

UCD CSN Technical Instruction #402D

Troubleshooting

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DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications
3/10/2020	SGC	9	Added steps 10, 11, 12. Minor update on terminology for consistency
5/11/2021	RY, AB	9	Provided details on FID battery change in list step 1. Inserted information in list step 2 describing the procedure when calibration area is consistently low

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1. PURPOSE AND APPLICABILITY

The subject of this technical instruction (TI) is troubleshooting issues encountered during carbon analysis of quartz fiber filters.

2. SUMMARY OF THE METHOD

Procedures for resolving common issues that may arise during daily operation of the carbon analyzers are outlined.

3. DEFINITIONS

- **Flame Ionization Detector (FID):** The detector used in the carbon analyzer instruments
- **He+Ox:** Oxygen premixed with helium gas
- **Helium (He):** Gas used for carbon analysis
- **Hydrogen (H):** Gas used for carbon analysis

4. HEALTH AND SAFETY WARNINGS

Not applicable.

5. CAUTIONS

Not applicable.

6. INTERFERENCES

Not applicable.

7. PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING

Only trained lab personnel designated by the Laboratory Manager may operate instrumentation in the carbon laboratory.

8. EQUIPMENT AND SUPPLIES

Not applicable.

9. PROCEDURAL STEPS

1. FID turns off during sample analysis

Click on **Cancel Run** on the software and re-ignite the FID. If the analysis is at “Safe to put in a new sample”, “Purging offline”, or “Purging online” stages; the sample temperature should register no increase from room temperature. However, double check to confirm. If no increase from room temperature, the filter punch is not destroyed and is good for new analysis after FID re-ignition. If FID shuts off *after* the “Purging online” stage, a new punch should be loaded into the instrument. Make a

note in the paper logbook stating “FID turned off during Sample xxxxx (ID #). A new punch is (or is not) taken.” Add the lab flag “LE-1” and add a comment to the filter through the CSN web app.

Note: Check the Hydrogen pressure if FID keeps turning off. Check or change the FID battery when needed. FID battery is located on the upper left corner of the methanator oven. AA Alkaline battery is used for replacement.



2. Calibration area is consistently low

The FID performance is assessed by monitoring the Calibration Area of all sample and QC analyses. The daily average of each instrument should be consistent with previous days within $\pm 5\%$. The calibration area of individual analysis should also be within $\pm 10\%$ of the daily average value. If a repeated low Calibration area (< 150000) is observed, this is usually an indication of degraded FID component (e.g. high voltage power supply).

Note: A calibration area that is approximately $\frac{1}{4}$ of the normal value is ok. In cases of high loading samples, a secondary FID channel (FID2) is used to avoid signal saturation. FID2's signal range is approximately $\frac{1}{4}$ of the primary FID channel (FID1).

3. Oven pressure below minimum threshold values

The software displays a warning message if the minimum initial pressure threshold or the pressure at the “Purging Online” stage is not met. The analysis pauses at this point. Re-adjust the ball joint and the clamp to ensure a good seal and the analysis will resume automatically once the threshold value is met.

4. Sample punch dropped outside Sample Punching Tray

Carefully pick up the punch with metal forceps and load it to the instrument. Add the lab flag “LE-2” and comment to the filter through the CSN web app.

5. Quartz sample holder dropped during loading/unloading

Carefully examine if the sample holder is broken. If there is no damage, slide the sample holder into the instrument without a filter punch and close the oven ball joint. Click on **Action** menu and select **Clean oven**. After finishing, slide the sample holder backwards into the oven and repeat the “Clean oven” cycle. If the sample holder is damaged, notify the lab supervisor for a replacement sample holder. Clean the new sample holder as stated above before analysis.

6. Quartz sample holder flips and sample punch dropped inside the oven during loading/unloading

Try to retrieve the sample punch with the sample holder. If successful, clean the sample holder as specified in Step 4. Take a new punch from the same filter and start the analysis. Add the lab flag “LE-2” and detailed comment to the filter through the CSN web app. If filter punch retrieval is not successful, do not proceed and notify the lab supervisor.

7. Typed in wrong Sample Name, Punch Area, or Operator’s initial

The operator will add a detailed comment to the filter through the CSN web app. The lab supervisor will correct the wrong information in the database after finishing analysis.

8. Filter integrity issues

Add the lab flag “FI-1/2/3/4” and detailed comment about the issue to the filter through the CSN web app. If possible, avoid the area that has filter integrity issues when taking the punch.

9. Oven ball joint accidentally opened while oven is above room temperature

Immediately click on **Cancel Analysis** and leave the oven ball joint open. The lab supervisor or trained staff will examine if any water condensation occurs near the quartz oven opening and carefully wipe clean any condensed water. Remove the front oven thermal couple following manual instructions and let the instrument dry out for at least 30 minutes. **DO NOT** put the quartz oven ball joint cap back on until the dry-out process is completed.

10. Laser replacement

Follow shut-down instructions to turn off the methanator oven, back oven, and the analyzer. Take off the top cover of the analyzer and set the methanator on top of the analyzer, making sure the laser safety shut off switch is depressed so that the laser readings remain on. Loosen the set screw that secures the laser in its housing, disconnect the laser wiring, and remove the old laser. Install the new laser and reconnect the wire, keeping the set screw loose. Turn on the main switch of the analyzer, keeping the back oven and methanator oven off. Watch the reflectance and transmittance signal readings on the software and slightly rotate the laser head to

optimize the readings. The laser readings with a clean filter punch in the oven should be > 10000. Once that is achieved, tighten the set screw and put the cover back on. Follow the start-up procedures to restart the back oven and methanator oven.

11. Laser housing alignment

If the reflectance and transmittance values are not > 30000 with no sample holder in the oven, it is likely the laser is misaligned. Take off the top cover of the analyzer and set the methanator on top of the cover switch on the analyzer so that the laser readings remain on. Watch the reflectance and transmittance signal readings on the software and slightly rotate the laser housing until the readings are optimized. One can also loosen the set screw holding the laser in the housing and adjust the laser itself. If the transmittance and reflectance readings remain low, there might be debris in the laser pathway or the oven laser window may be frosted.

12. Cleaning the laser pathway

Take off the top cover of the analyzer and set the methanator off to the side. Disconnect the wire attached to the laser housing and remove said laser housing from the oven laser pathway by gently rotating and simultaneously pulling up on the housing. Look into the laser pathway while illuminating the oven with a flashlight from the oven exhaust vent. If debris is present, remove it with a long cotton swap or a gentle blast of compressed air.

10. QUALITY ASSURANCE AND QUALITY CONTROL

Not applicable.

11. REFERENCES

Not applicable.