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Determination of total sulphur in fertilizers by high temperature combustion

Dosage du soufre total dans les engrais par combustion à haute température

Reference number
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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document was created out of a need for newer and faster laboratory techniques to determine the total sulphur in fertilizer materials. There are numerous documented and validated methods available for determining total sulphur, but they are time-consuming and, in some cases, require the use of hazardous chemicals (e.g. bromine, perchloric acid). These methods also rely on the competency of the chemist/analyst and the laboratory technique is a critical component for producing accurate and reproducible results.

Combustion as an analytical tool has made great strides in recent years and, in some laboratories, this is a commonly used technique. Various detectors have been coupled to a furnace (combustion chamber) and the ensuing gases are measured for the analyte in question.

Determination of total sulphur in fertilizers by high temperature combustion

1 Scope

This document specifies a method to measure the total sulphur content in fertilizer and soil conditioner materials.

This method is applicable for measuring total sulphur concentration in solid and liquid fertilizers and its raw inputs in the range of 0,1 % to 97 %.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8157 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

This procedure involves conversion of sulphur (S) species from fertilizers and chemical standards into SO_2 through combustion at a temperature >1 100 °C followed by measurement with thermal conductivity detection (TCD) or infrared (IR) detection reported as weight/weight percentage (w/w %). In the case of thermal conductivity detection and where simultaneous measurements of additional elements, such as carbon (C), hydrogen (H), or nitrogen (N), are performed, an intermediate SO_2 separation by thermal adsorption/desorption is necessary.

5 Apparatus, material and reagents

5.1 General

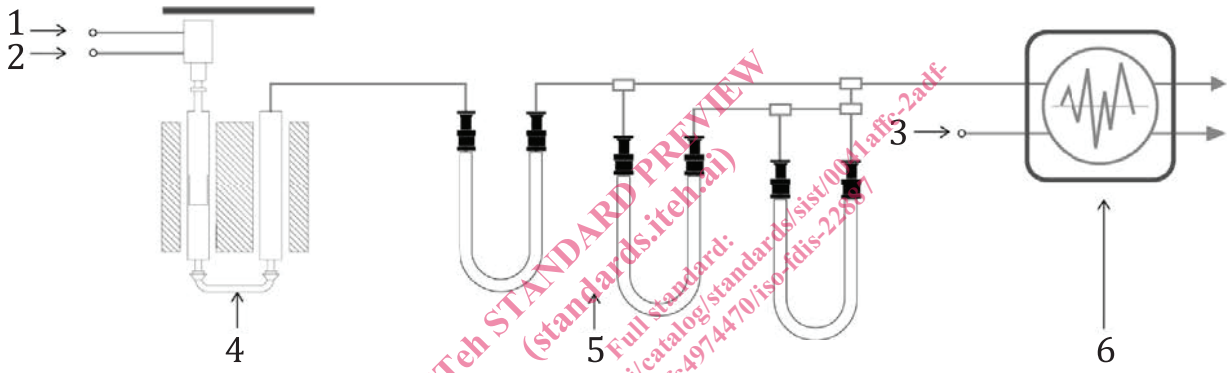
CAUTION — Incorrect handling during the elemental analysis using combustion can lead to the risk of burns as certain instrument components are heated during the method. Even after switching off the instrument, some components stay hot for long periods of time. Serious burns can occur if working carelessly with the instrument. Follow the manufacturer's specific operating instructions to ensure safe handling of equipment.

Total sulphur measurements can be performed via variable apparatus types depending on detection method of choice.

5.2 Apparatus

5.2.1 Apparatus A: Combustion followed by thermal conductivity detection

For Apparatus A type instruments, shown in Figure 1, sulphur as SO_2 is determined by TCD with helium or argon carrier gas allowing for multi-element analysis. With this setup, the test portion should be introduced into the combustion zone in a way such that atmospheric contamination is removed. Oxygen is added over the test portion at a temperature >1 100 °C converting all elements to their fully oxidized gaseous specie. A catalyst, such as tungsten (VI) oxide (WO_3), inside the combustion tube is used to aid oxidation. Following combustion, gases pass through a reducing environment and halogen scrubber in order that NO_x species be converted to N_2 and removal of halogen contaminants, respectively. Other resulting combustion gas components CO_2, H_2O, and SO_2 are scrubbed or adsorbed on analyte-specific thermal adsorption/desorption columns. N_2 is not adsorbed and flows directly to the thermal conductivity detector. Each CO_2, H_2O, and SO_2 are desorbed sequentially following the previous elements complete measurement by the TCD allowing for clear separation of the analyte species. Scrubbing materials, such as chemical or physical absorbers, may be placed between the furnace and detector to remove CO_2 and/or H_2O if determination of either C and/or H is undesired. With the help of a calibration curve, software processing converts the SO_2 peak signal into a w/w percentage of S in the sample.



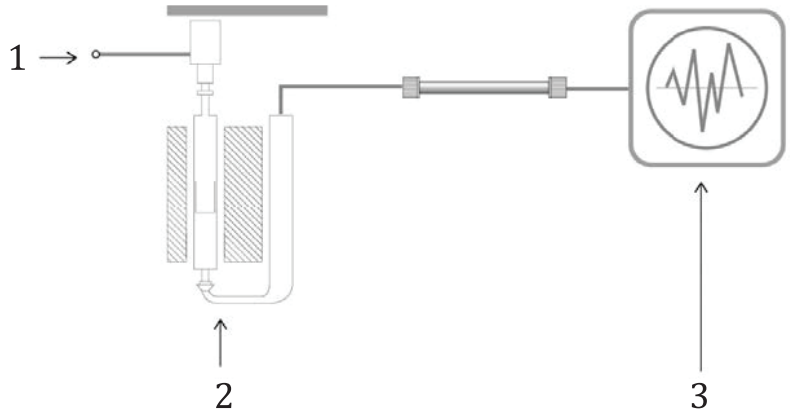
Key

1 He/Ar carrier gas
2 oxygen inlet
3 He/Ar reference gas
4 combustion and reduction
5 gas separation
6 detection (TCD)

Figure 1 — Typical multi-element measuring combustion system using adsorption/desorption separations and TCD

5.2.2 Apparatus B: Combustion followed by single-range infrared detection

For Apparatus B type instruments, shown in Figure 2, sulphur as SO_2 is determined by a sulphur-specific IR detector with oxygen carrier gas. The test portion is introduced into the combustion zone where oxygen in combination with a temperature >1 100 °C converts S to SO_2. A catalyst, such as tungsten (VI) oxide (WO_3), inside the combustion tube is used to aid oxidation. The gas stream is dried before entering the detector. With the help of a calibration curve, software processing converts the SO_2 peak signal into a w/w percentage of S in the sample. For best results using this apparatus type, follow special instructions in Clause 6.



Key

1 oxygen carrier gas
2 combustion
3 detection (IR)

Figure 2 — Typical sulphur only measuring combustion analyser using SO_2-specific IR detection

5.3 Materials, reagents and consumables

5.3.1 Materials

a) Analytical balance, resolution to at least 0,1 mg;

b) Test portions containers, typically tin foil or ceramic crucible;

c) Hand pellet press, for pelletizing powder materials;

d) Capsule sealing press, for making a gas-tight cold seal on tin capsule holding liquid materials.

5.3.2 Reagents

5.3.2.1 Helium or Argon, minimum 99,995 % purity.

5.3.2.2 Oxygen, minimum 99,5 % purity.

5.3.2.3 Tungsten (VI) oxide (WO_3) granulate, grain size approx. 0,5 mm to 2 mm, minimum 99,7 % purity — supplied by the instrument manufacturer.

5.3.2.4 Tungsten (VI) oxide (WO_3) powder as sample additive, minimum 99,7 % purity — supplied by the instrument manufacturer.

5.3.2.5 Copper wires, approx. 0,5 mm length — supplied by the instrument manufacturer.

5.3.2.6 Copper oxide wires, approx. 0,5 mm length — supplied by the instrument manufacturer.

5.3.2.7 Pt catalyst, 5 % on Al_2O_3, pelletized — supplied by the instrument manufacturer.

5.3.2.8 Corundum balls (inert), 3 mm to 5 mm diameter — supplied by the instrument manufacturer.