



Standard Operating Procedure
Soil Carbon and Nitrogen Determination by Dry Combustion
Soil Environmental Chemistry Program, The Ohio State University
Version 5

1.0 SCOPE

- 1.1 This is an instrumental dry combustion method for determining total Carbon (Nelson and Sommers, 1996) and Nitrogen (Bremner, 1996) in plant and soil like media

2.0 DEFINITIONS

- 2.1 Laboratory Control Sample: The laboratory control sample used for carbon and nitrogen analysis goes through the same preparation procedure as the samples. The concentration of carbon and nitrogen in the sample has been determined through repeated intralaboratory measurements.
- 2.2 Duplicate Samples: A duplicate test involves splitting a sample into sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.

3.0 EQUIPMENT AND SUPPLIES

- 3.1 NC2100 soil analyzer CE instruments (Lakewood, NJ).
- 3.2 Atropina calibration standard (CE instruments, Lakewood, NJ).
- 3.3 Sulphanilamide calibration check standard (CE instruments, Lakewood, NJ).
- 3.4 Tin sample capsules (CE instruments, Lakewood, NJ).
- 3.5 ≥ 18 M Ω deionized water.
- 3.6 Ultra high purity helium.
- 3.7 Ultra high purity oxygen.
- 3.8 Compressed air.

4.0 PROCEDURE

- 4.1 Oven dry samples at 55°C and grind to allow for a homogeneous subsample to be taken out for analysis.
- 4.2 Instrument set up and calibration:



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- 4.3 Perform four point linear calibration curve using an atropine standard (4.84% N, 70.055% C) weighed to the nearest 0.01mg. The instrument linear calibration range is approximately 1mg to approximately 7mg of atropine, corresponding to:
- 0.0484mg N - 0.339mg N
0.7055mg C - 4.938mg C
- 4.4 Weigh samples into tin capsules to the nearest 0.01mg and record sample mass.
- 4.5 The mass chosen for the sample should not exceed 100mg and should put the sample C and N within the calibration range.
- 4.6 Example calibration range for %C and %N if utilizing 50 mg sample weight:
- 0.0484mg N/50mg sample = 0.0968 %N
0.339mg N/50mg sample = 0.678 %N
0.7055mg C/50mg sample = 1.411 %C
4.938mg C/50mg sample = 9.876 %C
- 4.7 Input sample masses into Eager 200 software, which allows for results to be given in %C and %N.
- 4.8 Record Run ID in Carbon Analyzer log.
- 4.9 Start analysis.
- 4.10 Maintenance
- 4.10.1 Soil: Change crucible every 25 samples
 - 4.10.2 Perform routine maintenance in between analytical runs at intervals specified by the manufacturer or when chromatographic quality is suspect.

5.0 QUALITY CONTROL

- 5.1 Instrument calibration: $r^2 > 0.995$ shall be established for carbon and nitrogen.
- 5.2 Laboratory Control Sample: The laboratory control sample must fall within $\pm 20\%$ of the known value.
- 5.3 Sample Duplicates: The relative percent difference (RPD) must be no more than 20%.

$$RPD = 100 \times \frac{|S - D|}{S}$$



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Avg. (S,D)

- 5.4 Initial calibration verification (ICV) is an independent sulphanilimide standard run immediately after calibration. Standards must fall within $\pm 10\%$ of certified value.
- 5.5 Continuing calibration verification (CCV) is the independent sulphanilimide standard run interspersed within samples. Standards must fall within $\pm 10\%$ of certified value.

6.0 REFERENCES

- 6.1 Nelson D.W. and Sommers L.E. (1996) Total carbon, organic carbon, and organic matter. In Sparks, D. L. Methods of Soil Analysis. Part 3 - Chemical Methods. SSSA Book Series 5. Soil Science Society of America, Madison, WI, 961-1010.
- 6.2 Bremner J.M. Nitrogen-total. (1996) In Sparks, D. L. Methods of Soil Analysis. Part 3 - Chemical Methods. SSSA Book Series 5. Soil Science Society of America, Madison, WI, 1085-1121.
- 6.3 United States Environmental Protection Agency. (1995) Document number ILM04.0b. Contract Laboratory Program Statement of work for inorganic analysis, multi-media, multi-concentration. U.S. EPA: Washington, DC.