User's Guide

Center for Crystallographic Research

Chemistry Facility

Michigan State University

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Dr. Richard J. Staples
Manager
418A
353-1074

Small Molecule Crystallographic Guide
Material Characterization Guide

Updated 2-2-2025

Small Molecule Crystallographic Laboratory

Services Provided

The laboratory provides crystal and molecular structure determination from single crystal samples excluding large macromolecules. Services include data collection, structure solution and refinement.

User Input

The user should provide solid crystalline samples for data collection and structure determination. See attached paper by Peter Jones for crystal growing and quality details.

Sample specifications:

Type	: single crystal solid
Molecular size	: up to 400 non-hydrogen atoms (larger molecules see manager)
Smallest dimension	: 0.02 mm on side
Clarity	: non-opaque

Results

Upon completion of the structure determination users are provided:

(1) Complete details of the data collection and structure determination in publication format.

(2) Comprehensive graphics output including publication quality plots and figures.

(3) Complete tables of bond lengths, bond angles, atomic coordinates, Thermal parameters, Torsion angles, Metric parameters and observed and calculated structure factors all in publication format.

(4) An ASCII file in the CIF format will also be available for electronic submission of the data.

(5) In many cases the data is submitted to the CCDC to receive a CCDC number used in publication of the structure.

General Policy and Services

The Laboratory is maintained by Michigan State University Department of Chemistry. The facility is open to all students, faculty, and staff of the University. Users who wish to participate in the structure determination are free to do so provided that they have undergone the appropriate training courses and/or course work.

Four levels of Service have been established.

Level one	(full)	: Data Collection, Structure Determination
Level two	(data only)	: Data Collection only
Level three		: Structure Determination only (prior approval necessary)

The user can choose any level of service.

All users must submitted a submission form from the web site prior to trying to run a crystal and MUST HAVE CRYSTAL IN hand.

Synergy S Dual Diffractometer Reservation Rules

There is an online waitlist for this instrument. Once you submit a form you can add your name to the waitlist or wait for the manager to add it. The manager can be listed as often as necessary. The manager will use the instrument (generally) during normal business hours and users can use the instrument when their turn comes up. Sign the login sheet (paper) to confirm you are using the instrument and provide when the data set is done. Completion of the data set gives authorization for the next user to use the diffractometer. REMEMBER to measure the crystals prior to data collection or collect a video stream for face indexing. Failure to be courteous and polite to other users will result in loss of privileges.

Weekends: It has been found that the weekends is a first come first serve basis. The waitlist should be used, when possible, but it is more important to have the instrument in use.

It is anticipated that if you are told you can use the instrument in the morning, you will sign the log by 9:30 A.M and get started with the experiment.

X-ray Service Fees

The Flat Fee

Universities, colleges and academic institutions.

A flat fee will be charged to package services users. The package service includes: Crystal examination, unit cell determination, data collection, data reduction, structure solution, structure refinement, graphics interaction, and final written summary. **The flat fee will include low temperature charges**.

Fees

Data collection, structure solution and refinement	:	$60
Unit Cell Determination	:	No Charge
Data Sets (data collection only)	:	60.00
Structure Solution, Refinement and Graphics Interaction	:	100.00
Instrument Time non-standard experiments	:	10.00 / hr

These facilities fees are currently based on the estimated cost for laboratory operation, supplies and low temperature operation and can change over time.

This facility strongly encourages participation by the users in the structure determination, refinement and graphics interaction of molecular structure determinations. Help, advice and software can be provided for this task through software provided for your PC. For information please contact the manager. Generally, no additional fees are necessary for this service.

Industrial users.

The flat fee service is as above; however, cost of these services varies. Inquires are to be directed to the laboratory manager. See supplement description of services.

The Submission Form

Users are required to complete the submission form (https://www2.chemistry.msu.edu/Facilities/Crystallography/) found on the Centers web site. Once complete, the user will be notified of their place on the waitlist, (they can add their name directly at the web site) or crystals can be dropped off to Room 434.

Instructions

Please provide all information on the form: name, date, major advisor and email address where asked. Please provide your **sample #** and **Account** to be charged. Provide all information no matter what level of service you are requesting. Effective 1-2018, all potential authors should also be listed on request form.

Please provide the chemical formula, formula weight. Draw the proposed structure and label all chiral centers and upload the file where indicated. Be sure to indicate which centers are known and which are to be determined. If the crystal contains a racemic mixture please indicate it as such. If the sample is sensitive to air, water or light please say so. It is also important to state what solvent(s) was used for crystallization techniques.

The data can be collected at variable temperatures and will routinely be collected at -100C. Please make a note if this is a problem.

Sample Submissions:

Crystal quality is probably the single most important factor in determining the final precision for a given x-ray structural investigation. A high precision structural result requires high-quality crystals. In general crystals for an x-ray study must be single and of the proper size and shape. The crystal should be 0.05 to 0.2 mm on an edge and as equal-dimensional as possible. Needle- or plate-shaped crystals can usually be done if they are at least 0.05 mm thick.

Users Guidelines

To become a user:

Those who wish to collect the data on their own crystalline samples need to meet the following guidelines. See website for current details.
https://www2.chemistry.msu.edu/Facilities/Crystallography/

1) YOU MUST HAVE DONE THE RADIATION SAFETY TRAINING ONLINE COURSE!!!!!!!
ORCBS Radiation Safety Training

2) YOU MUST HAVE AND WEAR A RADIATION RING WHEN YOU USE THESE INSTRUMENTS!!!!!!!!
ORCBS Dosimetry Page

3) YOU MUST BE REGISTERED TO RESERVE INSTRUMENT TIME
YOU MUST BE A TRAINED USER TO GAIN ACCESS TO THE ROOM (or under going training)
APPLICATION FORM ***You will need your MSU net ID login.***

Take Chem. 913, special topics course in X-ray Diffraction.

4) Must follow Rules established for the operation and organization of the laboratory.

5) The user becomes responsible to ensure that data collected is adequate. If you collect a data set, you will be billed. The user is responsible to back-up the data. *The manager will back-up the raw frames from the data collection. The manager has the ability to remove any and all files at all times from the computer in the laboratory.*

6) It is expected that if you collect data, solution and refinement will be done on your own or your group's computer.

Rules and Regulations for use of the Small Molecule Facilities

Your badge is required for the operation of the instrument!

1) The lab is potentially open from 9 to 5:00 every day the university is in session.

2) The use of the facility is for authorized users only. Only authorized personnel are allowed to be in the lab during off hours, i.e. no kids, or friends.

3) Keep the lab clean and organized.

4) Do not allow users to use your key to access the room. Those requiring access contact the manager.

5) Violation of the rules will mean loss of after hour use.

6) Authorization to use the X-ray Diffractometer after hours must come from the Manager and with the following rules.

   a) You must have been trained in the operation and safety of the diffractometer.

   b) You must follow the guidelines of the calendar reservation system to ensure no conflict of use or violation of calendar rules. Be courteous and polite to other users.

   c) Sign the logbook and note activity and problems. Signing in signifies that you are using, then once done or know when done put the time or make done in last column. Notify the next person on waitlist. (see rules, pg 3.)

   d) If your collection results in the completion of a structure that has already been determined and is in the CSD or other available data bases, your advisor will be billed. As users, you will be bill for all data collections.

   e) If you need to call the manager, call before 10:00 PM, unless it is an emergency. Calling to see if you can collect ahead of someone in the que is not an emergency and should be assumed you will need to wait.

   f) During heavy use periods the failure to show within a reasonable time will result in loss of time.

IT IS THE AIM OF THESE REGULATIONS TO PROVIDE THE BEST OVERALL OPERATION OF THE DIFFRACTION FACILITIES.

Rules regarding Submitting Structure request.

1) **You must have crystals!** No signing up in hopes for crystals.

2) You can submit as many crystals at one time as you may need to run, but you will be limited to 1 data set each time on the instrument dependent on instrument usage and time data set requires to run. Data sets less than 1 hour can be followed by another crystal.

3) There is a separate Waitlist to use the instrument, this list will provide the means of knowing who the next user of the instrument is.

2) You must be a registered user.

4) When you start to use the instrument, Log in the date, initials, group and crystal owners name (initials).

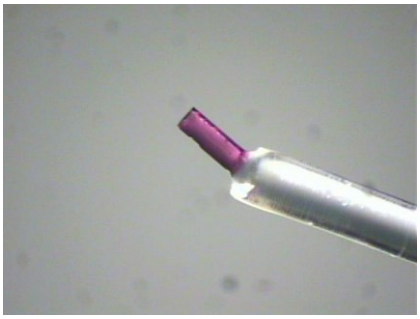
5) When you have finished, then place done, or if collecting data, Time instrument is done.

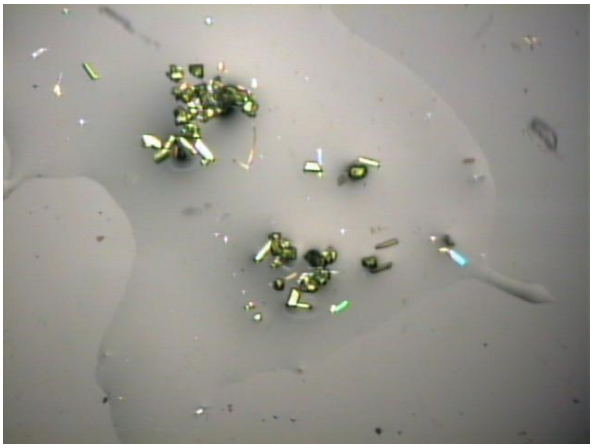
6) As a courtesy, if you evaluate your crystals, and they do not work out, Contact the Manager or You can use the waitlist and check you off of server and click the next person in line to use the instrument.

7) If you are next, and the person ahead of you has used the instrument and there is either a done, or filename, then check the instrument to see if it is in use collecting the data. Please be courteous and try and determine if the data set is fully completed before you take off someone else's crystal., you can take off the crystal (SAVE IT ON A Separate slide labeled) and begin your experiment. If in doubt, contact the manager.

9) Be Courteous and considerate of others work.

10) **KEEP THE INSTRUMENT AND MOUNTING AREA CLEAN AND ORGANIZED**.





The Center for Material Characterization

Services Provided

The laboratory provides access to a DaVicni D8 Powder diffractometer and software to analyze the resulting diffraction pattern. Services include data collection, and some help with interpretation.

User Input

The user should provide the material to be analyzed.

Sample specifications:

Type	: Most often powder
Size	: Powders should be ground and sieved for unity
Holder	: There is a wide range of holders available.

Results

(1) Complete details of the data collection

(2) An ASCII file that can be used in various programs to allow for analysis of the data.

General Policy and Services

The Laboratory is maintained by Michigan State University Department of Chemistry. The facility is open to all students, faculty and staff of the University. User's who wish to Run their own data may do so, provided that they have undergone the appropriate training courses and/or course work.

Two levels of Service have been established.

Level one	(data only)	: Data Collection only
Level two	(data only by service personnel)	: Data Collection Only

The user can choose any level of service.
Analysis of the data is not supplied, although you can ask manager for help on where to find answers.

X-ray Service Fees

The Hourly Fee

Universities, colleges and academic institutions.

An hourly fee will be charged based on the 1/4 hour time use of the instrument.

Fees

Internal Chemistry Department: $10 per hour instrument time
Internal Chemistry ran by XRD personnel: $32 per hour instrument time
MSU academic Account number: $10 per hour instrument time

MSU academic Account number run by XRD personnel: $32 per hour instrument time
Other academic: $75 per hour

The Time Block from 6 P.M to 8 A.M. is considered an over night run and billed as one 4 hour block. ($40)

Weekends will be billed as either per hour or as a block time, maximum charge on a given day will be $100

Added Costs
Use of Low or High temperature add $10 per hour
Liquid Nitrogen costs if beyond the $15/hour.
Capillaries supplied at costs.

Caution, abuse of reserving time and not using it will result in loss of privileges.

Industrial users.

Instrument Time you run:	$85 /hour (1 hour Minimum)
Instrument Time Run by personnel:	$125/ hour (1 hour Minimum)

To become a user:

Welcome to the Center for Crystallographic Research Reservation System. This is the main page to access calendar for the reservation system for the X-ray diffraction instrumentation under the control of the Center for Crystallographic Research.

Before reserving instrument time, be sure all of the following conditions are met.

OR If YOU NEED TRAINING, DO THESE THREE THINGS FIRST.

1 YOU MUST HAVE DONE THE RADIATION SAFETY TRAINING ONLINE COURSE!!!!!!!

ORCBS Radiation Safety Training

2 YOU MUST HAVE AND WEAR A RADIATION BADGE WHEN YOU USE THESE INSTRUMENTS!!!!!!!!

ORCBS Dosimetry Page

3 YOU MUST BE REGISTERED TO RESERVE INSTRUMENT TIME
YOU MUST BE A TRAINED USER (or undergoing training)
APPLICATION FORM ***You will need your MSU net ID login.***

Once you have done these three things, reserve time, with your sample ready, and email manager to confirm that he can make the time you have reserved. You need to reserve at least 1 hour.

Take Chem. 913, special topics course in X-ray Diffraction.

4) Must follow Rules established for the operation and organization of the laboratory.

5) The user becomes responsible to ensure that data collected is adequate. If you collect a data set, you will be billed. The user is responsible to back-up the data. *The manager has the ability to remove any and all files at all times form the computer in the laboratory. The Manager does not back-up raw powder diffraction data.*

Rules regarding Calendar Reservation System.

4) **You must have a sample**! No signing up in hopes of generating the sample!

5) You can only sign up for three time periods in a given Week. You can sign again when your turn is over.

6) There will be separate calendar to reserve the instrument. I reserve the right to bump anyone to get other samples run.

2) You must be a registered user.

3) You must sign up for use of the instrument on the calendar.

4) Remember to adjust the calendar reservation based on actual time used.

5) As a courtesy, if your sample runs shorter or longer than reserved try and contact the next person in the calendar reservation system.

6) If you are next, and the person ahead of you has used the instrument Make sure the data collection is finished. If your reservation time has started, save the experiment via a date for the filename and continue. Then remove the sample and label it.

7) Be Courteous and considerate of others work.

8) KEEP THE INSTRUMENT AND SAMPLE PREP AREA CLEAN AND ORGANIZED.

Rules and Regulations for use of the Center for Material Characterization.
Your RING is required for operation of the instrument!

1) The lab is locked, and access is by keycard (MSU ID).

2) The use of the facility is for authorized users only. Only authorized personnel are allowed to be in the lab during off hours, i.e. no kids, or friends.

3) Keep the lab clean and organized.

4) Do not allow users to use your ID to access the room. Those requiring access contact the manager.

5) Violation of the rules will mean loss of use.

6) Authorization to use the Powder Diffractometer, DaVinci must comes from the Manager and with the following rules.

   a) You must have been trained in the operation and safety of the diffractometer.

   b) You must follow the guidelines of the calendar reservation system to ensure no conflict of use or violation of calendar rules. Be courteous and polite to other users.

   c) Reserve time in the calendar program.

   d) As users, you will be bill for time use of the instrument, regardless of the results.

   e) If you need to call the manager, call before 10:00 PM, unless it is an emergency. Calling to see if you can collect ahead of someone in the calendar is not an emergency and should be assumed you will need to wait?

   g) During heavy use periods the failure to show within a reasonable time will result in loss of time.

IT IS THE AIM OF THESE REGULATIONS TO PROVIDE THE BEST OVERALL OPERATION OF THE DIFFRACTION FACILITIES.

Since one can not always predict the length of time required for an experiment, please be courteous of others and allow them extra time if need. Adjust the reserved time accordingly, and if possible, contact those that will be affected by the change in reserved time.