Determination of Carbon and Nitrogen collected on Whatman glass fiber filters (GFF).

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NAME: Perkin Elmer CHNS/O Analyzer 2400 Series II
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I. Introduction and Summary

The PE 2400 is a commercially available elemental analyzer that uses a combustion method to convert the sample elements to simple gases (CO_2, H_2O, and N_2). The sample is first oxidized in a pure oxygen environment. Elements such as halogens and sulfur are removed by scrubbing reagents in the combustion zone. The resulting gases are homogenized and controlled to exact conditions of pressure, temperature, and volume. The homogenized gases are allowed to de-pressurize trough a column where they are separated in a stepwise, steady-state manner and measured as a function of their thermal conductivities.

II. Calibration

Accurately weighed samples of a known standard, usually acetanilide (C_8H_9NO), are first analyzed to calibrate the analyzer in milligrams of carbon and nitrogen. A calibration curve is generated to calculate the carbon (C) and nitrogen (N) content of unknown samples.

The instrument uses a chromatographic approach to separate the measured gases, as they elute, each gas separates as a steady-state step, with each subsequent gas added to the previous one. Consequently, each step becomes the reference for the subsequent signal (Fig. 1).
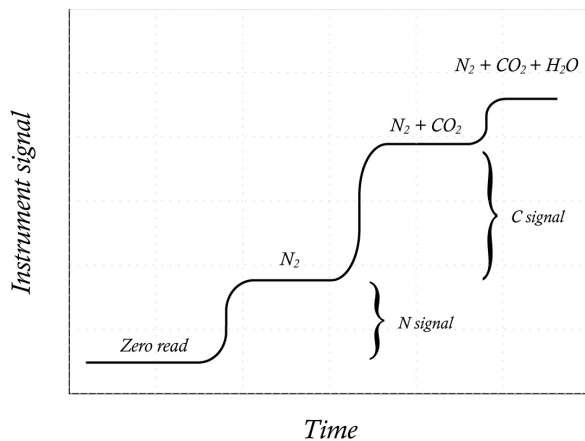

Figure 1. Schematic of the separation of gases and their corresponding instrument signal during a sample run in the Perkin Elmer CHN 2400.

The instrument signal that relates to the N content in the sample, the N signal (*NS*), equals the difference between the N read (*NR*) and the Zero read (*ZR*) (eq.1); the C signal (*CS*) equals the C read minus the N read (eq.2). The instrument also generates a hydrogen signal (*HS*), but in this context that signal is ignored.

$$NS = NR - ZR \qquad\qquad \text{eq. 1}$$

$$CS = CR - NR \qquad\qquad \text{eq. 2}$$

Least squares regressions are performed on the analytical blank-corrected C and N signals signals (*NS, CS*) for acetanilide standard runs versus their corresponding C and N content in milligrams at 10^{-3} mg precision. The C and N content of the acetanilide standards is equal to their weight multiplied by the C and N weight fraction in acetanilide, 0.7109 and 0.1036, respectively.

Blanks

Various types of blanks are performed during a typical run on the PE 2400. A few "air blanks" (i.e. empty run) are performed at the beginning of a run to stabilize the instrument. The usual number of air blanks is between 3 and 6; the instrument is deemed stable when the air blanks are reproducible to *CS, HS* and *NS* values ± 30, 100, and 16, respectively. Air blanks are excluded during analytical calibration calculations.

Once the instrument is stable, acetanilide standards (<2 mg) wrapped in 5x8 mm tin capsules (PE N241-1255) are run alternatively with "analytical blanks", which involve running an empty tin capsule. Usually, three pairs of analytical blanks and standards are run, after which two check acetanilide standard runs and two NIST standard reference material (Buffalo River Sediment, NIST #8704) runs are performed for initial quality control (QC). Approximately every 6 to 8 regular sample runs, check standards are run, and every 10 samples, additional acetanilide standards calibration run are introduced.
During the course sample run, three or more "method blanks" are performed. Method blanks are GFF filters that are processed as field samples in the laboratory. Average values from method blanks are subtracted from the C and N values found for samples.

III.   Sample Processing

Water samples are filtered through 2.5 cm pre-combusted Whatman GFF filters using an all-glass filtering funnel. Once enough material has been accumulated on the filter the samples are stored in Nalgene 1.8 mL cryovials at -80°C until analysis. For shipping, samples are stored in liquid nitrogen dry-shippers.

Prior to analysis samples are thawed and dried overnight at 65°C inside the uncapped cryovials. Dry samples in the uncapped vials are transferred overnight to a desiccator saturated with HCl fumes to remove particulate inorganic carbon present in the

sample. The air in the desiccator is kept saturated by leaving concentrated HCl in an open container in the lower compartment of the desiccator. Acid–treated samples are transferred back to a 65°C oven overnight, after which can be stored capped in cryovials for extended periods prior to analysis in elemental analyzer.

For analysis the samples are wrapped in tin foil discs (PE N241-1363) and pelletized on a Parr Pellet press.

The samples are analyzed in the Perkin Elmer CHNS/O Analyzer 2400 Series II. The manufacturer's data management software for the elemental analyzer is set up so that the analysis report includes the zero read (ZR), and the carbon and nitrogen signals (CS, NS) from the instrument for each analyzed sample.

IV.　　Data process and calculations

Carbon and nitrogen content in milligrams in each sample filter is calculated from the linear regressions estimated during calibration calibration, which relate the instrument signals for each element (CS, NS) to the C and N content by weight of the acetanilide calibration standards. The regression estimated C and N values are corrected by subtracting the average "method blank" values for each element. If the method blank values are negative, no correction is applied. To obtain actual POC and PON values, the values generated above are normalized by the volume filtered and the average of the replicates collected at each sampling location is reported.

Data processing is performed in Matlab using a script that generates a data report (Appendix 1) that includes the C and N content in mg for each filter replicate sample.

V. Quality Control

Analytical performance is evaluated by performing an error analysis on the Buffalo River sediment standard reference material runs (Appendix 1).

Appendix 1: Data processing script