### **TEST METHODS MANUAL**

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

# TM-LSD-04-10

SECTION : S	SOIL CHEMIST	RY			Issue No.: 3	Effective date: April 15, 2024
SUBJECT:	ORGANIC	CARBON	(WALKLEY-BLACK	-	Revision No.: 2	Page 1 of 7
COLORIMETRIC METHOD)						

#### SCOPE

This method is for the determination of organic carbon in soils. This method is not applicable to soils containing significant amounts of carbonized materials.

#### PRINCIPLE

The organic matter (OM) in soil results from the decomposition of plant residues, roots, and soil organisms. It exists in a variety of forms ranging from simple sugars and carbohydrates to more complex proteins, fats, waxes and organic acids. Carbon as soil organic matter is considered as an important component in soils as it is known to affect its physical, chemical and biological properties.

The amount and type of organic matter is known to influence soil moisture holding capacity, the storage and supply of exchangeable cations and other plant nutrients (e.g. nitrogen and phosphorus), and the maintenance of stable soil structure and aeration, which are important in enhanced plant productivity. Organic matter also results in less soil erosion through stable soil structure, thus helping in the nutrient retention on land and water quality protection. It is also known to play a role in minimizing leaching of hazardous chemicals through the soil profile by providing sites for anthropogenic chemicals.

Soil organic matter can be estimated by the determination of soil organic carbon (OC) content using the Walkley-Black Method. Organic matter in soil is oxidized with potassium dichromate in concentrated sulfuric acid without necessitating external heating. The heat of reaction raises the temperature which is sufficient to induce substantial oxidation. Chemical reaction is as follows:

 $2 \operatorname{Cr}_2 \operatorname{O_7}^{2-} + 3 \operatorname{C^0} + 16 \operatorname{H^+} \longrightarrow 4 \operatorname{Cr}^{3+} + 3 \operatorname{CO_2} + 8 \operatorname{H_2O}$ (orange-red) (green)

Although the method has been shown to lead to incomplete oxidation of organic carbon, it is adequate when Cr<sup>6+</sup> is in excess at the end of wet oxidation.

The green color of chromic ions ( $Cr^{3+}$ ) produced from the reaction is in direct proportion to the carbon oxidized which is measured by colorimetric estimation. The extent of oxidation can be determined by measuring the concentration at wavelength near 600 nm. The  $Cr^{3+}$  ion has two broad maxima in the visible range (near 450 and 600 nm) but is measured near 600 nm as dichromate ion absorption maximum coincides near 450 nm. The calculation of the amount of organic carbon is based on the oxidation of sucrose, under the same conditions.

#### **TEST PRECAUTIONS**

Samples must be air-dried or oven-dried to prevent over estimation of OC/OM, which may occur when significant amount of iron is present. Large inorganic particles (>2 mm) such as gravel, pebbles and rocks present in the sample may also be generally removed with little concern due to their lack of contribution to soil OC/OM and chemical inertness.

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Sulfuric acid should be used fresh from the bottle and not left standing in a beaker, as it rapidly picks up moisture from the air. It is satisfactory until the strength falls to <96%. Potassium dichromate, on the other hand, should be prepared fresh every 30 days. During analysis, these reagents must be delivered from a calibrated dispenser to reduce error from manual pipetting and to minimize contact of analyst with chemicals. Sulfuric acid must be added to the sample as quickly as possible since the reaction depends on the heat of dilution of the acid. During mixing of solution, avoid excessive swirling that would result in organic particles adhering to the sides of the flask out of the solution.

If using sipper as an accessory for UV-Vis spectrophotometer, allow for at least 20 s for the washing of compartment using each sample before reading. Run water blank after reading the highest standard or a highly colored sample.

Allow the digested samples and standards to stand overnight for 24 hours. This is to ensure that the soil samples fully settled at the bottom of the flask. If the samples are to be transported from preparation room to Instrument Room, stand the samples for at least 2 hours before reading in the UV-Vis Spectrophotometer to allow any disturbed soil sample to settle and prevent clogging of the instrument.

#### EQUIPMENT

- a) Analytical balance
- b) Spectrophotometer suitable for measuring absorbance at 600 nm wavelength
- c) Oven able to reach a temperature of 105°C

#### LABORATORY WARE

- a) Centrifuge or conical tubes, 16 mm (ID) x 100 mm; about 50 mL capacity
- b) Dispensing or volumetric pipettes, 5 mL
- c) Graduated pipettes; 1mL, 2 mL
- d) Calibrated dispenser; 2 mL, 5 mL, 10 mL
- e) Glass rod
- f) Volumetric flasks; 100 mL, 500 mL
- g) Beaker; 100 mL, 250 mL

#### CHEMICALS AND REAGENTS

- a) Deionized water/distilled water, it should have an EC <  $1.5*10^{-3}$  dS m<sup>-1</sup>
- b) Potassium dichromate, 10% (0.34 M). Dissolve 50 grams of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 500 mL deionized water.
- c) Sulfuric acid, concentrated (not less than 96%): If Cl<sup>-</sup> is present in soil, add Ag<sub>2</sub>SO<sub>4</sub> to the acid at the rate of 15 g per liter.
- d) Sucrose standard, 4 mg C/mL. Weigh 0.95 g sucrose (dried at 105°C for two hours) and dissolve in 100 mL deionized water.

#### 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.

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Chemicals used in the analysis, particularly potassium dichromate and sulfuric acid must be carefully and properly handled to avoid potential skin and/or eye contact, ingestion, or inhalation. Mixing of incompatible materials must also be avoided to reduce the risks of fire and explosion inside the laboratory.

Potassium dichromate is a highly corrosive chemical and a strong oxidizing agent, which emits toxic chromium fumes upon heating. It is a known human carcinogen by the inhalation route of exposure. It is not combustible but enhances combustion of other substances, with risk of fire and explosion on contact with combustible substances. Sulfuric acid, on the other hand, is corrosive to all body tissues and inhalation of vapor may cause serious lung damage. There is a risk of fire and explosion on contacts, oxidants, reducing agents or water.

PROCEDURE (GLOSOLAN-SOP-02, 2019

## Limited Preview

#### Preparation of Standards

- For full access, kindly fill out the Document Request
- 2. To each tube, add 2 mL K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution and mix Form.
- 4. Add 18 mL deionized/distilled H<sub>2</sub>0. M Google Form link:
- 5. Read the absorbance of the calibration standards in a spectrophotometer set at 600 nm wavelength.
- 6. Prepare a standar **https://forms.gle/RoggCdA54prtTS6gN7** curve is equal or greater than 0.9990, proceed with the analysis of samples. Otherwise, verify that the standards and reagents were correctly prepared, the instrument is functioning properly, and that the instrument set-up is correct. Corrective actions must be taken and the calibration standards must be reanalyzed.

Thank you!	

able 1.Standard preparation.

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