SIST

SLOVENSKI STANDARD
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01-marec-2020

Trdna alternativna goriva - Določevanje kalorične vrednosti (ISO/DIS 21654:2019)

Solid recovered fuels - Determination of calorific value (ISO/DIS 21654:2019)

Feste Sekundärbrennstoffe - Bestimmung des Brennwertes (ISO/DIS 21654:2019)

Combustibles solides de récupération - Détermination du pouvoir calorifique (ISO/DIS 21654:2019)

Ta slovenski standard je istoveten z: prEN ISO 21654

ICS:

75.160.10	Trda goriva	Solid fuels

oSIST prEN ISO 21654:2020 **en,fr,de**

DRAFT INTERNATIONAL STANDARD
ISO/DIS 21654

ISO/TC **300** Secretariat: **SFS**

Voting begins on: **2019-12-19** Voting terminates on: **2020-03-12**

Solid recovered fuels — Determination of calorific value

Combustibles solides de récupération — Détermination du pouvoir calorifique

ICS: 75.160.10

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 21654:2019(E)

Contents

Foreword

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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 fuels*.

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 is based on EN 15400.

The result obtained is the gross calorific value of the sample analysed at constant volume with all the water of the combustion products as liquid water. In practice, solid recovered fuels are burned at a constant (atmospheric) pressure and the water is either not condensed (removed as vapour with the flue gases) or condensed. Under both conditions, the operative heat of combustion to be used is the net calorific value of the fuel at constant pressure. The net calorific value at constant volume can also be used; equations are given for calculating both values.

General principles and procedures for the calibrations and the solid recovered fuels experiments are presented in the normative text, whereas those pertaining to the use of a particular type of calorimetric instrument are specified in Annexes A to C. Annex D contains the formula to calculate the removed ash contributors. Annex E contains checklists for performing calibration and fuel experiments using specified types of calorimeters. Annex F gives examples to illustrate some of the calculations.

Solid recovered fuels — Determination of calorific value

WARNING — Strict adherence to all of the provisions specified in this document should ensure against explosive rupture of the bomb, or a blow-out, provided that the bomb is of standard design and construction and in good mechanical condition.

1 Scope

This document specifies a method for the determination of gross calorific value of solid recovered fuels at constant volume and at the reference temperature 25 °C in a bomb calorimeter calibrated by combustion of certified benzoic acid.

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.

EN 15296, *Solid biofuels — Conversion of analytical results from one basis to another*

EN 15358, *Solid recovered fuels — Quality management systems — Particular requirements for their application to the production of solid recovered fuels*

EN 15440, *Solid recovered fuels — Methods for the determination of biomass content*[1]

EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*[2]

EN ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1)*

ISO/DIS 21637, *Solid recovered fuels — Terminology, definitions and descriptions*

ISO/DIS 21660-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

3 Terms and definitions

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

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

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

[1] ISO/CD 21644:2019 "Solid recovered fuels — Method for the determination of biomass content" is currently being processed for the preparation of the "DIS"-enquiry.

[2] ISO/CD 21646:2019 "Solid recovered fuels — Sample preparation" is currently being processed for the preparation of the "DIS"-enquiry.

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

3.1
gross calorific value at constant volume
absolute value of the specific energy of combustion, in Joules, for unit mass of a solid recovered fuel burned in oxygen in a calorimetric bomb under the conditions specified

Note 1 to entry: The products of combustion are assumed to consist of gaseous oxygen, nitrogen, carbon dioxide and sulphur dioxide, of liquid water (in equilibrium with its vapour) saturated with carbon dioxide under the conditions of the bomb reaction, and of solid ash, all at the reference temperature.

3.2
net calorific value at constant volume
absolute value of the specific energy of combustion, in Joules, for unit mass of a solid recovered fuel burned in oxygen under conditions of constant volume and such that all the water of the reaction products remains as water vapour (in a hypothetical state at 0,1 MPa), the other products being, as for the gross calorific value, all at the reference temperature

3.3
net calorific value at constant pressure
absolute value of the specific heat (enthalpy) of combustion, in Joules, for unit mass of a solid recovered fuel burned in oxygen at constant pressure under such conditions that all the water of the reaction products remains as water vapour (at 0,1 MPa), the other products being as for the gross calorific value, all at the reference temperature

3.4
reference temperature
international reference temperature for thermo-chemistry of 25 °C is adopted as the reference temperature for calorific values (see 8.7)

Note 1 to entry: The temperature dependence of the calorific value of solid recovered fuels is small [less than 1 J/(g · K)].

3.5
effective heat capacity of the calorimeter
amount of energy required to cause unit change in temperature of the calorimeter

3.6
corrected temperature rise
change in calorimeter temperature caused solely by the processes taking place within the bomb calorimeter

Note 1 to entry: It is the total observed temperature rise corrected for heat exchange, stirring power etc. (see 8.6).

Note 2 to entry: The change in temperature can be expressed in terms of other units: resistance of a platinum or thermistor thermometer, frequency of a quartz crystal resonator etc., provided that a functional relationship is established between this quantity and a change in temperature. The effective heat capacity of the calorimeter can be expressed in units of energy per such an arbitrary unit. Criteria for the required linearity and closeness in conditions between calibrations and fuel experiments are given in 9.3.

Note 3 to entry: A list of the symbols used and their definitions is given in Annex G.

3.7
Removed ash contributors
rac
coarse inert material (i. e. metals, glass, stones, tiles etc.) removed from the sample before preparation, in order to avoid damage to the preparation equipment

Note 1 to entry: The removed ash contributors (rac), after sample pre-drying, are taken into account for the calculation of the ash, carbon, hydrogen, nitrogen and sulphur content in the analysed sample.

Note 2 to entry: See Annex D for further information.

4 Principle

4.1 Gross calorific value

A weighed portion of the analysis sample of a solid recovered fuel is burned in high-pressure oxygen in a bomb calorimeter under specified conditions. The effective heat capacity of the calorimeter is determined in calibration experiments by the combustion of certified benzoic acid under similar conditions, accounted for in the certificate. The corrected temperature rise is established from observations of temperature before, during and after the combustion reaction takes place. The duration and frequency of the temperature observations depend on the type of calorimeter used. Water is added to the bomb initially to give a saturated vapour phase prior to combustion (see 8.2.1 and 9.2.2), thereby allowing all the water formed, from the hydrogen and moisture in the sample, to be regarded as liquid water.

The gross calorific value is calculated from the corrected temperature rise and the effective heat capacity of the calorimeter, with allowances made for contributions from ignition energy, combustion of the fuse(s) and for thermal effects from side reactions such as the formation of nitric acid. Furthermore, a correction is applied to account for the difference in energy between the aqueous sulphuric acid formed in the bomb reaction and gaseous sulphur dioxide, i. e. the required reaction product of sulphur in the solid recovered fuel. The corresponding energy effect between aqueous and gaseous hydrochloric acid can be negligible for solid recovered fuels of mainly biomass origin.

The corresponding energy effect between aqueous and gaseous hydrochloric acid depends on the sample characteristics, e. g. the content of inorganic and organic chlorine, mineral composition and the actual pH-value in bomb liquid. At the present time no values are available for this chlorine correction. Attention should be paid to the extremely high chlorine content in the test sample because e. g. PVC fractions can affect the calorific value significantly.

Automatic equipment (such as gravimetric analyses) may be used if the method is validated by parallel measurements. This automatic equipment shall fulfil all the requirements regarding sample size, heating procedure, temperature, atmosphere, and weighing accuracy. Deviations from this paragraph shall be reported and justified.

4.2 Net calorific value

The net calorific value at constant volume and the net calorific value at constant pressure of the solid recovered fuel are obtained by calculation from the gross calorific value at constant volume determined on the analysis sample. The calculation of the net calorific value at constant volume requires information about the moisture and hydrogen contents of the analysis sample. In principle, the calculation of the net calorific value at constant pressure also requires information about the oxygen and nitrogen contents of the sample.

5 Reagents

5.1 Oxygen, at a pressure high enough to fill the bomb to 3 MPa, pure with an assay of at least 99,5 % volume fraction, and free from combustible matter.

NOTE Oxygen made by the electrolytic process can contain up to 4 % volume fraction of hydrogen.

5.2 Fuse

5.2.1 Ignition wire, of nickel-chromium 0,16 mm to 0,20 mm in diameter, platinum 0,05 mm to 0,10 mm in diameter, or another suitable conducting wire with well-characterized thermal behaviour during combustion.

5.2.2 Cotton fuse, of white cellulose cotton, or equivalent, if required (see NOTE 1 of 8.2.1).

5.3 Combustion aids, of known gross calorific value, composition and purity, e. g. benzoic acid, n-dodecane, paraffin oil, combustion bags or capsules.

5.4 Standard volumetric solutions and indicators, only for use if analysis of final bomb solutions is required.

5.4.1 Barium hydroxide solution, $c[Ba(OH)_2] = 0{,}05$ mol/l.

5.4.2 Sodium carbonate solution, $c(Na_2C0_3) = 0{,}05$ mol/l.

5.4.3 Sodium hydroxide solution, $c(NaOH) = 0{,}1$ mol/l.

5.4.4 Hydrochloric acid solution, $c(HCl) = 0{,}1$ mol/l.

5.4.5 Screened methyl orange indicator, 1 g/l solution: Dissolve 0,25 g of methyl orange and 0,15 g of xylene cyanole FF in 50 ml of ethanol with a volume fraction of 95 % and dilute to 250 ml with water.

5.4.6 Phenolphthalein, 10 g/l solution: Dissolve 2,5 g of phenolphthalein in 250 ml ethanol with a volume fraction of 95 %.

5.5 Benzoic acid, of calorimetric-standard quality, certified by (or with certification unambiguously traceable to) a recognized standardizing authority.

NOTE 1 Benzoic acid is the sole substance recommended for calibration of an oxygen-bomb calorimeter. For the purpose of checking the overall reliability of the calorimetric measurements, test substances, e. g. n-dodecane, are used. Test substances are mainly used to prove that certain characteristics of a sample, e. g. burning rate or chemical composition, do not introduce bias in the results.

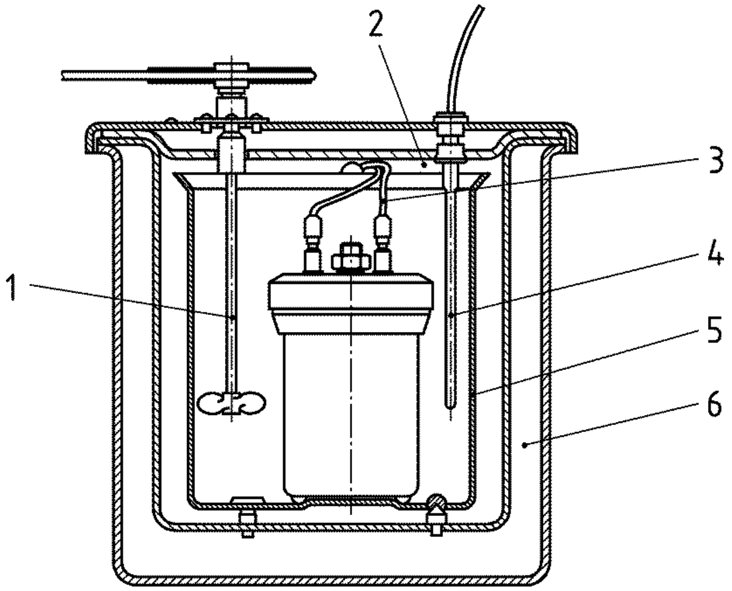
NOTE 2 The benzoic acid is burned in the form of pellets. It is usually used without drying or any treatment other than pelletizing; the sample certificate provides information. It does not absorb moisture from the atmosphere at relative humidities below 90 %.

The benzoic acid shall be used as close to certification conditions as is feasible; significant departures from these conditions shall be accounted for in accordance with the directions in the certificate. The energy of combustion of the benzoic acid, as defined by the certificate for the conditions utilized, shall be adopted in calculating the effective heat capacity of the calorimeter (see 9.2).

6 Apparatus

6.1 General

The calorimeter (see Figure 1), consists of the assembled combustion bomb (6.2.1), the calorimeter can (6.2.2) (with or without a lid), the calorimeter stirrer (6.2.3), water, temperature sensor, and leads with connectors inside the calorimeter can required for ignition of the sample or as part of temperature measurement or control circuits. During measurements the calorimeter is enclosed in a thermostat (6.2.4). The manner in which the thermostat temperature is controlled defines the working principle of the instrument and hence the strategy for evaluation of the corrected temperature rise.



Key

1 stirrer (6.2.3)
2 thermostat lid
3 ignition leads
4 thermometer
5 calorimeter can (6.2.2)
6 thermostat (6.2.4)

Figure 1 — Classical-type bomb calorimeter with thermostat

In aneroid systems (systems without a fluid) the calorimeter can, stirrer and water are replaced by a metal block. The combustion bomb itself constitutes the calorimeter in some aneroid systems.

In bomb calorimetric instruments with a high degree of automation, especially in the evaluation of the results, the calorimeter is in a few cases not as well-defined as the traditional, classical-type calorimeter. Using such an automated calorimeter is, however, within the scope of this document as long as the basic requirements are met with respect to calibration conditions, comparability between calibration and fuel experiments, ratio of sample mass to bomb volume, oxygen pressure, bomb liquid, reference temperature of the measurements and repeatability of the results. A print-out of some specified parameters from the individual measurements is essential. Details are given in Annex C.

As the room conditions (temperature fluctuation, ventilation etc.) can have an influence on the precision of the determination, the manufacturer's instructions for the placing of the instrument shall always be followed.

Equipment, adequate for determinations of calorific value in accordance with this document, is specified in 6.2 to 6.8.

6.2 Calorimeter with thermostat

6.2.1 Bomb calorimeter, capable of withstanding safely the pressures developed during combustion. The design shall permit complete recovery of all liquid products. The material of construction shall resist corrosion by the acids produced in the combustion of solid recovered fuels. A suitable internal volume of the bomb would be from 250 ml to 350 ml.

WARNING — Bomb parts shall be inspected regularly for wear and corrosion; particular attention shall be paid to the condition of the threads of the main closure. Manufacturers' instructions and any local regulations regarding the safe handling and use of the bomb shall be observed. If more than one bomb of the same design is used, it is imperative to use each bomb as a complete unit. Swapping of parts can lead to a serious accident.

