TI 276C: Performing HIPS Analysis

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1. PURPOSE AND APPLICABILITY

The purpose of this standard operating procedure is to describe the process of performing light absorption analysis on routine IMPROVE samples and field blanks with the Hybrid Integrating Plate/Sphere System.

2. SUMMARY OF THE METHOD

The HIPS system is warmed up overnight before being used. A calibration verification is performed using a set of 15 selected IMPROVE samples serving as standards. In addition, reanalysis of a selected set of previously analyzed IMPROVE filters is done to monitor system stability. After the standards and reanalysis set has been measured and it has been ensured that the results meet designated criteria, one sampling month's-worth of IMPROVE filters are then analyzed. This task can take approximately 2-3 days. An automation macro is used to record the data for reanalysis and routine IMPROVE filters.

3. PERSONNEL QUALIFICATIONS

A trained laboratory technician, under the supervision of the spectroscopist and/or the laboratory manager, performs all analyses utilizing the HIPS system, including calibration verifications, adjustments (if necessary), reanalysis and analysis of routine IMPROVE samples.

4. CAUTIONS

Make sure that the correct slide tray is being loaded for analysis. Loading the wrong tray will cause the recorded data to be incorrect.

Confirm that the correct macro is being used and that the switch is in the proper orientation for reanalysis and for routine IMPROVE samples.

5. EQUIPMENT AND SUPPLIES

The following equipment and supplies are used for HIPS analysis:

- Hybrid Integrating Plate/Sphere System (HIPS)
- Prepared HIPS log files for the sampling month to be analyzed
- Standards set
- Reanalysis set
- Slides with IMPROVE filters
- Computer connected to HIPS

6. Procedure

6.1 Preparation

1. Turn on the HIPS system at least 12-24 hours prior to intended use.
2. Before starting analysis, make sure that the air compressor system is functional. In the current set-up (at the time this SOP was written), the air compressor system is located in the Chemistry Laboratory in Mezzanine 203 of Jungerman Hall.

6.2. Logging into the HIPS Server

1. The current server for HIPS is the newer CNL server. All programs and applications on this server are current versions. Specifically, the server used for HIPS has Microsoft Excel 2010, so HIPS log files are saved as ".xlsm."
    a. The username for the HIPS server is: ou\cl-hips. This is not case sensitive.
    b. The password is Laserh1pster. This *is* case sensitive.

6.3 Calibration Verification and Adjustments

1. Go to U:\IMPROVE_Lab\LASER\Standards and open the document called "laser_standards.xlsx." Place the current date in the next available cell in column A of the spreadsheet. Record all reflectance (R) and transmittance (T) data taken during standards for the day in this row.
2. Set the Control Box Mode switch to Manual.
3. Retrieve the standards slide tray and place it in the instrument. The current tray (at the time this SOP was written) is labeled "Laser Standards, December 2011."
4. Using the current tray, calibrate with position 3. Set the Reflectance to 332 (read from Labsphere radiometer) and Transmittance to 437 (read from Oriel Detection System). In the current standards tray (at the time this SOP was written) the sample in position 3 is ACAD1 07/07/10.
5. Go back to position 1 and record the values of positions 1 and 2 in the spreadsheet. If by the time position 3 is analyzed again and the R and T values are *not* within ±1% of 332 and 437 respectively, return to step 4 and repeat until the values are correct for position 3.
6. Continue with all positions until finished with the tray.
7. Once finished, record the measurement for R and T from the Neutral Density Material (NDM) in the Rref and Tref columns. These Rref and Tref values are what the

reflectance and transmittance values are when no tray is loaded in the arm and the NDM is exposed to the laser beam.

8. Check the integrity of the data by looking at the absorbance spreadsheet "b" and the graphs on the subsequent spreadsheets that will automatically be generated. Make sure all the values are within the set criterion. For details about the criterion for the standards set, refer to TI 276D, "Quality Assurance/Quality Check of $PM_{2.5}$ Loaded Filters Using Hybrid Integrating Plate/Sphere (HIPS) Method for Measuring Light Absorption."
9. When finished, save and close the spreadsheet. This same spreadsheet will be used for every calibration, with new values appended each time, until a new set of standards is implemented.

6.4 Reanalysis

1. In U:\IMPROVE_Lab\LASER\Templates, open the document named "template_reanal worksheet.xlsm." Enable macros by pressing "Enable content" if prompted. The column labels "Day 1," "Day 2," and "Day 3" refer to the calendar dates on which the reanalysis data is collected before running routine IMPROVE samples.
2. After opening, rename the file to represent the data set that is later going to be analyzed. The format to follow is "Month Year Reanalysis.xlsm." For example, if the filters that are going to be analyzed were sampled during January 2012, the file name would be "January 2012 Reanalysis.xlsm." Save it in a folder named by the month and the year. The folder format is "NumberofMonth Month Year." For instance, the above example would have a folder named, "01 January 2012." These folders should also contain the prepared HIPS log files.
3. To prepare this spreadsheet for the "Log Once" macro, go to U:\IMPROVE_Lab\LASER\M401_Automatic and open the file Log_Once_Template.xlsm. Make sure to enable macros by pressing "Enable content" if prompted.
4. Press ALT+F11. A Visual Basic Application will open and a window with lines of code will appear. Copy all of this code and then close the file.
5. Go back to the reanalysis spreadsheet and press ALT+F11.
6. On the left-hand side, in the "Project-VBA Project" box, double-click on the option labeled "This Workbook," which should be in the drop-down menu for the folder entitled "Microsoft Excel Objects." When "This Workbook" is double-clicked, an empty window should appear.
7. Paste the lines of code into the empty window. Save the application. Then, close the application by going to the "File" menu and choosing "Close and return to Microsoft Excel."

8. In U:\IMPROVE_Lab\LASER\M401_Automatic, open the file M40xVB41_Log_Once.xlsm. Enable macros by pressing the "Enable content" button if prompted.
9. In the top menu bar on the far right, click on M40x Tools, then Connect.
10. Wait until "Ready" is registered on the bottom left of the Excel file.
11. Return to the Reanalysis workbook and make sure that the correct day has the proper date filled in. Then, double-click on the cell that data should be placed in first. Also confirm that the mouse pointer is in the cell area of the Excel file, but not on column F. It is safest to move it to the right-hand side of the screen.
12. With the control box switch set to Manual mode, load the reanalysis tray into the system and set it for position 1. Make sure to use the tray forward button to align the sample. Note that the tray advance button must be used while the mode switch is set to manual mode, otherwise the slide arm will run into the tray. The current tray (as of the time of this SOP) is labeled "Laser Reanalysis, December 2011."

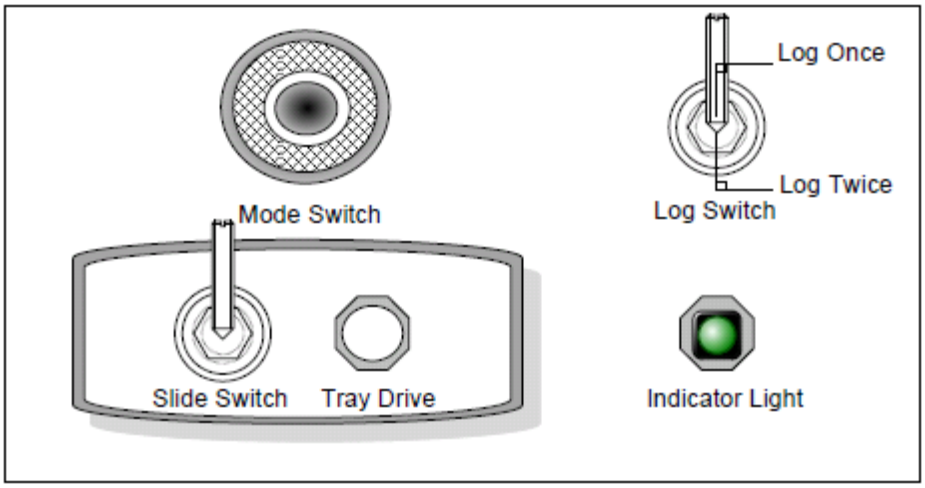

Figure 1. Control Box Layout

13. Next, make sure the Log switch is set to Log Once and change the mode from Manual to Auto.
14. Press the big green button to start analyzing.
15. As the tray is analyzing, make sure that the data is filled in each cell in the proper order and that the values make sense for the current filter being analyzed.
16. Switch the mode switch to single cycle once the last data point has been collected to stop the Auto cycle.
17. When the reanalysis tray is complete, check the integrity of the data by looking at the automatically generated graphs in the subsequent spreadsheets. For details about the criterion for the reanalysis set, refer to TI 276D.

18. When finished, go back to the M40x file and disconnect from the macro using the M40x Tools drop-down menu. Then, close the M40x file.
19. In the Reanalysis workbook, press ALT+F11 to get back to the Visual Basic Application. Delete the lines of code that were pasted in the window earlier. Then, save and close both the application and the worksheet.

6.5 IMPROVE Samples

1. IMPROVE samples are run in much the same way as reanalysis filters except with a few key differences:
    a. Use the prepared HIPS log files that were created for the IMPROVE samples and field blanks. If there is no prepared worksheet, there are instructions for creating those files in U:\IMPROVE_Lab\LASER\IMPROVE Laser SOPs.
    b. Instead of copying code from the file Log_Once_Template.xlsm into the Log workbook, copy and use the code from the file Log_Twice_Template.xlsm.
    c. Instead of using the macro from M40xVB41_Log_Once.xlsm, use the macro from M40xVB41_Log_Twice.xlsm. When acquiring data with this template, always make sure that the "active cell" is the cell where the sample's T value should be written to. The sample's R value will be written to the right of the T measurement.
    d. The Log switch should be set to Log Twice. This enables HIPS to take a reading of the NDM between each filter in order to confirm the stability of the system.
    e. Before analyzing a tray, make sure the contents have been tray-checked and that the slides in the tray match the order of the log file list.
    f. When a tray is finished, simply load the next tray and press the green button to continue. Keep an eye on the R and T measurements taken from the NDM. Note that the R value from the NDM can drift about 20 points, so the transmittance value is a more accurate measure of the system's stability.
    g. If the system jams, just give the slide arm a gentle push or pull. The system should continue on as if nothing happened. Measurements from the NDM may fluctuate, but do not do anything about this unless both the R and T has changed significantly and are not within + 5% of average from the first 200 measurements of the NDM. For details, refer to TI 276D.
    h. Every five trays, readjust the HIPS system using the standard in position 3 from the standards tray by setting the R and T values to 332 and 437 respectively. After, take T and R measurements from the instruments without a slide. A user can do this by turning the Mode Switch from Auto to Manual. Then, pressing the slide switch to manually push the slide arm in.

i. Follow the same procedure for field blanks, making sure to use the prepared Excel file for field blanks.
j. At the end of all analyses for the month, turn off the HIPS system.
k. If new tabs were created in the log workbook for routine IMPROVE filters or for 11^{th} sample dates, make sure to recombine those data with the data in the first tab so that everything is in the same place. Make sure to sort by date and site.

7. References

1. TI 276D, "Quality Assurance/Quality Check of $PM_{2.5}$ Loaded Filters Using Hybrid Integrating Plate/Sphere (HIPS) Method for Measuring Light Absorption."