

GEOSCIENCES DEPARTMENT
STANDARD OPERATING PROCEDURE

TITLE: Weighing Samples for EA IRMS Analysis

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APPROVAL

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Dept. Representative _____ **Date:** _____

Effective Date: _____

1. PURPOSE

This document describes the procedures that will be used to weigh and prepare standards and samples for EA IRMS analysis.

2. SCOPE

This SOP is applicable to those standards and samples for EA IRMS analysis.

3. RESPONSIBILITY

All persons assigned the task of preparing and/or weighing standards and samples for EA IRMS analysis will understand and implement these procedures.

4. DEFINITIONS

4.1. Blank - A sample tin prepared for analysis but contains no sample.

4.2. Double Blank - No sample and no sample tin.

4.3 International (Primary) standard – A standard (from a vendor such as NIST, NBS, Costech, etc) which has been assigned a value (e.g., $\delta^{13}\text{C}$, ^{15}N , etc.) through rigorous testing and is supplied with a certificate of analysis stating the certified value (and to what scale, e.g., VPDB, etc.) and tolerances. Primary standards are typically expensive, in limited supply and not for use on a day to day basis. Primary standards are typically used to assign values (δ , wt. %, etc.) for individually chosen Laboratory (Secondary) standards through rigorous testing.

4.4. Laboratory (Secondary) standard – A daily use standard that has a value (δ , wt. %, etc.) that has been assigned through comparison/validation with Primary standards. Laboratory standards are used in the day to day running and analysis of unknowns.

4.5. Sample – Generically refers to both an unknown sample and a Standard (Primary or Secondary).

- 4.6. Sample Tin – A container, which comes in various sizes, used to contain the sample for analysis and which also aids the combustion.
- 4.7. Sample Tray – Typical a 48 or 96-well plate used to contain weighed samples prior to sample loading.

5. PROCEDURES

- 5.1. Before weighing standards and/or samples, verify what the approximate analysis weight of the standards and/or samples should be. The amount of material to analyze depends on the estimated/known amount of carbon, oxygen, nitrogen, etc. in the sample. Enough material needs to be analyzed to attain adequate signal-to-noise ratios, but not so much as to saturate the detector response.
- 5.2. To keep the weighing process orderly, use a copy of the form **Sample Weighing Form** to record the ID's and corresponding weights of samples. This list should include the name of the standard and/or sample, the target weight, the actual weight, and the location of the weighed sample. (Note: target weight is just that, a target, generally the weight of the material for analysis should be +/- 20% of target weight.)
- 5.3. Before the weighing process begins, inspect the area where the process will occur. Clean any clutter and dust from the area.
- 5.4. Wear appropriate gloves (oils from your skin will contaminate the samples).
- 5.5. Before weighing the first sample of the day, verify the function/accuracy of the balance by weighing the precision scale weights (1mg, 10mg, and 50mg). Record the values in the calibration logbook. This is not a calibration of the balance, it is a verification of calibration. The verification weights should be within +/- 0.5% of the certified values. If these values are not within tolerance, stop the process and alert GeoSciences or Instrumentation faculty or staff as soon as possible. Do not continue until appropriate faculty or staff has addressed the issue and given authorization to continue.
- 5.6. Clean the tools used for weighing before you begin. This could be as simple as wiping the forceps, spoon, preparation block, etc. with kimwipes™. Remember, the slightest contamination **WILL** compromise the test results!
- 5.7. Now you are ready to begin. Select an appropriately sized sample tin. Using a forceps, place the empty sample tin on the balance, slide the door closed and press the TARE button. Wait a few seconds for the display to read all zeros and the balance to “beep”. Slide open the door and carefully remove the tin and place it in the sample preparation block.
- 5.8. Using an appropriate utensil, place some sample to be weighed into the tin. When you think the amount of sample in the tin is near the target weight, place the tin with the sample back on the balance, slide the door closed. Wait for the reading on the balance to stabilize, typically “mg” will appear on the right side of the display, when this occurs, the reading indicates the weight of the material you placed in the tin. Record this weight if the target weight range was achieved and continue with the next sample. Otherwise, proceed to Step 5.9.
- 5.9. If the weight is too low, carefully remove the tin from the balance and add sample until the proper weight is achieved. If the weight is too large, carefully remove sample from the tin and re-weigh until the proper weight is achieved. Handle the sample and tin carefully, if the tin is dropped or the integrity of the sample or tin is in question, discard the sample and tin and start

over per step 5.7 **IMPORTANT:** If weight adjustment is needed, do not re-tare the balance between re-weighings. If the tare button is accidentally pressed, discard the sample and the tin and re-start weighing per step 5.7.

- 5.10. Using forceps, carefully fold the sample tin into a ball shape. The procedure for folding the tin will vary depending on the size of the tin and the amount of material. For most sample sizes, fold the open end closed with a forceps, and carefully remove excess air in the tin by gently squeezing the tin from the contents up. Using both forceps, form the top edge of the tin into a “Z” shape (if looking down on the open end of the tin) and lightly squeeze the fold until flat. Next slightly fold the same end over to seal in the contents. Gently bend and fold over the tin again and lightly squeeze the tin until it is a ball shape, there should be no edges or openings in the tin. If sample falls out of the tin during this process, discard the entire tin and sample and start again as in step 5.7. For larger sample sizes, carefully fold the tin into a tight ball shape. Again, if sample falls out of the tin during this process, discard the entire tin and sample and start again as in step 5.7. The goal of this preparation is to form a spherical specimen and eliminate as much residual air in the sample and the tin as possible.
 - 5.11. Lightly drop the tin with the sample onto the preparation block, if any sample appears to have leaked out, discard the tin and sample and start again as in step 5.7.
 - 5.12. Weigh the folded sample once again to verify the weight, if there is a significant difference from the first recorded weight, discard the sample and start again as in step 5.7.
 - 5.13. Verify that the correct weight was entered on the **Sample Weighing Form**. Also record any comments about the sample on this form.
 - 5.14. Place the sample in a clean sample tray and record on the form the location of the sample in the tray.
 - 5.15. Before continuing to the next sample, make sure the preparation area and balance are clean of any residual material from the previous sample. Note: the slightest bit of contamination will render an analysis inaccurate.
 - 5.16. If you cannot finish weighing all the samples during one session (or the weighing is complete but they will not be analyzed that day) place the lid on the sample tray and wrap it with a rubber band. Make sure the sample tray can be easily identified. Store the sample tray in a desiccator. Keep your sample records in a safe location
6. REFERENCE DOCUMENTS
 - 6.1. Sartorius Balance Manual
 - 6.2 Sample Weighing Form
 - 6.3 Balance Calibration Log Book
 7. REVISIONS AND REASONS
 - 7.1. Original