



**SLOVENSKI STANDARD**  
**SIST EN 15170:2009**

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Characterization of sludges - Determination of calorific value

Charakterisierung von Schlämmen - Bestimmung des Brenn- und Heizwertes

Caractérisation des boues - Détermination du pouvoir calorifique

Ta slovenski standard je istoveten z: **EN 15170:2008**

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## Characterization of sludges - Determination of calorific value

Caractérisation des boues - Détermination du pouvoir  
calorifiqueCharakterisierung von Schlämmen - Bestimmung des  
Brenn- und Heizwertes

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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## Foreword

This document (EN 15170:2008) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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## Introduction

This method is a simple way to evaluate the amenability of sludge and sludge products to be treated by thermal processes. In this European Standard some thermo-chemical corrections are not considered. For detailed descriptions of analytical procedures and theoretical background see ISO 1928 or CEN/TS 14918.

The result obtained is the gross calorific value of the sample at constant volume with both the water of the combustion products and the moisture of the sludge as liquid water. The net calorific value can be obtained by calculation from the gross calorific value. For this either the hydrogen content of the sludge or the amount of water found in the combustion test has to be determined.

Sludges usually contain much water and (un-burnable) solids. Therefore their calorific value – especially on the “as received” basis – is quite low. For many purposes it may be sufficient to determine the gross calorific value only, and not the net calorific value for which additional determinations are necessary. The calculation of the net calorific value at constant volume is described here only, for calculation at constant pressure refer to either ISO 1928 or CEN/TS 14918.

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## 1 Scope

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

The result obtained is the gross calorific value of the sample at constant volume with both the water of the combustion products and the moisture of the sludge as liquid water. In practice, sludges are burned at constant (atmospheric) pressure and the water is not condensed but is removed as vapour with the flue gases. Under these conditions, the operative heat of combustion to be used is the net calorific value of the fuel at constant pressure. In this European Standard the net calorific value at constant volume is described as it requires less additional determinations.

This method is applicable to all kinds of sludges.

## 2 Normative references

The following referenced documents are indispensable for the application 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 12832:1999, *Characterization of sludges — Utilization and disposal of sludges — Vocabulary*

EN 12880, *Characterization of sludges — Determination of dry residue and water content*

EN ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis (ISO 16720:2005)*

ISO 651, *Solid-stem calorimeter thermometers*

ISO 652, *Enclosed-scale calorimeter thermometers*

ISO 1770, *Solid-stem general purpose thermometers*

ISO 1771, *Enclosed-scale general purpose thermometers*

ISO/TS 12902, *Solid mineral fuels — Determination of total carbon, hydrogen and nitrogen — Instrumental methods*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12832:1999 and the following apply.

### 3.1

#### **gross calorific value (at constant volume)**

absolute value of the specific energy of combustion, in Joules, for unit mass of a solid sludge burned in oxygen in a calorimetric bomb under the conditions specified. 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

### 3.2

#### **net calorific value (at constant volume)**

absolute value of the specific energy of combustion, in Joules, for unit mass of a solid sludge burned in oxygen under conditions of constant volume and such that all the water of the reaction products remains as

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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 energy of combustion, in Joules, for unit mass of a solid sludge 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), the other products being as for the gross calorific value, all at the reference temperature

**3.4 corrected temperature rise**  
change in calorimeter temperature caused solely by the processes taking place within the combustion bomb. It is the total observed temperature rise corrected for heat exchange, stirring power etc.

**4 Principle****4.1 Gross calorific value**

A weighed portion of the analysis sample of the solid sludge 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 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, 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 sulfuric acid formed in the bomb reaction and gaseous sulfur dioxide, i. e. the required reaction product of sulfur in the sludge. The corresponding energy effect between aqueous and gaseous hydrochloric acid can be neglected due to the usually low chlorine content of most sludges.

**4.2 Net calorific value**

The net calorific value at constant volume of the sludge is 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.

**NOTE** The main difference between the gross and net calorific values is related to the physical state of water in the reaction products.

**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 may contain up to 4 % (V/V) 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.

It is necessary to use a fuse with the same length and sections both in the calibration step and in the measurements.

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

**5.4 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 pelletizing; consult the sample certificate.

## 6 Apparatus

### 6.1 General

The calorimeter (see a typical example in Annex A), consists of the assembled combustion bomb, the calorimeter can (with or without a lid), the calorimeter 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 European Standard 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.

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

### 6.2 Calorimeter with thermostat

**6.2.1 Combustion bomb**, 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 sludges. 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.

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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  $\pm 0,1$  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.

As alternatives to the traditional mercury-in-glass thermometers, suitable temperature sensors are platinum resistance thermometers, thermistors, quartz crystal resonators, etc. which together with a suitable resistance bridge, null detector, frequency counter or other electronic equipment provide the required resolution. The short-term repeatability of this type of device shall be 0,001 K or better. Long-term drift shall not exceed the equivalent of 0,05 K for a period of six months. For sensors with linear response (in terms of temperature), drift is less likely to cause bias in the calorimetric measurements than are non-linear sensors.

Mercury-in-glass thermometers shall conform to ISO 651, ISO 652, ISO 1770 or ISO 1771. A viewer with magnification about  $5 \times$  is needed for reading the temperature with the resolution required.

A mechanical vibrator to tap the thermometer is suitable for preventing the mercury column from sticking. If this is not available, the thermometer shall be tapped manually before reading the temperature.

**6.2.6 Ignition circuit**

The electrical supply should be 6 V to 12 V alternating current from a step-down transformer. It is desirable to include a pilot light in the circuit to indicate when current is flowing.

**6.3 Crucible, of silica, nickel-chromium, platinum or similar un-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.

**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 from 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, indicating minutes and seconds****6.6 Balances**

**6.6.1 Balance for weighing the sample, fuse etc.**, with a resolution 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 (see 7.2.4).

**6.6.2 Balance for weighing the calorimeter water**, with a resolution of 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 having a diameter about 13 mm and a mass of  $(1 \pm 0,1)$  g.

## 7 Procedure

### 7.1 General

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

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

The procedure for both above determination is essentially the same. In fact, the overall similarity is a requirement for proper cancellation of systematic errors caused, for example, by uncontrolled heat leaks not accounted for in the evaluation of the corrected temperature rise  $\theta$ .

It consists in carrying out quantitatively a complete combustion reaction (in high-pressure oxygen in the bomb) and of measuring the change in temperature caused by the total bomb process. The combustion of the calibrant makes it possible to determine the effective heat capacity of the calorimeter.

Certain sludges may persistently burn incompletely, leaving residues that contain significant amounts of unburned sample or soot. By adding known amounts of an auxiliary material (e. g. benzoic acid, n-dodecane or paraffin oil), by using bags or capsules or cotton fuse, or by omitting the distilled water from the bomb, or by using a lower oxygen filling pressure, a clean combustion can in most instances be achieved.

The temperature measurements required for the evaluation of the corrected temperature rise  $\theta$  are made during a fore period, a main (reaction) period, and an after period (see 7.7.3 and Annex B).

### 7.2 Sample preparation

**7.2.1** Measure the dry residue of the sample following EN 12880, so that the matter obtained has a mass sufficient for the following test. If the sludge is visibly wet or contains too much moisture dewater (e. g. by pressing) or dry carefully at a temperature not exceeding 40 °C or by freeze-drying following EN ISO 16720. Repeat measuring the moisture content of the sample to be analysed.

Note: Drying of samples to e. g. 5% moisture is advantageous as less sample has to be weighed for combustion experiments compared to non-dries ones.

**7.2.2** Grind the sample to powder with a particle size of about to 0,2 mm nominal topsize.

**7.2.3** The sample shall be well-mixed and in reasonable moisture equilibrium with the laboratory atmosphere. The moisture content shall either be determined simultaneously with the weighing of the samples for the determination of calorific value, or the sample shall be kept in a small, effectively closed container until moisture analyses are performed, to allow appropriate corrections for moisture in the analysis sample.

Calibration experiments will give a temperature rise of about 2 K to 3 K (depending on the type of calorimeter, see Clause 8). In order to have the same range of temperature rises for the combustion of samples their mass should be sufficient to ensure this. If the temperature rise is too small, dry the sample (see 7.2.1) and/or increase the sample mass and/or use a combustion aid or combustion bags and repeat the combustion. Otherwise, the accuracy of measurements may be insufficient.

NOTE 1 It is difficult to give suitable sample masses due to varying solids and moisture contents. However, an indication is given. Experiments in an isoperibolic calorimeter gave a temperature rise of 1 K using 1 g of a sludge from an