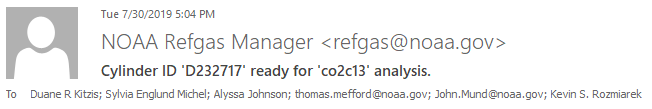
  **Protocol: Incoming Calibration Cylinders**

This document describes the procedure for incoming external cylinders for calibrations at INSTAAR’s Stable Isotope Lab. This lab calibrates cylinders for stable isotopes of CO2 (δ13CCO2 and δ 18OCO2 in air). We also provide informational data for carbon isotopes of methane (CH4). Calibration procedures for each are described in additional documents but handling of cylinders is the same.

This *does not* include how to calibrate the cylinder for specific species, but explains how to properly handle and document incoming cylinders.

Most of the cylinders needed for external calibrations arrive from NOAA where the orders are initiated through. NOAA’s Reference Gas Manager website provides a ‘to-do’ list for what type of analysis is needed on specific cylinders along with order information (i.e. lab/facility, date, location etc.). This information can be found at: <https://om.cmdl.noaa.gov/rgm/index.php>. A username and password is required. Additionally, an email will be sent to the designated personnel who are responsible for analyzing the cylinder.

Email example:



Cylinders arrive at SIL as they become available for analysis. The NOAA delivery personnel will deliver cylinders when they bring flasks and PFP’s (MWF).

The following steps describe the procedure once tanks arrive at SIL:

1. Check that the serial number matches on the cylinder and paperwork attached to tank. Add a necktie to the tank so we can easily distinguish it from our internal tanks.
2. Check in the cylinder on RefGas Manager so the people at NOAA can see that we have received it.
3. Enter cylinder and concentrations into *Session Builder*. You can get this information from the paperwork on the tank or from RefGas Manager. Log into PuTTY, cd /projects/co2c13/flask/sb…then ‘nedit’ corresponding file (i.e. for CO2 external calibrations -> nedit reference\_external.co2c13, then add cylinder to bottom of the list). It is important to make sure the cylinder does not already exist in session builder to ensure all data for specific cylinder is in one file.
4. Find appropriate regulator and put on cylinder. Make sure the regulator is seated properly and flush 3X before closing it off. Mark the pressure on both regulator gauges. Leave overnight. If there’s no change to the pressure, it’s ok to begin running. If the pressure dropped at all (any amount), re-seat regulator and re-test overnight.
5. The procedure for calibrating tanks for CO2 isotopes is to analyze a total of at least thirty measurements. The informational methane isotope analysis is about the same. In general, we are looking for ~30-40 sufficient data points that meet our quality standards. Periodically check data using ‘tank\_view’ program in IDL. Try to spread out the runs a bit (i.e., don’t do 5 tank runs in a row).
6. Once enough data is collected, tank reports are to be completed for each cylinder (following section).

*Completing Tank Reports*

Tank reports are completed for every external calibration cylinder measured. Once the data is compiled and meets our quality parameters, the report is completed, transferred onto the NOAA vortex2 network and the final data is reported in Reference Gas Manager.

1. First open template for species being calibrated (i.e. CO2 calibration letter or CH4 informational letter) both found on Carbonito under ‘Tank Calibrations’ folder. Save-as tank name and species (i.e. ‘CB11025silco2 or CB11025silch4) in ‘Tank Calibrations’ folder under created tank folder. This is the document you will copy and paste final data into and provide to customer.
2. Sign on to Network Connect and log into X11 to access IDL. Run the ‘tank\_view’ program using the following commands:

*For CO2:*

->tank\_view, tank= ‘CB11025’, filldate= ‘02252014’, savegraphs =0

*For CH4:*

->tank\_view, tank= ‘CB11025’, filldate= ‘02252014’, spec=’ch4c13, savegraphs =0

A figure will pop up which allows you to look at all the data run for that cylinder.

1. Run the same command in tank\_view, except savegraphs=1. This will output a data file on FileZilla in vortex2 (found in /projects/co2c13 (or ch4c13)/cals.) Drag file onto Carbonito/Organico and left click to open. Examine the data and look for possible flier/bad runs and ensure the stdev and uncertainties are within our specifications. Additionally, a .psc file (graph) will be created. This file needs to be converted into a .png file. In PuTTY: cd /projects/co2c13/cals/external\_cyl/stats
2. If hand flagging data is necessary, open raw file (cd /projects/co2c13/cals/external\_cyl) and nedit raw file (nedit ‘filename.co2c13’). Here, you can add a ‘!’ flag for bogus data on points that are not representative of that cylinder (i.e. bad run, bad tank line, tank not open etc.)
3. Once satisfied with the data, run tank\_view again and create a new raw data file. Drag into folder created for specific cylinder on FileZilla, save-as Excel file. Once excel file is created, fill in table on calibration letter document by copying and pasting into template (see below). Fill out the remaining information on document (prepared for, date, cylinder id, fill date).

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| **Certificate of Isotopic Calibration** | | | | | | Stable Isotope Lab INSTAAR, Univ. of Colorado, Boulder | | | |
| **Cylinder ID** |  | | | | | | | | |
| **Date filled** |  | | | | | | | | |
| **Date Analyzed** | **n 13C** | **Daily Mean 13C** | **Daily113C** | **13C Daily Uncertainty** | **n 18O** | | **Daily Mean 18O** | **Daily 1**  **18O** | **18O Daily Uncertainty** |
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|  | **Total n 13C** | **13C** | | **Std error** | **Total n 18O** | | **18O** | | **Std error** |
| **Final Value** |  |  | |  |  | |  | |  |

1. Complete calibration report by inserting graph of data at the end of report. This is done by converting .psc figure in IDL to .png (command: convert CB11037.09152011.co2c13.psc CB11037.png) Run the tank\_view program again, savegraphs=1 and the .png file will appear under stats folder in PuTTY (found in /projects/co2c13 (or ch4c13)/cals/external\_cyl/stats).
2. Double check the data was copied and pasted correctly, then file ->save as ->TANKID.report (or silch4) as a .pdf file in tank folder (FileZilla->tank calibrations).
3. Using a secure transfer file system (i.e. FileZilla) copy the tank reports to vortex/the NOAA database (/projects/co2c13 (ch4c13)/cals/external\_cyl/reports).
4. Send Duane Kitzis (Duane.R.Kitzus@noaa.gov) an email with attached copies of tank reports.

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1. Lastly, add final values to NOAA’s Reference Gas Manager website <https://om.cmdl.noaa.gov/rgm> and enter values in internal spreadsheet on the to do list link (Calibration Cylinder Log.xls found on Carbonito/Organico).

Once reports are completed and sent out, regulators can be removed from cylinders, capped and sent back to NOAA via cart people. Please call ahead to let them know there are cylinders (and how many) at INSTAAR that need to be picked up and returned to NOAA. (303)497-3405