Contact

- **Location:**
  47 Noyes Laboratory

- **Hours:**
  8:30 a.m. — 5:00 p.m.
  Monday through Friday

- **Telephone:**
  217/333-3095

- **E-mail:**
  micro@scs.illinois.edu

- **Web:**
  http://scs.illinois.edu/microanalysis/

Rudiger Laufhutte
Lab Manager
laufhutl@illinois.edu
217/333-3095
Contact

Rudiger Laufhutte
Lab Manager
laufhut@illinois.edu
217/333-3095

Ratna Dutta
Research Specialist in Chemistry
rdutta@illinois.edu
217/333-3095

Elizabeth Eves
Research Specialist in Chemistry
bleves@illinois.edu
217/333-3095

Not pictured: Charles Nash, the Halide Guy
Scope Of Services

The Microanalysis lab provides two types of services: elemental and thermal analysis. NOTE: For details on all procedures please see sections on each instrument. The elements selected by the user determine which instruments the lab uses.

Elemental Composition Calculator

Sample Login

ELEMENTAL ANALYSIS
The lab uses various instruments for elemental analysis:

1. CHN Analysis - *Exeter Analytical CE 440* and *Perkin Elmer 2440, Series II* (for measuring carbon, hydrogen, and nitrogen in compounds)

2. ICP Analysis - *ICP-MS* and *ICP-OES* (for measuring all metallic elements including S and P)

3. Halide Analysis - Titration & ISE (for measuring halides)
THERMAL ANALYSIS

1. **Microcalorimeter** - The measurement of heat generated or absorbed in liquid samples as a result of mixing two or more reactants. **NOTE:** Only liquids can be used in this instrument.

2. **Thermogravimetric Analyzer (TGA)** - The TGA analyzer measures weight changes of sample over a given temperature and pressure range under specific environmental conditions. **NOTE:** No corrosive gas can be used so far.

3. **Differential Scanning Calorimeter (DSC-7)** - The DSC 7 is used for the calorimetric measurement, characterization, and analysis of thermal properties of materials. This instrument is best used for studies at temperatures of 400° C and higher.

4. **Differential Scanning Calorimeter (DSC-Diamond)** - The DSC is used for the calorimetric measurement, characterization, and analysis of thermal properties of materials. This instrument is best used for studies at temperatures of 400° C and lower.
Instruments And Techniques

All results for elemental analysis are calculated based on a known value of a standard. A computer then calculates the ratio of the sample output to the standard output to arrive at a value. The standards used are all traceable back to NIST primary standards and all instruments are checked with a NIST primary standard on a regular basis to assure day to day accuracy of results.

All results, unless noted otherwise, will be reported as % by weight.

INSTRUMENTS

- CHN Analysis
  - Exeter Analytical CE 440
  - PerkinElmer 2440, Series II

- ICP Analysis
  - PerkinElmer Elan DRCe ICP-MS
  - PerkinElmer Optima 2000DV ICP-OES

- Halide Analysis
  - Thermo Scientific "Orin" Ion Selective Electrodes
    - Bromide Combination Probe
    - Chloride Combination Probe
    - Fluoride Combination Probe
  - Titration
    - Kolbenburette/Piston Burette/Metrohm Ag Horesau
  - Fisher Scientific Accumet pH/Ion meter 25

- Thermal Analysis
  - Microcalorimeter
  - Thermogravimetric Analyzer (TGA)
  - Differential Scanning Calorimeter (DSC -7)
  - Differential Scanning Calorimeter (DSC-Diamond)

Submission Of Samples To The Microanalysis Lab

Below are links to information that is important in the sample submission process.

- Sample Login
- Sample Preparation
- Unstable Samples
- Sample Submission
- TTA
- Results
Microanalysis Lab Sample Submission Guidelines

Sample Purity:
The determination of the concentration of elements in your sample is based upon the direct weight of the the material sampled. Therefore, it is very important that samples be dry, free of foreign substances such as dust, rust, hair, aluminum foil, parafilm, and paper filter fibers (the most common contaminant). Microscopic inspections have shown that over half of the samples submitted are obviously contaminated.

Most journals require 0.40% for each element. For comparison, NIST standards are 0.03%. Typical compounds submitted have errors ranging from 0.05% to 0.45%. These compounds have all gone through several stages of purification. Let’s say while putting your sample into a vial a spec of graphite from your pencil fell into the vial and blended in with your yellow crystals. Let’s also say that the spec of graphite weighs 100 µg (that’s really a large spec by the way). The nominal sample size for CHN is 2000 µg. If the spec is taken as part of the sample, then there is a 5% error in the weight. So you see, it doesn’t take much to throw the result off.

Sample Size:
The actual amount of sample needed depends on the concentration of elements to be detected. To avoid confusion, listed below are the requirements for minimum amount of sample needed per analysis.

<table>
<thead>
<tr>
<th>SERVICE</th>
<th>AMOUNT</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHN (routine)</td>
<td>2.0 – 3.0 mg recoverable material/single analysis in flat bottom ½ dram(12x35mm)(32843901)</td>
</tr>
<tr>
<td>CHN (routine, duplicate)</td>
<td>5.0 – 6.0 mg recoverable material/duplicate analysis in flat bottom ½ dram(12x35mm)(32843901)</td>
</tr>
<tr>
<td>CHN (routine, soil)</td>
<td>10.0 – 15.0 mg recoverable material/single analysis</td>
</tr>
<tr>
<td>CHN (soil, duplicate)</td>
<td>20.0 – 25.0 mg recoverable material/duplicate analysis, this will depend on the theoretical values of the carbon, hydrogen and nitrogen in the actual sample submitted for analysis</td>
</tr>
<tr>
<td>CHN (liquid (non-aqueous), non-routine/air sensitive/unstable)</td>
<td>It is recommended the student bring at least 2 vials of liquid material in the following vials: V-vial (0.3ml) (13x32mm)(32NLD890)(reactive vial) OR Flat bottom ½ dram vial(5x35mm)(32843901)</td>
</tr>
</tbody>
</table>
Unstable Or Air-Sensitive Samples

Some samples may remain at room temperature as submitted for 0.5 to 24 hours before an analysis is started. If your sample is unstable there are several steps that can be taken to avoid agony in the results. (SEE SPECIAL NOTE below)

1. Submit sample in air-sensitive double vialed capsule. (Sn for CHN; AI for METAL /HALIDE, unless the metal is AI, then use Sn capsule) Once the capsule is taken out of the vial, it is only protected as well as the seal made on the capsule. Make sure capsule is sealed and folded over.

2. Leave sample in desiccator or freezer. Enter on the online submission form where sample is located.

3. Submit sample vial inside a larger vial that is partially filled with Drierite and has wool keeping any possible specs of Drierite away from sample vial.

4. For extremely sensitive samples, make an appointment for drop off of sample for processing immediately.

SPECIAL NOTE FOR AIR SENSITIVE CAPSULES
To use air-sensitive bottles, take the appropriate one from the shelf located just inside the lab. Fill the capsule and crimp it in your dry box. After crimping gently place a fold in the crimped section to form a good seal. Reseal in both vials. Drop off bottle in vacuum desiccator and write down bottle letter and/or number on submission record card. There are a limited number of bottles for air-sensitive samples. Be considerate of your fellow researchers and take a bottle only if you are going to be submitting a sample in the next couple of days. If a bottle gets broken, please bring back the remains and let us know which bottle and if the capsule has been contaminated.
Submit Your Sample  “Samples are checked in when they arrive”

(There are special requirements for CHN, Halide, and ICP. If you have any questions, please ask Rudiger Laufhutte (laufhutt@illinois.edu)

To submit samples for microanalysis, you must fill out the online sample submission form (https://www-s.scs.illinois.edu/micro/) and submit it.

For University of Illinois, UC users, fill out the sample submission online form and bring the samples to Room 47 Noyes Lab and place in the appropriate service tray. Use the computer in this room to check them in. Samples must be checked in so that lab staff know the samples have been brought to the lab. Your samples will not be run unless they are properly submitted online and checked in.

Samples that require multiple services should be placed in the service tray to be performed first. If a sample requires multiple analyses, the customer is also asked to submit one sample for each area of analysis. For example if the customer requests CHN & ICP analysis the customer should submit at least a minimum of 2 samples for analysis. Customer is also asked to review the "Sample Preparation" section for the proper amount of sample to place in each vial. Samples that are moderately sensitive should be placed in the appropriate container such as vacuum desiccator, regular desiccator, high humidity container, or freezer. (The freezer is maintained at below 0 °C). Enter on your on-line submission form where the sample is located. Users from outside the University of Illinois need to fill out a Technical Testing Agreement before sending samples. Samples should be mailed in protected containers to:
Your Results Will Be Sent To You By E-mail

The estimated turnaround time (SCS = School of Chemical Sciences faculty):

**CHN** –

a. for SCS samples, 24 hours (not including weekends and holidays), providing analyst has the sample in hand by 10:00 a.m. on the day the sample is submitted

b. All samples cannot be returned. Inside customers can request the samples be retained by staff, providing it is documented on the customer’s request

**ICP** – for SCS samples, 3-10 working days

**Halides** – for SCS samples, 3-5 working days

These times will be longer if there are instrumental problems or maintenance or if the staff is reduced due to sickness, vacation or meetings. When your sample is finished, the results will be sent to you via auto generated e-mail, possibly as PDF attachments.

For users outside of University of Illinois, UC, the samples will not be returned unless you make a comment in the comment box of the online submission form telling us to return the samples. You will be billed for shipment.
Sample Submissions

The sample submission forms can be created on computers in your lab. Checking in your sample(s), which assigns a Work Order Number and creates a line in our In Progress system, can only be done at the Check In computer in room 47. If you wish to have multiple techniques run on your sample, please submit multiple vials of your sample. (Ex. Both CHN and ICP - two vials submitted.)

Routine samples:

- **SOLIDS:** Preferably in small pieces/ground up fine/homogeneous
  - Flat-bottomed ½ dram or 1-dram vial
- **LIQUID, NON-SOLID TYPE MATERIALS, or EXTREMELY SMALL AMOUNTS OF SAMPLE:**
  - V-vial 0.3 ml
  - Flat-bottomed ½ dram or 1-dram vial
- **PRE-DISSOLVED, AQUEOUS ICP SAMPLES:**
  - Any non-leaking container is acceptable.

Non-routine samples: **Air-sensitive, extremely moisture sensitive or unstable samples:**

Jars containing pre-weighed capsules are located in the upper right drawer of the check-in bench. Make sure that the filled capsules are free from any debris on the outside of the capsule and are completely crimped/sealed, as holes or gaps can affect your sample and your result.

For CHN or Al ICP-analysis, please use the **RED** jars with the appropriate amount of material in each sealed tin capsule. (CHN - 2 - 2.5mg of sample; ICP - at least 10mg of sample)

For non-Al ICP analysis and **Halide** analysis, please use the **BLUE** jars with the appropriate amount of material in each sealed aluminum capsule. (Both techniques - at least 10mg of sample.)
What does Recoverable Material mean?
RECOVERABLE MATERIAL is defined as the analyst’s ability to recover that amount of material from the vial. You may want to consider submitting more material to ensure the analyst can recover the amount needed for analysis. Sticky/gooey/fluffy materials will need more sample submitted in your vial, as sometimes these are quite difficult to extract from the sample vial. If you are looking for trace elements, you may need to submit more sample than specified to ensure better results.

ICP Specifics
2 - 10mg of recoverable material
Turn-around time: 10 business days, depending on the number of requests, number of samples & elements on the request forms.

Halide Specifics
5 - 10mg of recoverable material
Turn-around time: 3 - 5 business days, depending on the number of requests in queue to be analyzed

CHN Specifics
Aqueous solutions cannot be run via CHN analysis.
5 - 6mg of recoverable material
8 - 10mg of recoverable material, if the sample is liquid/goo/oil.
Turn-around time:
Routine: 24 - 48 hours, providing samples are dropped off at the laboratory by 10:30 AM. (SEE NOTES BELOW!)
Non-routine: Air/moisture-sensitive capsules are considered non-routine and should be discussed with the technician before for when the samples should be dropped off for analysis.
Liquid/oil/goo samples are also considered non-routine and may take longer for completion than routine samples.

If a sample is dropped off after 10:30 AM, the sample might not be analyzed until the next day/run set unless something has already been arranged with the technician. We will try to accommodate our clients as much as possible.

The 24 hour turn-around is also dependent on the number of samples in the queue to be analyzed previous to these samples submitted, the maintenance needs of the instrument (ex. trap or tube replacement) and the success of the previous day's run.
If 24 hour-or-less turn-around time is needed, talk to the technician first!
If your samples are not Checked In, they WILL NOT BE RUN!!!

There is a difference between creating a submission form (can be done at any computer) and Checking In your sample (can only be done at the Micro computer).

Until they are Checked In on the computer, WE CAN’T SEE THEM IN OUR SYSTEM.

If you just drop your samples off, we can’t see whose they are. We have no information for them at all. And if you don’t talk to us about your samples, we also can’t notify you that you forgot to Check In.

If you do not follow procedure, we cannot be held responsible for you not getting your data in a timely manner!

Your samples will not be placed in the que to be run until they have been Checked In!
I’m checking in my sample(s)!

Great! Now talk to the technician who will be analyzing your sample(s).

They may be in the CHN section, the ICP section, the back office or in room 54. Or they may be out.

In the cases of the different sections of room 47 and room 54, remember to KNOCK LOUDLY!

Unless there is a DO NOT DISTURB sign.

ICP: Rudi (laufhutt@illinois.edu) and/or Ratna (rdupta@illinois.edu)
CHN: Beth (bleves@illinois.edu)
Halides: Charles (cnsh@illinois.edu)

But I’ve submitted samples before! / They’ve run this for me before.

Thank you for continuing to use the MicroAnalysis Lab! Still, talk to the technician.

Why?

First, it’s part of the sample submission instructions.
Second, it lets us know that you have dropped off your sample(s).
Third, it lets us talk with you about any possible sample issues/sensitivities and gives us a chance to tell you about any possible delays. (ex. maintenance or times when the technician will not be available/able to run samples)

The technician I need isn’t here.

Then talk to any of the technicians who are in. If your samples are routine, then you will probably be told it’s okay to finish checking in. You may also be advised to email your technician (see above) to let them know that you’ve dropped off your sample(s) and any specific sensitivities that they may have.
If your samples are not Checked In, they WILL NOT BE RUN!!!

There is a difference between creating a submission form (can be done at any computer) and checking in your sample (can only be done at the Microcomputer).
Until they are Checked In on the computer, WE CANT SEE THEM IN OUR SYSTEM.

If you just drop your samples off, we can't see whose they are. We have no information for them at all. And if you don't talk to us about your samples, we also can't notify you that you forgot to Check In.
If you do not follow procedure, we cannot be held responsible for you not getting your data in a timely manner!
Your samples will not be placed in the queue to be run until they have been Checked In!
Any Questions?