  - 

- Is it the right stuff?
  Once you have the unit cell you can guess the Laue group and the Lattice. From this and the unit cell volume you should calculate the density.

*Z(1)* = non-centrosymmetric*: Z(-1)* = centrosymmetric

L = Lattice

| System | Laue | L | *Z(1)* | *Z(-1)* |
|---|---|---|---|---|
| Triclinic | -1 | P | 1 | 2 |
| Monoclinic | 2/m | P | 2 | 4 |
| Monoclinic | 2/m | C | 4 | 8 |
| Orthorhombic | mmm | P | 4 | 8 |
| Orthorhombic | mmm | C | 8 | 16 |
| Orthorhombic | mmm | A | 8 | 16 |
| Orthorhombic | mmm | I | 8 | 16 |
| Orthorhombic | mmm | F | 16 | 32 |
| Tetragonal | 4/m | P | 4 | 8 |
| Tetragonal | 4/m | I | 8 | 16 |
| Tetragonal | 4/mmm | P | 8 | 16 |
| Tetragonal | 4/mmm | I | 16 | 32 |
| Trigonal | -3 | P | 3 | 6 |
| Trigonal | -3 | R | 9 | 18 |
| Trigonal | -3m | P | 6 | 12 |
| Trigonal | -3m | R | 18 | 36 |
| Hexagonal | 6/m | P | 6 | 12 |
| Hexagonal | 6/mmm | P | 12 | 24 |
| Cubic | m-3 | P | 12 | 24 |
| Cubic | m-3 | I | 24 | 48 |
| Cubic | m-3 | F | 48 | 96 |
| Cubic | m-3m | P | 24 | 48 |
| Cubic | m-3m | I | 48 | 96 |
| Cubic | m-3m | F | 96 | 192 |

$$Density = \frac{FW \times Z}{0.6023 \times V}$$

| Type | Density Limits g/ml |
|---|---|
| C-H | 1.1 – 1.3 |
| + N | 1.1 – 1.4 |
| + O | 1.2 – 1.6 |
| + S | 1.2 – 1.7 |
| 1st row TR | 1.4 – 1.9 |
| + Br | 1.4 – 2.0 |
| 2nd row TR | 1.4 – 2.1 |
| + I | 1.5 – 2.3 |
| 3rd row TR | 1.5 – 2.4 |
| +Pb | 1.6 – 2.9 |

For example our sugar molecule with a formula weight of 342 is monoclinic Laue group of 2/m P. The molecule is chiral so the space group will be non-centrosymmetric and *Z* =2. The cell volume is 715 which affords a density of 1.59 g/ml. The density fits the range shown in the table.

Data Collection

point to Acquire/EditMulti

Generic Data Collection Strategy

| # | 2Theta | Omega | Phi | Chi | Axis | Range | # | Time |
|---|---|---|---|---|---|---|---|---|
| 1 | -28 | -28 | 0 | 54.9 | 2 | -0.3 | 600 | 20 |
| 2 | -28 | -28 | 90 | 54.9 | 2 | -0.3 | 600 | 20 |
| 3 | -28 | -28 | 180 | 54.9 | 2 | -0.3 | 400 | 20 |
| 4 | -28 | -28 | 270 | 54.9 | 2 | -0.3 | 400 | 20 |

About a 12 hour data collection

How fast can you go

Collect a few frames. Acquire/Multirun

If you do not see spots near the right hand edge of the frame then slow the data collection to 30 secs and repeat the collection.

Load the correct dark current for your data collection time

point to Detector/Dark Current and Load

the look for *****020.*** for 20 secs ***30.*** for 30 etc.

Start Data Collection

point to Acquire/MultiRun

set the JOB name (normally just DATA) and point to OK.