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Sludge recovery, recycling, treatment and disposal — Determination of calorific value of sludge

Valorisation, recyclage, traitement et élimination des boues — Détermination du pouvoir calorifique des boues

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Foreword

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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).

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This document was prepared by the European Committee for Standardization as EN 15170:2008 and was adopted, without modification other than those given below, by Technical Committee ISO/TC 275, *Sludge recovery, recycling, treatment and disposal*.

— mercury-in-glass thermometers are replaced by resistance thermometers;

— subclause 7.7 was renumbered as Clause 8, and subsequent Clauses and subclauses were renumbered accordingly;

— the calculation of the net calorific value at constant volume after determination of water amount in the test (subclause 10.3 in EN 15170:2008) was removed;

— Annex B was removed, and Figure 1 was added in 8.2.2 instead;

— Annex C in EN 15170:2008 is Annex B in this document.

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

The method specified in this document is a simple way to evaluate the amenability of sludge and sludge products to be treated by thermal processes. In this document, some thermo-chemical corrections are not considered. For detailed descriptions of analytical procedures and theoretical background see ISO 1928.

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 derived from the gross calorific value. For this the hydrogen content of the sludge is determined.

Sludge usually contains much water and (un-burnable) solids. Therefore, the calorific value – especially on the “as received” basis – is quite low. For many purposes it can 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 only is described here; for calculation at constant pressure refer to ISO 1928.

Sludge recovery, recycling, treatment and disposal — Determination of calorific value of sludge

1 Scope

This document 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, sludge is 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. The net calorific value at constant volume can also be used, equations for the calculation are given only as this requires less additional determinations.

This method is applicable to all kinds of sludge.

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 11465, *Sludge, treated biowaste, soil and waste — Determination of dry residue or water content and calculation of the dry matter fraction on a mass basis*[1)]

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

ISO 29541, *Coal and coke — Determination of total carbon, hydrogen and nitrogen — Instrumental method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
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

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.

1) Under preparation. Stage at the time of publication: ISO/DIS 11465.

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)

Note 1 to entry: The other products are in the same state 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)

Note 1 to entry: The other products are in the same state as for the gross calorific value, all at the reference temperature.

3.4
corrected temperature rise
θ
change in calorimeter temperature caused solely by the processes taking place within the combustion bomb

Note 1 to entry: This 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 sludge.

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 % volume fraction and free from combustible matter.

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.

If a fuse is used, it shall have 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. For example, benzoic acid, n-dodecane, paraffin oil, combustion bags or capsules can be used.

5.4 Benzoic acid, $C_7H_6O_2$, CAS RN® 65-85-0[2], 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 CRM certificate. The benzoic acid does not absorb moisture from the atmosphere at a relative humidity below 90 %, but it should be stored in a moisture-free environment (e.g. a desiccator) until use.

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. A test substance should have a certified purity and a well-established energy of combustion.

6 Apparatus

6.1 Overview

The calorimeter (see a typical example in Annex A, Figure A.1), 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 way 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 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.

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

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2) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same result.

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 sludge. A suitable internal volume of the bomb is from 250 ml to 350 ml.

WARNING — Combustion vessel parts shall be inspected regularly for wear and corrosion; particular attention shall be paid to the condition of the threads of the main closure. The user shall follow manufacturer's instructions and shall be aware of any local regulations regarding the safe handling and use of the combustion vessel. When more than one combustion vessel of the same design is used, each combustion bomb (body and screw lid) shall be used as a complete unit. Colour coding is recommended. Swapping of parts can lead to a serious accident.

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 Resistance thermometer, 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.

It should be noted that resistance thermometers have a non-linear characteristic. The calibration range should be chosen so that the function of the characteristic curve has practically no influence on the measurement result.

6.2.6 Ignition circuit.

The electrical supply should be 6 V to 25 V alternating current from a step-down transformer. A pilot light should be included in the circuit to indicate when current is flowing.

NOTE Modern calorimeters have the ignition circuit already built in, then no external transformer is required.

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 approximately 20 mm deep. Silica crucibles should be approximately 1,5 mm thick and metal crucibles approximately 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.