



TEST METHODS MANUAL

Laboratory Services Division
Bureau of Soils and Water Management
Department of Agriculture

TM-LSD-04-22

SECTION : SOIL CHEMISTRY

Issue No.: 3

Effective date: April 15, 2024

SUBJECT : **TOTAL CARBON, TOTAL NITROGEN AND TOTAL SULFUR (DUMAS DRY COMBUSTION METHOD)**

Revision No.: 1

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SCOPE

This method is for the simultaneous determination of Total Carbon, Total Organic Carbon, Total Nitrogen and Total Sulfur in soil samples using an autoanalyzer.

PRINCIPLE

The sample weighed in a tin capsule container (silver capsules if total organic carbon is to be determined) and stored in the auto sampler, is dropped into the combustion reactor. The container, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. In the combustion process (furnace at 1000 °C), carbon is converted to carbon dioxide; hydrogen to water; nitrogen to nitrogen gas/ oxides of nitrogen and sulphur to sulphur dioxide. If other elements such as chlorine are present, they will also be converted to combustion products, such as hydrogen chloride. A variety of absorbents are used to remove these additional combustion products as well as some of the principal elements, sulphur for example, if no determination of these additional elements is required. The combustion products are swept out of the combustion chamber by inert carrier gas such as helium and passed over heated (about 600 °C) high purity copper. The function of this copper is to remove any oxygen not consumed in the initial combustion and to convert any oxides of nitrogen to nitrogen gas. The gases are then passed through the absorbent traps in order to leave only carbon dioxide, water, nitrogen and sulphur dioxide. The eluted gases are conveyed to the thermal conductivity detector (TCD). The electrical signals generated by the detector are properly processed by software providing carbon, hydrogen, nitrogen and sulfur percentages.

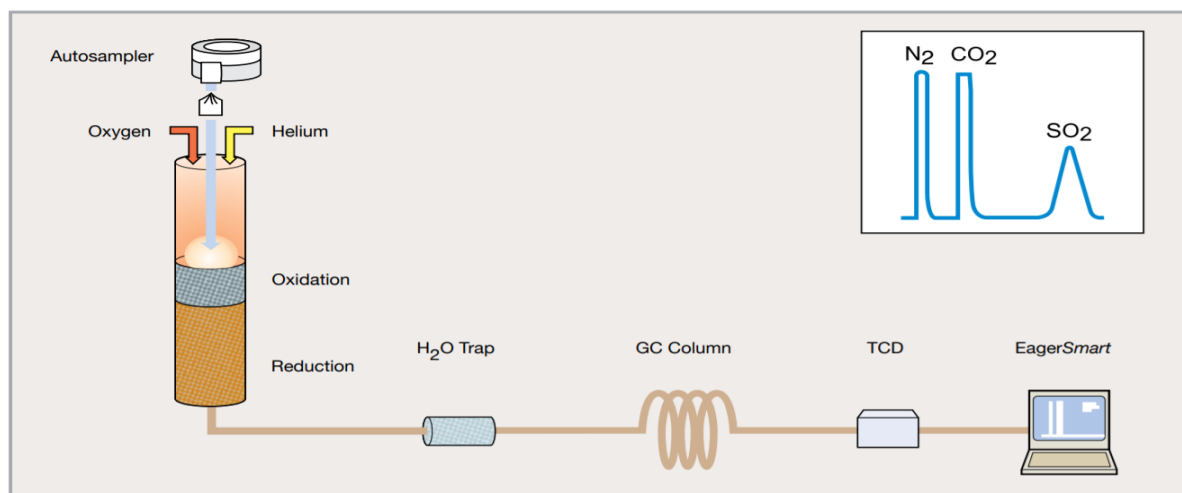


Figure 1. FlashSmart NCS Configuration.

TEST PRECAUTIONS

- Soil samples must be porfirised (grind fine and homogeneously) and was previously treated (dried and sieved to 2 mm).
- It is necessary to add about 10 mg of vanadium pentoxide (V_2O_5) to ensure complete oxidation of all forms of sulfur.

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- c) The nitrogen content in soil samples is generally very low (0.1%) thus set a high sensitivity of integration and use only oxygen of maximum purity grade.
- d) Use silver capsules container for determination of total organic carbon (TOC).
- e) It is good practice to conduct "component check" for NCS every 5 batches to check if instrument drifts and affects retention time.

EQUIPMENT

- a) CNS Autoanalyzer
- b) Analytical Balance, ± 0.0001 g
- c) Milling system ideal for 2 mm soil samples

LABORATORY WARE

- a) Tweezer
- b) Spatula
- c) Sample holder
- d) Dropper or micro-syringe
- e) Pipette
- f) Beaker

CHEMICALS AND CONSUMABLES

- a) Prepacked reactor.
- b) Magnesium perchlorate.
- c) Vanadium pentoxide.
- d) Tin capsule containers.
- e) Silver capsule containers.
- f) Certified Reference Material (CRM) with known N, C, S content to calibrate the autoanalyzer (Aspartic acid, EDTA, acetanilide, or soil samples with certified total NCS content).
- g) Hydrochloric acid, 1:1 ratio. Add 5 mL of concentrated HCl solution into 5 mL deionized water then mix
- h) Oxygen gas of maximum purity (greater than 99.99%).
- i) Helium gas of maximum purity (greater than 99.99%).

HEALTH AND SAFETY

Wear proper personal protective equipment (PPE) when conducting the analysis. Use laboratory coat, closed shoes, gas mask or dust mask, and chemical gloves to mitigate the harmful effects of exposure on chemicals.

Vanadium pentoxide is harmful by inhalation and if swallowed. Irritating to the respiratory system.

Catalyzer residues are toxic and must be disposed of properly. Take necessary precautions when handling compressed gasses and high-temperature equipment.

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