UCD CSN Technical Instruction #402F Main Oven Temperature Calibration

Chemical Speciation Network Air Quality Research Center University of California, Davis

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DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications
5/11/2021	AB	9	Minor updates on "Temperature Calibration" procedure

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1. PURPOSE AND APPLICABILITY

The subject of this technical instruction (TI) is to describe the procedures for calibrating the temperature of the oven.

2. SUMMARY OF THE METHOD

Temperature plateaus in the IMPROVE A protocol and used for the analysis of CSN samples are measured by temperature calibration device provided by the Sunset Laboratory Inc. A temperature calibration is performed every six months or as needed after replacement of main oven or adjustment of insulation.

3. **DEFINITIONS**

Not applicable.

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 receive and inventory CSN samples.

8. EQUIPMENT AND SUPPLIES

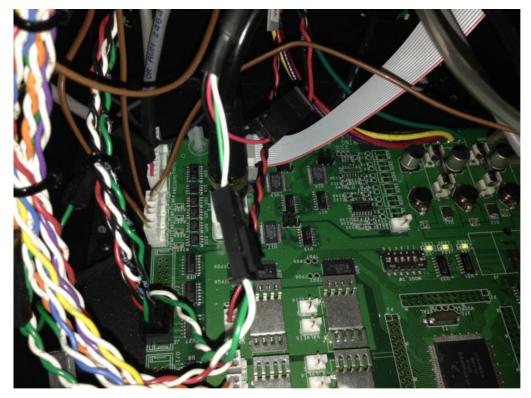
- 1. 7/16 inch open-ended wrench
- 2. ½ inch open-ended wrench
- 3. Phillips screw driver
- 4. Temperature Calibration device
- 5. Flashlight
- 6. Sunset Laboratory Temperature Calibration Template

9. PROCEDURAL STEPS

The ovens must be at room temperature prior to performing the temperature calibration. Refer to *UCD TI #402E: Instrument Startup and Shutdown* for instructions on shutting

down the analyzer. Use a Phillips screw driver to remove the cover screws from the main instrument. Remove the main instrument cover.

- 1. Turn off the power switches to the main instrument, the main oven and the methanator.
- 2. Disconnect the power cord from the methanator to prevent damage to the instrument.
- 3. Remove the front oven cap and use the 7/16" wrench to remove the Swagelok fitting from the front of the instrument. Place the front oven cap assembly on a laboratory wipe with a label indicating which instrument it belongs to.
- 4. Remove the sample holder from oven and place on a laboratory wipe with a label indicating which instrument it belongs to.
- 5. Remove the corresponding temperature calibration device from box and place the Oring over the thermocouple.
- 6. Carefully insert the thermocouple into the main oven via the ball joint opening and secure with the clamp.
- 7. Disconnect the laser housing reflectance connector and carefully remove the laser housing by gently rotating back and forth while simultaneously pulling up.
- 8. Verify the tip of the thermocouple is in the center of the laser pathway by looking down the oven laser tube with a flashlight directed through the side vent of the oven. If this is not the case then adjust the insertion length of the thermocouple by loosening the locking nuts on the thermocouple fitting using both wrenches.
- 9. Reconnect the laser housing to the oven port.
- 10. Connect the tubing from the Temperature Calibration device to the Front oven port using the 7/16" wrench. Do not over-tighten.
- 11. Disconnect the methanator thermocouple connector from the CPU board and attach the temperature Calibration device to the same CPU board connection. The correct connection is shown in the picture below.



- 12. Place the main instrument cover over the main instrument. Turn on the instrument (only power switch and the main oven power switch) using *TI402E- Instrument Startup and Shutdown* instructions on startup the analyzer.
- 13. **Do not connect the methanator**. Note the Sunset manual requests turning on the FID, however there is no need as the methanator is off and disconnected.
- 14. Take the instrument in the "Out of Standby" position
- 15. <u>Conditioning Run</u>: Open the Sunset OCEC software. From the parameter drop down menu select **Temperature Calibration of NIOSH 930.par**.
- 16. In the Output Raw Data file, select the folder U:\CSN\Carbon analyzers\Temp Cal and name the file by "instrument name_conditioning_date.txt" (e.g., alpha_conditioning_02012019.txt).
- 17. In the instrument parameters file update the analyzing pressure to 0.20, make note of original value. Save the file and reload the parameters file by expanding the Status window. Click "Reload Params" on the "Status" window of the software.

- 18. Verify the Desired Methanator Oven temperature is at ~20 °C. Then minimize the "Status" window.
- 19. Use Conditioning Run for SAMPLE ID #, analyst initials in Analyst, and select **1.00** sq cm for Punch Area.
- 20. Check the back oven is at 870 ± 10 °C if not wait until the temperature has stablized before continuing to the next step.
- 21. Select **Start analysis** and click **OK** to the "Laser is too high!" error message.

 Note: the methanator is off, there will be no Calibration peak at the end of the run.
- 22. <u>Temperature Calibration Run:</u> After the conditioning run is completed, from the parameters drop down menu select **temperature calibration of combined**niosh_improvea_eussar2. The temperature calibration will be performed for all three protocols.
- 23. Name the Output Raw Data file as "instrument name_tempcal_date.txt" (e.g., alpha_tempcal_02012019).
- 24. Type **TEMP CAL** in the SAMPLE ID #, type the analyst's initials in "Analyst" and select **1.00 sq cm** for Punch Area.
- 25. Start the run by clicking on **Start analysis**.
- 26. After run has completed, open the text file in Excel, check the **delimited with commas** box.
- 27. Open the Sunset Laboratory Temperature Calibration Template for Combined procedures. The template is located in: U:\IMPROVE_Lab\Carbon Analysis Lab\Daily Operation files\Maintenance\Temp Cal\Sunset Laboratory Temperature Calibration Template Combined NIOSH930-IMPROVEA-EUSAAR2.xlsx
- 28. Copy and paste the Temp cal data to the "All data" worksheet for the combined procedure template. Save the file as Temp Cal Instrument name_date (e.g., Temp Cal alpha_02012019).
- 29. Check the Summary worksheet for calculated offsets for each protocol.
- 30. Update temp offsets summary.xlsx in U:/IMPROVE_lab/Carbon Analysis Lab/Daily Operation files/Maintenance/Temp Cal

- 31. Open the IMPROVE A parameters file and update the temperature offsets. Save the file as improve a date.txt (e.g., improve a 02012019). Close the parameters file.
- 32. <u>Verification Run:</u> In the Sunset OCEC software select the most updated IMPROVE A protocol (e.g., improve a 02012019) as the parameter file.
- 33. Open the status window and update "Desired CH4 oven temperature" to room temperature, **25**. Then minimize the status window.
- 34. Name the Output Raw Data file, as instrument name_verification_date.txt (e.g., alpha verification 02012019).
- 35. Type **verification** in the SAMPLE ID #, analysts initials in Analyst and select **1.00 sq cm** for Punch Area.
- 36. Start the run by clicking on **Start analysis**.
- 37. Open the verification file in Excel. Then copy and paste the values from the verification file into the IMPROVE A verify worksheet. Offsets must be within \pm 10 degrees. If the offsets are higher, check for errors in data entry. If offsets are still outside \pm 10 degrees boundaries then restart at step 22, from the "Temperature Calibration Run".
- 38. Once all verification offsets are within \pm 10 degrees then the Temperature Calibration has been sussessful and the oven can be used in normal operation.
- 39. Follow *UCD TI #402E: Instrument Startup and Shutdown* for shutting down the instrument.
- 40. Disconnect the Temperature Calibration device and remove the O-ring. Connect the methanator thermocouple and re-attach the tubing to the front oven. Insert the sample holder back in the oven and use the O-ring to seal the front oven. Reattach the main oven cover with the screws.
- 41. Finally, update the analyzing pressure to the original value in the "Instrument Parameters" file. Save and close the file. Reload parameters file in the "Status" window if the Sunset OCEC software is open.
- 42. Follow startup procedures to start the analyzers.

10. QUALITY ASSURANCE AND QUALTY CONTROL

Not applicable.

11. REFERENCES

1. Sunset Laboratory Inc. Organic Carbon/ Elemental Carbon (OCEC) Laboratory Instrument Manual. Model 5L Revision 8.051.