CORESUPDATE

Center of Research & Educational Support Newsletter

December 2020

HOLIDAY Closures

All CORES facilities will be closed from December 24, 2020 through January 4, 2021.

Additionally, Graphic Services will be closed beginning December 21 through January 4.

The CORES facilities appreciate your customer support throughout a challenging 2020 and look forward to many successful collaborations in the new year.

We extend our best wishes to you for the holiday season and the new year.

The Machine Shop is ready to put your project in motion.

The Machine shop is now open from 7:30 am to 3:30 pm, Monday through Friday.

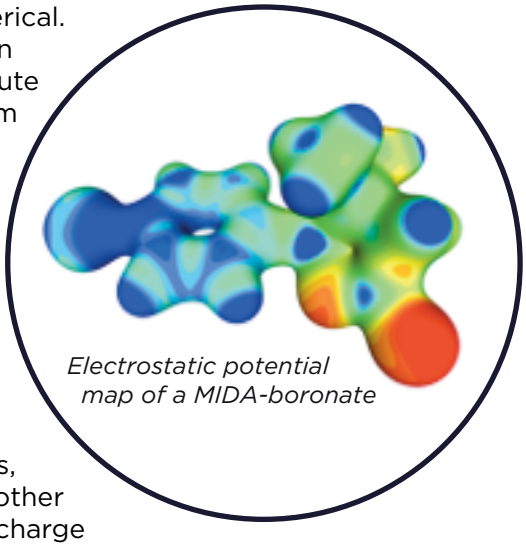
We can assist with consultation, design, and fabrication of most large or small projects.

Currently our Work Order backlog is relatively low, so now is a great time to contact us via email. We can schedule a Zoom Meeting to discuss any projects you have or need designed.

Email:
scs-machineshop@illinois.edu

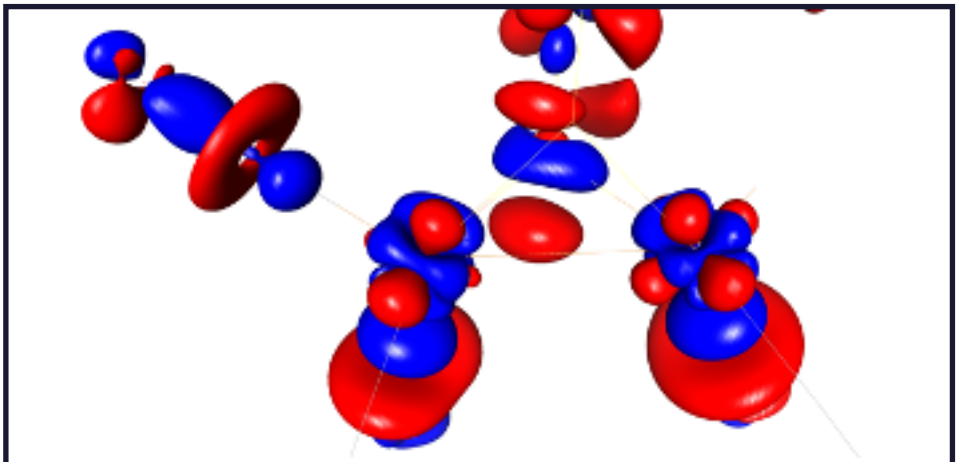
Charge Density Analysis now available in the X-ray lab

A new detector was recently installed on the DUO diffractometer in the X-ray lab. This new detector allows us to collect extremely accurate diffraction data to very high resolution. A routine structure determination from our lab uses the approximation that the electron density of all atoms is completely spherical. The capability to collect high resolution diffraction data allows us to de-convolute the spherical core electron density from the aspherical bonding density in a structure. Having an experimentally determined picture of the bonding density allows a host of molecular properties to be determined: partial charge on an atom, degree of charge transfer/charge separation, lone pair orientation, degree of covalency in a bond, presence of hyperconjugation, presence of agostic interactions, electrostatic potential, dipole moments, d-orbital populations, and almost any other property that is a function of how the charge (electrons) are distributed in a molecule.



Electrostatic potential map of a MIDA-boronate

This type of analysis requires very good crystals that are capable of diffracting to high resolution. Additionally, the structure cannot have any crystallographic problems such as disorder or twinning; neutral molecules are the most straightforward to analyze but charged species can be accommodated.



Aspherical bonding density in a di-iron carbonyl complex

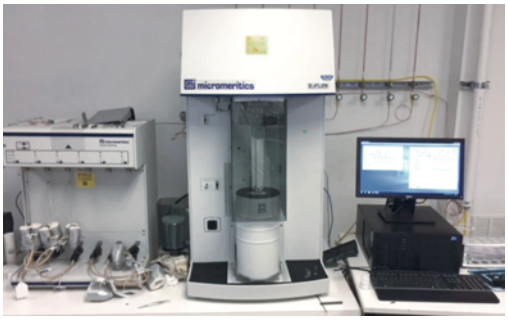
Please contact Toby Woods (tobyw@illinois.edu) if you want more information or are interested in pursuing a charge density project.

Instrument TiPS

Microanalysis Lab Surface Analysis

How to Maximize the Information You Receive with a Physisorption Experiment Request

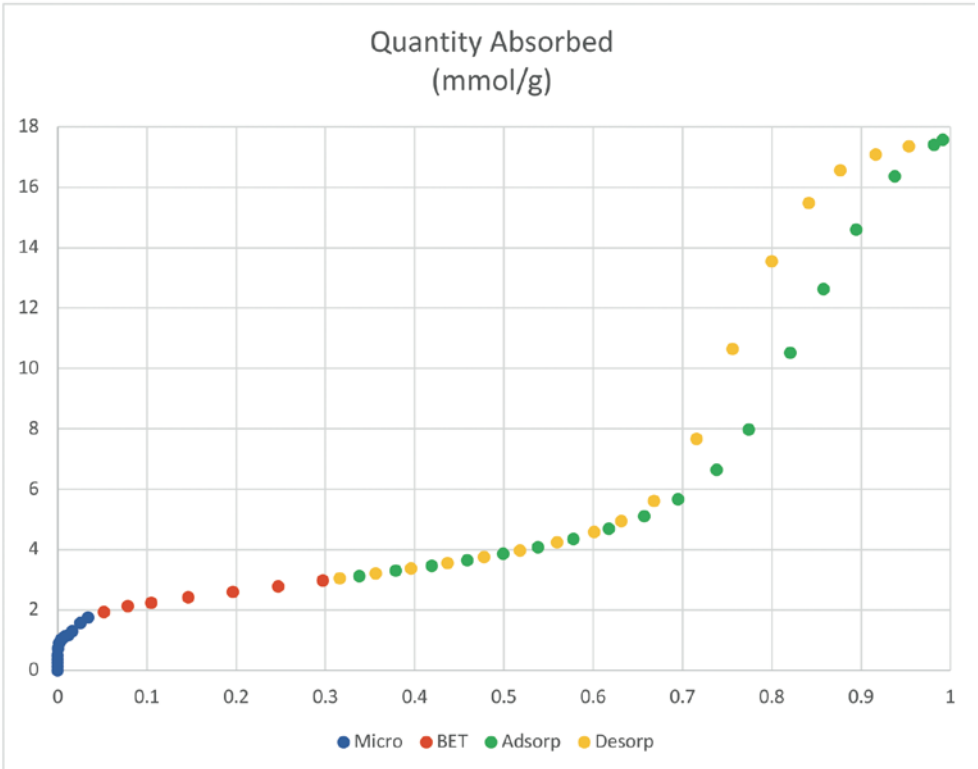
The Microanalysis Lab has not only elemental analysis and thermoanalysis available, but also surface area analyses. We have a MicroMeritics 3Flex instrument, capable of running physisorption and chemisorption on a variety of compounds. We ask that sample compounds for these analyses be bigger than 100 µm in particle size.



Students can be trained in the use of the instruments if they will be running experiments on a regular basis, otherwise we recommend having the tech run the sample analysis. Contact scs-microlab@illinois.edu with questions.

What information can a Physisorption experiment give you?
A common physisorption surface area request is "I want BET." The client is then confused when they receive only BET surface area, aka the meso surface area, when they had actually wanted surface area and pore size information.

Let's take a look at a surface analysis isotherm:



Micro region:

If a sample contains or might contain micropores (~0.35-2nm), this region is run. This region gives the micropore surface area, the micropore size and the micropore volume. Gas choice is important when investigating micropores; CO2 can often fill micropores better than N2 and so a separate micropore analysis must often be run. A CO2 micropore analysis will only yield micropore information.

BET region:

This region will provide mesopore surface area.

Adsorption/desorption regions:

These regions will provide mesopore pore size (~2nm - 50nm) and volume information.

Be informed when asking for a Physisorption experiment. Don't just ask for BET or you might not get the information you are looking for.

BET analysis: will only get you the mesopore surface area

Micropore BET with CO_2: will only get you micropore information

Micropore BET with N_2: will get you Miropore information, and mesopore surface area

Mesopore sorption/desorption: will get you mesopore surface area, pore size and volume

Micropore sorption/desorption with N_2: will get you micropore information and all the mesopore information.

Adding a second cycle to the sorption isotherms to gain information about the stability of the surface structure under pressure and loading.