



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L208

ThermoFisher Delta V IRMS – General Instrument Use

Standard Operating Procedure

SOP Number	SOP-L208
Revision	1.1
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Date	2026-06-26
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1. Purpose

This SOP describes the general operation, care, and policies governing the ThermoFisher Delta V Isotope Ratio Mass Spectrometer (IRMS) and its four front-end peripherals in the Oxtoby Environmental Isotope Lab (Coplen, 2011; Werner & Brand, 2001). It is the single instrument-level reference for all IRMS work. Individual application SOPs (e.g., [SOP-L414](#) for soil CN analysis, [SOP-L415](#) for carbonate isotopes) build on this SOP and should be read in conjunction with it.

2. Scope

- **Applies to:** The Delta V IRMS and all peripherals (ConFlo IV, Flash IRMS EA, TC/EA, GasBench II) in the Oxtoby Isotope Lab, SGM Rm 135, Pomona College.
- **Does not apply to:** Application-specific sample preparation, data reduction, or QC criteria. See the relevant application SOP.
- **Location:** David W. and Claire B. Oxtoby Environmental Isotope Lab (SGM Rm 135). Sample preparation: wet lab (SGM Rm 133).

3. Responsibilities

Role	Responsibility
Lab PI	Approve SOP; authorize new users
Lab Manager / Super-User	Certify new users; maintain reactors and gas supply
Certified User	Operate instrument per this SOP; log all sessions
Student Researcher	Operate only under supervision until certified

4. Definitions

- **ConFlo IV:** Interface hub connecting peripherals, reference gases, and the IRMS; handles reference-gas dilution and peak-center switching.
- **Delta V IRMS:** The ThermoFisher Delta V Isotope Ratio Mass Spectrometer housed in the Oxtoby Isotope Lab, SGM Rm 135.
- **Flash IRMS EA:** Elemental analyzer peripheral that combusts samples at 1020 °C to produce N₂, CO₂, and (optionally) SO₂ for simultaneous $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ analysis.
- **GasBench II:** Low-flow peripheral for headspace gas sampling from sealed vials; used for carbonate CO₂, DIC, and water $\delta^{18}\text{O}$.
- **H₃⁺ Factor:** Correction applied to $\delta^2\text{H}$ data for H₃⁺ ions formed in the source that interfere with HD⁺ at m/z 3.
- **Isodat:** ThermoFisher software used to design analytical sequences, control the instrument, and export raw isotope data.
- **Linearity:** The dependence of measured δ on signal amplitude; a well-tuned instrument shows minimal linearity offset across the working signal range (Werner & Brand, 2001).
- **Super-User:** Staff or faculty member certified for IRMS operation and minor maintenance (gas replacement, reactor exchange).
- **TC/EA:** Thermal Conversion/Elemental Analyzer peripheral; pyrolyzes samples at ~1450 °C to produce H₂ and CO for $\delta^2\text{H}$ and $\delta^{18}\text{O}$ analysis.
- **User:** Student, staff, or faculty member certified to prepare samples and operate the IRMS without direct supervision.
- **Zero-Enrichment Test:** Series of reference-gas on/off pulses run before analysis to verify the IRMS produces consistent δ values independent of signal amplitude (Coplen, 2011).

5. Safety and Hazards

Hazards

- **Compressed gases:** He, O₂, CO₂, N₂, SO₂ (where used). Cylinders must be chained; O₂ must be stored away from flammables.
- **High-voltage electronics:** The IRMS source operates at high voltage. Do not open the source housing without authorization.
- **High temperatures:** Flash EA reactor operates at 1020 °C; TC/EA at ~1450 °C. Allow to cool before maintenance.
- **Chemical:** Tin capsules contain sample material; some samples may be hazardous. Handle per the sample-specific SOP.

5.1. Required PPE

- ☐ Safety glasses
- ☐ Lab coat
- ☐ Nitrile gloves (during sample loading and reactor maintenance)

5.2. Required Training

- [SOP-L002 Laboratory Safety](#)
- This SOP (Section on User Certification) before any independent use

6. Equipment and Materials

Item	Location	Notes
Delta V IRMS	SGM Rm 135	Do not power off without authorization
ConFlo IV interface	SGM Rm 135	Always on when IRMS is on
Flash IRMS EA	SGM Rm 135	Application-specific reactor config
GasBench II	SGM Rm 135	Requires dedicated Isodat method
TC/EA	SGM Rm 135	High-temperature; Super-User only for maintenance
He supply (cylinder)	SGM Rm 135	Check pressure before each session
Reference gas cylinders	SGM Rm 135	CO ₂ , N ₂ , H ₂ , SO ₂ as needed
Tin capsules (5 x 9 mm)	SGM Rm 133	Sample prep area
Lab logbook	SGM Rm 135	Sign in/out for every session

7. Procedure

7.1. User Certification

Before operating the IRMS independently, personnel must complete the Training Record (Section 13). Contact the lab manager to schedule certification.

7.2. Pre-Analysis Checklist

1. Sign in to the lab logbook.
2. Verify He carrier gas pressure (minimum 1500 psi on the regulator).
3. Check reference gas pressures (CO₂, N₂ as applicable).
4. Confirm Isodat is running and the instrument status screen shows no error flags.
5. Verify the correct peripheral is selected in Isodat.
6. Run a Zero-Enrichment Test (3 on/off pulses) and confirm δ values are stable within $\pm 0.1\%$.
7. Run an on/off linearity check if not done within the past 48 h.

7.3. Analytical Sequence Design

1. Open the Isodat Sequence Editor.
2. Load the appropriate method file for the peripheral and analyte.
3. Build the sequence: blank(s), conditioning samples, reference materials, unknowns, check standards, blanks.
4. Reference material placement: minimum 1 RM per 10 unknowns; two-point normalization requires at least 2 different RMs.
5. Save the sequence file using the naming convention: `YYYYMMDD_Project_SeqID_User.seq`

7.4. Starting and Monitoring a Run

1. Load samples into the autosampler or carousel per the application SOP.
2. Press **Start** in the Isodat Sequence Editor.
3. Monitor the first 3–5 injections on-screen to confirm peaks are present, within expected amplitude, and baseline-resolved.
4. Do not leave the instrument unattended during the first 30 min.

7.5. Shutdown

1. After the sequence completes, export raw data per the application SOP.
2. Remove all samples and consumables from the autosampler.
3. Log gas usage and any issues in the lab logbook.
4. Leave the instrument in standby per the lab manager's current instructions (the Delta V and ConFlo IV are typically left powered on continuously).

8. Troubleshooting

Symptom	Possible Cause / Action
High m/z 28 background	Air leak; check fittings and reactor seals
High m/z 18 background	Moisture; extend bake-out time
High m/z 40 background	Air leak (Ar); check fittings
Poor standard reproducibility	Capsule sealing; exhausted reactor
No peaks in chromatogram	Gas flow interrupted; check He supply
Sequence stops mid-run	Autosampler jam; check carousel alignment
On/off tests fail repeatedly	Source needs tuning or maintenance
Red/yellow lights on IRMS panel	Check Isodat error log; contact lab manager

If you cannot resolve an issue, **stop the run** and contact the lab manager. Do not attempt reactor replacement or major maintenance unless you are a certified Super-User.

9. Quality Control

- Run at least two certified reference materials per analytical sequence for two-point scale normalization (Paul et al., 2007).
- Reference material δ values must recover within $\pm 0.2\%$ of certified values; flag sequences that exceed this threshold.
- Blanks (empty tin capsules) must produce no detectable N_2 or CO_2 peak above the noise baseline.
- Archive all raw Isodat sequence files per [SOP-L801](#).

10. References

- Coplen, T. B. (2011). Guidelines and recommended terms for expression of stable-isotope-ratio and gas-ratio measurement results. *Rapid Communications in Mass Spectrometry*, 25(17), 2538–2560. <https://doi.org/10.1002/rcm.5129>
- Paul, D., Skrzypek, G., & F'orizs, I. (2007). Normalization of measured stable isotopic compositions to isotope reference scales – a review. *Rapid Communications in Mass Spectrometry*, 21(18), 3006–3014. <https://doi.org/10.1002/rcm.3185>
- Werner, R. A., & Brand, W. A. (2001). Referencing strategies and techniques in stable isotope ratio analysis. *Rapid Communications in Mass Spectrometry*, 15(7), 501–519. <https://doi.org/10.1002/rcm.258>

11. Related SOPs

SOP	Title	Relationship
L002	Laboratory Safety	Required prerequisite

SOP	Title	Relationship
L414	Soil CN by Flash EA-IRMS	Application SOP
L415	Carbonate Isotopes by GasBench-IRMS	Application SOP

12. Training Record

Before using the IRMS independently, personnel must:

1. Read this SOP in full
2. Complete [SOP-L002 Laboratory Safety](#)
3. Read the application SOP for the peripheral(s) they will use
4. Observe at least one complete analysis session by an experienced user
5. Complete supervised practice runs
6. Demonstrate a start-to-finish analysis to the satisfaction of the lab technician or a Super-User
7. Be signed off by the lab manager or PI