

UCD CSN Technical Instruction #277B

Performing HIPS Analysis

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DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications
4/28/2020	LMK	All	Changed procedures to reflect new software for slide tray generation and analyzing samples, update figures.
4/29/2021	LMK	6.4, 6.5	Added end of day QC check.

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1. PURPOSE AND APPLICABILITY

The purpose of this technical instruction (TI) document is to describe the process of performing light absorption analysis on routine CSN samples and field blanks with the Hybrid Integrating Plate/Sphere System.

2. SUMMARY OF THE METHOD

The HIPS system is stabilized at least four hours prior to use. The detectors are registered to a set value. A detector registration verification is performed using a set of 15 pre-selected samples serving as verification filters. In addition, reanalysis of a selected set of previously analyzed filters is performed to monitor system stability. After the verification and reanalysis filters have been measured and the results meet designated criteria, one sampling month's-worth of CSN filters are then analyzed. This task can take approximately 2-3 days. A LabVIEW program is used to collect data and write it to the database.

3. PERSONNEL QUALIFICATIONS

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

4. CAUTIONS

Make sure the correct slide tray is being loaded for analysis by verifying the first and last BarcodeID on the program with the first and last BarcodeID marked for the slide tray in the chain of custody. Loading the wrong tray will cause the recorded data to be incorrect.

Do not forget to write to database after each tray has been analyzed, as the values recorded on the LabVIEW program will be written over as soon as analysis of the next tray begins. There is also an option to “Write Tray Data to File” which will only be used under special circumstances.

5. EQUIPMENT AND SUPPLIES

The following equipment and supplies are used for HIPS analysis:

- Hybrid Integrating Plate/Sphere System
- Registration filter and registration verification set

- Reanalysis set
- Slides with CSN filters
- Computer connected to HIPS

6. PROCEDURAL STEPS

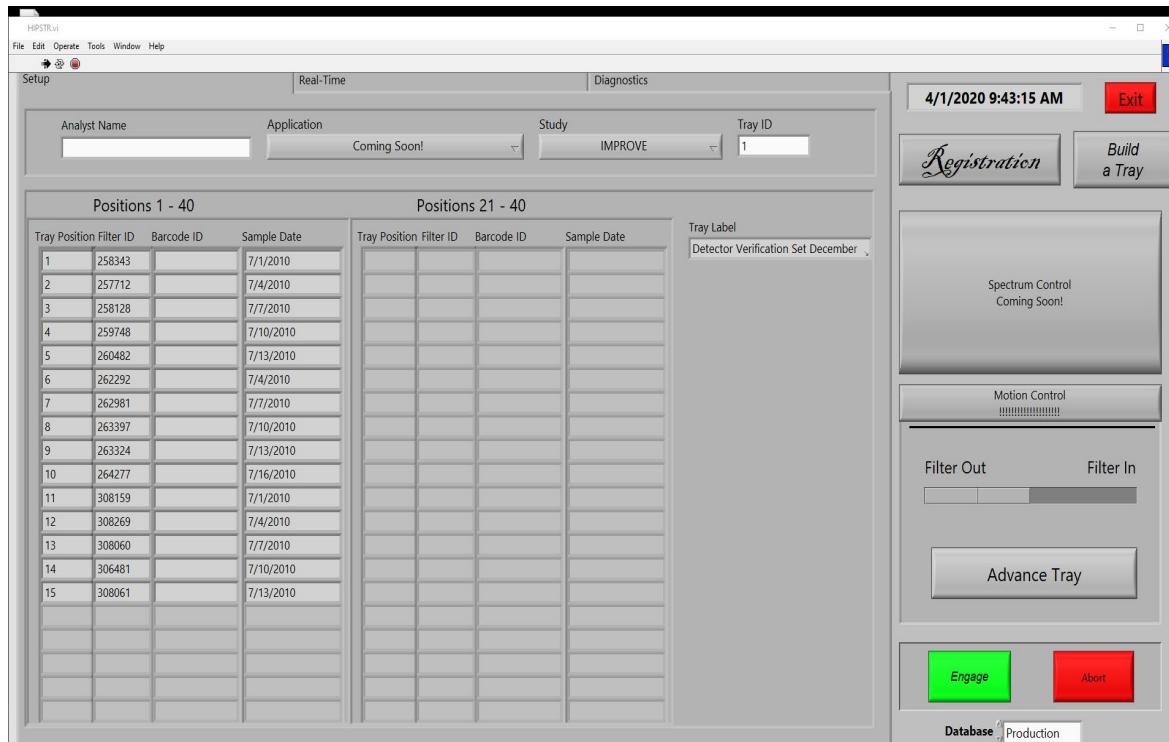
6.1 Preparation

- 1) Turn on the HIPS system at least four hours prior to intended use so the laser and detectors can stabilize.
- 2) Before starting analysis, make sure that the air compressor system is functional.

6.2 Registration of HIPS Detectors

- 1) Go to C:\HIPS\Applications\HIPSTRv2.exe

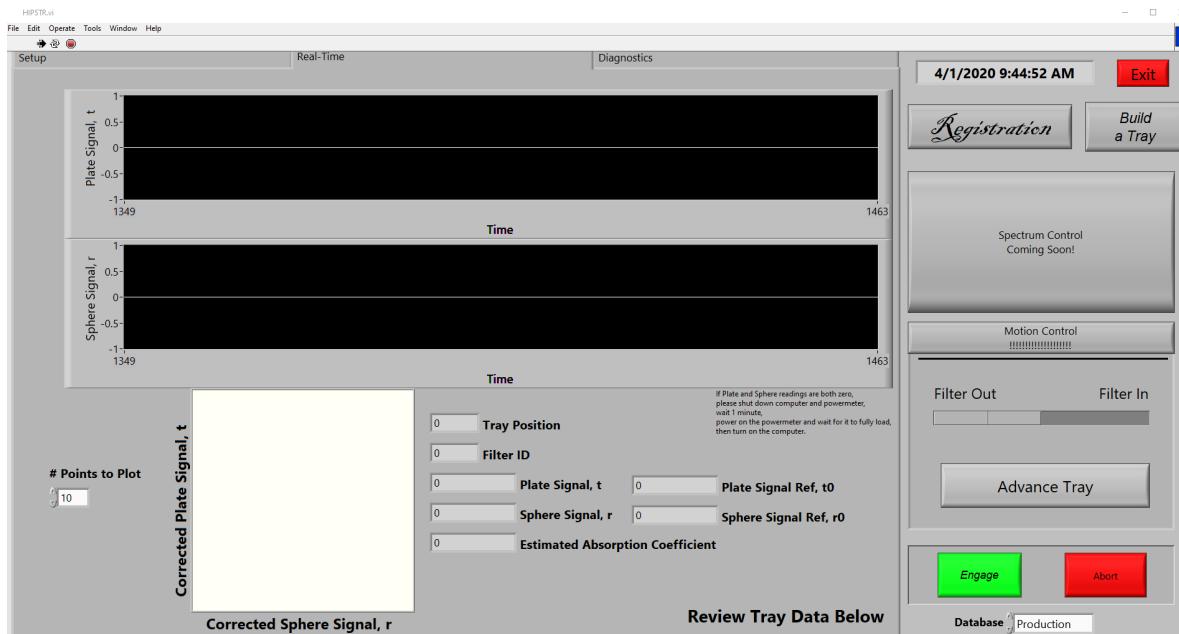
Figure 1. HIPS- LabVIEW main page.



- 2) Click the white arrow in the top left corner to engage the program (the arrow changes to black when the program is engaged).
- 3) Enter your initials in the “Analyzer name” box.

- 4) Navigate to the “Real-Time” tab.

Figure 2. LabVIEW Real-Time tab.



- 5) Retrieve the verification slide tray and place it in the instrument, the tray is labeled “Detector Verification Set December 2011”. Ensure the slide tray is in the correct orientation and properly aligned, refer to Figure 7 below (numbers 1-40 will be visible on the side of the slide tray). Line position 1 up with the slide arm. Slide position 1 (first filter in the slide tray) in, click “Filter In”, to slide this filter back out click “Filter Out”. If the arm jams while attempting to load position 1, gently push or pull on the arm to correct it. The slide tray can be unloaded from the instrument and reloaded if misaligned. Once position 1 has been properly aligned proceed to step 6.

Figure 3. Slide tray orientation.



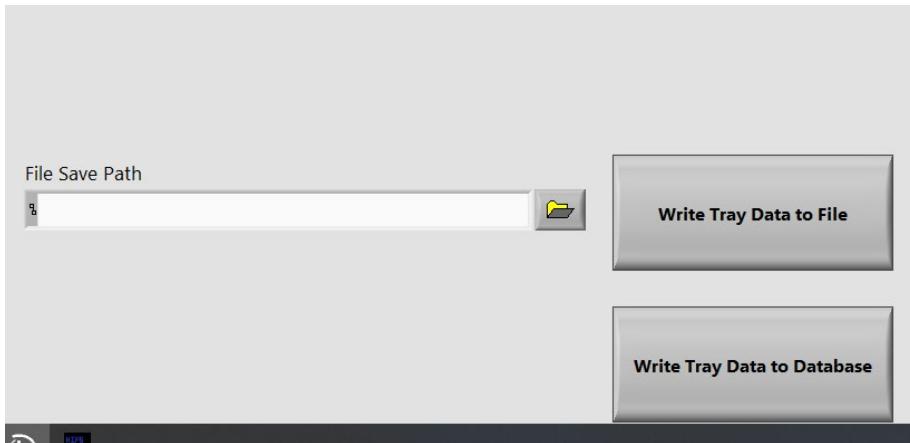
- 6) Click the “Advance Tray” button to move the tray one position at a time until position 3 is aligned to load into the instrument (the filter in position 3 is ACAD1 07/07/10). This is the current registration filter.
- 7) Once position 3 is lined up slide the filter in by clicking “Filter in”.
- 8) With position 3 inserted wait a few seconds for the Transmittance (T) and Reflectance (R) values on the screen to stabilize. Once the values stabilize, click the “Registration” button. The T and R values should now read 437 and 332, respectively. If they are not within $\pm 1\%$ of these values, click “Registration” again.
- 9) Slide the position 3 filter out and back in and verify the values are still within $\pm 1\%$ of 437 and 332. If not, click the “Registration” button again and repeat. Once complete, remove the tray from the instrument.

6.3 Detector Registration, Verification, and Adjustments

- 1) Navigate to the “Set-up” tab, from the Study dropdown box select “IMPROVE” and type a ‘1’ in “Tray ID” field.
- 2) The filter information will auto-populate and the tray label on the screen will read “Detector Verification Set December 2011.”

- 3) Place the verification set slide tray in the instrument and line up position 1. Refer to step 5 in section 6.2 “Registration of HIPS Detector” if further instructions are needed for proper alignment.
- 4) Select the green “Engage” button and the system will automatically analyze all filters in the slide tray. Scroll down to see the data recorded in real-time.
- 5) When the tray is finished analyzing, click “Write Tray Data to Database.”

Figure 4. Write tray data to database.



- 6) Perform a QC check on the verification results to ensure the system is suitable for use. See TI 277C, “Quality Assurance/Quality Check of PM_{2.5} Loaded Filters Using Hybrid Integrating Plate/Sphere (HIPS) Method for Measuring Light Absorption” for details.

6.4 Reanalysis

- 1) Navigate to the “Set-up” tab and from the Study dropdown box select “IMPROVE” and type a ‘2’ in “Tray ID” field.
- 2) The filter information will auto-populate and the tray label on the screen will read “Reanalysis Set June 2010”.
- 3) Retrieve the reanalysis slide tray and place it in the instrument lining up position 1, the tray is labeled “Reanalysis Set June 2010”. Refer to step 5 in section 6.2 “Registration of HIPS Detector” if further instructions are needed for proper alignment.
- 4) Select the green “Engage” button and the system will automatically analyze all filters in the slide tray. Scroll down to see the data recorded in real-time.
- 5) When the tray is finished analyzing, click “Write Tray Data to Database”.

- 6) Perform a QC check on the reanalysis results to ensure the system is suitable for use. See TI 277C, “Quality Assurance/Quality Check of PM_{2.5} Loaded Filters Using Hybrid Integrating Plate/Sphere (HIPS) Method for Measuring Light Absorption” for details.
- 7) Steps 1-6 are repeated following the completion of sample analysis each day as an end of day QC check.

6.5 CSN Samples

- 1) Before beginning analysis of routine CSN samples, the samples need to sit at room temperature for approximately 15 minutes.
- 2) Verify T and R values for the registration filter, position 3 of the verification set before beginning routine analysis. Retrieve the verification slide tray and align position 1. Navigate to the “Real-Time” tab. Then click the “Advance Tray” button on the screen twice to align position 3, then click “Filter In”. If the values are within $\pm 1\%$ of 437 (for T) and 332 (for R) then proceed with routine analysis. If the values are not within $\pm 1\%$ of 437 (T) and 332 (R) press the registration button on the screen. The values will reset to 437 (T) and 332 (R). Slide the filter out by clicking “Filter Out” and remove the verification slide tray.
- 3) Navigate to the “Set-up” tab of the LabVIEW program.
- 4) In the “Analyst Name” field, fill in your initials.
- 5) From the study dropdown box select “CSN”.
- 6) Select the first tray for analysis and enter the LabVIEW tray number indicated on the tray label, into the “Tray ID” field. The filter ID information will auto-populate on the screen.
- 7) Verify the first and last filter barcode IDs on the computer with those marked for this particular slide tray in the chain of custody. Additionally, verify the manufacturer serial number for the first and last filter with the chain of custody.
- 8) Load the slide tray to the instrument, lining up position 1.
- 9) On the screen click “Engage” and the tray will run automatically. Scroll down to see the data recorded in real time.
- 10) When the tray finishes analyzing, verify T and R values were recorded for every filter in the slide tray, once verified click “Write Tray Data to Database.”
- 11) Remove the tray from the HIPS instrument, place in a sealed plastic bag and return the samples to the refrigerator.
- 12) Repeat steps 6-11 for analyzing additional routine CSN samples.
- 13) Every 200 filters (typically 5 trays), the registration filter needs to be re-registered. Refer to section 6.2 Registration of HIPS Detector for step-by-step

procedure. Additionally, if the HIPS instrument is left idle for more than 10 minutes the registration filter will need to be re-registered.

- 14) Following the completion of routine sample analysis each day the reanalysis set is reanalyzed. Data for the reanalysis set is immediately ingested and exported for end of day QC. Refer to section 6.4 Reanalysis for step-by-step procedure.
- 15)

6.6 Troubleshooting

- 1) If the system jams, just give the slide arm a gentle push or pull. The system should continue on as if nothing happened. It may be necessary to reanalyze samples immediately following a jam of the slide arm. If the arm bumps into the system aggressively this can shift the alignment and adjustments may be needed.
- 2) To check the alignment after a slide arm jam, load position 3 of the verification set and verify T and R values, are within $\pm 1\%$ 437 (T) and 332 (R). If the values are within 1 % continue with analysis, if not additional troubleshooting is required.
- 3) If the values for position 3 (registration filter) are not within $\pm 1\%$ of 437 (T) and 332 (R) then the Registration of HIPS Detectors will need to be done again. Refer to TI277B Section 6.2 for detector registration steps. Once this step is complete confer with the spectroscopist before resuming analysis of routine samples.
- 4) If a slide tray is not aligned properly start by removing the slide tray from the slide changer. Place the tray back in the slide changer and use the slide arm as a guide. When properly aligned the number 1 on the side of the slide tray will be covered by the slide arm, refer to Section 6.3, figure 1. Slide the first filter in from the program. Once the arm moves slide 1 in without jamming you may proceed with analysis.
- 5) If the software freezes please wait a couple of minutes before getting a lab lead.
- 6) If the software has an error during analysis, the entire tray will need to be reanalyzed.
- 7) If filter information between LabVIEW, the COC and the physical slide tray does not match, stop analysis and get a lab lead.

7. REFERENCES

UCD TI 277C: Quality Assurance/Quality Check of PM_{2.5} Loaded Filters Using Hybrid Integrating Plate/Sphere (HIPS) Method for Measuring Light Absorption