GEOSCIENCES DEPARTMENT

STANDARD OPERATING PROCEDURE

TITLE: Loading Samples in the EA Autosampler

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APPROVAL

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Effective Date: _____

1. PURPOSE

This document describes the procedures that will be used to load standards and samples in the Elemental Analyzer (EA – Costech) autosampler for IRMS analysis.

2. SCOPE

This SOP is applicable to persons who will analyze standards and samples for EA-IRMS analysis.

3. RESPONSIBILITY

All persons assigned the task of loading standards and samples for EA-IRMS analysis will understand and implement these procedures.

4. DEFINITIONS

4.1. 50 Sample Zero-Blank Autosampler – Has positions for 50 samples, but in reality can be used for 49 samples. Due to the large diameter of the sample port in the 50 sample autosampler it must be used for larger samples which cannot fit (or are of a questionable fit) in the 100 position autosampler.

4.2. 100 Sample Zero-Blank Autosampler - Has positions for 100 samples, but in reality can be used for 99 samples. Due to the smaller diameter of the sample port in the 100 sample autosampler, the physical size of the prepared sample will be the determining factor for using this autosampler.

5. PROCEDURES

5.1. Isolate the EA and Conflo from the mass spectrometer by closing the needle valve on the front of the mass spectrometer. The needle valve is delicate and can be easily damaged by applying too much force to close it, finger tight should be enough.

5.2. Turn off the ionization filament and turn on the helium diluter (from either the instrument control or instrument acquisition programs).

5.3. Determine which autosampler will be used (based on sample physical size), if the appropriate autosampler is already installed, continue to Step 5.4. If the autosampler needs to be changed, notify laboratory management. Note: Exchanging autosamplers is beyond the scope of this SOP and shall be performed by laboratory management personnel.

5.4. Verify that the mass spectrometer needle valve is closed, the helium diluter is on, and the filament is off.

5.5. To vent the EA autosampler to atmosphere, slowly open the vent valve on the top of the autosampler, you should hear a whistling sound as the EA vents.

5.6. Once vented, loosen the three screws holding the three clamps that secure the lid of the autosampler. Move the clamps out of the way and open the lid of the autosampler.

5.7. Examine the autosampler for cleanliness. Verify there are no samples or remnants of samples remaining from the previous analysis.

5.8. Check the alignment of the autosampler over the combustion tube. When looking down into the combustion tube from above through the autosampler sample port, you should see a complete circle of the sample port in the combustion tube, or a slight flat area in circle on the left side of the sample port. This will allow the sample to drop into the combustion tube without catching on the lip of the autosampler base as the autosampler increments itself.

5.9. To adjust the alignment, press the down arrow key on the front panel display on the EA until "EL. AUTOSAMPLER" is displayed. There are three choices on this menu:

1. Go

2. Jog+

3. Jog-

"Go" will increment the autosampler as if it were advancing to drop the next sample. "Jog+" will allow the autosampler to move clockwise (forwards) continuously at slow speed until stopped by the user, "Jog-" will allow the autosampler to move counter-clockwise (backwards) continuously at slow speed until stopped by the user. "Jog+" and "Jog-" are used for adjusting the alignment of the next sample port on the autosampler to ensure that the sample port is centered over the combustion tube and that the sample is unencumbered from dropping.

5.10. To adjust the alignment, down arrow to "Jog+", then right arrow once and press the "ENTER" key, this will start the autosampler rotating slowly in a clockwise direction. This rotation will continue until the "ENTER" key is pressed again. If there is slack in the autosampler, it may take a few seconds before the autosampler starts to turn, this is normal. Note: When going from "Jog+" to "Jog-", or "Jog-" to "Go", there will be slack to be taken up in the autosampler. Always finish an alignment with a "Jog+" or "Go".

5.11. Look down through the sample port into the combustion tube as the autosampler rotates, when the alignment is acceptable, press the "ENTER" key to stop the rotation. This process will

take some practice and may need to be repeated several times, this is why it is performed before any samples are placed in the autosampler.

5.12. Once aligned, to increment the autosampler one sample port at a time, press the right arrow to "Go". Each time the "ENTER" key is pressed the autosampler will increment through one sample. Increment the autosampler until the port you want for your first sample is to the right of the port which drops through to the combustion tube. Note: Your first sample does not have to be in sample port '1", however it can make loading 50 or 100 samples easier if the autosampler is rotated until port labeled "1" is the first sample to be tested.

5.13. As long as the autosampler is not accidently moved, it will remain in alignment. If it is moved while loading samples, alignment will need to be verified and possibly adjusted before preceding.

5.14. Begin loading your samples. Using an appropriate utensil (typically forceps), pick up a sample from the sample tray and carefully place the sample in the correct sample port. Be very careful not to drop any samples, do not accidentally place two samples in one port.

5.15. When all samples have been loaded, check the o-ring seal on the autosampler. Verify the o-ring is seated properly, rub you finger around the o-ring to remove any dust or particles from the sealing surface. If any cracks or breaks are observed in the o-ring, replace it.

5.16. Carefully close the lid of the autosampler, press the lid onto the o-ring. Place the three clamps back on the autosampler lid and tighten the three thumb screws on the clamps equally and securely. You should hear gas coming from the vent valve on the lid of the autosampler (as in step 5.5).

5.17 After three to five minutes (this purges residual air from the system that entered when sample loading), close the vent valve sealing the system.

5.18. If the sequence method has not been entered into the Instrument Acqusition program yet, this is a good time to create the new test sequence for this analysis. You could edit an existing sequence with the information, or create a new one.

5.19. Slowly open the needle valve on the mass spectrometer to allow EA gases to be sampled.

5.20. Turn on the ion source filament.

5.21. Turn off the helium diluter.

5.22. Allow the instrument to stabilize for at least fifteen minutes before starting the analysis.

5.23. When the analysis of all the samples in the autosampler is complete, the instrument should be placed in stand-by. Isolate the mass spectrometer by closing the needle valve, turn off the ion source filament and turn down the reference gases on the Conflo to ~0.5 bar.

5.24. Since the samples are consumed during the analysis, the autosampler does not need to be opened (vented) until the next group of samples are ready for analysis.

6. REFERENCE DOCUMENTS

6.1. Costech ECS 4010 manual, Sections 3.5 and 10.0.

7. REVISIONS AND REASONS

7.1.	Original