



Chemical Hygiene Plan Section C: Standard Operating Procedures and Work Practices

I. Brief Description of Research

Characterization equipment located in room MM107 include a Raman 300 microscope – Witec , an atomic force microscope (AFM) MFP-3D Origin 300 - Asylum Research, and an electron paramagnetic resonance (EPR) - Bruker. This equipment is used in the characterization and post evaluation of inorganic and organic materials, and includes novel thin films, functional ceramics and composites, and polymers among others.

MM107 is a characterization facility. Sample preparation is facilitate in a hood when this involve solvents or volatile compounds. Gloves, eye protection must be wear.

II. Standard Operating Procedures

- No personnel will use instrumentation without proper instruction.
- No machines will be left unattended while in operation.
- Complete shutdown procedures will be understood.
- Protective gloves, goggles, glasses, etc. will be worn as necessary.
- All users of this lab have taken safety and waste management training.
- Laser safety training is required for some instruments.
- All personnel will be aware of the gases and chemicals in the facility.
- All chemicals shall be handled in the fume hood areas.

III. Chemicals

Gases: N/A

Full Chemical Name	Location	Comments

Chemicals

Full Chemical Name	Location	Comments
Ethanol	MM107	Solvent to rinse tweezers, clean sample holders

Chemical are used for research and stored in these labs when not in use.

IV. Location of Protective Equipment

- Gloves are located in the bench close to the sink in room MM107.



- Each authorized user carries his/her own protective glasses.
- Additional safety glasses, and shields, are located in label shelves for the particular application .
- First aid emergency kits are located in room MM107.
- MSDS sheets are located adjacent to the chemical hygiene plan.



STANDARD OPERATING PROCEDURES
DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
LABORATORY SAFETY MANUAL

SOP Part 2, List of Common Chemicals – Provide a list of the most commonly used chemicals in the laboratory along with hazard information, compatibility, and special handling instructions for each chemical listed. An example of this type of information may be obtained at the following URL:

http://www.brown.edu/Administration/restricted/example_sop2.doc

Assistance and resources for determining hazard information, and compatibility, may be obtained by contacting the University Chemical Hygiene Officer at 863-1737 or by browsing the resources available at the Brown University EHS website:

<http://www.brown.edu/Administration/EHS/lab>

This form allows for the listing of 25 chemicals. If you have more than 25 commonly used chemicals, additional templates may be obtained at the following URL:

http://www.brown.edu/Administration/restricted/sop2_template.doc

SOP PART 2 - LIST OF COMMON CHEMICALS



LIST OF COMMON CHEMICALS - CONTINUED

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Ethanol	Hazardous in case of skin contact (irritant), of eye contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of ingestion.	Reactive with oxidizing agents, acids, alkalis	<p>Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, acids, alkalis, moisture.</p> <p>Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame). Do not store above 23°C (73.4°F).</p>



STANDARD OPERATING PROCEDURES
DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
LABORATORY SAFETY MANUAL

SOP Part 3, Equipment - Provide information relevant to any piece of equipment or instrument that requires special safety considerations to operate. This may include, but not be limited to, instructions for conducting high pressure reactions, using compressed gas cylinder manifolds, distillations, calibration of safety equipment, work with high voltage equipment, power tools, etc. An example of this type of information may be obtained at the following URL:

<http://www.brown.edu/Administration/restricted/sop3.doc>

Assistance and resources for determining which pieces of equipment may require special instruction within the SOP may be obtained by contacting the University Chemical Hygiene Officer at 863-1737.

SOP PART 3 – EQUIPMENT

Witec Raman 300 Microscope (Basic Operation Instructions)

Standard Operation Procedure Instructions provided to authorized user's of the WITEC =A300M+; Confocal Raman Microscope

NOTE: this is a basic operation instruction, for more information, please refers to the manual.

1. Users must reserve the instrument time in advance on the IMNI-Core Facility Raman Calendar. Sign into the logbook located in MacMillan 107, next to the instrument. Report equipment malfunctions to Hector Garces. Indicate any unusual instrument behavior on the logbook.
2. Turn on the laser (shutter should be closed) from the switch at the back of the power supply by turning the key to the ON position. It will take some minutes for the laser to stabilize, wait for the power light to illuminate.
3. Check initial microscope settings: BF left position, camera IN, and microscope coupler with filter set or in white light.
4. The instrument is operated using the Witec Control software on the desktop (Control Five). Log in into the computer using your Brown USERNAME and PASSWORD. Open Control Five software.
5. Upon opening the software, the instrument will go through a self-check. The events will be displayed on the dialogue box in the upper central part of the window. Each operation should be checked as successful to ensure the instrument works well. If any of the initialization steps are recorded as unsuccessful, **DO NOT** try to run the instrument, inform the lab supervisor.
6. After successful initialization, calibrate instrument using a silicon sample and observing the LO phonon line at about 521cm^{-1} .
 - Under Witec control, check configuration/Raman and choose CCD1 or CCD2 (CCD1: 457 nm, 532 nm, 633 nm. CCD2: 785 nm).
Note: when using the 785 nm laser the NIR 400 coupler should be IN (push rod out)
 - Place the sample on the stage.
 - Focus the sample moving the microscope Z control. For safety purposes move the Z control up first to avoid crashing the objective. Focus your sample using the small magnification

objective first and progress to high magnification objectives. The camera should be in.

- Press Auto-brightness (right side video screen). While focusing look at the sample on the video screen to determine if you are in focus.
 - Installed the **LASER GUARD** on the alpha 300 system. The laser guard is designed to shield the user from stray light scattered by a sample during confocal Raman measurements. The laser guard is magnetically held; make sure it is attached properly.
 - Once focused, turn the filter wheel to the corresponding laser filter, move BF to the right, and push out the rod (camera out) to allow the laser light to illuminate the sample and the light to be collected back into the spectrophotometer. **LASER SHUTTER** is still close and the **LASER GUARD** installed on the alpha 300 system.
 - Click on run oscilloscope and open laser shutter. Determine if you are getting any peaks. Optimize the counts by adjusting the focus (z up and down).
 - Once satisfied with the number of CCD counts, stop the oscilloscope mode, acquire a single spectrum. Close **LASER SHUTTER** and remove the sample. You are now ready to run your samples.
7. Place sample to test on the sample stage and focus by observing the image on the video screen. Once in focus, move BF to the right, choose the Raman filter, and pull out the push rod (camera out) and run the oscilloscope. Change parameters on the left hand of the screen until the signal/peaks observed are satisfactory.
8. Most common runs on the instruments:
- Single scans: choose how many scans you want to accumulate, integration time, and then click on acquire single scan.
 - Image scans: choose your integration times, geometry, type of scan and then click on acquire scan. To acquire images prior knowledge of the characteristics peaks of the sample is needed. Create filters for the peaks in the filter box and lock it to allow integration of the peaks for the images.
9. Once done with the experiments:
- Raise up the microscope using the focusing button
 - Save file on your personal folder. Do not save your files locally. Save your data files into the I:Drive. This can be found by navigating to the File Explorer > This PC.
 - Once you have completed your work on the Raman, go to any non-instrument connected computer, where you can save/move your data to your own storage, such as your own Google Drive or network storage. (If you are using wireless or off-campus, you will first need to connect to Brown VPN). (For external users it is advised to save the data at the desktop, email the data, and delete the data collected after done with the experiments).
 - Close out the software.
10. Turn **LASER OFF** by turning the key to the off position (switch at the back of the power supply). Shutter should be closed at this point
11. Remove sample from microscope stage
12. Sign out on the logbook

NOTE 1: When switching lasers align the pinhole (top of video camera) using front and left knobs to move positions in **X** and **Y** directions. Use silicon chip to maximize the signal.

PRECAUTIONS:

The alpha 300 microscope may use different lasers during operation. Most of these laser are class 3B due to their power or wavelength, the free beam will never appear outside the microscope if the system is operated correctly at all times. Additional, personal protective equipment should be wear. The safe use of lasers requires that all laser users, and everyone near laser systems, are aware of the dangers involved during operation.

Direct eye contact with the output beam from a laser will cause serious damage to the eye. In addition to the main beam, smaller beams can be present at various angles near the laser system formed by specular reflections of the main beam at polished surfaces such as lenses or beam splitters. While weaker than the main beam, these reflections can still be sufficiently intense to cause eye damaged.

Laser sources are power enough to burn skin, and ignite flammable substances such as alcohols, gasoline, and other solvents. The user is advised to follow the precautions below:

- Observed all safety precautions in the operation manual, and use extreme caution when using solvents in the area of the laser.
- Limit access to the laser and microscope to qualify users with complete the safety lasers guidelines and are aware of the dangers involved during operation of lasers.
- Never look directly into the laser source, optical fibers, or laser light scattered from any reflective surface.
- Never open the laser housing.
- All experimental setups must be maintained below eye level to prevent beam-eye contact.
- Laser must be use in an enclosed room. Use curtain to isolate the area during operation of the instrument.
- Post warnings signs in the area of the laser beam at all times to alert those present.
- Advise all users of these precautions.
- The laser is operated in the IMNI/NanoTools Facility , MacMillan Hall Room 107. This room is card access, which is provided to authorized users, after satisfactorily trained by the Lab Supervisor, and EHS training compliant. Do not intent to work with someone that is not authorized to be in the room. Only authorized users are allowing to operate the instrument.
- While wearing laser safety glasses be extremely careful since they can prevent the operator from seeing the beam
- Identify the signs that alerts the operator to the danger of exposure to hazardous laser radiation
- Identify the warning label that indicates the position of the aperture as well as the direction in which the laser beam will be emitted
- Identify the classification of the laser product

Orientation MM107

Orientation in the NanoTools facility MM107

- No personnel will use instrumentation without proper instruction
- No machines will be left unattended while in operation
- Complete shutdown procedures will be understood
- Protective gloves, goggles, glasses, etc. will be worn as necessary
- All users of this lab have taken safety and waste management training
- Laser safety training is required for some instruments. Requirements and recommendations for proper use of lasers must be follow at all times
- All personnel will be aware of the gases and chemicals in the facility

- All chemicals shall be handled in the fume hood area
- Chemical hygiene plan location
- Location eye wear
- Basic operation instructions (Witec Raman)
- Practices approved by laser safety officer
- Start up and shut down procedures of instrumentation
- Use of the analysis software
- Resources for sample preparation: hood, optical microscope, tweezers, etc

At Orientation/Training:

- Log in into the computer using your Brown USERNAME and PASSWORD.
- After successful training, the NanoTools Lab Supervisor will request Brown Card access to MM107. User will receive a confirmation when their card access has been provided by Brown PSO. (expect 24-48 hours)

Calendar – Reserve Instrument

- Authorized new user will receive an email (from imni-core-facilities@brown.edu) inviting to view the NanoToolsFacility Instrument Calendar. User should select the ‘View Your Calendar’ link. If using Gmail, the calendar will automatically add to your Google calendar. Otherwise reply asking for link.

Instrument Google Group (Listserve)

- Authorized new user

BRUKER EMX-PLUS EPR

Turning on the EPR at Room Temperature

1. Turn on H₂O valves by moving the yellow handles behind the console.
2. Switch on the heat exchanger. Temperature reader should appear at front of the instrument digitally.
3. Switch on the console by pushing the blinking blue button. The blue light will become steady
 - a. The bridge on the desk will turn on after the console is turned on: the power switch on the bridge can be maintained always on. When the blue light is stable, the bridge is on standby mode.
4. The cavity should already be connected to the bridge. If not:
 - a. Screw on the four gold screws from the bridge to the top of the cavity. Place the black wedges along the side of the cavity arm to add extra support.
 - b. Plug the modulator to the cavity. This port align directly to the cavity. Once aligned, turn the silver cap clockwise to lock the modulator into place.
 - c. Place the iris arm into the iris screw. This may require moving the iris arm until it can be aligned properly. Using software, bring the iris motor to the lowest stopping point: Tuning panel/options/iris rundown/disconnected (yes). Attach to iris screw. Once the arm is in the iris screw, you must tighten the arm in place using the gold screw connected to the table and also the iris screw (plastic white screw) should be tightened as well (clockwise).
5. Magnet can be turned on when you are ready to use the EPR. The magnet is located below the console.

Turning on the EPR at Low Temperature

1. Turn on H₂O valves by moving the yellow handles behind the console.
2. Switch on the heat exchanger. Temperature reader should appear at front of the instrument

- digitally.
3. Switch on the console by pushing the blinking blue button. The blue light will become steady
 - a. The bridge on the desk will turn on after the console is turned on: the power switch on the bridge can be maintained always on. When the blue light is stable, the bridge is on standby mode.
 4. The cavity will be connected to a cryostat. The cryostat should have been placed under vacuum the night before usage. If using $\text{He}_{(l)}$, cryostat should be removed from vacuum at this point. If $\text{N}_{2(l)}$, leave cryostat on pump.
 - a. The cavity and cryostat system should already be connected to the bridge. If not, slide the cryostat down the silver ramp until the two pins are inserted into the corresponding sockets. When the pins are in, screw the cryostat onto the ramp using the two gold screws.
 - b. Connect the cavity to the bridge using the four gold screws from the bridge to the top of the cavity. You do not need the black wedges along the side of the cavity arm to add extra support.
 5. Plug in the modulator to the cavity. This port aligns directly to the cavity. Once aligned, turn the silver cap clockwise to lock the modulator into place.
 6. If you need to attach the cavity, see section Room temperature #4.
 7. Connect the sensor cord to the cryostat. This plug is located in the back. The sensor cord can only be inserted one way so make sure to align the pins first.
 8. Connect the waveguide to the house nitrogen port. Open house nitrogen half way once the tube is connected.
 9. Connect the controller cable to the transfer line. The controller cable has a red dot that will align with the red dot on the transfer line.
 - a. At this point, we need to turn on the controller.
 - i. The gas flow light will be blinking red. Wait until the red blinking stops. When it stops, turn off controller and turn it back on to make sure the gas flow light really stopped blinking.
 - b. We need to open the gas flow all the way to 99.9
 - i. Double click on the button underneath gas flow and use the up arrow to check the value to 99.9.
 - ii. Wait until the valve is completely open.
 - c. Slowly place the transfer line into liquid helium or liquid nitrogen dewar. For $\text{N}_{2(l)}$, should see some pressure in balloon before opening dewar. Make sure the brass nut is loose on the dewar.
 - i. Take the protective shield off the transfer line
 - ii. Slowly lower the transfer line into the dewar, until you see that there is gas flow at the tip. (Have a vial of ethanol nearby to check).
 - iii. Once you see bubbles flow, continue lowering the transfer line until it is all the way in the dewar. Lowering should take about 2-3 min, go slowly.
 - iv. Check to see if there is still gas flow. If there isn't, that means the tip may have had some moisture frozen it. If there are still bubbles, tighten the brass nut on the dewar.
 - d. Insert the tip into the cryostat. Do not tighten.
 - e. Connect the clear pump tubing to the clear tubing on the transfer line. Turn pump on. The silver knob on the pressure box should be completely open. You should notice the pressure dial go to low pressure.
 - f. Wait until the pressure starts increasing (the needle will move to the right, the gas flow (red) and exhaust (silver) markers will shoot up, and the pump will change its sound). Once the pressure is positive, tighten the connection between the transfer line and the cryostat.
 - g. Turn on PID to auto.
 - h. Turn on the heater to auto.
 - i. Click set and use the arrows to set it to the temperature you want (10 K for $\text{He}_{(l)}$, 82 K for $\text{N}_{2(l)}$, etc).
 - i. Wait for ~ 45 – 60 minutes until the temperature drops and stabilizes at where you set it. As the temperature is dropping, you will need to continue tightening the connection between the

transfer line and the cryostat.

- j. After about 1 hour, if the temperature has stabilized, change the gas flow to auto.
10. Magnet can be turned on when you are ready to use the EPR. The magnet is located below the console.

Computer Program – Xenon

The computer program we will use to take EPR spectra is called Xenon. There is a shortcut located on the desktop. Double click to open.

1. Click on acquisition/connect to spectrometer
Click the ok button to connect (make sure server name is localhost). Sometimes you may get an error message; this is normal, just click ok again to connect. If the problem still persists, restart the computer and try to connect to the spectrometer again.
2. Click the tuning button located on the bottom left corner. This will open the microwave bridge tuning dialog box.

The current status of the bridge should be on standby. To begin tuning, click the tune button.

You should see a dip in the box; if you cannot find it, move the frequency to 9.8 Hz. Once the dip is visible, click the Auto Tune button (Up or Down). This function will automatically tune the frequency, the current diode, and APF.

If you get an error message, click on the operate mode to check the diode current. If it is at 400, the value is too high. Move the iris down by clicking on the down arrow. Click tune, and try auto tuning again. Continue to lower the iris in operate mode if the error continues to occur.

Once auto tune is complete, you are ready to place a sample into the cavity.

If auto tune does not work, you can manually tune. Open the microwave bridge tuning dialog box and follow the procedure below:

1. Switch the microwave to tune mode.
 2. Attenuation should be moved to 25dB for a good dip.
 3. Turn the reference arm off.
 4. Try to center the dip with the center line by changing the frequency.
 5. If the mode pattern amplitude is too small, increase the microwave power in 1dB steps by decreasing the attenuation. Try to get the tune dip to fill much of the display.
 6. Insert sample into cavity.
 7. Maximize the depth of the dip by lowering or raising the iris.
 8. Retune the dip by adjusting the frequency.
 9. Set the attenuation to 30 dB and switch to operator mode. The diode current should be near zero (2-10 uA). Do this by decreasing the attenuation in 1 dB steps. If the diode current starts to rise, click either the up or down iris button to maintain minimum diode current.
 10. Adjust the bias by turning the attenuation to 60 dB. Click the reference arm on. Adjust the bias until diode current is 200 uA.
 11. Decrease the attenuation to 30 dB and switch to tune mode. While the dip is in the center of the display, adjust the signal phase slider until the dip is negative.
 12. Fine-tune the microwave source frequency by clicking operate and fine tune.
3. Taking a spectrum at room temperature

On the microwave bridge tuning dialog box, click tune. You should again see the dip.

Once you place your sample in the cavity, you should see the dip shift. From here, click on operate/fine tune.

Depending on what you are analyzing, click on organic radical or transition metals. Then, hit the play button located on the left side of the screen.

To save the file, hit the button containing the red swirl next to the FS (full size) button. You can either save onto disk or store in memory. It is best to save onto disk.

Filenames cannot have spaces or any special characters; the title may contain a more detailed description of your experiment.

4. Taking a spectrum at low temperature

All steps are the same as in 3 (taking a spectrum at room temperature) but you will need to use a top hat to ensure that there is no air leakage:

- i. Take your EPR tube and place the top hat on. Loosen the screw so the EPR tube can be moved up and down. Place the EPR tube in a small dewar containing liquid nitrogen to freeze the sample.
- ii. When you are ready to place the sample into the cavity, you must turn off the pump. Wait until the pressure on the pressure reader is positive (red marker will jump up). At this point, remove current sample or top hat and quickly place your sample in. remember to wipe the sample with kimwipes to prevent frost. Place top hat down all the way and then slide EPR tube down the cavity. When the EPR is inserted all the way to the bottom, tighten the screw cap clockwise. Turn on the pump and wait until the temperature is stabilized.
- iii. Once the temperature is stabilized, auto tune, take spectra, and save your sample the same way you would with a room temperature sample.

Turning off the EPR at low temperature

1. Turn pump off. When pressure is positive, take sample and top hat out and put black stopper in sample holder.
2. Put bridge in standby, using computer software
3. Turn off magnet.
4. For N_{20} , remove cryostat pump by first closing valve on cryostat, then on vacuum and then detach metal tube connecting it to pump (careful not to drop o-ring).
5. Loosen connection between transfer line and cryostat. Remove transfer line from cryostat.
6. Detach transfer line from pump via clear tubing.
7. Put protector on transfer line.
8. Using cryogloves and two people, remove transfer line from cryogen. Set on floor for the time being. When finished, put on hook.
9. Turn off controller. Remove black modulation cable from transfer line. If removing cryostat, see step 11.
10. Disconnect software. Turn off console, heat exchanger, cold water, house nitrogen.
11. If using the next day, reconnect cryostat to pump. Turn on pump power. Open valve on vacuum. When pressure reaches $< 1.0 \times 10^{-5}$, open valve on cryostat. Vacuum overnight.
12. If you need to remove cryostat:
 - a. After removing modulation cord from transfer line, disconnect iris motor.
 - b. Disconnect house nitrogen. Loosen waveguide screws all the way. Move bridge farther to the left.
 - c. Loosen cryostat. Hold rear valve; begin to pull cryostat out. Easier to hold bottom of cryostat

when it's most of the way out. Remove completely.

Turning off EPR at room temperature

1. Same as for low temperature, except for all steps related to cryostat, cryogen.

MFP-3D Origin AFM Asylum Research (Basic Operation Instructions)

This is a basic operation instruction, for more information, please refer to the manual.

I. Loading the cantilever

1. Load tip holder into loading apparatus; it offers stability when loading tips. Tip holder has a machined ball bearing port that snaps into place using the lever on the apparatus and head port. The tip holder also works well for rinsing the tip holder after imaging.
2. Slide cantilever chip under tongue of tip clamp. Position cantilevers so that they are centered in the clear trapezoidal shaped window. The tip holder was designed to be resilient to scratching from tweezers. DO NOT push tip chip substrate all the way back in the pocket: it can cause the chip to lift off the floor of pocket which then compromises the deflection signal
3. Tighten the screw in the center of the stainless steel tongue assembly (finger tight).
4. Put tip holder into the MFP-3D head. Make sure the tip holder is parallel to the top of the head; otherwise it is not properly seated.
5. Open the MFP-3D software; the launch icon is on desktop
6. Program is loaded when Igor says its ready (lower left hand side of window). Do not interrupt the loading, wait for program to be fully loaded.

II. Laser Alignment via top view optics CCD

1. Turn CCD camera on (look for ccd icon on lower left of MFP-3D software); focus camera on tip using focus adjustment wheel on back of AFM head. The two knobs in the back of the head are for x- and y- position adjustment of the camera; the large wheel in the housing is for focus. CCW focuses down, CW focus up (in the plane of the tip).
2. Follow the laser spot in the camera. Move the laser back onto the support chip to locate it in the camera view, and then move it towards to the end of the desired cantilever. This results in a 'Sum' signal on S & D meter. Move the laser in x- & y- to maximize the sum.
3. Adjust Photodetector (PD). Use the thumbwheel on the left side of the head to adjust the PD, whose value is seen in the Sum & Deflection meter. If you have red in the deflection meter, you need to go negative to adjust to ca. zero. The top of the head is labeled to tell you what way to go (positive or negative). When aligning the laser, just pay attention to the Sum and Deflection meter voltage values. For contact mode imaging, the deflection should be slightly negative (slightly blue); for AC mode, it should be zero (no color). At any point, clicking the 'Withdraw' button will disengage the tip, retracting the piezo to a reading of 0.0V (mostly blue on the meter).
4. At this point, you are ready to engage the tip, depending on what imaging mode or application you are doing. If you are working in fluid, you may have to wait out an equilibration period. You will see significant drift in the deflection value without equilibration (seen in deflection value of the S & D meter).

NOTE: When adjusting any of the alignment thumbwheels, if you feel resistance turning them, don't force it. It's probably at the end of its travel. If you over torque it, it becomes very difficult to reverse its direction.

III. Contact Mode Operation:

1. From Main tab in Master Panel, select 'Contact' mode from the imaging mode pull-down menu. Image channel selection: Choose what kind of data you want shown during imaging (i.e image channels).
2. Click on a channel tab in the Master Channel Panel. At the 'Input' pull down menu to choose what data you want for contact mode imaging (i.e, height, deflection, lateral, etc). If you want lateral data, but don't change these tabs from the default AC mode imaging windows (i.e, Ht, AM & Ph; seen below), you are just going to have some noisy looking image that isn't representative of your surface.
3. Adjust deflection value to slightly negative for contact mode (deflection meter is a little blue, like 0 to -0.20 V). This is done to compensate for a ca. zero deflection once tip is engaged.
4. On the Main tab, adjust these parameters (dependent on sample): Set point (force): minimum of whatever the deflection value on the S&D meter is (gentle, low force) to 1.0 V (higher force; the larger the value, the larger the applied force); if you need to know what kind of force you are applying, you should determine the spring constant (k) using the procedure described in Section IV.
5. Integral gain (I): 5 to 10,
6. Scan rate: 1 Hz.
7. Scan angle, resolution (scan points & lines) and image size is up to you.
8. Tip Engagement: click engage in the Sum and Deflection window. You will notice the z-piezo voltage maximizing all the way red (150 V), indicating the z- piezo is fully extending in anticipation of engagement of feedback servo.
9. Slowly turn the thumbwheel counter-clockwise (towards the surface) until the deflection value on the S&D meter equals your user defined set point, AND you here a chime sound (computerspeaker volume must be on). You will also notice the z-piezo voltage meter goes from 'railed' in the red (150 V), to some lesser value. If you have the volume up on the transducer (front of controller, headphones plugged in), you can hear a frequency change immediately before engagement occurs.
10. Carefully adjust the front thumbwheel down to around zero (~ 70 V; no color in z-voltage meter). This indicates the piezo is in the middle of its z- range (~ 7.5 μm standard head; ~ 14 μm extended head). At this point the tip is engaged and just sitting on the surface- it does not begin rastering until you tell it.

NOTE: If the z-piezo voltage is railed all the way blue (-10 V, upon clicking simple engagement), the piezo is fully retracted because it thinks it has crashed. This indicates a false engagement and adjustment must be made. Do this by clicking 'Stop' and adjusting the set point voltage or drive amplitude to allow extension of the piezo. This often occurs when imaging in fluids/ high humidity.

11. To begin scanning, click the 'Do Scan' button on the Main Tab. After a brief moment imaging will begin.
12. Tune the parameters with set point force, integral gain (I) and scan rate. You can use the arrow clickers to adjust parameters, opposed to typing values in.

NOTE: Image quality can be monitored by image resolution and amount of noise in the line traces (located below images). Rotating the scan angle results in counter clockwise rotation with positive rotation angle values.

When finished scanning, see shut down procedure below

IV. Shut down.

1. Raise tip from nanoposition head
2. Turn off light source
3. Turn off the vibration isolation unit

4. Turn laser to the off position using the key
5. Close the asylum research program
6. Sign in the logbook

STANDARD OPERATING PROCEDURES

DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
LABORATORY SAFETY MANUAL

SOP Part 4, Carcinogens and Reproductive Toxins - List all Select Carcinogens and Reproductive Toxins used within the laboratory and identify a designated area for use (i.e., such as one particular chemical exhaust hood) and specific control methods to be used to limit exposure.

Definition of Select Carcinogen - Select Carcinogen means any substance which regulated by OSHA as a carcinogen; or it is listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (NTP - latest edition); or it is listed under Group 1 ("carcinogenic to humans") by the International Agency for research on Cancer Monographs (IARC)(latest editions); or it is listed in either Group 2A or 2B by IARC or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:



- After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10mg/m³;
- After repeated skin application of less than 300 (mg/kg of body weight) per week; or
- After oral dosages of less than 50 mg/kg of body weight per day.

Definition of a Reproductive Toxin – Reproductive Toxin means chemicals which affect the reproductive capabilities which affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).

SOP PART 4 – CARCINOGENS AND REPRODUCTIVE TOXINS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Designated Area</i>	<i>Control Measures</i>

STANDARD OPERATING PROCEDURES

DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN & LABORATORY SAFETY MANUAL

SOP Part 5, Spill Equipment - List or the location of spill equipment available to your laboratory personnel for small incidental spills.

SOP PART 5 – SPILL EQUIPMENT

SOP Part 6, Emergency/safety Equipment - List the location of emergency eyewash stations, drench showers, fire alarm pull stations, fire extinguishers, first aid kits and other safety related devices available

to the laboratory staff.

SOP PART 6 – EMERGENCY/SAFETY EQUIPMENT

Location of nearest Eyewash Station: MM107, by the main door

Location of nearest Emergency Drench Shower: MM107, by the main door

Location of nearest Fire Alarm Pull Station: N/A

Location of nearest Fire Extinguisher: N/A

Location of nearest First Aid Kit: MM107, shelf

Other Emergency/Safety Equipment Locations:

- Emergency shower/eye wash sign, location: MM107
- Fire extinguisher sign, location: N/A
- Empty container disposal brochure guide, location: MM107



STANDARD OPERATING PROCEDURES
DEVELOPED TO SUPPLEMENT THE BROWN UNIVERSITY CHEMICAL HYGIENE PLAN &
LABORATORY SAFETY MANUAL

SOP Part 7, Housekeeping - Provide information concerning the housekeeping rules specific to this laboratory including, but not limited to, methods for laundering laboratory coats.

SOP PART 7 – HOUSEKEEPING

1. Glass disposal container, location: N/A
2. Sharps container, location: N/A



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SOP Part 8, Additional Information - Provide additional safety information relevant to the work being conducted in this laboratory that is not already covered in the Brown University Chemical Hygiene Plan or in Parts 1-7 of this SOP. Include a complete list of chemicals used in this laboratory as an attachment (if not already provided).

SOP PART 8 – ADDITIONAL INFORMATION



INFORMATION AND INSTRUCTION

Signatures - All persons working (paid or unpaid) in the laboratory must sign the cover page of Section 3. By signing Section 3 the individual is acknowledging that he or she has read and understood the entire Brown University Chemical Hygiene Plan and Standard Operating Procedures.

Examples and Template - The Office of Environmental Health & Safety has published two example SOP's at the following URL:

http://www.brown.edu/Administration/EHS/restricted/sop_001.pdf

http://www.brown.edu/Administration/EHS/restricted/sop_002.pdf

In addition, the Office of Environmental Health & Safety has provided a template for use in developing laboratory specific SOP at the following URL:

http://www.brown.edu/Administration/EHS/restricted/sop_003.pdf

Assistance in Developing the Standard Operating Procedures – The University Chemical Hygiene Officer 863-1737 is available to assist in the development of SOP's. In addition, the Brown University Office of Environmental Health & Safety offers several resources to aid in the development of the laboratory specific SOP at the following URL:

<http://www.brown.edu/Administration/EHS/lab>

Renewal Instructions - Laboratory specific SOP's are required to be reviewed and if necessary, revised annually. If procedural changes have not occurred or if new chemicals have not been introduced to the research the renewal process may be as simple as completing a new cover page along with the required signatures, and resubmitting the entire package to the EHS office to the attention of the University Chemical Hygiene Officer, Box 1914. Notifications for annual SOP renewal will be sent automatically to each Laboratory Supervisor by the Office of Environmental Health & Safety.

Distribution and Availability – All laboratory employees shall have available the contents of this manual, the laboratory specific SOP, a copy of the Laboratory Safety Standard (29 CFR 1910,1450), and all appendices.



Chemical Hygiene Plan Section C: Standard Operating Procedures and Work Practices

I. Brief Description of Research

Characterization equipment located in room PL104 includes a Bruker D8 Discover high-resolution x-ray diffractometer and Bruker 2D D8 Discover x-ray diffractometer. These equipment are used in the characterization and post evaluation of inorganic and organic materials, and includes novel thin films, functional ceramics and composites, coatings, powders, and polymers among others.

PL104 is a characterization facility. Gloves, eye protection must be wear as necessary.

II. Standard Operating Procedures

- No personnel will use instrumentation without proper instruction.
- No machines will be left unattended while in operation.
- Protective gloves, goggles, glasses, etc. will be worn as necessary.
- All users of this lab have taken safety and waste management training.
- All personnel will be aware of the gases and chemicals in the facility.

III. Chemicals

Gases:

Full Chemical Name	Location	Comments
Nitrogen (N ₂)	PL104	Inert gas used for some applications.

Chemicals: N/A

IV. Location of Protective Equipment

- Gloves are located in the bench in room PL104.
- Each authorized user carries his/her own protective glasses.
- Additional safety glasses, and shields for the particular applications.



STANDARD OPERATING PROCEDURES
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SOP Part 2, List of Common Chemicals – Provide a list of the most commonly used chemicals in the laboratory along with hazard information, compatibility, and special handling instructions for each chemical listed. An example of this type of information may be obtained at the following URL:

http://www.brown.edu/Administration/restricted/example_sop2.doc

Assistance and resources for determining hazard information, and compatibility, may be obtained by contacting the University Chemical Hygiene Officer at 863-1737 or by browsing the resources available at the Brown University EHS website:

<http://www.brown.edu/Administration/EHS/lab>

This form allows for the listing of 25 chemicals. If you have more than 25 commonly used chemicals, additional templates may be obtained at the following URL:

http://www.brown.edu/Administration/restricted/sop2_template.doc

SOP PART 2 - LIST OF COMMON CHEMICALS



LIST OF COMMON CHEMICALS - CONTINUED

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Incompatibilities</i>	<i>Handling Instructions</i>
Ethanol	Hazardous in case of skin contact (irritant), of eye contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of ingestion.	Reactive with oxidizing agents, acids, alkalis	<p>Keep locked up.. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, acids, alkalis, moisture.</p> <p>Store in a segregated and approved area.Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame). Do not store above 23°C (73.4°F).</p>



STANDARD OPERATING PROCEDURES

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SOP Part 3, Equipment - Provide information relevant to any piece of equipment or instrument that requires special safety considerations to operate. This may include, but not be limited to, instructions for conducting high pressure reactions, using compressed gas cylinder manifolds, distillations, calibration of safety equipment, work with high voltage equipment, power tools, etc. An example of this type of information may be obtained at the following URL:

<http://www.brown.edu/Administration/restricted/sop3.doc>

Assistance and resources for determining which pieces of equipment may require special instruction within the SOP may be obtained by contacting the University Chemical Hygiene Officer at 863-1737.

SOP PART 3 – EQUIPMENT

Bruker 2D-XRD (Basic Operation Instructions)

1. Sign in logbook
2. Open diffract measurement center
3. Set current and voltage to 40 kV and 40 mA respectively
4. Set start angle and stop angle for the scan (do not use a start angle lower than 20, 2 Theta)
5. Click start to initiate the scan
6. Save the scan when done
7. If done with measurement, set voltage and current to 20 kV and 5 mA
8. Close diffract measurement program
9. Sign in the logbook

Bruker Hi-Res XRD (Basic Operation Instructions)

1. Sign in logbook
2. Open diffract measurement center
3. Set current and voltage to 40 kV and 40 mA respectively
4. Set the correct high of the sample by performing z and a rocking curve scans
5. Set start angle and stop angle for the scan
6. Click start to initiate the scan
7. Save the scan when done
8. If done with measurement, set voltage and current to 20 kV and 5 mA
9. Close diffract measurement program
10. Sign in the logbook



STANDARD OPERATING PROCEDURES

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SOP Part 4, Carcinogens and Reproductive Toxins - List all Select Carcinogens and Reproductive Toxins used within the laboratory and identify a designated area for use (i.e., such as one particular chemical exhaust hood) and specific control methods to be used to limit exposure.

Definition of Select Carcinogen - Select Carcinogen means any substance which regulated by OSHA as a carcinogen; or it is listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (NTP - latest edition); or it is listed under Group 1 ("carcinogenic to humans") by the International Agency for research on Cancer Monographs (IARC)(latest editions); or it is listed in either Group 2A or 2B by IARC or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:

- *After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10mg/m³;*
- *After repeated skin application of less than 300 (mg/kg of body weight) per week; or*
- *After oral dosages of less than 50 mg/kg of body weight per day.*

Definition of a Reproductive Toxin – Reproductive Toxin means chemicals which affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).

SOP PART 4 – CARCINOGENS AND REPRODUCTIVE TOXINS

<i>Full Chemical Name</i>	<i>Hazards</i>	<i>Designated Area</i>	<i>Control Measures</i>

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SOP PART 6 – EMERGENCY/SAFETY EQUIPMENT

Location of nearest Eyewash Station: N/A

Location of nearest Emergency Drench Shower: N/A

Location of nearest Fire Alarm Pull Station: N/A

Location of nearest Fire Extinguisher: PL104 close to main door

Location of nearest First Aid Kit: N/A

Other Emergency/Safety Equipment Locations:

- Fire extinguisher sign, location: PL104



STANDARD OPERATING PROCEDURES
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SOP PART 7 – HOUSEKEEPING

1. Chemical hygiene plan and laboratory safety manual
2. Basic operation instructions for instrumentation



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SOP PART 8 – ADDITIONAL INFORMATION

A large, empty rectangular box intended for providing additional safety information and a list of chemicals used in the laboratory.



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