



ASAC 1101 (2-1-0)

FUNDAMENTALS OF SOIL SCIENCE

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PRACTICAL MANUAL







EX No.11

Date:

DETERMINATION OF ORGANIC CARBON IN SOIL BY WALKLEY AND BLACK METHOD

Aim: To estimate oxidizable organic carbon in soil by Walkley & Black method

Principle:

The organic carbon in the soil is oxidized by chromic acid (mixture of potassium dichromate and sulphuric acid) by utilizing heat of dilution of sulphuric acid. The excess of chromic acid not reduced by the organic carbon of the soil is determined by back titration either with ferrous ammonium Sulphate or ferrous Sulphate.

Oxidation:

$$2K_2Cr_2O_7 + 8H_2SO_4 + 3C \longrightarrow 2K_2SO_4 + 2Cr_2(SO_4)_3 + 8H_2O + 3CO_2$$

2K₂Cr₂O₇
$$\longrightarrow$$
 3C (588g of K₂Cr₂O₇ oxidises 36g of C)

1000 mL of 1 N K₂Cr₂O₇ oxidises 3g of C

1 mL of 1 N K₂Cr₂O₇ solution oxidises 0.003g of C

<u>Titration</u> reactions:

$$2\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2 \text{O} + \text{H}_2 \text{SO}_4 \longrightarrow (\text{NH}_4)_2 \text{SO}_4 + \text{Fe} (\text{SO}_4)_3 + 3\text{H}_2 \text{O}_4$$

Apparatus: Burette, conical flask (500 mL), 10mL pipette, measuring cylinder, beaker, balance, droppers.

Reagents:

- 1. 1 N potassium dichromate (K2Cr2O7): Dissolve 49.04 g in 1 lit distilled water.
- 2. <u>0.5 N Ferrous Ammonium Sulphate (FAS):</u> Dissolve 196g in distilled water, add 15mL of conc.H₂SO₄ and make up to 1 litre.
- 3. Diphenyl amine indicator: Dissolve 0.5 g of DPA in 100mL of conc. H₂SO₄.
- 4. Concentrated H₂SO₄
- 5. Concentrated H₃PO₄
- 6. Sodium fluoride

Procedure:

- 1. Take 1.0 g of soil sample in 500 mL conical flask (0.5 mm sieved soil sample).
- 2. Add exactly 10mL of 1 N K₂Cr₂O₇ with a pipette.
- 3. Add 20 mL of conc. H₂SO₄ with measuring cylinder. Shake it for 5 minutes gently and keep for 30min for digestion at room temperature (1:2).

- 4. Add 100 mL of distilled water, 10 mL of ortho-phosphoric acid and pinch of sodium fluoride and 1 mL of diphenylamine indicator. Contents turn into violet blue colour.
- 5. Fill in the burette with 0.5N FAS and titrate the contents till the violet blue colour changes to dark green. The colour change is sudden; hence titration should be carried out carefully.
- 6. Note down the burette reading and repeat the experiment till two values coincide.
- 7. Run a blank titration also without the soil.

Rating:

% organic carbon	Description	
< 0.30	Very low	
0.30 - 0.50	Low	
0.50 – 0.75	Medium	
0.75 – 1.0	High	
> 1.0	Very high	

Result:

The present organic ca	rbon in given soil sample is	
It falls under	category.	

(1N K₂Cr₂O₇ + soil) vs 0.5 N FAS (DPA indicator)

S.No.	Volume of 1N K ₂ Cr ₂ O ₇ taken	Burette reading (mL)		End point	
	(mL)	Initial	Final	Titre value	
1	10.0				Violet blue to dark green
2	10.0				

Calculation:

% oxidizable organic carbon (OC) =

Volume of 1N
$$K_2Cr_2O_7 \times \left(\underbrace{(B-S)}_{B}\right) \times 0.003 \times \frac{100}{w}$$

Where, B = Blank titre value

S = Sample titre value

% oxidizable organic matter (OOM) in soil = % OC \times 1.724

Total organic matter (TOM) in soil = % OOM \times 1.30

Note: Organic matter on an average contains 58% carbon. The percent organic matter can be obtained by multiplying the % organic carbon with 100/58 or 1.724 which is 'Van Bemelon factor'.