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Trdna alternativna goriva - Določanje samosegrevanja - 1. del: Izotermalna kalorimetrija (ISO 21911-1:2022)

Solid recovered fuels - Determination of self-heating - Part 1: Isothermal calorimetry (ISO 21911-1:2022)

Feste Sekundärbrennstoffe - Bestimmung der Selbsterhitzung - Teil 1: Isotherme Kalorimetrie (ISO 21911-1:2022)

Combustibles solides de récupération - Détermination de l'auto-échauffement - Partie 1: Détermination calorimétrique isotherme (ISO 21911-1:2022)

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Solid recovered fuels — Determination of self-heating —

Part 1: Isothermal calorimetry

Combustibles solides de récupération — Détermination de l'auto-échauffement —

Partie 1: Détermination calorimétrique isotherme

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 300, *Solid recovered materials, including solid recovered fuels*.

A list of all parts in the ISO 21911 series can be found on the ISO website.

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

There is a continuous global growth in the trading and use of solid recovered fuels (SRFs). This has resulted in an increased probability of fires, which has consequences for the handling, transporting and storage of SRFs.

SRFs can generate heat spontaneously by exothermic biological, chemical and physical processes. The heat build-up can be significant in large storage volumes if the heat conduction in the material is low. In some conditions the heat generation can lead to pyrolysis and spontaneous ignition. The potential for self-heating varies considerably for different types and qualities of SRFs and it is important to be able to identify SRF fractions with high heat generation potential to avoid fires in stored materials.

The increasing number of incidents is a clear indicator that safety needs to be prioritized, first of all for human safety and environmental concerns but also because interruptions in energy supply will have significant consequences. SRF fires throughout the supply chain will also, in addition to safety and environmental issues and direct economic losses, have a negative impact on the confidence in the SRFs as a reliable energy source. They can also lead to difficulties in obtaining insurance coverage.

It is difficult for SRF producers, logistics providers, SRF users, equipment suppliers and manufacturers, consultants, authorities and insurance providers to determine reasonable safety measures and an appropriate level of protection due to a lack of standards and recommendations.

As part of the determination and the assessment of risks for SRFs, defined test methods and standards are established or need to be developed. However, ageing and degradation due to the handling and storage of SRFs in actual environments affects their characteristics, so safety margins should be established in relation to actual analysis results.

The test method described in this document, isothermal calorimetry, is a method where the heat flow generated from the test portion is measured directly. The operating temperature for an isothermal calorimeter is normally in the range of 5 °C to 90 °C (some calorimeters can reach even higher temperatures) and can therefore measure low-temperature reactions, such as those from bacteria and other microbes. However, isothermal calorimetry is used for monitoring the thermal activity or heat flow of chemical, physical and biological processes. The technique is most commonly used in the fields of pharmaceuticals, energetic materials and cement. Isothermal calorimetry has also been applied for the measurement of heat flow from the self-heating of solid biofuel pellets[6]–[10].

For investigating heat generation at high temperatures, other types of test methods, such as basket heating tests, are possibly more suitable.

Data on spontaneous heat generation determined using this document are only associated with the specific quality, composition and age of the sample material.

The information derived using this document is for use in quality control and in hazard and risk assessments.

Solid recovered fuels — Determination of self-heating —

Part 1:
Isothermal calorimetry

1 Scope

This document specifies an analytical method for quantification of the spontaneous heat generation from solid recovered fuels using isothermal calorimetry.

This document gives guidance on the applicability and use of the specified analytical method. It further establishes procedures for sampling and sample handling of solid recovered fuels prior to the analysis of spontaneous heat generation.

The test procedure given in this document quantifies the thermal power (heat flow) of the sample during the test. It does not identify the source of self-heating in the test portion analysed.

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 21637, *Solid recovered fuels — Vocabulary*

ISO 21645, *Solid recovered fuels — Methods for sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637 apply.

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
analysis temperature
temperature of the analysis environment, i.e. the calorimeter temperature

3.2
self-heating
rise in temperature in a material resulting from an exothermic reaction within the material

[SOURCE: ISO 13943:2017, 3.341, modified — “<chemical>” removed from the definition.]

3.3
test portion
sub-sample of either a *laboratory sample* (3.5) or a *test sample* (3.4) required for the specific measurement

3.4
test sample

laboratory sample (3.5) after an appropriate preparation made by the laboratory

3.5
laboratory sample

part of the sample sent to or received by the laboratory

3.6
thermal power

rate of heat flow produced by the sample during the test

Note 1 to entry: Expressed in W or J/s.

Note 2 to entry: Commonly also expressed as specific thermal power with reference to the unit mass of solid recovered fuel in W/g or J/(s·g).

4 Principle

Isothermal calorimetry is one of the sensitive techniques for studying heat production or heat consumption from samples of different kinds. It is non-destructive and non-invasive to the sample. Heat production due to any physical, chemical or biological changes in a sample can be measured. When heat is produced or consumed by any process, a temperature gradient is developed across the sensor. This will generate a voltage, which is proportional to the heat flow across the sensor and to the rate of the process taking place in the sample ampoule. The signal is recorded continuously and in real time.

NOTE 1 A commercial instrument for isothermal calorimetry normally has multiple channels and can thus be used for measurements of several samples simultaneously.

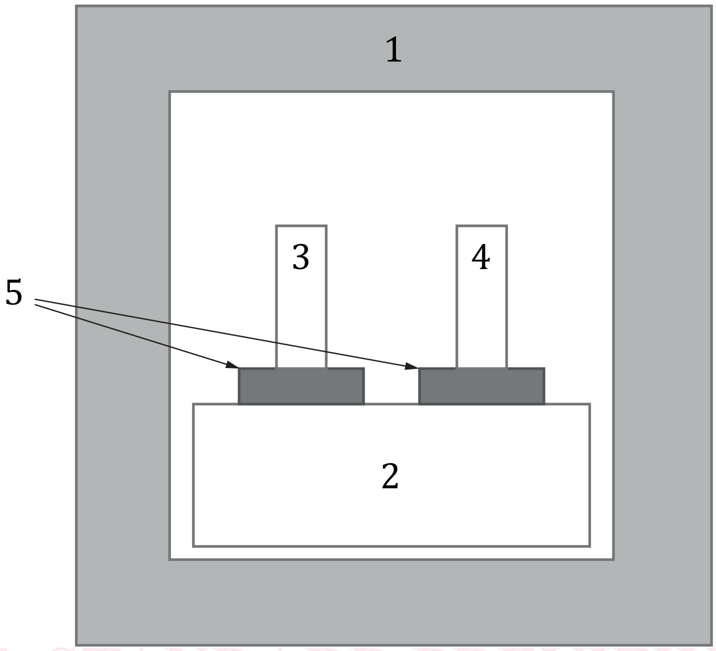
For each sample (channel) there is an inert reference that is on a parallel heat-flow sensor. During the time that the heat flow is monitored, any temperature fluctuations entering the instrument will influence both the sample and the reference sensors equally. This architecture allows a very accurate determination of heat that is produced or consumed by the sample alone while other non-sample-related heat disturbances are efficiently removed. The measured heat flow is normalized against the mass of the sample and the result is expressed in mW/g.

NOTE 2 The operating temperature for an isothermal calorimeter is typically in the range of 5 °C to 90 °C. However, there are calorimeters with a somewhat higher span for operating temperature.

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

5.1 Isothermal calorimeter, consisting of a sample holder for the sample vial and the reference vial, each thermally connected to heat-flow sensors, which are thermally connected to a constant temperature sink. See example in Figure 1.



Key

1	thermostat	4	reference
2	heat sink	5	heat-flow sensors
3	sample		

Figure 1 — Schematic drawing of an isothermal calorimeter

The calorimeter shall be calibrated for the analysis temperature according to Annex A, Clauses A.1, A.2 and A.3. The analysis temperatures for the screening test procedure are 50 °C and 70 °C.

The data acquisition equipment shall be capable of performing continuous logging of the calorimeter output measured at time intervals of less than or equal to 10 s.

The baseline shall exhibit a low random noise level and be stable against drift, according to Annex A, Clause A.4.

The minimum sensitivity for measuring power output shall be 100 µW.

5.2 Sample vial, made of glass with a minimum volume of 20 ml and provided with an airtight lid with an inert seal. Vials with volumes larger than 20 ml can be used if the sample loading is scaled accordingly (see 7.2.2). In such cases, this deviation from the standard procedure is noted in the test report.

5.3 Balance, which should have a resolution of at least 10 mg.

6 Sample handling

6.1 General

Sample handling is important in maintaining the physical, chemical and biological properties of solid recovered fuel samples. The transport and storage (see 6.3) is of especial importance for self-heating