

Characterization of sludges — Determination of calorific value

ICS 13.030.20

National foreword

This British Standard is the UK implementation of EN 15170:2008.

BSI, as a member of CEN, is obliged to publish EN 15170 as a British standard. However, attention is drawn to the fact that during the development of this European Standard, the UK committee voted against its approval as a European Standard.

Determination of calorific value has been standardized for many other materials, such as coal and oil. In the opinion of the UK committee, in this standard, it should only be necessary to refer to exceptional analytical problems that apply specifically to sludges.

This standard sets out how the two types of calorimeter (static jacket and adiabatic) are assembled, operated and calibrated and the method for correcting for heat loss in one type. In the opinion of the UK committee this is not necessary because such theory and practice are contained in the operating instructions for the equipment, almost without exception.

The UK committee believes that information on the different types of equipment that can be used, the standard for calibration (but not the precise method of application as this is equipment dependent) and the potential for errors can be found in such British Standards as BS 1016-105:1992 and BS 7420:1991, as well as in guides produced by the oil companies. Without exception, the reference material for calibration is benzoic acid, because it can be dried to a water content of 0.01%, is chemically stable and is not prone to bacterial attack. It has also been subject to assessment for at least 60 years.

The UK participation in its preparation was entrusted to Technical Committee EH/5, Sludge characterization.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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English Version

Characterization of sludges - Determination of calorific value

Caractérisation des boues - Détermination du pouvoir
calorifique

Charakterisierung von Schlämmen - Bestimmung des
Brenn- und Heizwertes

This European Standard was approved by CEN on 11 October 2008.

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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.

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

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.