



SLOVENSKI STANDARD
SIST ISO 10694:1996

01-september-1996

**Kakovost tal - Določevanje organskega in celotnega ogljika po suhem sežigu
(elementna analiza)**

Soil quality - Determination of organic and total carbon after dry combustion (elementary analysis)

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Qualité du sol - Dosage du carbone organique et du carbone total après combustion sèche (analyse élémentaire)

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ICS:

| | | |
|-----------|--------------------------|-----------------------------------|
| 13.080.10 | Kemijske značilnosti tal | Chemical characteristics of soils |
|-----------|--------------------------|-----------------------------------|

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INTERNATIONAL
STANDARD

ISO
10694

First edition
1995-03-01

**Soil quality — Determination of organic
and total carbon after dry combustion
(elementary analysis)**

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*Qualité du sol — Dosage du carbone organique et du carbone total après
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Reference number
ISO 10694:1995(E)

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 10694 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annexes A and B of this International Standard are for information only.

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Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)

1 Scope

This International Standard specifies a method for the determination of the total carbon content in soil after dry combustion. The organic carbon content is calculated from this content after correcting for carbonates present in the sample. If carbonates are removed beforehand, the organic carbon content is measured directly.

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

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 revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 10390:1994, *Soil quality — Determination of pH*.

ISO 10693:1995¹⁾, *Soil quality — Determination of carbonate content — Volumetric method*.

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

1) To be published.

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

3 Principle

The carbon present in the soil is oxidized to carbon dioxide (CO₂) by heating the soil to at least 900 °C in a flow of oxygen-containing gas that is free from carbon dioxide. The amount of carbon dioxide released is then measured by titrimetry, gravimetry, conductometry, gas chromatography or using an infrared detection method, depending on the apparatus used. When the soil is heated to a temperature of at least 900 °C, any carbonates present are completely decomposed. For the determination of the organic carbon content, any carbonates present are previously removed by treating the soil with hydrochloric acid. Alternatively, if the carbonate content of the examined samples is known and corrections are made for the carbonates present when the organic carbon content is calculated.

NOTE 1 When the pH-CaCl₂ is less than 6,5, the presence of carbonates is unlikely. For example, this combination of low pH and presence of carbonates is only possible in recently limed soils.

4 Reagents

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

4.1 Water, with an electrical conductivity not higher than 0,2 mS/m at 25 °C (grade 2 water according to ISO 3696).

4.2 Calibration substances, e.g. acetanilide (C_8H_9NO), atropine ($C_{17}H_{23}NO_3$), calcium carbonate ($CaCO_3$), spectrographic graphite powder (C) and potassium hydrogen phthalate ($C_8H_5KO_4$).

4.3 Hydrochloric acid, $c(HCl) = 4 \text{ mol/l}$.

Dilute 340 ml of concentrated hydrochloric acid ($\rho = 1,19 \text{ g/ml}$) to 1 000 ml with water (4.1).

NOTE 2 Depending on the method of detection used, reagents and/or catalysts may be required for reduction, oxidation, removal and/or fixing of combustion gases which interfere with the analysis. The manufacturer's manual should be consulted for the apparatus to be used.

5 Apparatus and glassware

5.1 Standard laboratory glassware.

5.2 Analytical balance (capable of weighing accurately to 0,1 mg) or **microbalance** (capable of weighing accurately to 0,01 mg).

5.3 Apparatus for determination of the total carbon content, by combustion of the sample at a temperature of at least 900 °C, including a detector for measuring the carbon dioxide formed.

NOTES

3 The following detection methods are currently available: titrimetry, gravimetry, conductometry, gas chromatography and infrared measurement.

4 Some modern instruments are capable of determining organic and inorganic carbon separately in one run, by increasing the temperature gradually and continuously measuring the carbon dioxide.

5 Some instruments are capable of simultaneously determining total nitrogen and total carbon in soils.

5.4 Crucibles, made of porcelain, quartz, silver, tin or nickel, of various sizes.

NOTE 6 Tin or nickel crucibles are not acid-resistant.

6 Laboratory sample

Use the fraction of particles less than 2 mm of air-dried soil samples pretreated according to ISO 11464. Use part of the laboratory sample to determine the water content according to ISO 11465 and, if necessary, the carbonate content according to ISO 10693.

7 Procedure

The procedure consists of the determination of either

- the total carbon content including that carbon present as carbonate; or
- the organic carbon content after removal of carbonate.

NOTE 7 The total organic carbon content may be calculated by determining the total carbon content and subtracting the carbon present as carbonate which can be determined according to ISO 10693.

7.1 Calibration of the apparatus

Calibrate the apparatus as described in the relevant manual. For calibration or establishing a calibration graph, use one of the substances listed in 4.2.

7.2 Determination of the total carbon content

The amount of test portion taken for analysis depends on the expected total carbon content and on the apparatus used. Weigh out m_1 g of the air-dried sample in a crucible (5.4). Carry out the analyses in accordance with the manufacturer's manual for the apparatus.

When the organic carbon content is to be determined, the carbonates present shall be removed first. In this case, follow the procedure described in 7.3.

7.3 Determination of the organic carbon content

Add an excess of hydrochloric acid (4.3) to the crucible containing a weighed quantity of air-dried soil (see 7.2) and mix. Wait 4 h and dry the crucible for 16 h at a temperature of 60 °C to 70 °C. Then carry out the analysis in accordance with the manufacturer's manual for the apparatus.

NOTE 8 The quantity of hydrochloric acid to be added depends on the weighed amount of test portion and on the carbonate content. In all cases, an excess of hydrochloric acid should be added, which can be estimated by assuming that the quantity of the sample to be analysed consists of 100 % carbonates.

SAFETY PRECAUTIONS — The crucibles used should be large enough to avoid problems when transporting crucibles containing samples to which hydrochloric acid has been added.

8 Calculations

8.1 Total carbon content

Calculate the total carbon content of the sample, on the basis of oven-dried soil, using the following equation:

$$w_{C,t} = 1\,000 \times \frac{m_2}{m_1} \times 0,272\,7 \times \frac{100 + w_{H_2O}}{100}$$

where

$w_{C,t}$ is the total carbon content, in grams per kilogram, on the basis of oven-dried soil;

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of carbon dioxide released by the soil sample;

0,272 7 is the conversion factor for CO₂ to C;

w_{H_2O} is the water content, expressed as a percentage by mass, on a dry mass basis, determined according to ISO 11465.

8.2.2 Organic carbon content (direct determination)

If the carbonates are removed beforehand (following the procedure described in 7.3), the organic carbon content is determined according to 8.1.

8.3 Organic matter content

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

$$w_{om} = f \times w_{C,o}$$

where

w_{om} is the organic matter content of the soil, in grams per kilogram, on the basis of oven-dried soil;

$w_{C,o}$ is the organic carbon content of the soil, in grams per kilogram, on the basis of oven-dried soil;

f is a conversion factor.

NOTE 9 The conversion factor depends on the type of organic matter and, for agricultural soils, may vary between 1,7 and 2,0.

8.2 Organic carbon content

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8.2.1 Organic carbon content (indirect determination)

Calculate the organic carbon content of the sample, on the basis of oven-dried soil, using the following equation:

$$w_{C,o} = w_{C,t} - (0,12 \times w_{CaCO_3})$$

where

$w_{C,o}$ is the organic carbon content, in grams per kilogram, on the basis of oven-dried soil;

$w_{C,t}$ is the total carbon content, in grams per kilogram, on the basis of oven-dried soil, calculated according to 8.1;

0,12 is the conversion factor;

w_{CaCO_3} is the carbonate content of the soil, in grams per kilogram, expressed as calcium carbonate equivalent on the basis of oven-dried soil, determined according to ISO 10693.

9 Repeatability

The repeatability of the determination of the carbon content carried out in two separate consecutive executed measurements shall satisfy the conditions given in table 1.

Table 1 — Repeatability

| Carbon content g/kg | | Acceptable variation |
|------------------------|------------------------|-------------------------|
| greater than | up to and including | |
| 0,0 | 2,5 | 0,25 g/kg absolute |
| 2,5 | 75 | 10 % relative |
| 75 | | 7,5 g/kg absolute |

The results of an interlaboratory trial for the determination of total carbon and organic carbon in five soils are given in annex A.

10 Test report

The test report shall contain the following information:

- a) a reference to this International Standard;
- b) a reference to the method used;
- c) all information necessary for complete identification of the sample;
- d) the result of the determination for total carbon and/or organic carbon, in grams per kilogram, calculated on the basis of oven-dried soil; when the organic carbon content is determined, it shall be stated whether the carbonate content was determined, or whether carbonates were removed prior to the determination;
- e) 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.

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Annex A (informative)

Results of interlaboratory trials

An interlaboratory trial was organized in 1993 by the Wageningen Agricultural University, in the Netherlands, to verify the procedures specified in this International Standard.

For this interlaboratory trial, the determination of the organic and total carbon content in five soil samples was carried out by nine laboratories. Results were received from eight laboratories for organic carbon and also for total carbon.

The types of soils used and their origin are listed in table A.1.

The repeatability (r) and the reproducibility (R) of the results of the analyses obtained by the laboratories are presented in tables A.2 and A.3.

The values were calculated according to ISO 5725-2:1994, *Accuracy (trueness and precision)*

of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

Table A.1 — Types of soils used for the interlaboratory trial and their origin

| Soil No. | Soil type | Origin |
|----------|--------------------|-------------|
| 1 | Muck soil | Poland |
| 2 | Andosol | Indonesia |
| 3 | Garden soil | Netherlands |
| 4 | Loess under forest | Switzerland |
| 5 | Sandy soil | Mali |

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Table A.2 — Results of the interlaboratory trial for the determination of organic carbon in soil

| Parameter | Results | | | | |
|--|----------|--------|--------|--------|--------|
| | Soil No. | | | | |
| | 1 | 2 | 3 | 4 | 5 |
| Number of laboratories retained after eliminating outliers | 7 | 8 | 8 | 8 | 8 |
| Number of outliers (laboratories) | — | — | — | — | — |
| Number of accepted results | — | — | — | — | — |
| Mean value (g/kg dry soil) | 410,42 | 63,3 | 83,88 | 41,537 | 2,47 |
| Standard deviation of the repeatability (s_r) | 4,318 | 1,225 | 4,275 | 1,045 | 0,272 |
| Relative standard deviation of the repeatability (%) | 1,052 | 1,935 | 5,096 | 2,515 | 10,998 |
| Repeatability limit ($r = 2,8 \times s_r$) | 12,090 | 3,43 | 11,969 | 2,925 | 0,761 |
| Standard deviation of the reproducibility (s_R) | 127,413 | 11,957 | 19,376 | 5,523 | 1,555 |
| Relative standard deviation of the reproducibility (%) | 11,087 | 18,888 | 23,098 | 13,297 | 62,92 |
| Reproducibility limit ($R = 2,8 \times s_R$) | 45,505 | 33,48 | 54,253 | 15,465 | 4,355 |

Table A.3 — Results of the interlaboratory trial for the determination of total carbon in soil

| Parameter | Results | | | | |
|--|----------|--------|--------|-------|--------|
| | Soil No. | | | | |
| | 1 | 2 | 3 | 4 | 5 |
| Number of laboratories retained after eliminating outliers | 8 | 8 | 8 | 8 | 8 |
| Number of outliers (laboratories) | — | — | — | — | — |
| Number of accepted results | — | — | — | — | — |
| Mean value (g/kg dry soil) | 439,46 | 72,86 | 98,56 | 45,61 | 2,63 |
| Standard deviation of the repeatability (s_r) | 6,622 | 1,314 | 2,387 | 1,131 | 0,127 |
| Relative standard deviation of the repeatability (%) | 1,507 | 1,803 | 2,422 | 2,481 | 4,833 |
| Repeatability limit ($r = 2,8 \times s_r$) | 18,544 | 3,679 | 6,686 | 3,169 | 0,356 |
| Standard deviation of the reproducibility (s_R) | 25,246 | 5,92 | 9,483 | 2,387 | 1,391 |
| Relative standard deviation of the reproducibility (%) | 5,744 | 8,124 | 9,620 | 5,234 | 52,751 |
| Reproducibility limit ($R = 2,8 \times s_R$) | 70,69 | 16,577 | 26,552 | 6,685 | 3,895 |

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