Section 5.19 Title: Using and Maintaining the Infrared Spectrometer

Revision Date: 11/01/19

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Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No

___ Carcinogen ___ Reproductive Toxin ___ High Acute Toxicity

Does this procedure require medical surveillance? No

Does this require use of a fit-tested respirator? No

Brief Description of Procedure:

Collection of IR spectra. Maintenance of the IR instrument.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*

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Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Ethanol	Flammable, toxic

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

N/A

Exposure Controls: *(Check all that apply)*

PPE: _X_ Safety Glasses ___ Face Shield ___Chemical Splash Goggles

___Chemical Apron _X_ Gloves (Nitrile) _X_ Lab Coat

___Respirator (type) ___Other:

Engineering Controls:

___ Fume Hood ___Biosafety Cabinet ___ Glove box

___ Vented gas cabinet ___Other:

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

N/A

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills of EtOH may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training is required. Training is performed by the group member(s) responsible for this apparatus or another lab member they have approved. The procedure will be demonstrated at least once and new members will be supervised their first time.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry Signature: _____ Date: _____

Using and Maintaining the Infrared Spectrometer

IR Use:

ATR (Solid and Liquid Samples) Setup

1. Open the blue hatch on the Bruker Vertex 70 IR.
2. Remove the flat plate by pressing the lever while firmly pulling up on the front edge of the plate. Place the plate aside in a place where it won't be damaged.
3. Attach the ATR base (found in the black case in the cabinet below the Vertex 70 IR) where the plate was by lining up the prongs with the sockets and pressing down to snap it into place; you should hear a beep. Do not touch the screws on the ATR base as this can cause the beam to go out of alignment. If mistakenly adjusted, see the section "Troubleshooting: Peak Location Error" below.
4. Extend the black cylinders from each side of the base to press up against the windows.
5. Attach the overhead arm (also found in the black case) by placing it onto the base and screwing in both screws.
6. Wet a Kim Wipe with EtOH and wipe off the ATR crystal where sample will be placed. Also wipe off the bottom of the tip of the overhead arm. Do NOT use any fibrous cloth other than a Kim Wipe to wipe off the ATR crystal.
7. Allow the crystal and overhead arm to air dry to make sure no EtOH remains.
8. Open OPUS 7.0 on the desktop. Click "yes" to allow the program to make changes. The login password is OPUS.

Liquid Cell Setup

1. Open the blue hatch on the Bruker Vertex 70 IR.
2. Remove the flat plate by pressing the lever while firmly pulling up on the front edge of the plate. Place the plate aside in a place where it won't be damaged.
3. Attach the base plate with a liquid cell mount (found outside of and next to the black case in the cabinet below the Vertex 70 IR) where the plate was by lining up the prongs with the sockets and pressing down to snap it into place; you should hear a beep.
4. Bring a liquid cell into the glovebox along with two small Teflon stoppers. Do NOT touch the faces of the KBr plates so as not to create smudges.
5. Dispense your liquid sample into one of the holes on the liquid cell; do not dispense too much so as not to overflow.
6. Firmly press both Teflon stoppers into the two holes and bring the liquid cell outside of the Glovebox.
7. Attach the liquid cell to the liquid cell mount by lining up one of the metal plates of the cell with the groove on the mount and sliding the cell into it.

Collecting Data

1. After initializing, click the button with a green test-tube depicted on it.
2. Make sure that "MIR-ATR.XPM" is the loaded experiment for ATR samples. Under the "Basic" tab, which should be the first screen that opens, type an appropriate description for the sample, and specify the sample form (liquid or solid).
3. Under the "Advanced" tab, specify the filename (typically the same name as the sample description). Select an appropriate path for the data to be saved by pressing the ellipsis button next to the "Path" field.

4. The parameters under the “Advanced” tab should be as follows for the MIR-ATR experiment.
    - Resolution: 4 cm^{-1}
    - Sample Scan Time: 16 Scans
    - Background Scan Time: 16 Scans
    - Save Data From: 4500 to 600 cm^{-1}
    - Result Spectrum: Transmittance
5. If, for some reason, any of these parameters are changed, it is recommended that the MIR_ATR.XPM experiment be re-loaded to ensure all default parameters are set appropriately.
6. Click on the "Check Signal" tab and make sure that the interferogram shows a signal.
7. Check that your experimental parameters are acceptable for your measurement.
8. Go back to the “Basic” tab. Press the “Background Single Channel” button (you may choose to either leave the overhead arm up or down base on your preference). In the bottom right corner, the progression of the scans will be displayed in green. If you encounter an error in the collection, see the “Troubleshooting” section below.
9. Once all 16 background scans have been collected, deposit a small quantity of your sample onto the ATR crystal; for solid samples, do NOT touch the crystal directly with your spatula, as this may scratch it. Ensure that as much as the surface of the crystal is covered by your sample as possible. For liquid samples, 1-2 drops with a Pasteur pipet should be sufficient.
10. For solids, once the sample has been appropriately deposited, rotate the black knob on the overhead arm clockwise to press the tip of the arm against the ATR-crystal. Continue screwing the knob until a distinct click is felt. Do not attempt to screw the knob further down. For liquids, the overhead arm should be left up.
11. Press the “Sample Single Channel” button to collect the IR spectrum.

Data Analysis

1. Wait until all 16 scans have been collected before continuing.
2. At the top of the screen, press the “Baseline Correction” button (the mouse will display the function of the button when hovering the cursor over it). Press the “Correct” button at the bottom of the window.
3. At the top of the screen, press the button “Peak Picking” button. Adjust the sensitivity threshold appropriately; it should be at a transmittance value slightly less than the minimum intensity peak. Alternatively, press the “Start Interactive Mode” button to click and drag the threshold to an appropriate value. Press the “Peak Picking” button near the bottom to automatically peak pick the spectrum.
4. If some peaks were missed during the auto peak picking procedure, right click on the IR spectrum and press the “Single Peak Pick” option. Hover the mouse directly on top of the desired peak and left click to manually peak pick it. Right click anywhere on the spectrum to exit the "Single Peak Pick" mode.
5. If need be, the transmittance scale of the IR spectrum can be adjusted by right clicking on the spectrum and pressing the “Properties” option. Adjust the Transmittance axis display limits to your desired range; the wavenumber axis display may also be adjusted as desired.

6. Once you are happy with the IR spectrum, save the data as an OPUS format file (can be opened by OPUS again later if further analysis is required). Additionally, save the data as a .dpt file; click on the “Mode” tab to adjust the output file format to “data point table”. This is an ASCII format and can be used to manually plot your IR spectrum in Origin or equivalent software. Note that the raw data is saved as "filename".0, and your processed data will increment the file extension and save as "filename".1.
7. Finally, under “Print Setup”, set the printer to “Adobe PDF”, change the orientation to “Landscape”, and press “OK”. Press the “Quick Print” button at the top of the screen (looks like a purple printer) to save the file in a .pdf format.

Clean Up

1. Close out of the OPUS software.
2. ATR Clean Up
    a. Unscrew the black knob of the overhead ATR arm in a counterclockwise direction. Keep unscrewing until there is adequate space for cleaning the ATR crystal and the ATR arm.
    b. Wet a Kim Wipe with EtOH, and wipe off the ATR crystal, as well as the tip of the overhead ATR arm. Make sure that both the crystal and the arm tip are completely clean.
    c. Unscrew both screws that attach the overhead ATR arm to the base and place the overhead arm back into the black case where you found it.
    d. Contract both black cylinders pressed against the windows of the compartment. Remove the ATR base by pressing in on the lever and pulling up on the front edge firmly. Place the ATR base back into the black case where you found it and place the black case back into the cabinet below the Bruker Vertex 70 IR.
3. Liquid Cell Clean Up
    a. Remove the liquid cell from the liquid cell mount by sliding the cell out of the groove of the mount.
    b. Remove both Teflon stoppers and dispose of liquid sample appropriately.
    c. Rinse out the liquid cell with an appropriate solvent through one of the holes. Do not use H_2O, as this will dissolve the KBr plates. Do not use alcohol solvents to rinse out the cell for the same reason. Acetone is the recommended solvent unless acetone will cause precipitation to occur.
    d. Remove the liquid cell mount base by pressing in on the lever and pulling up on the front edge firmly. Place the liquid cell mount base back next to the black case housing the ATR base in the cabinet below the Bruker Vertex 70 IR.
    e. Blow a gentle stream of N_2 through one of the holes of the liquid cell to dry out any residual solvent before the next use.
4. Attach the flat plate you put aside at the beginning by lining up the prongs with the sockets and then pressing down on the plate to snap it into place.
5. Close the blue hatch on the compartment and sign the log book (kept on top of the IR instrument) with the date, your initials, what setup you used (ATR-IR, liquid cell, etc.), and putting a checkmark in the “clean?” column.

Troubleshooting: Peak Location Error

1. If you get an error during data collection that says “Peak location out of range, please save peak position : 577 Measurement file not found.”, follow the next steps.
2. Press the “Check Signal” tab.
3. At the top of the screen, press the “Full Scan” button, and wait a moment or two.
4. On the left of the screen, press the “Save Peak Position” button. You should now be all set to collect data without encountering any further errors.
5. Peak location: ~64000 cm^{-1}

Troubleshooting: Liquid Cell Disassembly/Assembly

1. It may be necessary to disassemble the liquid cell to properly clean it. To do so, unscrew the four metal screws found on the face of one of the plates of the cell.
2. Making sure to wear gloves, carefully separate the plates, making sure to not lose the KBr plates; only directly touch the KBr plates by the edges, not the faces.
3. Use a KimWipe wet with acetone (or a different appropriate solvent; note: no H_2O or alcohol solvents should be used to clean KBr plates) to wipe the plate clean. Repeat with the Teflon spacer as well as the other plate.
4. When dry, create a sandwich by placing the Teflon spacer in between both KBr plates.
5. Replace the KBr plate sandwich into the metal pegs of the back plate of the liquid cell.
6. Place the top metal plate of the liquid cell on top.
7. Gently use paper clips to line up the KBr plate holes properly by inserting the paper clips into the holes for injecting liquid into the cell.
8. Once lined up, leave the paperclips in. Tighten the four screws on the face of the front plate of the cell down. It is only necessary to tighten these finger tight; do NOT overtighten these screws, or the KBr plates could crack!
9. Remove the paperclips.

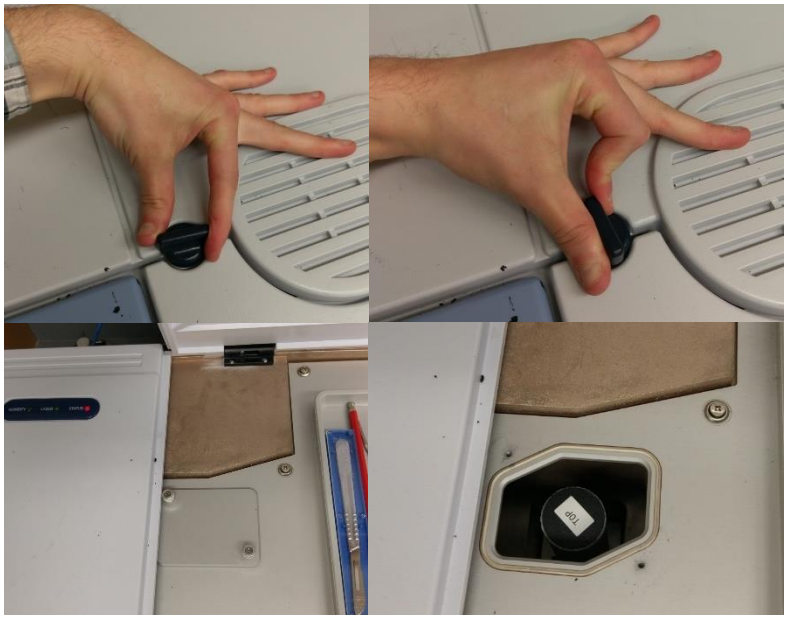
IR Maintenance

Changing the Sieves

1. The instrument has three LED lights to indicate the status of the instrument. If the Humidity LED is red, it is time to change the sieves.
2. Open the lid (far left of the instrument, below the LED indicator - it should have a blue ellipse on the top) to the compartment with the white porous cylinder containing the sieves. See pictures below:

Lid to first sieve compartment. Left: lid in closed position, middle: lid in open position, right opened lid compartment. Circled in red is the cylinder containing the sieves.

3. Use a spatula to carefully pry the black lid off the white cylinder. Pour the sieves into a 400-mL beaker and heat them in the oven overnight. It is unnecessary to use a regenerator for the sieves, since they will not be used for solvents, only for keeping the air inside the instrument dry. Use of the regenerator will cause the sieves to degrade more quickly.
4. Repeat for the other white porous cylinder; this is located underneath the lid to the right of the LED indicator. Underneath this lid, on the far left, there is a compartment sealed by two slotted screws; open this compartment to get to the second white cylinder with sieves and add the sieves to the beaker for drying. See pictures below:



Lid to second sieve compartment. Top left: lid in closed position, top right: lid in open position, bottom left closed sieve compartment, bottom right: open sieve compartment.

5. Once the sieves are dry, place the beaker in a desiccator to allow them to cool. Once cool, pour the sieves back into the porous white cylinders, replace the black lids, and replace the cylinders in the instrument. Close all the compartment lids when finished.

Changing the Laser (Do with a technician present)

1. Go to 10.10.0.1/config/report.htm to check the status of the HeNe laser. The life of the laser is 25000 hours (~2.85 years) and should be replaced if it has been running for longer than this.
2. Turn off the instrument power by flipping the power switch on the back of the instrument. Disconnect the power cable.
3. Open the compartment lid to the right of the LED indicator.
4. Grasp the LED indicator panel, gently pull up on the lid, and remove it from the hinge holding the lid down at the top. Do NOT pull up too far on the lid, as there is a cable connecting to the LEDs underneath the lid.
5. Disconnect the LED indicator cable and remove the lid completely.
6. There are 15 spring-loaded fasteners on the edges of the metal plate in the compartment; loosen all of them with a screwdriver. Once loosened, first lift up the

left side of the metal plate and then remove the metal plate entirely to reveal the interferometer.

7. In the interferometer compartment, you should see a module that says “LASER RADIATION DO NOT STARE INTO BEAM”. There is a retention plate holding the module in. Loosen the screw of the retention plate and rotate the plate away from the laser module.
8. Use both hands to pull up on the laser module to remove it from the interferometer compartment. This will require some force. Do NOT pull up on it too far, as there are cables connected to it still.
9. The power supply cable for the laser is held in place by two screws; loosen these screws and remove the power cable.
10. Plug the power cable into the new laser, tighten the two screws to keep the power cable in place, and push the laser module into the holder.
11. Rotate the retention plate over the laser module and tighten the screw to hold it in place.
12. Replace the metal plate back over the interferometer compartment and tighten all spring-loaded fasteners down with a screwdriver.
13. Plug the LED indicator cable into the slot on the LED lid and replace the lid.
14. Close the lid to the compartment to the right of the LED indicator panel.
15. Plug in the power cable to the instrument and turn on the power.
16. In the OPUS software, click the “Measure” menu and click the “Optics Diagnostics” button. The “Instrument Status” window should open. Click on the HeNe laser icon and click on “Service Info”. Click on “Reset” to reset the counter.
17. Go to the “Validation” menu, click the “Setup OVP” button, and click “OVP Test Channel Setup”. Since the sample compartment is empty, select “IT1: Sample Compartment”. Click on “Measure LWN”. Note that this step should be done any time you change a component.
18. Go to the “Validation” menu, click the “Setup OVP” button, and click “OVP Test Channel Setup” tab. Under the “PQ and OQ” tab, you can select which tests you would like to run; select all tests by default.
19. The PQ test uses references, so remeasure your references prior to running the PQ test. Click “Measure Reference Spectra” under “OVP Test Setup”. Then click on “Extract Reference Spectra” and run the PQ test.

Aligning the Interferometer (Do with a technician present)

1. Click on the “Measurement” menu and click the “Check Signal” button. On the display with the graph, there should be a sharp peak. If the amplitude is > 20000 then there is no need to do anything further.
2. Open the compartment lid to the right of the LED indicator.
3. Grasp the LED indicator panel, gently pull up on the lid, and remove it from the hinge holding the lid down at the top. Do NOT pull up too far on the lid, as there is a cable connecting to the LEDs underneath the lid.
4. Disconnect the LED indicator cable and remove the lid completely.
5. There are 15 spring-loaded fasteners on the edges of the metal plate in the compartment; loosen all of them with a screwdriver. Once loosened, first lift up the

left side of the metal plate and then remove the metal plate entirely to reveal the interferometer.

6. The interferometer is in the center of this compartment. There should be three knobs on a plate shaped like a right triangle on the interferometer, two on the diagonal of the triangle and one on the off-diagonal. Do NOT touch the off-diagonal knob. Adjust the knobs on the diagonal and wait after each adjustment.
7. The mirrors in the detector compartment may also need aligning. Adjust them by opening the compartment below the LED indicator panel. For one of the mirrors, only use the on-diagonal knobs to align it. For the other mirror, all three knobs may be utilized.
8. Make sure the amplitude is > 20000; do not saturate the detector. Once this condition is met, you may close all the compartments.

Changing the MIR Source

1. Click on the "Measurement" menu and click the "Check Signal" button. On the display with the graph, there should be a sharp peak. Check that the amplitude is > 20000; if it is less, it may be necessary to change the MIR source. It's possible that the interferometer simply needs to be realigned (see the "Aligning the Interferometer" section), so check to make sure that this is not the case first. The MIR source life is 45000 hours (~5.13 years) and should be replaced if it has been used for longer than this. The other light source next to the MIR source should not ever need to be changed.
2. Turn off the power on the back of the instrument.
3. Open the compartment to the right of the LED indicator panel.
4. Loosen the grew thumb screw on the release lever of the MIR source (should be the one on the right labeled "MIR").
5. Gently push down on the MIR source and rotate the release lever so that the MIR source can be removed.
6. Pull up on the light source and remove it from the instrument.
7. Replace the MIR source by placing a new unit into the source holder.
8. Gently push down on the MIR source and rotate the release lever to secure the MIR source.
9. Tighten the grey thumb screw on the release lever of the MIR source.
10. Close the compartment lid and turn the instrument power back on.
11. Click on the "Measurement" menu and click the "Check Signal" button. On the display with the graph, there should be a sharp peak. Check that the amplitude is > 20000.

Reconfiguring ATR Tests

1. Mount the ATR according to the "ATR (Solid and Liquid Samples) Setup" section.
2. Only do steps 3 and 4 if you have changed the laser or realigned the interferometer.
3. Click on the "Measurement" menu and click the "Check Signal" button. On the display with the graph, there should be a sharp peak. Check that the amplitude is > 20000. If it is less, turn both knobs on either side of the ATR to increase the amplitude to the desired level. Wait for a little bit after turning each knob.

4. Go to the “Validation” menu, click the “Setup OVP” button, and click “OVP Test Channel Setup”. Enter an appropriate title for the configuration. Since the sample compartment contains the ATR, select “IT2: Pike Miracle”. Specify that you’re using the “MIR” source. For the instrument configuration, select “Sample Compartment RT-DLaTGS”. For the accessory, select “MIRacle, Multiple Crystals”. For the crystal, select “Ge”. Click on “Measure LWN” and click “yes” when you see a warning window. Click “Save” and then exit.
5. Run the OQ test and then the PQ test using the same steps described in the “Changing the Laser” section.
6. Note that all previous test results are stored under C:\OPUS\Validation\Reports in case you need to check previous test results.

Other Useful Information

For inquiries, reference the BIO number found underneath the cover hatch.

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