IMSERC User Manual for Raman

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INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/pcm-instruments.html. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/pcm-instruments.html by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please ***stop***, and ***get help***. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found next to room DB033A and outside the DB area in the hallway, respectively. Gloves should be removed when using the computer.

Please collect all waste such as samples, gloves, kimwipes, etc. and dispose of them properly in your lab. There are no containers for hazardous chemical waste in DB022.

DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied on your group folder on 'imsercdata.northwestern.edu' under 'others/Raman' once every hour. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

Data acquisition and real time plotting are performed with the 'LabSpec' software. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please ensure that you have exported your data as TXT files for using 3rd party data processing software for smoothing and background subtraction. You have the option to use the instrument computer for analysis, but you must reserve instrument time through NUcore

DEFAULT INSTRUMENT STATUS

The default measurement mode using the Raman spectrometer is **room temperature measurements of bulk liquid or solid samples.** Please notify the appropriate staff member well in advance if you would like to run an experiment in a different mode. For the full list of modes and capabilities, please check at http://imserc.northwestern.edu/pcm-instruments.html#raman. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement.

The default working condition of the Raman is as follows:

1. The computer screen is turned off by default. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore
2. The default 'Raman' user account should be logged in. In case the computer was restarted, the password for the 'Raman' account is (see hardcopy by the instrument)

3. Acquisition software (LabSpec) should not be running. Close the acquisition software when you are done with the measurement
4. Key for the shutter must be at the 'Disable' position and the 'Laser Out' and 'Signal In' ports must be plugged as shown in Figure 4
5. Power on the spectrometer should not be on. Spectrometer must be turned off at the end of the measurements
6. There should be no error messages on either the front panel of the instrument or the acquisition software. Please check the 'Troubleshooting' session for a potential solution before reporting the error

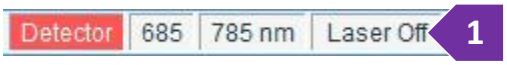
If there is an error or problem with the instrument that is not covered under the 'Troubleshooting' section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions

COLLECTING SPECTRA

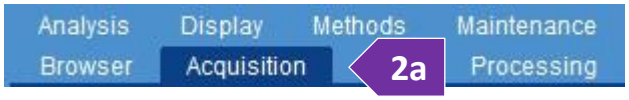
The following procedure should be followed by the users who want to use the default instrument configuration.

1. Turn on the spectrometer by flipping the power switch to the 'On' position. Power switch is located at the left side of the spectrometer next to the power cord. It takes about two minutes for the system to warm up and cool down the detector. If you start the software early, you may see that the detector is not ready with the red indicator at the bottom of the software window (Figure 1). Detector indicator should turn green in a couple of minutes. Please wait for the indicator to turn green before moving to the next step



2. Perform an autocalibration (AC) if the red indicator of the AC is on (Figure 2). If the 'AC' status is green, please proceed to the next step. To perform the autocalibration, follow the steps below:

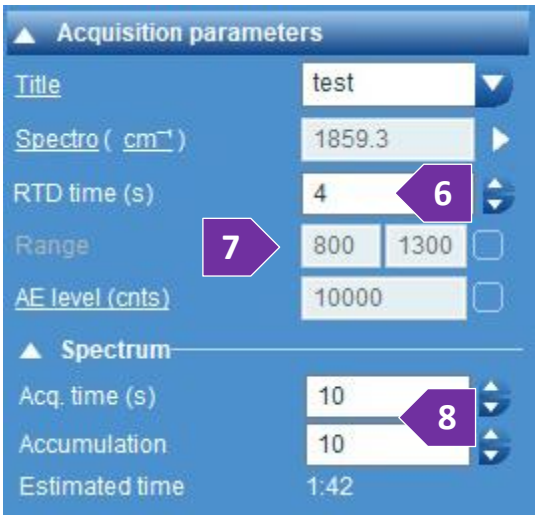
   a. Open the top lid of the blue compartment and put an empty plastic cuvette. Cuvettes are located on the top shelve above the spectrometer. The autocalibration procedure will look for the polyethylene peak at around 1,000 cm^{-1}
   b. Close the lid and turn the laser on by pressing on the 'Laser Off' status at the bottom of the window, see figure 1
   c. Select the 'Acquisition' tab at the top right of the window (Figure 2c)



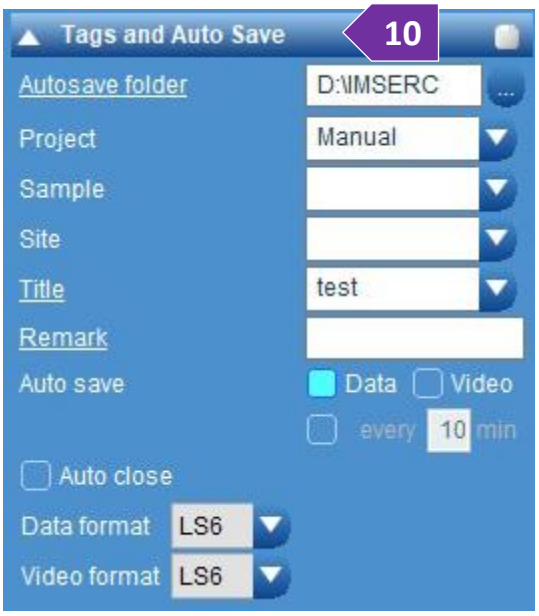
   d. Under the 'Instrument setup' panel on the 'Acquisition' tab, increase the laser power to 100% (figure 2d)

   e. Press on the red 'AC' status at the bottom of the window (figure 2) and press on the 'Start Autocalibration' button to start the process
   f. Calibration process should take only a few seconds, and you should see a window that tells you that the calibration was successful. In case you receive an error, ensure that you are using an empty plastic cuvette and that the power of the laser is set at 100%. If the error message is persistent, please contact a staff member
3. Open the top lid of the blue compartment, remove the empty plastic cuvette if you have performed the autocalibration step, and put your sample in
4. Close the lid, and reduce the laser power to 10% (figure 2d)
5. Turn the laser on by pressing on the 'Laser Off' status at the bottom of the window (figure 1)

6. Press on the play button at the top of the window to see a live acquisition view of the spectrum of your sample (figure 6). If the spectrum is too noisy (low signal-to-noise ratio), gradually increase the power of the laser and/or increase the number of seconds in the 'RTD time (s)' (Real Time Detection) field located under the 'Acquisition parameters' panel under the 'Acquisition' tab (figure 6). You have the option to let the software optimize the exposure by enabling the 'AE level (cnts)' option below the 'RTD' setting. To enable each option, check the checkbox next to the setting. A color-filled checkbox means that the option is enabled



7. Verify that the 'Range' covers the window of interest for your sample by adjusting the range values appropriately (figure 7)
8. Set the exposure time of the experiment by providing the number of seconds in the 'Acq. time (s)' option (figure 8)
9. You can improve the signal-to-noise ratio by averaging multiple spectra/collections of the same sample by providing the number of desired spectra under 'Accumulation' (figure 8)
10. Once you have determined the exposure and range, you need to select the output path for the collection file. Select the 'Tags and Auto Save' panel under the 'Acquisition' tab. Software gives you options to create up to four levels of directories that are called 'Autosave folder', 'Project', 'Sample', and 'Site' (figure 10):
    - The minimum information needed is your person folder under your group folder as 'Autosave folder', and the name of the data file as 'Title' in figure 8
    - Optionally, you can create another folder under your personal folder named as 'Project' folder
    - Optionally, you can create another folder under your 'Project' folder named as 'Sample' folder, and
    - Optionally, you can create another folder under your 'Sample' folder named as 'Site' folder



11. It is highly recommended to keep the 'Data format' and 'Video format' options to the native binary 'LS6' format of the acquisition software to save all metadata available.

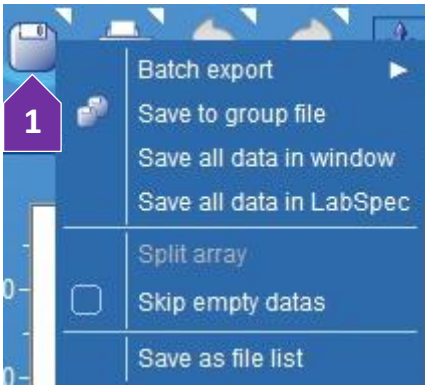
You'll be exporting the spectra to a TXT format after the measurement for easy plotting using standard 3rd party software

12. Once you have provided the file name and its path, start the data acquisition by pressing the record button located next to the play button (figure 12)

EXPORTING DATA

It is highly recommended to save your raw data using the native LS6 binary format, so that you can preserve all metadata after a collection. To export the spectra into an ASCII format that can be imported by 3rd party software which creates graphs:

1. Right press on the disk-like icon on the top right side of the window (figure 1)
2. Alternatively, if you have only one spectrum that you'd like to convert into ASCII, left press on the disk-like icon
3. Select the appropriate option and export as TXT file(s)



PUBLICATION

A. EXPERIMENTAL SECTION

Modify the text below according to the setup and conditions you used during the measurement:

"Raman spectra were collected at room temperature using a Horiba MacroRAM spectrometer equipped with a 785 nm laser. Sample was placed in a **plastic cuvette | quartz cuvette | quartz capillary | solid holder** and data were collected by using an exposure of **XX** seconds and averaging **YY** spectra."

B. ACKNOWLEDGEMENT

"This work made use of the IMSERC (RRID:SCR_017874) Physical Characterization facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University."

TROUBLESHOOTING

A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

B. COMPUTER REQUIRES LOGIN AND A PASSWORD

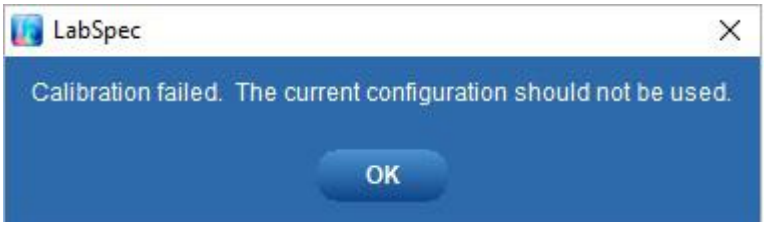
The default 'Raman' user account should be logged in. In case the computer was restarted, the password for the 'Raman' account is (see hardcopy by the instrument). See 'Default instrument status' section for more details.

C. AUTOCALIBRATION KEEPS FAILING

In case the 'Autocalibration' keeps failing, please ensure that:

- An empty plastic cuvette is installed in the sample compartment
- The laser is on, and
- The power of the laser is set at 100%

If all the above criteria are met, please see a staff member.



D. THERE IS AN ERROR/PROBLEM WITH THE INSTRUMENT THAT IS NOT ADDRESSED UNDER THE TROUBLESHOOTING SECTION

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html add place the 'Stop' sign near the instrument computer. 'Stop' signs are located on the shelf above the computers in BG51 and online at the link above. Email or talk to a staff member
3. Email or talk to a staff member

REVISIONS

v1.01 2025/09/12	• Release of original version of the user manual for the acquisition software LabSpec v6