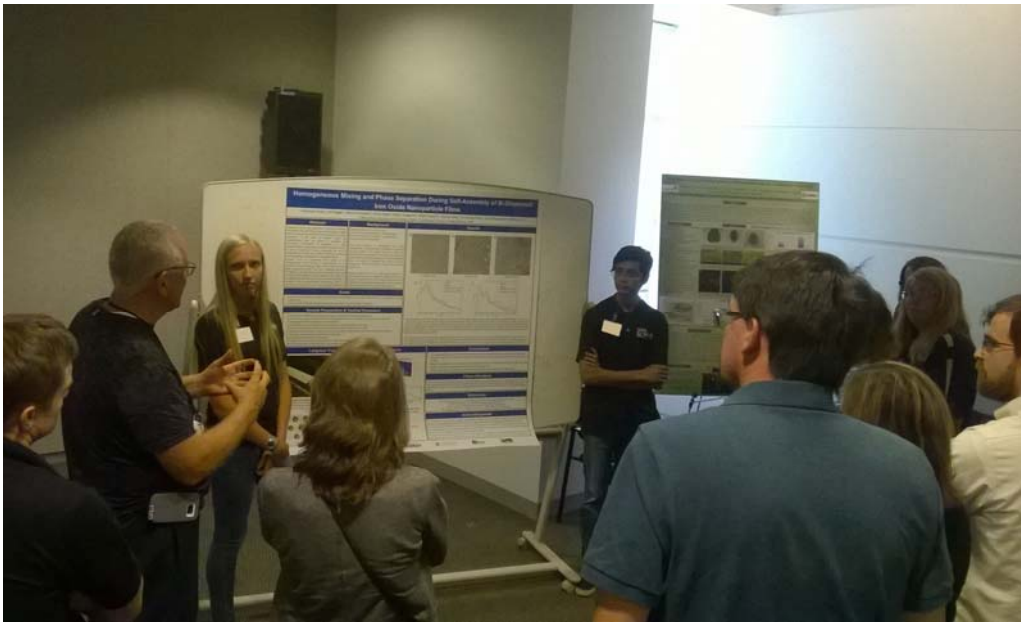
Hoffman Estates High School Engineering Students Return to ChemMatCARS For Argonne National Laboratory's ESRP 2016 – 2017

In August of 2016, Hoffman Estates High School engineering students began their second year in Argonne National Laboratory's Exemplary Student Research Program (ESRP). Scientists from ChemMatCARS, a national synchrotron facility dedicated to research in chemistry and materials research at the Center for Advanced Radiation Sources, University of Chicago, agreed to continue to work with the HEHS team. The 2016 team consisted of sophomore Zoey Aleksieva, juniors Allison Schrader and Matt Vlasaty and seniors Harpreet Auby, Jill Prigge and Miraj Shah. They were later joined by sophomore R.J. Webster.

On August 2, 2016 Jill Prigge and Miraj Shah presented at the ESRP Teacher-Scientist Workshop at Argonne's Advanced Photon Source (APS). The APS at the U.S. Department of Energy's Argonne National Laboratory provides ultra-bright, high-energy storage ring-generated x-ray beams for research in almost all scientific disciplines.



Jill Prigge and Miraj Shah present their research and answer questions at the Teacher-Scientist Workshop – August 2, 2016

In the afternoon, they met with Dr. Binhua Lin and Dr. Yu-Sheng Chen of ChemMatCARS at Sector 15 of the APS to discuss potential research using the Advanced Crystallography facility.

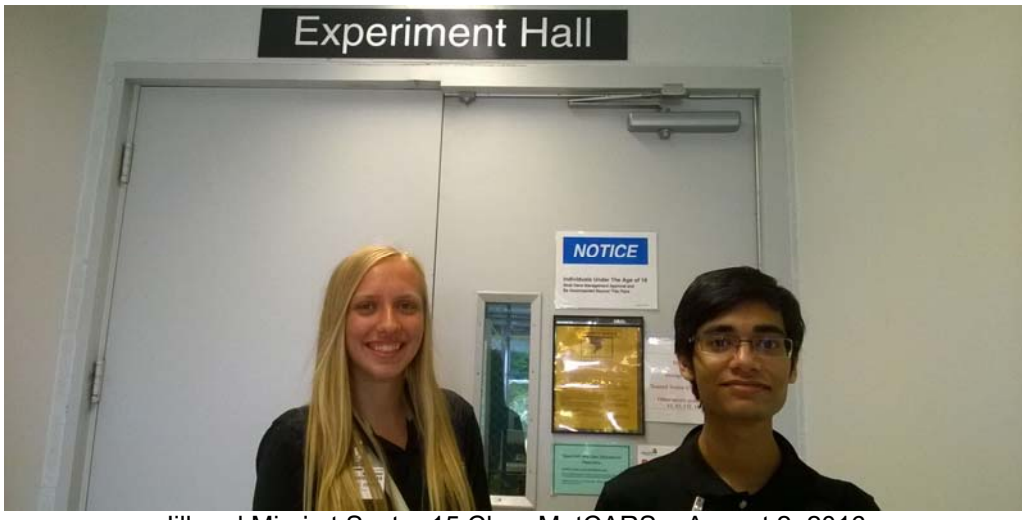
Under the guidance of Dr. Lin and Dr. Chen, it was determined that the team would learn the process of crystallography by testing a series of known and catalogued simple crystal structures. After learning the proper procedures using known crystal structures, the team would use their experience to test and analyze an unknown complex crystal structure.

Argonne ESRP

Using the world-class facilities at Argonne's Advanced Photon Source (APS), high school students and their teachers work with Argonne scientists to prepare a research proposal, design an experiment, set up the experiment, gather and analyze data, make conclusions, and prepare a final poster. In this program, school teams are considered to be part of the larger Argonne international synchrotron light source user community.

ChemMatCARS

ChemMatCARS operates as a national research resource for the chemistry and materials research communities. ChemMatCARS welcomes students at all levels – high school, undergraduate, graduate and post graduate, into our laboratories, giving them the opportunity to learn about x-ray science and instrumentation as well as to be involved in hands-on activities.



Jill and Miraj at Sector 15 ChemMatCARS – August 2, 2016

The team started writing an APS General User Proposal (GUP) and held a video conference with Dr. Chen on October 25, 2016 to help answer questions about the field of x-ray crystallography. On October 28, 2016 the team submitted a proposal requesting three 8 hour shifts for “Using X-Ray Diffraction to Confirm the Structure of Cataloged Crystals and Determine the Crystal Structure of an Unknown Molecule.”

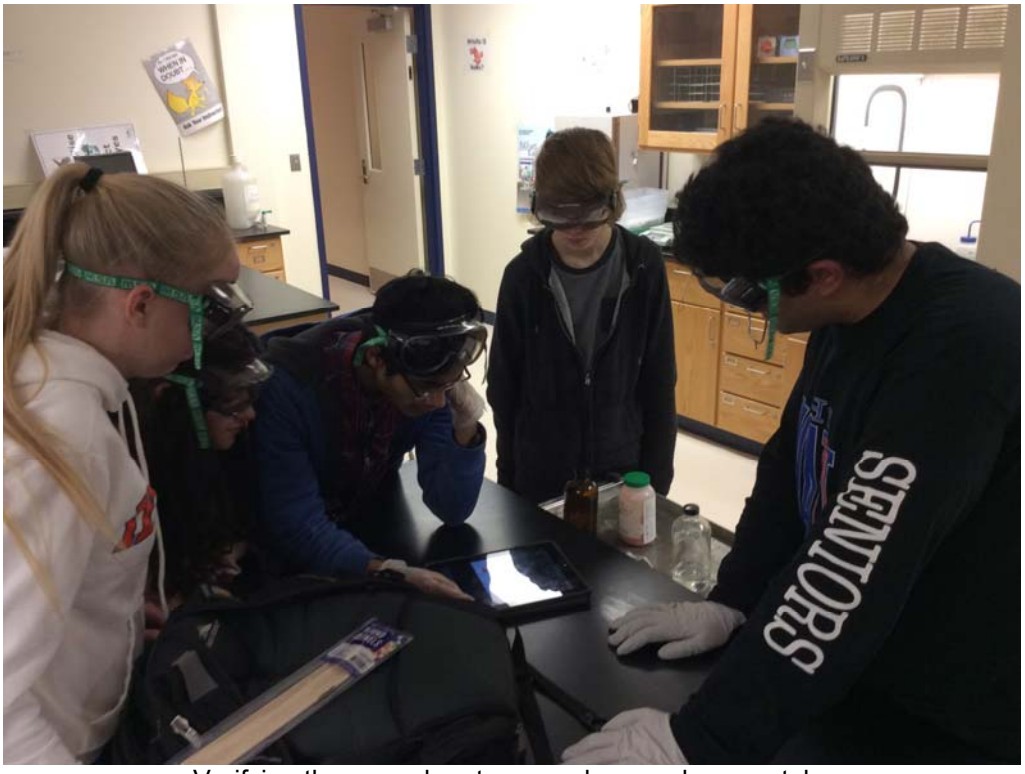
Video conference with Dr. Chen to discuss the research proposal – October 25, 2016

Researchers from around the world may submit proposals to work at one of the 35 sectors available at the APS. All proposals are judged by a panel of scientists. At the beginning of December the team was informed that they were approved for all three requested shifts. Their proposal earned a score of 1.5 on a scale of 1 to 5 where 1=Extraordinary, 2=Excellent, 3=Good, 4=Fair and 5=Poor.

Since three shifts are normally run consecutively in a 24 hour period, the team was scheduled to begin their first shift at 8:00AM on March 25, 2017 and end their third shift at 8:00AM on March 26, 2017. In order to work at the APS, experimenters must register as APS users, receive badge numbers and complete all required training sessions. In addition, an on-site sector orientation must be completed upon arriving for the first shift.

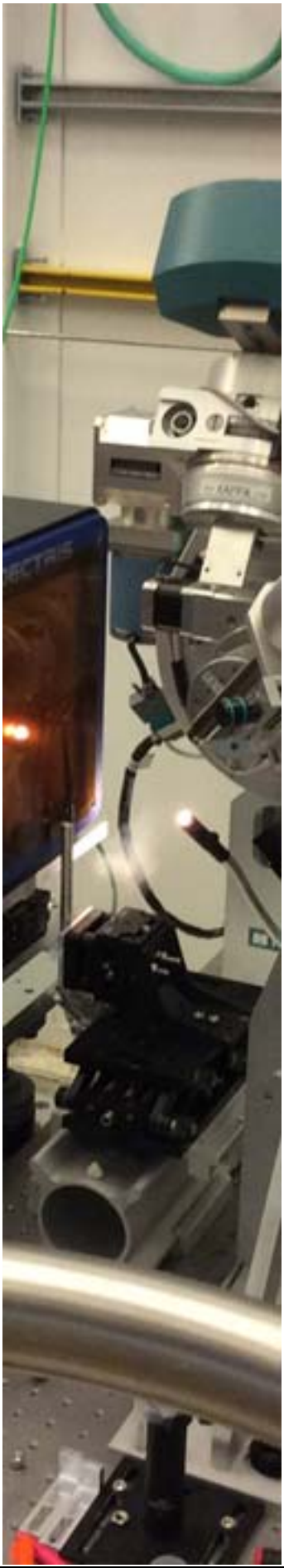
As part of the experiment, the team was tasked with growing the known simple crystal structures. After research, the team decided to grow salt, sugar, sodium bicarbonate, sodium borate, alum and copper(II) sulfate crystals.

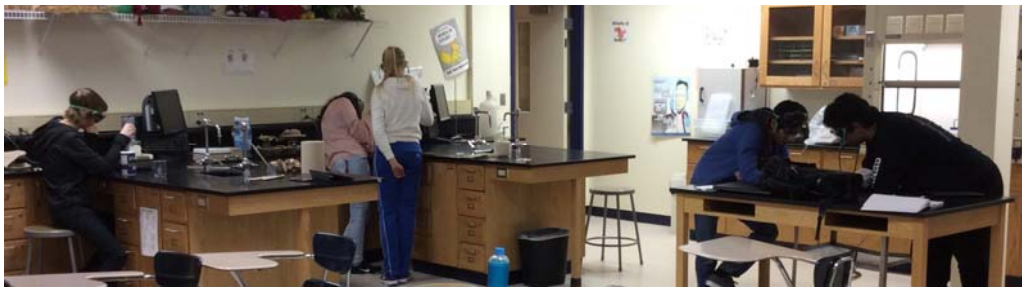
The team worked with Hoffman Estates High School science teachers Kristy LeVanti, Darcy Sowle and Tanya Katovich who shared their knowledge and provided use of their laboratory equipment to grow the crystals.



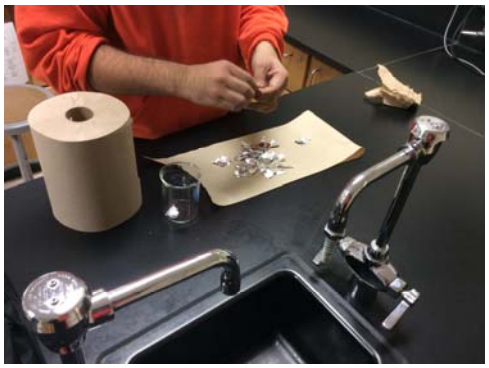
Verifying the procedure to grow chrome alum crystals.

The team members were able to apply their chemistry knowledge to grow crystals such as salt and sugar as well as follow more detailed procedures such as growing chrome alum crystals. The team dedicated three afternoon sessions to preparing the known crystal structures prior to visiting the APS.



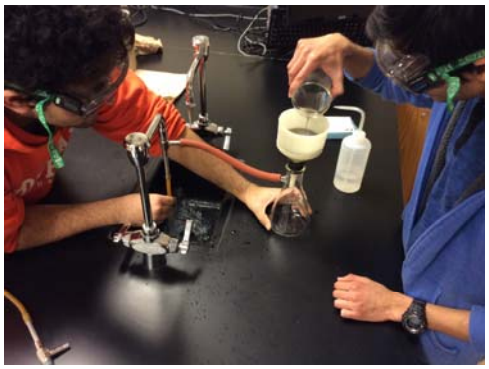


Team members working on various crystals in the lab.



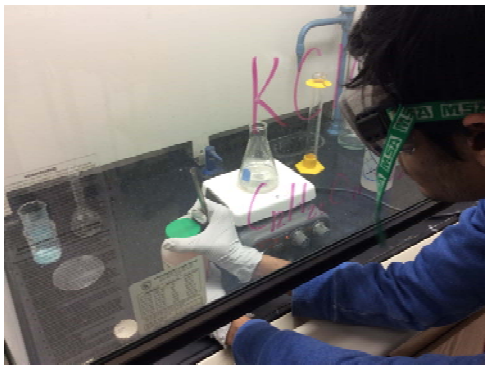
Preparing to make chrome alum.

Working under the vent hood

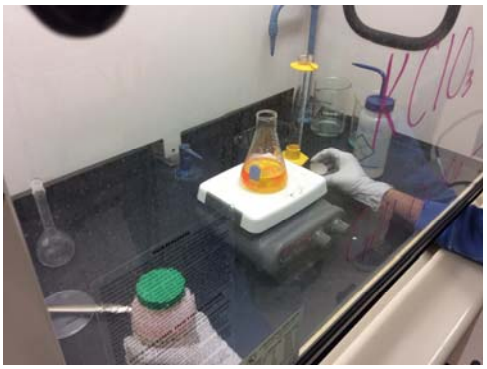


Harpreet and Miraj use a Büchner flask while creating chrome alum.

Preparing for filtration at the end of synthesization.



Preparing to add potassium dichromate to reagents on the hot plate.



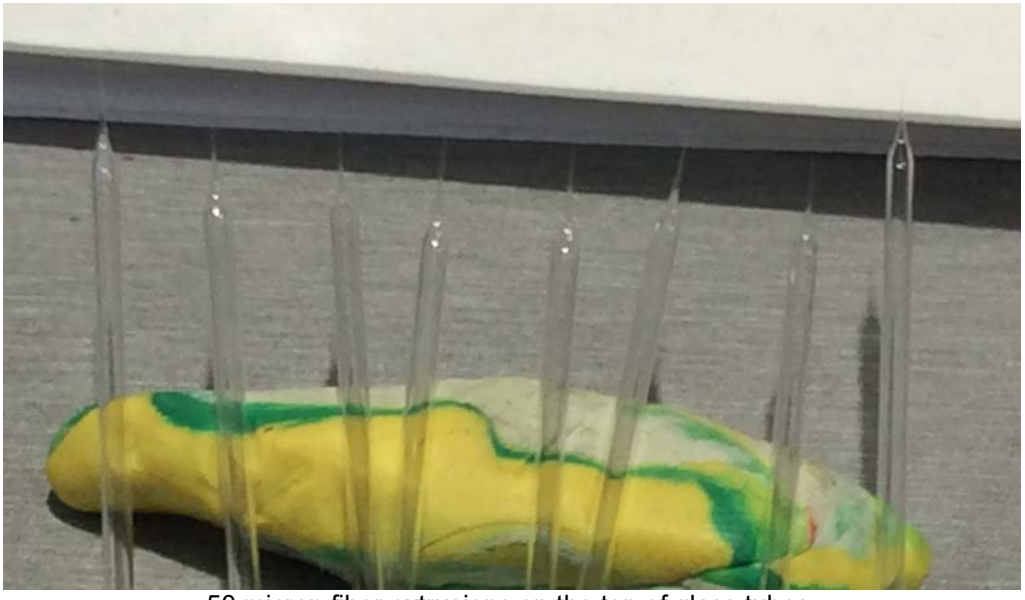
Mixing reagents under the vent hood for synthesis of chrome alum.

Photo captions courtesy of Harpreet Auby.

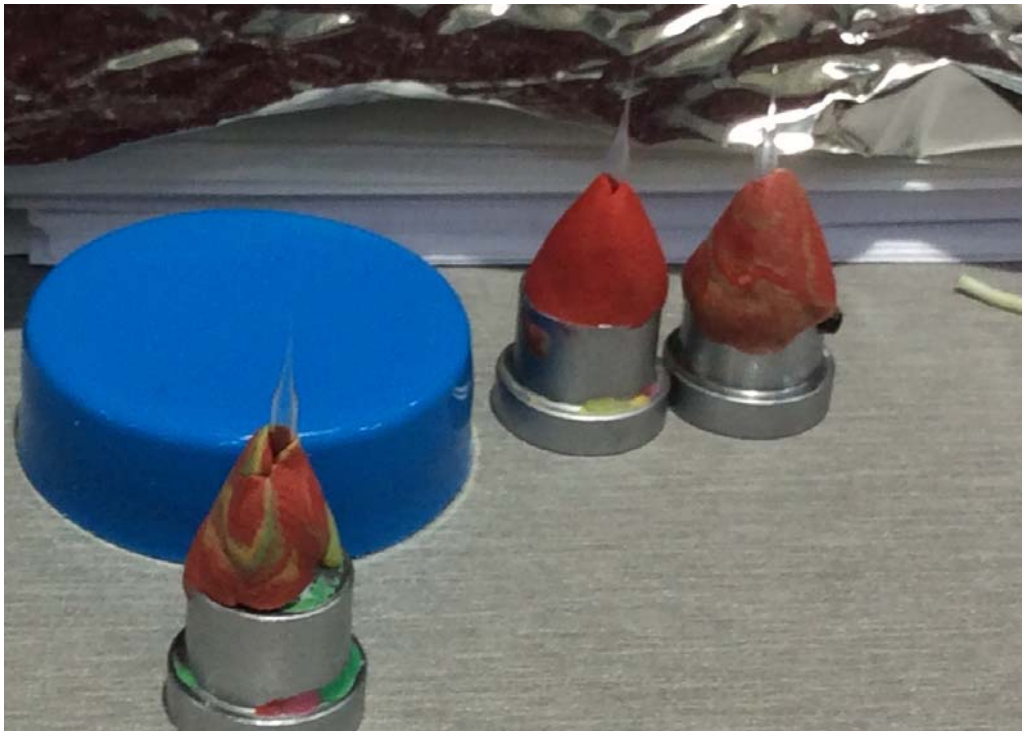
The team arrived on the morning of March 25, 2017 to begin their first shift. Dr. Chen gave the team an introductory lecture on synchrotron x-ray radiation and x-ray crystallography. Dr. Wang then led the team through the required sector training.

The team began working on sample preparation. The first step was to create 50 micron (.002”) diameter fibers to hold the crystals in the x-ray beam. Each team member was able to create fibers out of glass tubes. The fibers were then mounted on magnetic bases.

Making glass fibers.



50 micron fiber extrusions on the top of glass tubes.



Tubes mounted on magnetic bases.

After creating the fibers, the team began to work with their known crystal samples. The optimal crystal size for testing was between 100 and 150 microns (.004” and .006”). Using the microscope and computer monitoring, the team would measure and cut the crystals in order to obtain an appropriate sample for testing.

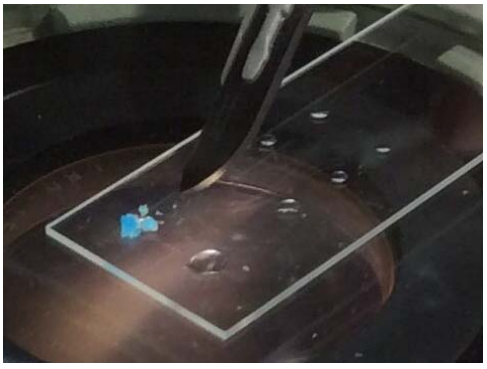
Dr. Chen inspecting crystal samples before demonstrating how to measure and cut.

The team working at the microscope stations at Sector 15.

Matt preparing a crystal sample.

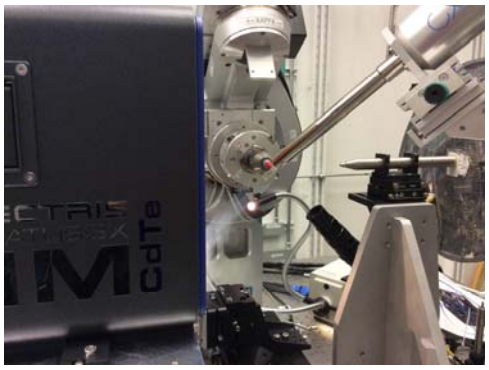
Harpreet preparing a crystal sample.

Miraj preparing a crystal sample. The slide in the image to the right is being magnified on the monitor above.

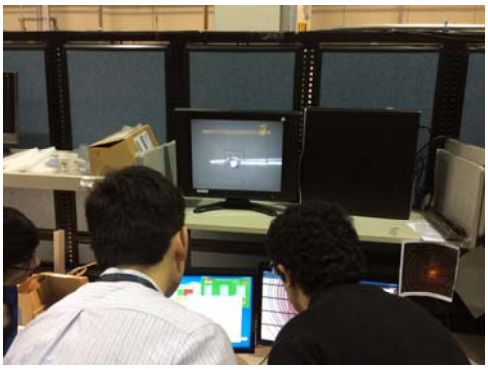


The white speck just below the scalpel tip is the dark spot in the middle of the monitor in the image to the left.

After measuring, cutting and mounting, the samples were ready to be tested. The magnetic base supporting the crystal was set in place on the goniometer, which is used to align the crystal in the path of the x-ray beam using a computer controlled system. After aligning the crystal, the hutch was cleared for testing.



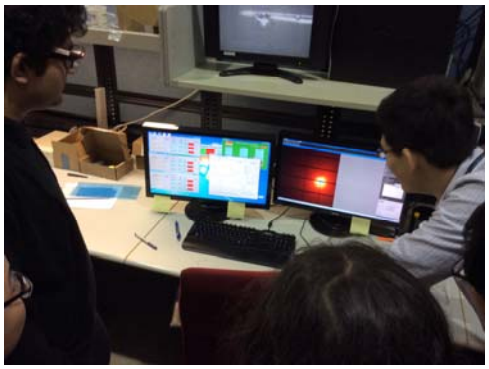
Crystal mounted on goniometer.
Note the red clay holding the fiber.



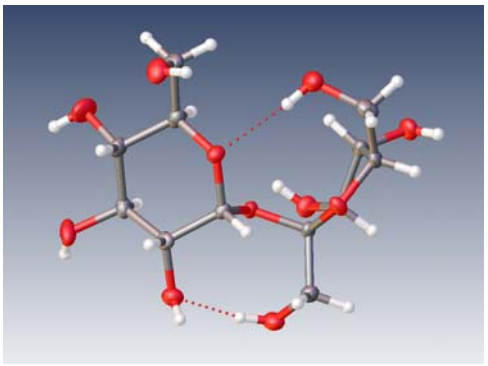
Aligning the crystal shown in the red box on the top monitor.

With the test hutch checked and cleared, the sample was exposed to photons from the storage ring. The team used a Dectris PILATUS3 X CdTe 1M hybrid pixel array detector to collect diffraction pattern data. This detector was one of only two in the world at the time. One benefit of this detector is the speed at which data was collected. The team planned to collect data on six known crystals and one unknown crystal during their three shifts. The speed of the detector enabled them to collect data on nine known and two unknown crystals in only two shifts.

APEX II and XPREP software was used to compile and integrate the diffraction intensity data collected by the detector. This data was then imported to Olex2 software. Olex2 was used to model the crystal structure based off of the output files from XPREP. The team determined the chemical structure, bond lengths, packed cell model and packed crystal lattice model for each of the eleven crystal samples.



Diffraction pattern data is shown on the bottom right monitor.

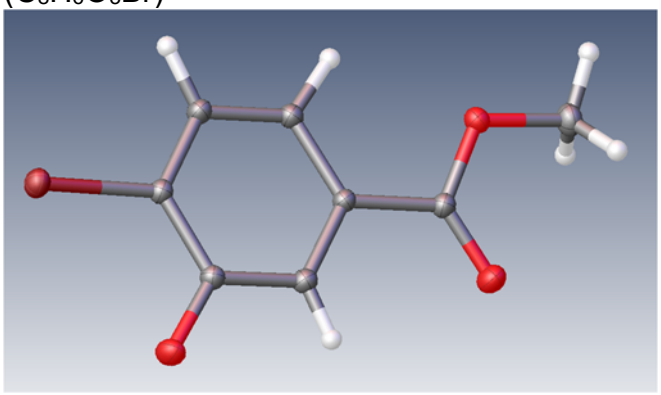


Software model of sucrose crystal.

The known crystals were used to check the validity and reliability of the procedures and equipment. The team verified the structure of the samples they grew as Alum ($KAl(SO_4)_4 \cdot 12H_2O$), Borax ($Na_2B_4O_7 \cdot 10H_2O$), Copper Sulfate ($CuSO_4$), Sodium Bicarbonate ($NaHCO_3$), Sodium Chloride (NaCl) and Sucrose ($C_{12}H_{22}O_{11}$). Samples provided by Dr. Chen were verified as Azobenzene ($C_{12}H_{10}N_2$), Hexamethylenetetramine ($C_6H_{12}N_4$) and Oxalic Acid ($C_2H_2O_4$).

After Dr. Chen was satisfied that the students had an understanding of the procedures, he asked them to test two unknown samples provided by Dr. Ting-Zheng Xie from the University of Akron. These samples were determined to have a chemical structure of ($C_8H_6O_3Br$) for Crystal 1 and ($C_{39}H_{27}O_3N$) for Crystal 2. The models are shown below.

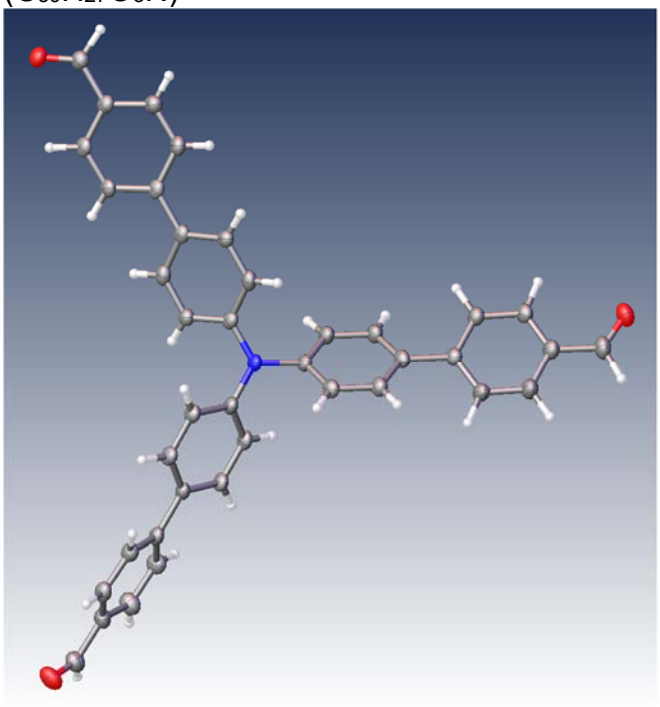
($C_8H_6O_3Br$)



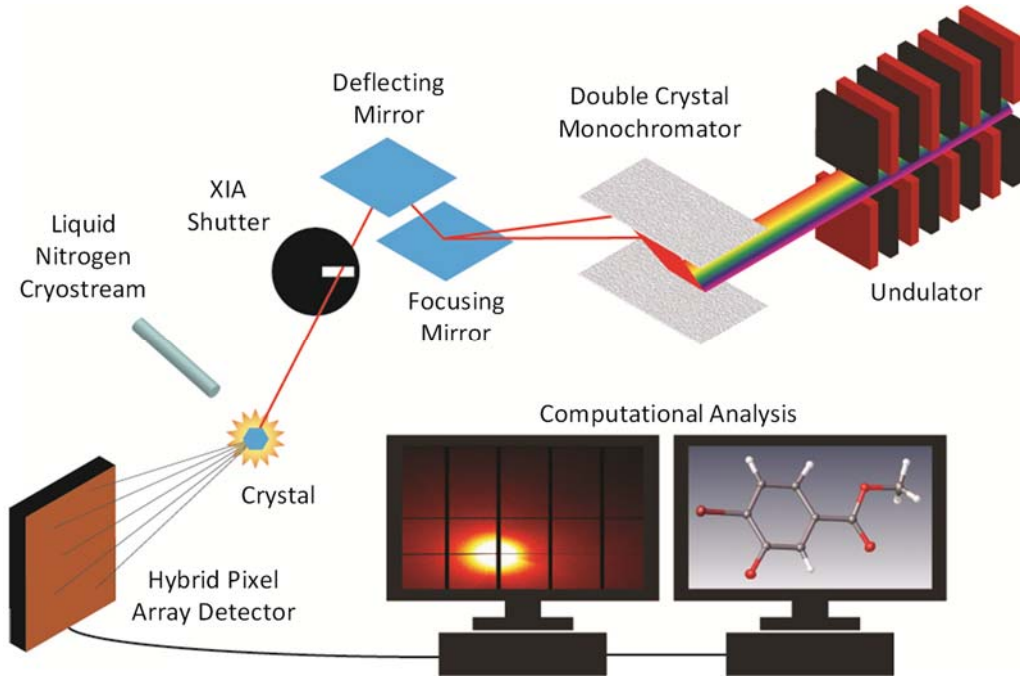
Bond Lengths

C-Br: 1.898 Å
C-O: 1.356 - 1.453 Å
C-C: 1.396 - 1.493 Å
C-H: 0.919 - 1.089 Å

($C_{39}H_{27}O_3N$)



Dr. Chen and Dr. Wang observe as Miraj collects data.



X-Ray Diffraction Crystallography Schematic
Advanced Photon Source Sector 15-ID-D

APEX II and XPREP software is used to compile and integrate diffraction intensity data collected from the detector. Olex2 software is used to model the crystal structure based off of the XPREP output files.

The Hoffman Estates High School ESRP Team:

Zoey Aleksieva, Sophomore
Rudolph Webster, Sophomore
Allison Schrader, Junior
Matt Vlasaty, Junior
Harpreet Auby, Senior
Jill Prigge, Senior
Miraj Shah, Senior
Wayne Oras, Faculty Sponsor

The Hoffman Estates High School ESRP Team would like to acknowledge:

Dr. Binhua Lin, Deputy Director, ChemMatCARS
Dr. Yu-Sheng Chen, Operations Manager, ChemMatCARS
Dr. SuYin Grass Wang, Jr. Beamline Scientist, ChemMatCARS
Dr. Ting-Zheng Xie, Department of Polymer Science, University of Akron
Constance Vanni, Assistant Manager, User Programs, APS
Tieyan Chang, Ph.D. Student, ChemMatCARS
Kimberly Simms, Sector User Coordinator, ChemMatCARS
Louis Harnisch, Educational Program Coordinator, Argonne National Lab
Tanya Katovich, Chemistry Teacher, Hoffman Estates High School
Kristy LeVanti, Chemistry Teacher, Hoffman Estates High School
Darcy Sowle, Chemistry Teacher, Hoffman Estates High School

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NSF National Science Foundation WHERE DISCOVERIES BEGIN
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