INTERNATIONAL STANDARD

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Soil quality — Determination of total sulfur by dry combustion

Qualité du sol — Dosage du soufre total par combustion sèche

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15178 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annex A of this International Standard is for information only.

Soil quality — Determination of total sulfur by dry combustion

WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to consult and establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard describes a procedure using dry combustion for the determination of total sulfur in soil samples. It is applicable to all types of air-dried soil samples.

NOTE High-temperature combustion methods might not determine total sulfur, only combustible sulfur. The difference between total and combustible sulfur is usually negligible in soils.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

3 Principle

The sulfur content of a soil sample, pretreated in accordance with ISO 11464, is determined by heating the sample to a temperature of at least 1 150 °C in a stream of oxygen-containing gas. Organically and inorganically bound sulfur is converted to SO_2. This reaction may in some cases need a higher temperature or the addition of catalysts, modifiers or accelerators. At temperatures $< 1\,350$ °C, SO_3 may be formed in the presence of excess oxygen. This SO_3 has to be reduced to SO_2 using an appropriate reagent, e. g. copper. The SO_2 arising from the combustion is measured by infrared spectrometry, thermal conductivity or other suitable detection technique. Gases resulting from the combustion which may interfere with the detection stage have to be removed from the stream of gas prior to detection.

This method is for use with commercially available sulfur analysers equipped to carry out the preceding operations automatically. These instruments shall be calibrated using chemical compounds or standard reference materials of known sulfur content based on the range of sulfur in each sample analysed.

NOTE Complete decomposition of sulfur-containing compounds may not occur at lower temperatures, especially in the presence of free alkaline or alkaline earth carbonates or sulfates. In such cases, temperatures $\geqslant 1\,350$ °C or the use of matrix modifiers such as WO_3 or V_2O_5 may be necessary. The efficiency of the quantitative recovery of sulfur from such soils can be tested using substances such as calcium sulfate.

4 Reagents

All reagents used should be of an analytical grade in accordance with the manufacturer's instruction manual for the instrument used.

4.1 Oxygen-containing combustion gas, free of sulfur and its compounds, in accordance with the instructions of the equipment manufacturer.

4.2 Calibration substances, such as sulfanilic acid ($C_6H_7NO_3S$) or other compounds of known sulfur content or soil samples with certified sulfur contents.

NOTE Standard reference materials (SRM) or other commercially available reference soils with a certified sulfur content can be obtained and used for calibration purposes in this test method. Examples of such materials are given in annex A. Users of SRMs are urged to use these materials to develop their own internal reference materials and associated quality assurance procedures.

5 Apparatus

5.1 Analytical balance.

The resolution of the balance should be less than or equal to 0,000 1 times the mass of sample or calibration substance in accordance with the following table:

Table 1

Mass of sample or calibration substance g	Balance resolution mg
$>$ 1	$\leqslant$ 0,1
1 to 0,1	$\leqslant$ 0,01
$<$ 0,1	$\leqslant$ 0,001

5.2 Measurement apparatus, to determine the total sulfur content of a sample by burning it at a temperature of at least 1 150 °C and detecting the SO_2 generated.

5.3 Accessories and consumables.

Accessories and consumables in accordance with the manufacturer's instructions.

6 Preparation of sample

Pulverize the sample to pass a 250 μm sieve and mix thoroughly in accordance with ISO 11464. A separate portion of the sample should be analysed for moisture content in accordance with ISO 11465 for recalculation of results to an oven-dry basis.

Care should be exercised to minimize, during handling, the probability of altering the moisture content of the SRM through desiccation or absorption of atmospheric moisture. All standard reference materials should be used in accordance with the information given in the certificate of analysis.

7 Procedure

7.1 Instrument preparation

Assemble the apparatus according to the manufacturer's instructions and condition it to eliminate drift in the sensitivity.

7.2 Calibration

Analyse sufficient samples or calibration substances (4.2) with sulfur contents covering the range of values to be expected in the unknown samples.

Widely accepted quality control procedures may be employed to establish the required number of calibrated samples and the precision and frequency of calibration. A minimum of four calibration points is strongly recommended.

7.3 Analysis

Weigh a test portion into a suitable crucible. Choose the mass of the test portion so that the expected sulfur content falls within the range of values covered by the calibration curve. If the sulfur content is greater than the maximum calibration value, repeat the analysis with a smaller mass of test sample.

Operate the instrument in accordance with the manufacturer's instructions.

8 Calculation and expression of the results

Calculate the total content of sulfur, w_{ts}, in grams per kilogram, on the basis of the oven-dried soil according to the following formula:

$$w_{ts} = 0{,}500\,5\ (m_2/m_1) \cdot (100 + w)\ /100$$

where

0,505 5 is the molar conversion factor from SO_2 to S;

m_1 is the mass of the soil sample, in grams;

m_2 is the mass of the sulfur dioxide released by the sample, in milligrams;

w is the mass fraction of water content (expressed as a percentage) on the basis of the oven-dried soil (clause 6).

NOTE Automatic instruments may give sulfur contents directly, although these data may need correction to an oven-dried basis.

9 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) all necessary information for the complete identification of the sample;

c) the results of the determination, in grams sulfur per kilogram of oven-dried soil;

d) details of any operation not specified in this International Standard or which is optional, as well as any factor which may have affected the results.

Report the result to the nearest 0,1 g/kg.

10 Precision and comparison with other methods

10.1 General

The quantitative limit of this method is estimated to be about 0,2 g/kg.

This method is highly dependent upon the calibration of the equipment. The reference materials should be as close as possible to the samples in sulfur content, chlorine content, iron content, etc.

10.2 Precision

This method was tested in an interlaboratory trial in 1998. Four soil samples were analysed by eight laboratories. In accordance with 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*, the results in Table 2 were obtained.

Table 2

Sample	Mean sulfur content	Repeatability conditions		Reproducibility conditions	
	g/kg	s_r	r	s_R	R
1	1,62	0,05	0,13	0,16	0,46
2	0,46	0,10	0,28	0,21	0,58
3	0,25	0,05	0,13	0,10	0,28
4	0,28	0,06	0,18	0,13	0,37

s_r is the repeatability standard deviation.

r is the repeatability value.

s_R is the reproducibility standard deviation.

R is the reproducibility value.

10.3 Comparison with other methods

The samples from the interlaboratory trial were analysed by two other methods, X-ray fluorescence, XRF, and inductively coupled plasma spectrometry, ICP. The ICP determination was performed after extraction with concentrated nitric acid. The results in Table 3 were obtained.

Table 3

	Sample			
Method	**1**	**2**	**3**	**4**
Mean value from ring test	1,62	0,46	0,25	0,28
XRF	1,50	0,56	0,27	0,46
ICP	1,52	0,45	0,20	0,32