Total Carbon, Organic Carbon, Nitrogen and Sulphur Analysis
Flash Combustion Method

Introduction

- Soil carbon is the largest terrestrial pool of carbon and plays a key role in the global carbon cycle.
- Soil organic carbon is also a key determinant of soil quality, affecting cation exchange capacity, supply of nutrients to plants, water holding capacity and soil physical structure.
- Soil carbon is also the principle energy source for microbes and sustains soil biodiversity.
- Soil organic nitrogen and soil organic sulphur are the predominant pools of nitrogen and sulphur in soils and their mineralization is the main supply of nitrogen and sulphur respectively for plant growth and development.
- Soil inorganic carbon accounts for about one third of the total carbon in soils globally and has considerable influence on the physical and chemical properties of soil.

Analytical Method

- Carbon, Nitrogen and Sulphur analysis is based on the flash combustion method.
- Flash combustion method produces complete combustion of the sample within a high temperature reactor followed by an accurate and precise determination of the elemental gases produced.

Figure 1: Schematic working principle of the Flash 2000

Combustion

R·N + O₂ →→ N₂ + NOₓ + O₂ + CO + CO₂ + CH₄ + X⁻ + SOₓ + H₂O

Oxidation

CO + CH₄ →→ CO₂ + H₂O

Reduction

NOₓ+O₂ +SO₃→CuO→ N₂+CuO +SO₂ (trace)

- Suitable for organic or inorganic samples, the method is rapid, about 4 minutes for a complete sample characterization, and easily automated with solid or liquid autosamplers.

Sample preparation

- Samples ground to 75 µm are weighed (20 mg for soils and 2 to 3 mg for plants) using a micro-balance and encapsulated in tin capsules.
- For TOC analysis silver capsules are used since they are not easily corroded by HCl.
- Upon acidification and drying, a silver capsule containing the sample is wrapped in a tin capsule to enhance complete combustion.

Figure 2: Micro balancing

Working Principle

- Samples placed inside the autosampler are dropped at a preset time into an oxidation/reduction column maintained at a temperature of 900-1000 °C.
- An exact amount of oxygen required for optimum combustion is delivered into the combustion reactor at a precise time.
- The reaction of oxygen with the tin capsule at elevated temperature generates an exothermic reaction which raises the temperature to 1800 °C for a few seconds.
- At this high temperature both organic and inorganic substances are converted into elemental gases.
- After further reduction, the elemental gases are separated by a chromatographic column and finally detected by a highly sensitive thermal conductivity detector (TCD).

Total Organic Carbon Determination

- Organic carbon is analyzed after inorganic carbon dissolution.
- This is achieved by acidification of the sample within a silver capsule.
- The carbonates are evolved in the form of carbon dioxide upon sample acidification.
- The silver capsule containing the sample is then encapsulated in a tin capsule and analyzed.
- This technique eliminates reweighing procedures, eliminates loss of acid soluble carbon and is free from matrix interferences.

Figure 3: Acidification and drying

Instrument calibration

- CNS data produced is primarily used to calibrate infrared spectroscopy models.

Figure 4: Calibration

Figure 5: Quality control

Figure 6: A typical Chromatogram showing Nitrogen, Carbon and Sulphur peaks

Results

Figure 7: Carbon Distribution in selected sites of the Africa Soil Information Service

AFSIS SITES

Key advantages of Flash Combustion

- Wide detection range of 0.01 to 100 %.
- The method is widely accepted as a standard due to its simplicity, unparalleled data reproducibility and truly quantitative results.
- Simultaneous determination of Carbon, Nitrogen and Sulphur.
- Environmentally friendly compared to other methods like Kjeldal for Total N and Walkley and Black for carbon analysis.
- Total Organic Carbon analysis by the acidification method is accurate and rapid.
- Flash combustion method is rapid, about 4 minutes for a complete sample characterization, and easily automated.
- The analyzer can be coupled with an Isotope Ratio Mass Spectrometer (IRMS) for isotope analysis and/or with a flame photometric detector for trace sulphur analysis which improves the sulphur detection limit to the nanogram level.

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