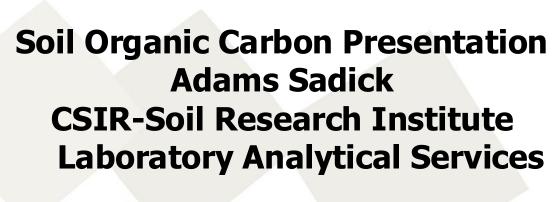




Raising confidence in quality measurements from soil laboratories in Sub-Saharan Africa

Dakar, 23-27 October

SOIL ORGANIC CARBON PRACTICAL WALKLEY-BLACK METHOD (Colorimetric Method)















Introduction

 Soil organic carbon (SOC) is the key element that determines soil quality, fertility and atmospheric carbon dioxide fixation.

• It is the key element of soil organic matter (SOM) that is readily measured quantitatively.

It has influence on:

- Physical
- Chemical and
- Biological properties of soil.



Introduction

- Soil organic matter includes:
- Fresh plant and animal residues
- Humus
- Soil organic matter is calculated from the organic-C content of the soil.
- SOM is assumed to be 58% carbon (SOM = $1.724 \times SOC$)



Principle in Colorimetric Method

- Suitable for all soils except with those where organic carbon is less than 0.2% (<0.2%)
- It is a wet oxidation procedure which follows the reaction below:

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$$2Cr_2O_7^{2-} + 3C^0 + 16H^+ \longrightarrow 4Cr^{3+} + 3CO_2 + 8H_2O$$

In the above reaction:

•
$$C^{\circ}$$
 \longrightarrow C^{4} : Carbon is oxidized



Principle in Colorimetric Method

 Measurement of organic carbon is based on the oxidation of carbon in an acidic medium

 Organic carbon is estimated from amount of chromic ion formed after oxidation using colorimetric procedure



Preparation of Soil Sample

 Air-dry the collected sample from the field in an open and ventilated shade and stirring from time to time.

• Grind the air-dried sample to crush the soil aggregate

• Sieve the sample through 2mm mesh sieve to remove gravels, roots, and any crop residues for only the fine particles to remain.

Keep the sample for laboratory analysis



Apparatus

- Analytical Balance
- Spectrophotometer
- Centrifuge Tube/glass conical tubes
- Volumetric pipettes
- Volumetric flasks
- Graduated pipettes
- Glass rod
- Beaker



Reagents (Colorimetric Method)

- Potassium dichromate, 10%: dissolve 10g in 100 ml de-ionized water.
- Sucrose
- Sulphuric acid, concentrated



Laboratory Analysis of Organic Carbon

Weigh 0.5g of the prepared soil sample

Add 2.0ml of 10% potassium dichromate solution

 Add 5.0ml of concentrated Sulphuric acid, allow it to cool down in an insulated sheet

 Add 20ml of deionized water and allow it to stand overnight in a fume chamber

Preparation of Standard Curve

Prepare a set of sucrose standard as shown in the table below

Mass of OC (mg)	0	1	2	3	4	5	6	7	8
Sucrose STD (4mgC/ml)	0.00	0.25	0.50	0.75	1.00	1.25	1.50	1.75	2.00
Distilled water	2.00	1.75	1.50	1.25	1.00	0.75	0.50	0.25	0.00



Preparation of Standard Curve

Add 2ml of 10% potassium dichromate to the stands

Add 5ml of Sulphuric acid, allow it to cool down and wait for 30 minutes inside an insulated sheet

 Add 20ml of distilled water, and allow it stand overnight in a fume chamber



Measurement

- Read the absorbance of the standards at 600nm
- Plot a graph of absorbance against standards
- Make sure that correlation coefficient of the curve is equal to or greater that 0.999
- Proceed with the reading of the samples at 600nm



Calculation

•
$$OC = \frac{f(A - A_{blank})}{Km} \times 1000$$
 in g/kg

- Where A = absorbance in the study solution
- A_{blank} =absorbance of blank
- K = coefficient of calibration function
- M = sample weight of the test soil, mg
- f = correction factor

• $OM = 1.724 \times OC \text{ in } g/kg$



Health and Safety

- Safety glasses, gloves and lab coat must be worn when handling any chemicals.
- Potassium dichromate: Highly corrosive and a strong oxidizing agent.
- Sulphuric acid: Keep away from naked flames/heat. Always add the acid to the water.



Quality Assurance/Quality Control

ACCURACY TEST

 Participate in interlaboratory proficiency testing program at least once a year.

PRECISION TEST

• Perform replicate analysis at most every 10% samples of a batch. Calculate the %RSD and compare the result with the target precision for the analyte concentration.







Thank you











