SLOVENSKI STANDARD
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Karakterizacija odpadkov - Določevanje sežigne vrednosti in izračun kurilne vrednosti

Characterization of waste - Determination of gross calorific value and calculation of net calorific value

Charakterisierung von Abfällen - Bestimmung des Brennwertes und Berechnung des Heizwertes

Caractérisation des déchets - Détermination du pouvoir calorifique brut et calcul du pouvoir calorifique net

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English Version

Characterization of waste - Determination of gross calorific value and calculation of net calorific value

Caractérisation des déchets - Détermination du pouvoir calorifique supérieur et calcul du pouvoir calorifique inférieur

Charakterisierung von Abfällen - Bestimmung des Brennwertes und Berechnung des Heizwertes

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (CEN/TS 16023:2013) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

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Introduction

This method is a quick and easy way to evaluate the suitability of waste to be treated by thermal processes.

The determination of gross calorific value is carried out without thermo-chemical corrections. These corrections typically result in minor changes of the result. The influence of these corrections is shown in Annex C.

The result obtained is the gross calorific value at constant volume with both the water of the combustion products and the moisture of the waste as liquid water.

The net calorific value is obtained by calculation from the gross calorific value. For the purpose of this Technical Specification, the value of the net calorific value does not need to be determined exactly. The correction for hydrogen is simplified by the use of typical hydrogen contents derived from table values of hydrogen contents in common types of waste.

This Technical Specification specifies a quick method to determine calorific value; a more comprehensive analysis is described in ISO 1928.

Waste can contain water and (unburnable) solids in large amounts. Therefore their calorific value – especially on the “as received” basis – can be quite low. For some purposes it may be sufficient to determine the gross calorific value only, and not the net calorific value.

WARNING — Strict adherence to all of the provisions prescribed in this Technical Specification should ensure against explosive rupture of the bomb, or a blow-out, provided that the bomb is of proper design and construction and in good mechanical condition. Anyone dealing with waste and sludge analysis is required to be aware of the typical risks of this kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction. Consequently, it is recommended that these samples be handled with special care. The gases that may be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

1 Scope

This Technical Specification specifies a simplified method for the determination of the gross calorific value of waste at constant volume and at the reference temperature of 25 °C in a bomb calorimeter calibrated by combustion of certified benzoic acid. This Technical Specification does not include thermo-chemical corrections.

This Technical Specification also specifies a simplified calculation of the net calorific value from the gross calorific value.

This Technical Specification is applicable for the evaluation of suitability of waste to be treated by thermal processes and for the energy to be recovered.

This Technical Specification is applicable to all kinds of waste.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13965-2:2010, *Characterization of waste - Terminology - Part 2: Management related terms and definitions*

EN 14346, *Characterization of waste - Calculation of dry matter by determination of dry residue or water content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 13965-2:2010 and the following apply.

3.1
corrected temperature rise
change in calorimeter temperature caused solely by the processes taking place within the combustion bomb

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

3.2
gross calorific value at constant volume
absolute value of the specific energy of combustion, in Joules, for unit mass of waste 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 sulfur 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.

3.3
net calorific value at constant pressure
absolute value of the specific energy of combustion, in Joules, for unit mass of waste burned in oxygen at constant pressure under such conditions that all the water of the reaction products remains as water vapour (in a hypothetical state at 0,1 MPa)

Note 1 to entry: The other products are assumed to remain at the reference temperature.

4 Principle

4.1 Gross calorific value

A weighed portion of the waste sample is burned in a high-pressure oxygen atmosphere in a bomb calorimeter under specified conditions. The effective heat capacity of the calorimeter is determined in calibration experiments by 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, 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, without corrections 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.

4.2 Net calorific value

The net calorific value at constant pressure is calculated from the gross calorific value at constant volume using typical values of hydrogen content. The calculation is made without corrections due to the oxygen and nitrogen content of the samples.

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 % (*V/V*), and free from combustible matter.

NOTE Oxygen made by the electrolytic process can contain up to 4 % (*V/V*) hydrogen.

5.2 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.

Knowing the gross calorific value of the fuse is necessary if an accurate calculation according to Annex C is to be carried out. This information is not required for the method described in this Technical Specification.

5.3 Cotton fuse, of cellulose cotton, or equivalent, if required.

Knowing the gross calorific value of the fuse is necessary if an accurate calculation according to Annex C is to be carried out. This information is not required for the method described in this specification. It is necessary to use a fuse with the same length and sections both in the calibration step and in the measurements.

5.4 Combustion aids, of known gross calorific value, composition and purity, such as benzoic acid, n-dodecane, paraffin oil, combustion bags or capsules may be used.

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

The benzoic acid is burned in the form of pellets. It is normally used without drying or any treatment other than pelletising; consult the sample certificate. The benzoic acid shall be used as close to certification conditions as is feasible; significant modifications to the conditions specified should be accounted for in accordance with the directions in the certificate.

5.6 Gelatine or acetobutyrate capsules.

6 Apparatus

6.1 General

The calorimeter (see Figure A.1), consists of the assembled combustion bomb, a can (with or without lid), stirrer, 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. 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.

In combustion 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 Technical Specification as long as the basic requirements are met with respect to calibration conditions, comparability between calibration and waste experiments, ratio of sample mass to bomb volume, oxygen pressure, bomb liquid, reference temperature of the measurements and repeatability of the results.

Equipment, adequate for determinations of calorific value in accordance with this Technical Specification, is specified below.

6.2 Calorimeter with thermostat

6.2.1 Combustion bomb, capable of safely withstanding the pressures developed during combustion. The design shall permit complete recovery of all liquid products. The material of construction shall resist corrosive acids resulting from the combustion of waste. A suitable internal volume of the bomb would be from 250 ml to 350 ml.

6.2.2 Calorimeter can, made of metal, highly polished on the outside and capable of holding an amount of water sufficient to completely cover the flat upper surface of the bomb while the water is being stirred.

6.2.3 Stirrer, working at constant speed.

The stirrer shaft should have a low-heat conduction and/or a low-mass section below the cover of the surrounding thermostat to minimize transmission of heat to or from the system; this is of particular importance when the stirrer shaft is in direct contact with the stirrer motor.

6.2.4 Thermostat (water jacket), completely surrounding the calorimeter, with an air gap of approximately 10 mm separating calorimeter and thermostat.

The mass of water of a thermostat intended for isothermal operation shall be sufficiently large to outbalance thermal disturbances from the outside. The temperature should be controlled to within ± 0,1 K or better throughout the experiment.

6.2.5 Temperature measuring instrument, capable of indicating temperature with a resolution of at least 0,001 K so that temperature intervals of 2 K to 3 K can be determined with a resolution of 0,002 K or better.

The absolute temperature shall be known to the nearest 0,1 K at the reference temperature of the calorimetric measurements. The temperature measuring device should be linear, or linearized, in its response to changes in temperature over the interval it is used.

6.3 Crucible, of silica, nickel-chromium, platinum or similar non-reactive material.

The crucible should be 15 mm to 25 mm in diameter, flat based and about 20 mm deep. Silica crucibles should be about 1,5 mm thick and metal crucibles about 0,5 mm thick. Base metal alloy crucibles are suitable if after a few preliminary firings the weight does not change significantly between tests.

6.4 Ancillary pressure equipment

6.4.1 Pressure regulator, to control the filling of the bomb with oxygen.

6.4.2 Pressure gauge, e.g. 0 MPa to 5 MPa, to indicate the pressure in the bomb with a resolution of 0,05 MPa.

6.4.3 Relief valve or bursting disk, operating at 3,5 MPa, and installed in the filling line, to prevent overfilling the bomb.

CAUTION — Equipment for high-pressure oxygen shall be kept free of oil and grease (high vacuum grease recommended by the manufacturer can be used according to the operating manual of the instrument). Do not test or calibrate the pressure gauge with hydrocarbon fluid.

6.5 Timer

6.6 Balances

6.6.1 Balance for weighing the sample, fuse etc., with an accuracy of at least 0,1 mg; 0,01 mg is preferable and is recommended when the sample mass is of the order of 0,5 g or less.

6.6.2 Balance for weighing the calorimeter water, with an accuracy of at least 0,5 g (unless water can be dispensed into the calorimeter by volume with the required accuracy).

6.7 Pellet press, capable of applying a force of about 100 kN, either hydraulically or mechanically, and having a die suitable to press a pellet with a diameter of about 13 mm and a mass of (1 ± 0,1) g.

7 Sample storage

Biologically active laboratory samples should be stored at about 4 °C and the analysis should be carried out within seven days after sampling. If this is not possible, the samples should be preserved further, for example by freezing or freeze drying in order to minimize biodegradation and loss of volatile compounds.

8 Sample preparation

The goal of any sample preparation procedure is to prepare a test sample in which the composition is not significantly changed compared to the laboratory sample. Due to the different properties of the various kinds of materials there is no general procedure available.

Recommendations for sample pretreatment are given in EN 15002.

Depending on the nature of the sample material a drying step might be required. If necessary, air-dry the complete sample or dry it in a ventilated drying oven at a temperature not exceeding 40 °C or in a freeze dryer. The drying time depends on the technique chosen and the type of sample.

For solid waste materials, one or more particle size reduction steps might be needed in order to achieve a homogeneous and representative test portion. The choice of the technique depends on the nature of the sample and on the particle size needed. Typically, particle size reduction is a multi-step operation that implies the use of a sequence of different techniques like crushing, cutting or grinding.

The particle size of the analysis sample material of solid waste samples for the determination of calorific value shall be less than 1 mm and preferably less than 0,2 mm.

Determination of the moisture content of the resulting analysis sample shall be carried out according to EN 14346.

9 Procedure

9.1 General

The calorimetric determination consists of two separate operations under the same specified conditions:

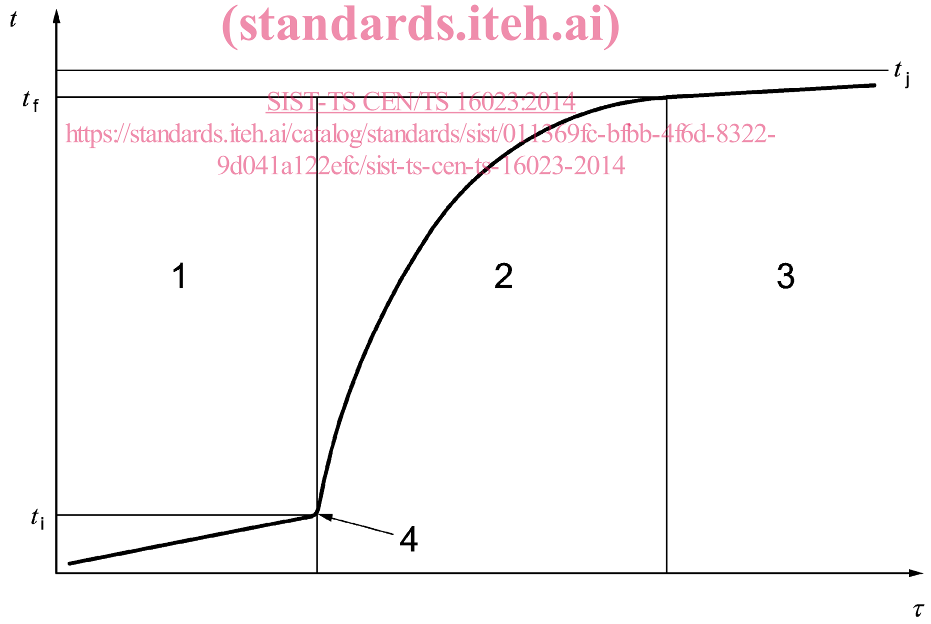
- combustion of the calibrant (benzoic acid);
- combustion of the sample.

The procedure for both operations is essentially the same.

In the calibration experiment, the “effective heat capacity” of the calorimeter is determined (see Clause 10).

Both experiments consist of carrying out quantitatively a combustion reaction (in high-pressure oxygen in the bomb) to defined products of combustion and of measuring the change in temperature caused by the total bomb process.

The temperature measurements required for the evaluation of the corrected temperature rise θ are made during a fore period, a main (= reaction) period, and an after period as outlined in Figure 1. For the adiabatic type calorimeter, the fore and after periods need, in principle, be only as long as required to establish the initial (firing) and final temperatures, respectively. For the isoperibol (isothermal jacket) and the static-jacket type calorimeters, the fore and after periods serve to establish the heat exchange properties of the calorimeter required to allow proper correction for heat exchange between calorimeter and thermostat during the main period when combustion takes place. The fore and after periods shall then be longer.



Key

1	fore period	t_i	initial temperature, in °C
2	main period	t_j	temperature of the jacket, in °C
3	after period	t_f	final temperature, in °C
4	ignition	τ	time, in min

Figure 1 — Time-temperature curve (isoperibol calorimeter)