

Total Nitrogen/Total Carbon by Dry Combustion Method Summary

- 1. Principle:** This method describes the determination of total N and/or total C in soil, sediments, plant, or biologic material by flash combustion. Complete combustion of the sample is achieved by dropping a known mass of sample, packed into a tin or silver capsule, into a combustion tube containing chromium (III) oxide and silvered cobaltous/cobaltic oxide catalysts. An aliquot of purified oxygen is added to the quartz tube generating a flash combustion reaction, increasing the reaction temperature from 1020°C to 1800-2000°C. The carbon in the sample is converted to CO₂, and the nitrogen is converted to N₂ and NO_x. These combustion gases are carried via UHP Helium through a reduction furnace filled with reduced copper wires, reducing NO_x species to N₂. Depending on the analysis requested, the gas stream now passes through either one or two sorbent traps to remove water (magnesium perchlorate) and, if only N₂ is being determined, CO₂ (EMASorb, formerly Carbasorb). The resulting N₂ gas and/or CO₂ gas is separated on a 2m x 6mm OD stainless steel Porapak QS 80/100 mesh packed chromatographic column and detected quantitatively by a Thermal Conductivity Detector (TCD). The integrated TCD peak signal in the resulting chromatogram is directly proportional to the amount of C and N present in the sample which, along with the sample weight, is used to calculate %C and %N (w/w).

For TOC analysis, samples are weighed in open silver capsules and acidified with sequential additions of 50uL 1M HCl to remove inorganic carbon. HCl is added until no further reaction is observed, as indicated by bubbles formed by the reaction of HCl and carbonate. The samples are then oven dried at 70°C overnight and sealed before analysis.

- 2. Instrument Used:** Thermo FLASH 2000 Organic Elemental Analyzer, Thermo Fisher Scientific Inc., Bremen, Germany 2016.
- 3. References:**
 - 3.1.** Official Methods of Analysis of AOAC International, 17th Edition (2000), AOAC International, Arlington, VA. Method 972.43, Micro-chemical Determination of Carbon, Hydrogen, and Nitrogen, Automated Method.
 - 3.2.** Methods of Soil Analysis, Part 3 – Chemical Methods, Soil Science Society of America Book Series #5, Soil Science Society of America, Inc., Madison, Wisconsin. Dumas Methods, Determination of Total Nitrogen and Total Carbon by Combustion.
 - 3.3.** Thermo FLASH 2000 Elemental Analyzer Operating Manual, 5th ed (P/N 31712052), Thermo Fisher Scientific Inc., Bremen, Germany 2014.
 - 3.4.** Schumacher, B.A. “Methods for the determination of total organic carbon (TOC) in soils and sediments”. NCEA-C-1282, EMASC-001. United States EPA, Las Vegas, **2002**.
- 4. Standards Used:**
 - 4.1. Calibration Standards:** Acetanilide (CH₃CONHC₆H₅), certified. Acetanilide theoretical value of 71.09 % (w/w) C and 10.36 % (w/w) N.
 - 4.2. Continuing Calibration Verification (CCV):** A known mass of the calibration standard (acetanilide) or external reference standard (atropine) is analyzed. Recovery must fall within the following acceptance limits: 95% - 105% (relative) of theoretical value.
 - 4.3. External Reference Standards:** Atropine, certified. Atropine theoretical value of 70.56 % (w/w) C and 4.84 % (w/w) N. Recovery must fall within the following acceptance limits: 95% - 105% (relative) of theoretical value.