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Soil quality — Determination of organic carbon by sulfochromic oxidation

*Qualité du sol — Dosage du carbone organique par oxydation
sulfochromique*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14235 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Determination of organic carbon in soil by sulfochromic oxidation

1 Scope

This International Standard specifies a method for the spectrometric determination of organic carbon content in soil by oxidation in a sulfochromic medium.

This International Standard is applicable to all types of air-dry soil samples.

This International Standard is not applicable to soils containing mineral-reducing compounds, e.g. Cl^- or Fe^{2+} . By convention the chloride content in the test portion shall not exceed 2 mg.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent additions of the standard indicated below. Members of ICE and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical use — Specification and test methods*.

ISO 10694:1995, *Soil quality — Determination of organic and total carbon after dry combustion (elemental analysis)*.

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analysis*.

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*.

3 Principle

The organic carbon present in the soil is oxidized in a mixture of potassium dichromate solution (in excess) and sulfuric acid at a temperature of 135 °C. The dichromate ions, which colour the solution orange-red, are reduced to Cr^{3+} ions which colour the solution green. The intensity of this green colour is measured spectrophotometrically. As it is assumed that the oxidation of one carbon atom of the organic matter produces four electrons, there is a direct relationship between the Cr^{3+} formed and the amount of organic carbon. The method is calibrated using glucose as a source of readily oxidizable carbon.

NOTE An amount of 2 mg of chloride present in the test sample corresponds to an apparent carbon content of about 0,7 mg.

4 Reagents

Use only reagents of recognized analytical grade and use distilled or deionized water for all solutions.

4.1 Water, having a specific conductivity not higher than 0,2 mS/m at 25 °C (grade 2 water in accordance with ISO 3696).

4.2 Sulfuric acid, H_2SO_4 , concentrated ($\rho = 1,84 \text{ g/cm}^3$).

4.3 Potassium dichromate solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,27 \text{ mol/l}$.

Dissolve 80 g of potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, in ca. 800 ml of water (4.1) in a 1000 ml volumetric flask and make up to the mark with water (4.1).

WARNING: The chromate ion present in potassium dichromate is potentially toxic. Laboratory personnel working with this chemical should take appropriate precautions to avoid contact with, or ingestion of, this chemical, following national or international safety regulations where applicable. Such regulations can extend to the disposal of solutions containing chromate ions, as these may also damage the environment. If in doubt, seek professional advice.

4.4 Glucose, anhydrous, $\text{C}_6\text{H}_{12}\text{O}_6$.

5 Apparatus and glassware

5.1 Standard laboratory glassware.

5.2 Analytical balance, capable of weighing accurately to 0,1 mg.

5.3 Volumetric glass tubes of 75,0 ml \pm 0,2 ml, adapted to fit the heating block (5.4).

5.4 Heating block, capable of maintaining a uniform temperature of 135 °C \pm 2 °C.

The block shall be drilled with holes such that, if h is the height, in millimetres, of the sulfochromic solution in the tube (5.3), the depth of the holes shall be at least $(h + 10)$ mm and the upper part of the tube emerging from the holes shall be at least 150 mm long. The temperature shall be measured with the tubes containing oxidation solution and a thermometer in the holes of the block. To minimize heterogeneity of the temperature within the block, all holes not utilized by tubes containing samples shall be occupied by tubes containing 15 ml of sulfuric acid (4.2).

5.5 Centrifuge.

5.6 Glass fibre filters.

5.7 Automatic pipette.

5.8 Spectrophotometer, equipped with a 10 mm cuvette and adjusted to a wavelength of 585 nm.

5.9 Water bath.

6 Laboratory sample

Use the fraction of particles < 2 mm of air-dry soil samples pretreated in accordance with ISO 11464. Use part of the sample to determine the water content in accordance with ISO 11465. For the determination of carbon, a representative subsample of the laboratory sample shall be milled until it passes a 250 μm aperture sieve in accordance with ISO 11464.

7 Procedure

7.1 General

The efficiency of the oxidation depends on the mass of the test portion, as well as the mass of carbon within it, even if the potassium dichromate remains in excess. Experience has shown that, under the conditions given in this International Standard, the mass of carbon in the test portion should not exceed 20 mg. Therefore the mass of the test portion used shall be in accordance with table 1.

Table 1 — Mass of test portion in relation with the estimated carbon content of the soil

Estimated carbon content g/kg	0 to 40	40 to 80	80 to 160	160 to 400	> 400
Mass of the test portion mg	400 to 500	200 to 250	100 to 125	45 to 50	20 to 25

7.2 Oxidation

Transfer the test portion to a volumetric tube (5.3). Using a pipette (5.7), add first 5,0 ml of the dichromate solution (4.3) and then 7,5 ml of sulfuric acid (4.2). Homogenize carefully.

Place the tubes in the heating block (5.4) which should be preheated to a temperature of 135 °C. Leave the tubes in the heated block for 30 min, then remove them and cool rapidly to room temperature in the water bath (5.9). Slowly add about 50 ml of water (4.1) to each tube and again cool rapidly in the water bath. Transfer the contents of the tube quantitatively into a 100 ml volumetric flask, make up to volume with water (4.1) and mix thoroughly.

Allow the suspension to settle for 1 h. Decant part of the supernatant into a centrifuge tube and centrifuge (5.5) for 10 min at 2000 *g*. If solid particles still remain in the suspension after centrifugation, filter the supernatant (5.6).

CAUTION: The addition of water to sulfuric acid solution is potentially hazardous, as is the use of dichromate solutions. Suitable protection, e.g. gloves, safety mask or goggles, and laboratory coat is essential. If in doubt, obtain professional advice.

7.3 Preparation of the standard series

Prepare a standard series according to table 2 by dissolving the mass of glucose indicated in 5,00 ml of dichromate solution (4.3) in a series of 100 ml volumetric tubes and mix to dissolve completely.

Add 7,5 ml of sulfuric acid as in 7.2 and continue the digestion step accordingly. The resulting solution should be completely clear, and should not require centrifugation or filtration. If this is not so, then the digestion has failed and shall be repeated.

Table 2 — Standard series of glucose in dichromate solution

Tube number	Mass of glucose g	Carbon content in 5 ml solution mg
1	0,00	0
2	0,25	5
3	0,50	10
4	0,75	15
5	1,00	20

7.4 Measurement

Measure the absorbance (5.8) of the solutions at 585 nm in the following order: standard series, blank and soil samples. Ensure that no cross-contamination occurs between the different samples measured.

Make a graph of the standard curve and determine the mass of organic carbon present in the blank and in the soil samples.

8 Calculation of organic carbon content

Calculate the organic carbon content on the basis of oven-dry soil using the following equation:

$$w_{oc} = (a/m) \cdot [(100 + w)/100]$$

where

- w_{oc} is the organic carbon content of the soil on the basis of oven-dry soil, in grams per kilogram;
- a is the mass of organic carbon in the test portion, in milligrams;
- m is the mass of the test sample, in grams;
- w is the water content, expressed as a percentage (mass fraction) on dry mass basis, determined in accordance with ISO 11465.

NOTE Comparison of carbon determinations in accordance with this procedure and the procedure given in ISO 10694 showed that, in a variety of soil samples, 95 % of the total organic carbon present was oxidized.

The organic matter content of the soil sample can be calculated from the organic carbon content using the formula:

$$w_{om} = f \cdot w_{oc}$$

where

- w_{om} is the organic matter content of the soil on the basis of oven-dry soil, in grams per kilogram;
- w_{oc} is the organic carbon content of the soil on the basis of oven-dry soil, in grams per kilogram;
- f is a conversion factor, the magnitude of which depends on the type of organic matter present and may vary between 1,7 and 2,5 for agricultural soils.

9 Repeatability

The repeatability of the determination of organic carbon content carried out in two separate but sequential measurements shall satisfy the conditions given in table 3.

Table 3 — Repeatability

Carbon content, g/kg		Acceptable variation
greater than	up to and including	
0	10	1 g/kg absolute
10	--	6,5 %

10 Test report

The test report shall contain the following information.

- A reference to this International Standard;
- a reference to the method used;
- all information necessary for the complete identification of the soil sample;
- the result of the determination for organic carbon, in grams per kilogram, to the nearest decimal point, calculated on the basis of oven-dried soil;
- a statement of the conversion factor used, if the results have been expressed as organic matter;
- details of any operation not specified in this International Standard or regarded as optional, as well as any factor which may have affected the results.

ICS 13.080

Descriptors: soils, quality, tests, soil testing, chemical analysis, determination of content, organic carbon, oxidation analysis, spectrometric method.

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