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THE USE OF CARLO ERBA MODEL 1112 FLASH ELEMENTAL ANALYZER FOR THE ANALYSIS OF TOTAL CARBON AND NITROGEN IN SEDIMENTS AND FILTERED PARTICULATE MATTER

Prepared By: Nathan Saxe

Approved By: _____ Date:_____

Robin S. Davis
Quality Assurance Unit

The Use of Carlo Erba Model 1112 Flash Elemental Analyzer for the Analysis of Total Carbon and Nitrogen in Sediments and Filtered Particulate Matter

1. METHOD.

   1.1. This is a method for analyzing total organic carbon and/or nitrogen content in sediment/algae or filtered particulate matter using a Carlo Erba Model 1112 Flash Elemental Analyzer or equivalent.

2. SUMMARY.

   2.1. The sample is carefully placed into a tin boat either by weight or by filtered volume. The boat is closed and, following the manufacturers guidelines, the sample is combusted at 1000°C. The resultant CO_2 and N_2 gas evolved is separated via GC and the mass is determined with a thermal conductivity detector. If the sample contains inorganic carbon, the inorganic carbon is removed by fuming HCl overnight, dried, then the sample is placed into a cleaned tin boat for combustion and analysis in the CHN.

3. APPARATUS.

   3.1. Carlo Erba Model 1112 Flash Elemental Analyzer or equivalent, autosampler rack and cover, combustion and reduction columns, and gases (UHP Helium and Oxygen). The instrument is set up following manufacturers guidelines.

   3.2. Eager 300 Software installed on a compatible computer.

   3.3. A constant temperature drying oven, capable of maintaining 60 ± 5˚C.

   3.4. Micro-balance accurate to at least 3 decimal places (e.g. 0.000 mg).

   3.5. Hydrochloric Acid (concentrated).

   3.6. Drying desiccator (glass or plastic).

   3.7. Acid-washed glass or plastic petri dishes (preferably glass).

   3.8. Acetone (reagent grade) and squirt bottle.

3.9. Acetone cleaned tin boats (3.5 x 5 mm).

3.10. Acetone cleaned pelletizer die and assorted micro-forceps.

3.11. Muffled aluminum foil.

3.12. Aspartic Acid, crystal form (dried in a low temperature oven and in desiccator).

3.13. SRM-CE Soil or Spinach (1570a).

4. PROCEDURE.

4.1. Filtration and collection of samples should follow SOPs for water filtration of organic particles or sediment collection. Filters need to be pre-combusted glass fiber filters (e.g., Whatman GFF) and all material should be cleaned prior to and during filtration. Sediment samples should be collected with cleaned sediment sampler.

4.2. Removal of inorganic carbon.

4.2.1. Filters or sediments are placed in acid-washed glass petri dishes, which are then placed in a large glass desiccator (without desiccant).

4.2.2. Fill a glass petri dish halfway with concentrated hydrochloric acid, carefully place dish in the center of the desiccator and quickly replace lid.

4.2.3. Samples are left overnight in the fuming HCl, in order to remove the inorganic C from the sample. The next day the filters and sediments are placed in a 60 ± 5˚C drying oven overnight.

4.3. Tin boat/instrument cleaning.

4.3.1. Place an appropriate number of tin boats in a clean 100-ml beaker, add enough acetone to completely cover the boats, swirl, decant, repeat two more times and place the beaker in the drying oven for a minimum of three hours. Remove the beaker, and cover with muffled aluminum foil.

4.3.2. Using acetone filled squirt bottle, liberally squirt pelletizer die and each micro-forceps, shake off excess, place on muffled foil and dry in the drying over for a minimum of three hours. Upon removal from the oven, wrap in muffled foil.

4.4. Sample/boat Preparation.

4.4.1. Remove a glass petri dish containing sample from the desiccator. Every other sample regardless of type will be a duplicate. Place a cleaned tin boat in the pelletizer die.

4.4.1.1. If analyzing particulate filters, using the pelletizer die as a work surface, the filter must be folded twice in order to fit the filter into the boat. Using forceps, place the folded filter into the boat and pack it down. Be careful not to lose any material from filter.

4.4.1.2. If analyzing sediments, tare the tin boat on the microbalance, place in die, using scoop tool, place a small amount of sediment in boat (low mg quantities), weigh, record weight.

4.4.2. Remove boat from die, z-fold the top, and fold over the top and pack the boat until it is a three-dimensional cube or sphere.

4.4.3. Place boat in labeled tray or autosampler and record sample location. Dip all forceps used into a 100-ml acetone filled beaker, to clean them. Using a Kimwipe, dipped in acetone, clean the pelletizer die. Repeat procedure for next sample. Be sure all acetone has evaporated away.

4.5. Blank and Standard Preparation.

4.5.1. Three types of blanks, gas, boat and filter, will be used during the analysis.

4.5.1.1. Gas blanks will consist of nothing more than an empty chamber on the autosampler rack. This will represent baseline C and N.

4.5.1.2. Boat blanks will consist of empty folded boats placed in the autosampler.

4.5.1.3. Filter blanks, during particulate analysis represent the lab/field blank (DI filtered through), sample preparation is the same as all of the other samples. This value will be subtracted from the samples as the blank.

4.5.2. Using a tared boat, weigh out a representative amount of the Aspartic Acid standard (~1mg)and record the weight. Also using a tared boat, weigh out a representative amount (~25 mg) of the SRM to be used and record the weight. Run blanks, SRMs and standards at intervals as outlined in the project protocol, usually every 10 samples.

4.5.3. Standard amounts for the curve and the standard unknowns should be calculated from the C and N expected in the samples. Here are typical weights used for calibration:

mg of Aspartic Acid	N (µg)	C (µg)
0.50	53	180
1.00	105	361
1.50	158	541
2.00	210	722
2.50	263	902

4.6. Sample Analysis.

4.6.1. Sample runs should consist of the following:

- A standard curve with a Bypass, Blank, and five standards
- An Aspartic, SRM and Boat blank as standard unknowns
- Unknown samples run in duplicate
- Run standard unknowns for all the samples (~ every 10 samples)

4.6.2. Follow manufacturers guidelines for the operation and set up of instrument.

5. REFERENCES.

5.1. Carlo Erba Manuals.

5.2. US. Environmental Protection Agency. 1992. Determination of carbon and nitrogen in sediments and particulates of estuarine/coastal waters using elemental analysis. Method 440.0 *In:* Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Samples. EPA/600/R-92/121 ORD, Washington DC.