



BSI Standards Publication

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

National foreword

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**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
inférieurCharakterisierung von Abfällen - Bestimmung des
Brennwertes und Berechnung des Heizwertes

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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 $\pm 0,1$ 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.

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 $(1 \pm 0,1)$ 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:

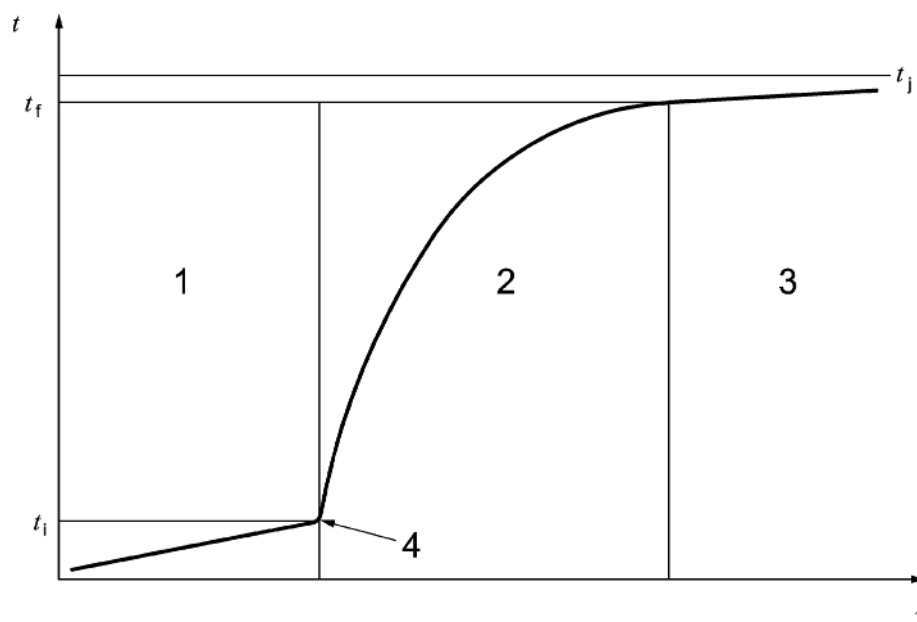
- combustion of the calibrant (benzoic acid);
- combustion of the sample.

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)

The power of stirring shall be maintained constant throughout experiments that call for a constant rate of stirring. An excessive rate of stirring results in an undesirable increase in the power of stirring with ensuing difficulties in keeping it constant. A wobbling stirrer is likely to cause significant short-term variations in stirring power.

During combustion, the bomb head will become noticeably hotter than other parts of the bomb, and it is important to have enough well-stirred water above it to maintain reasonably small temperature gradients in the calorimeter water during the rapid part of the rise in temperature.

9.2 General preparations, measurements and temperature corrections

9.2.1 Preparation of the bomb

Weigh the combustible fuse and/or ignition wire either with a resolution of at least 0,1 mg, or keep its mass constant, within specified limits, for all experiments (calibration and waste sample experiments). Fasten the ignition wire tautly between the electrodes in the bomb. Check the resistance of the ignition circuit of the bomb; for most bombs, it should not exceed 5 Ω to 10 Ω , measured between the outside connectors of the bomb head, or between the connector for the insulated electrode and the bomb head.

Tie, or attach firmly, the fuse to the ignition wire.

Place the crucible with its content (according to 9.3 or 9.4) in its support and bring the fuse into contact with the content. Make sure that the position of the crucible in the assembled bomb is symmetrical with respect to the surrounding bomb wall.

When the ignition wire is combustible as well as electrically conducting, an alternative procedure may be adopted. A longer piece of wire, enough to make an open loop, is connected to the electrodes. After mounting of the crucible, the loop is brought in contact with the sample pellet or capsule. (In some cases the ignition process is better controlled when the wire is kept at a small distance above the sample pellet.) Care should be taken to prevent any contact between ignition wire and crucible, in particular when a metal crucible is used since this would result in shorting the ignition circuit. A special fuse is superfluous under these conditions. The resistance of the ignition circuit of the bomb will be increased by a small amount only. For closer details of preparing the bomb also refer to the manufacturer's instructions.

Add a defined amount of distilled water to the bomb. The amount shall always be exactly the same in calibration and in sample experiments. As a main principle (1,0 \pm 0,1) ml distilled water is added into the bomb. For some waste samples (and some calorimeters) the complete combustion can be achieved by omitting the addition of distilled water to the bomb. In some cases the total absorption of the gaseous combustion products might require the use of a larger amount of distilled water (e.g. 5 ml).

Assemble the bomb.

Charge the bomb slowly with oxygen to obtain a pressure of (3,0 \pm 0,2) MPa. The same procedure shall be used both in calibration and in sample experiments. If the bomb is inadvertently charged with oxygen above 3,3 MPa, discard the test and begin again.

WARNING — Do not reach over the bomb during charging.

9.2.2 Assembling the calorimeter

Bring the calorimeter water to within \pm 0,3 K of the selected initial temperature and fill the calorimeter can with the required amount. This quantity of water in the calorimeter can shall be the same to within 0,5 g or better in all experiments.

Place the bomb in the calorimeter can.

Check the bomb for gas leaks as soon as its top becomes covered with water. If the gas valves are not fully submerged, check for leaks with a drop of water across the exposed opening.

Connect the leads for the ignition circuit.

WARNING — If gas escapes from the bomb, discard the test, eliminate the cause of leakage and begin again.

Cooling water, temperature controls, stirrers, etc. are turned on. Make sure that the stirrer works properly. A period of about 5 min is normally required for the assembled calorimeter to reach a steady-state in the thermostat, regardless of the type of calorimeter.

9.2.3 Combustion reaction and temperature measurements

Start taking temperature readings, to the nearest 0,001 K or better, as soon as the calorimeter has reached steady-state conditions. Readings at 1 min intervals normally suffice to establish the drift rate of the fore period. If a mercury-in-glass thermometer is used for the temperature measurements, tap the thermometer lightly for about 10 s before each reading and take care to avoid parallax errors.

At the end of the fore period, when the initial temperature t_i has been established, the combustion is initiated by firing the ignition wire. Hold the switch closed only for as long as it takes to ignite the wire.

NOTE Many calorimeters do this automatically.

Continue taking temperature readings at 1 min intervals. The time corresponding to t_i marks the beginning of the main period. During the first few minutes of this period, when the temperature is rising rapidly, readings to the nearest 0,02 K are adequate.

Resume reading temperature to the nearest 0,001 K or better as soon as practicable, but no later than 5 min after the beginning of the main period. Criteria for the length of the fore-, main-, and after periods, and hence the total number of temperature readings required, depend on the type of calorimeter (adiabatic, static jacket or isoperibol). For detailed descriptions, see ISO 1928 or EN 15400.

After beginning the after period, continue taking temperature readings at 1 min intervals for an additional 5 min.

9.2.4 Disassembling the calorimeter

At the end of the after period, when all the required temperature readings have been completed, remove the bomb from the calorimeter, release the pressure slowly (following manufacturers manual) and dismantle the bomb.

Examine the interior of the bomb, the crucible and any solid residue carefully for signs of incomplete combustion. Discard the test if unburned sample or any soot deposit is visible.

Remove and measure any unburned pieces of ignition wire.

9.2.5 Temperature corrections

9.2.5.1 Reference temperature

The temperature at the end of the main period, the final temperature t_f , is the reference temperature of the individual experiment.

9.2.5.2 Corrected temperature rise θ

The temperature at the end of the main period t_f gives, together with the initial temperature t_i the observed temperature rise $t_f - t_i$.

a) Isoperibol and static jacket calorimeters

In addition to the rise in temperature caused by the processes in the calorimetric bomb, the observed temperature rise contains contributions from heat exchange between calorimeter and thermostat and from stirring power. Allowance for heat exchange is made by the so-called heat-leak correction Δt_{ex} , which includes the contribution from stirring power:

$$t_f - t_i = \theta + \Delta t_{ex} \quad (1)$$

and hence

$$\theta = (t_f - t_i) - \Delta t_{ex} \quad (2)$$

In the Dickinson extrapolation method, the objective is to find a time τ_x such that

$$\Delta t_{ex} = (\tau_f - \tau_x) \cdot g_f + (\tau_x - \tau_i) \cdot g_i \quad (3)$$

where

Δt_{ex} is the heat-leak-correction and can be evaluated in various ways. See Annex B for the Dickenson approach;

τ_x is the time, where the change in temperature ($t_x - t_i$) is 0,6 times the observed temperature rise ($t_f - t_i$), in min;

t_x is the temperature corresponding to 0,6 times the observed temperature rise ($t_f - t_i$), in °C;

τ_i is the time at the beginning of the main period, in min;

τ_f is the time at the end of the main period, in min;

g_i is the drift rate at τ_i , in °C per min;

g_f is the drift rate at τ_f , in °C per min.

This is accomplished when the hatched areas a and b in Figure B.1 are of equal size. The corrected temperature rise θ becomes:

$$\theta = t_f - t_i - (\tau_f - \tau_x) \cdot g_f - (\tau_x - \tau_i) \cdot g_i = t_f^* - t_i^* \quad (4)$$

where

t_i^* is the corrected initial temperature, in °C;

t_f^* is the corrected final temperature, in °C.

For a combustion reaction, the time-temperature curve is close to being an exponential, which means that τ_x is the time associated with the temperature where the change in temperature ($t_x - t_i$) is 0,6 times the total (observed) temperature rise ($t_f - t_i$). The quantity $(\tau_x - \tau_i)$ varies with the kinetic behaviour of the combustion reaction of the type of sample studied.

Another method to determine the corrected temperature rise is the "Regnauld-Pfaundler method" (for details see ISO 1928 or EN 15400).

b) Adiabatic calorimeters

In adiabatic systems, heat exchange is by definition negligible. It is, however, common practice to compensate for the stirring power by an offset in temperature in the adiabatic control system (for details see ISO 1928 or EN 15400).

9.3 Calibration

Combustion of certified benzoic acid (5.5) under specified conditions to gaseous carbon dioxide and liquid water serves to change the temperature of the calorimeter by one unit interpretable in defined units of energy. The classical type of combustion calorimeter can be maintained unchanged over extended periods of time in terms of mass (heat capacity), geometry and heat exchange surfaces. This allows for calibration of the instrument to be carried out as a separate series of measurements, establishing the effective heat capacity ε of the calorimeter.

The certificate value for the energy of combustion of benzoic acid refers to a process where the mass of sample and initial water, respectively, is 3 g/l of free bomb volume, the initial pressure of oxygen is 3,0 MPa and the reference temperature is 25 °C. When calibrations are performed under different conditions, the certificate value shall be adjusted. A numerical expression to correct for such deviations is given in the certificate.

The design of the calibration experiment, regarding oxygen pressure, amount of bomb water, reference temperature, duration of the fore-, main-, and after periods, the temperature rise etc., defines the detailed procedure for subsequent waste sample combustions. For the ordinary series of calibrations, five satisfactory combustions of benzoic acid shall be carried out. The sample shall be burned as pellets. The mass of benzoic acid should be varied between 0,7 g to 1,3 g. The calorimetric procedure described in 9.2 shall be followed. The initial temperature shall be chosen such that the reference temperature of the determination is within the chosen range for the reference temperature.

For details concerning calibration conditions and deviations from the conditions see ISO 1928.

9.4 Samples

9.4.1 Setting up the test samples

Samples containing no volatile compounds 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.

Samples containing volatile compounds shall be carefully and rapidly homogenised and filled in to a capsule (5.6), in order to minimize losses of the volatile compounds.

The mass of the sample shall be chosen to give a temperature rise comparable to calibration experiments which typically give a rise of about 2 K to 3 K. If the temperature rise is too small 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 Automated calorimetric instruments are often aneroid systems (systems without a fluid) where the calorimeter can, stirrer and water are replaced by a metal block. Characteristically they have a small heat capacity, leading to large changes in calorimeter temperature, thus facilitating the measurement of θ with a relatively high resolution. Conversely, large values of θ tend to increase the risk for introducing systematic error, in aneroid systems aggravated by difficulties in

achieving uniform calorimeter surface temperature during combustion of the sample. A countermeasure is to limit the sample mass, bearing in mind that for smaller samples particular attention need to be given to their being representative.

Place the test portion in the crucible and determine the weight of the portion to the nearest 0,1 mg. In case of samples containing volatile compounds, weigh the capsule (5.6), fill in the sample and weigh again, both to the nearest 0,1 mg. Put the weighed fuse (weigh to nearest 0,1 mg) into the crucible.

In case of solid waste with low density press a pellet (6.7) with a suitable force to produce a compact, unbreakable test piece, containing the weighed fuse, weigh to nearest 0,1 mg and put it into the crucible.

The calorimetric procedure described in 9.2 shall be followed.

9.4.2 Use of combustion aid

9.4.2.1 General

Certain waste may persistently burn incompletely; leaving residues that contain significant amounts of unburned sample or soot.

In this case, a clean combustion can be achieved in most instances by one of the following methods:

- adding known amounts of an auxiliary material (e.g. benzoic acid, n-dodecane or paraffin oil);
- using bags, capsules or cotton fuse;
- by omitting addition of distilled water to the bomb;
- by using a lower oxygen filling pressure.

9.4.2.2 Liquid combustion aid

After the mass of the test portion has been determined, auxiliary liquid material shall be added drop by drop on the sample placed in the crucible (allowing the liquid to be absorbed), and the added amount shall be determined by weighing to the nearest 0,1 mg.

9.4.2.3 Solid combustion aid

After the mass of the test portion has been determined, solid combustion aid (benzoic acid recommended) should be added, mixed homogenously and the added amount shall be determined by weighing to the nearest 0,1 mg.

9.4.2.4 Combustion bags or capsules

Combustion capsules or bags, or combustible crucibles with precisely known calorific value (gelatine, acetobutyrate (5.6) or polyethylene) can be used as combustion aids (as such or with e.g. benzoic acid) according to the manufacturer's instructions. They shall be weighed precisely before filling. The sample and the combustion aid, e.g. benzoic acid, shall be mixed cautiously in the bag or capsule before testing.

10 Calculation of effective heat capacity

For systems where the quantity of water in the calorimeter can is kept the same in all experiments, the effective heat capacity ε is calculated by Formula (5):

$$\varepsilon = \frac{m_{ba} \cdot q_{V,ba}}{\theta} \quad (5)$$

where

- ε is the effective heat capacity of the calorimeter as determined in the calibration, in Joules per Kelvin (J/K);
- m_{ba} is the mass of benzoic acid, in grams (g);
- $q_{V,ba}$ is the certified gross calorific value of benzoic acid at constant volume, in Joules per gram (J/g);
- θ is the corrected temperature rise, in Kelvin (K).

NOTE Formula (5) is a simplified version of the correct calculation including thermo-chemical corrections and corrections of wires and fuses (see Annex C).

11 Gross calorific value

Calculate the gross calorific value at constant volume from the individual experiment by substituting into Formula (6):

$$q_{V,gr} = \frac{\varepsilon_{(n)} \cdot \theta - m_2 \cdot q_{V,2}}{m_1} \quad (6)$$

where

- $q_{V,gr}$ is the gross calorific value at constant volume of the sample as analysed, in Joules per gram (J/g);
- $\varepsilon_{(n)}$ is the mean value of the effective heat capacity of the calorimeter as determined in the calibrations, in Joules per Kelvin (J/K);
- m_1 is the mass of the waste sample, in grams (g);
- m_2 is the mass of the combustion aid (if relevant), in grams (g);
- $q_{V,2}$ is the gross calorific value at constant volume of the combustion aid (if relevant), in Joules per gram (J/g).

NOTE Formula (6) is a simplified version of the correct calculation including thermo-chemical corrections and corrections of wires and fuses, see Annex C.

The gross calorific value as analysed is calculated to dry basis according to Formula (7):

$$q_{V,gr,d} = q_{V,gr} \cdot \frac{100}{100 - M_{ad}} \quad (7)$$

where

- $q_{V,gr,d}$ is the gross calorific value at constant volume of the dry sample, in Joules per gram (J/g);
- $q_{V,gr}$ is the gross calorific value at constant volume of the sample as analysed, in Joules per gram (J/g);
- M_{ad} is the moisture content in the analysis sample, in percent by mass (%).

The gross calorific value at constant volume required for any particular moisture content basis, $q_{V,gr,M}$, is derived from Formula (8):

$$q_{V,gr,M} = q_{V,gr,d} \cdot \frac{100 - M}{100} \quad (8)$$

where

- $q_{V,gr,M}$ is the gross calorific value at constant volume of the moisture content basis, in Joules per gram (J/g);
- M is the moisture content for which the calorific value is required, in percent by mass (%).

12 Calculation of net calorific value

12.1 General

The main difference between the gross and net calorific values is related to the physical state of water in the reaction products (see 3.2 and 3.3). Water can derive from waste moisture and as a combustion product from hydrogen. The correction for hydrogen is simplified by use of typical hydrogen contents derived from Table D.1.

12.2 Net calorific value at constant pressure

The net calorific value on the “dry basis” in J/g is calculated by Formula (9):

$$q_{p,net,d} = q_{V,gr,d} - 212,2 \times \omega(H)_d \quad (9)$$

where

- $q_{p,net,d}$ is the net calorific value at constant pressure of the dry (moisture-free) fuel (waste), in Joules per gram (J/g);
- $\omega(H)_d$ is the hydrogen content, in percentage by mass (mass fraction), of the moisture-free (dry) waste (including the hydrogen from the water of hydration of the mineral matter as well as the hydrogen in the waste). From the knowledge of the type of waste, a value is taken from Annex D.

NOTE Formula (9) is a simplified version of the correct calculation as the small correction due to the oxygen and nitrogen contents in the waste is ignored. See ISO 1928 for the correct calculation.

Net calorific value at any moisture level in J/g is given by Formula (10):

$$\begin{aligned} q_{p,net,M} &= \left(q_{V,gr,d} - 212,2 \times \omega(H)_d \right) \times \frac{100 - M}{100} - 24,43 M \\ &= q_{p,net,d} \times \frac{100 - M}{100} - 24,43 M \end{aligned} \quad (10)$$

where

- $q_{p,net,M}$ is the net calorific value at constant pressure with moisture content M ;
- M is the moisture content for which the calculation is required, in percentage by mass (%).

On the dry basis, $M = 0$; on the air-dried basis, $M = M_{ad}$; on the as-sampled or as-fired (as received, ar) basis, $M = M_{ar}$ (total moisture content as received).

13 Expression of results

The results of the calculations stated in Clause 11 and Clause 12 are expressed in the basic SI unit for calorific values, J/g. It is, however, more convenient to report the calorific values in MJ/kg.

Hence, the results of the calculations should be converted into MJ/kg using $1 \text{ J/g} = 0,001 \text{ MJ/kg}$ and rounded to the next 1 MJ/kg for the purpose of reporting.

14 Quality control

For heterogeneous samples, it is recommended that the analysis is carried out in duplicate.

The performance of the system shall be checked by application of suitable reference materials (e.g. polyethylene powder). A convenient way to check a combustion system for an amount of, e. g. 1,0 g sample, is to use benzoic acid as a quality control sample.

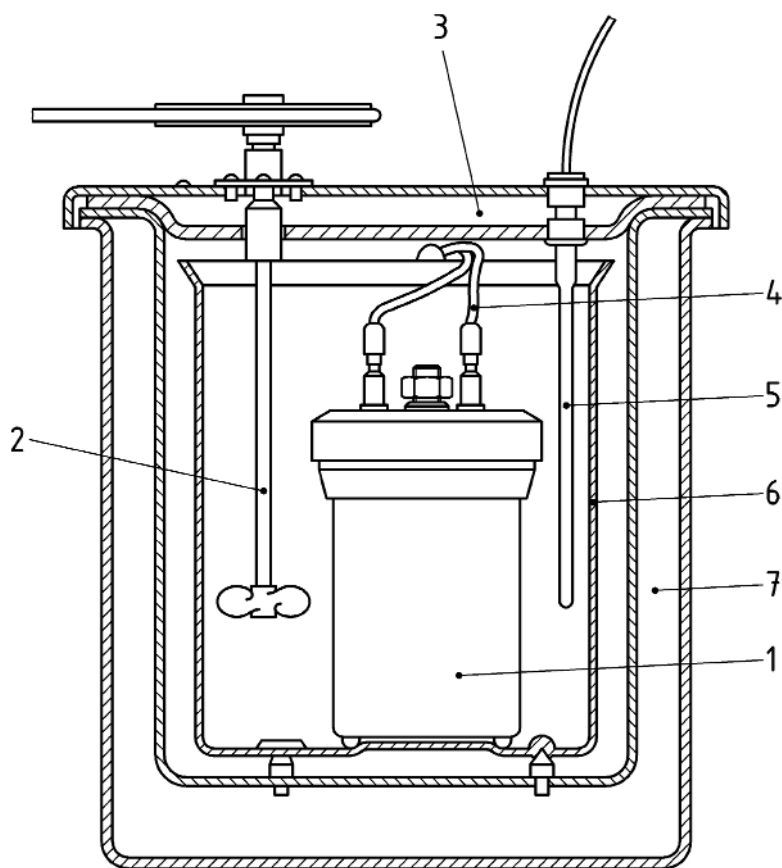
15 Test report

The test report shall include at least the following information:

- a) reference to this Technical Specification (CEN/TS 16023);
- b) all information necessary for the complete identification of the waste sample;
- c) details of sample preparation, if carried out;
- d) results of the determination with reference to the reporting basis valid for the calorific value(s);
 - 1) gross calorific value (at constant volume) of dry waste;
 - 2) net calorific value (at constant pressure) of dry waste;
 - 3) net calorific value (at constant pressure) of moist waste (moisture content as sampled or as received).
- e) information about the hydrogen value used for the calculation of the net calorific value;
- f) any details not specified in this Technical Specification and any other factor which may have affected the results.

Annex A (informative)

Example of a calorimeter



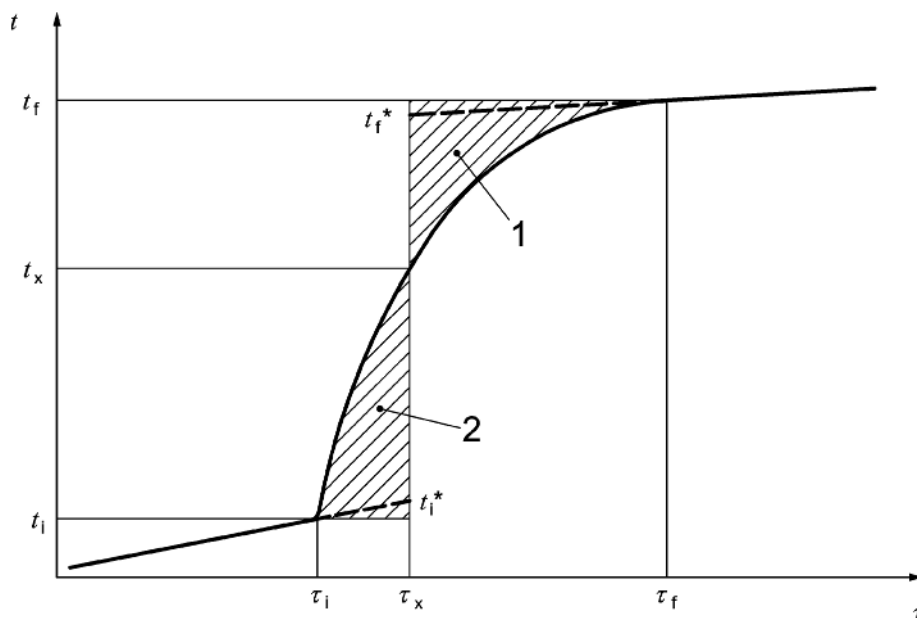
Key

- 1 combustion bomb
- 2 stirrer
- 3 thermostat lid
- 4 ignition leads
- 5 thermometer
- 6 calorimeter can
- 7 thermostat (water jacket)

Figure A.1 — Classical-type bomb combustion calorimeter with thermostat

Annex B (informative)

Temperature evolution



Key

- 1 area a
- 2 area b
- t temperature, in °C
- t_i initial temperature
- t_x temperature corresponding to 0,6 times the observed temperature rise $(t_f - t_i)$, in °C;
- t_f final temperature, in °C
- t_i^* corrected initial temperature, in °C
- t_f^* corrected final temperature, in °C
- τ time, in min
- τ_i time at the beginning of the main period, in min;
- τ_x time where the change in temperature $(t_x - t_i)$ is 0,6 times the observed temperature rise $(t_f - t_i)$, in min;
- τ_f time at the end of the main period, in min

NOTE Dickinson's method is only one of the methods for evaluating the Δt_{ex} .

Figure B.1 — Dickinson extrapolation

Annex C (informative)

Calculation of the gross calorific value – Correct calculation versus the simplified calculation

C.1 Correct calculation

The correct calculation of gross-calorific value at constant volume in J/g is according to ISO 1928:

$$q_{V,gr} = \frac{\varepsilon_{(n)} \cdot \theta - Q_{fuse} - Q_{ign} - m_2 \cdot q_{V,2} - \frac{Q_S}{m_1} - \frac{Q_N}{m_1} - \frac{Q_X}{m_1}}{m_1} \quad (C.1)$$

where

- $q_{V,gr}$ is the gross calorific value at constant volume of the sample as analysed, in Joules per gram (J/g);
- $\varepsilon_{(n)}$ is the mean value of the effective heat capacity of the calorimeter as determined in the calibrations, and calculated according to the Formula (C.2) below, in Joules per Kelvin (J/K);
- θ is the corrected temperature rise, in Kelvin (K);
- m_1 is the mass of the waste sample, in grams (g);
- m_2 is the mass of the combustion aid (if relevant), in grams (g);
- $q_{V,2}$ is the gross calorific value at constant volume of the combustion aid (if relevant), in Joules per gram (J/g);
- Q_S is the correction for taking the sulfur from aqueous sulfuric acid to gaseous sulfur dioxide, in Joules (J);
- Q_N is the correction from formation of nitric acid (from liquid water and gaseous nitrogen and oxygen), in Joules (J);
- Q_X is the contribution from formation of halogen acids (from ions in water to gaseous compounds), in Joules (J);
- Q_{fuse} is the contribution from oxidation of the fuse, in Joules (J);
- Q_{ign} is the contribution from oxidation of the ignition wire, in Joules (J).

The correct calculation of the effective heat capacity ε is according to ISO 1928:

$$\varepsilon = \frac{m_{ba} \cdot q_{V,ba} + Q_{fuse} + Q_{ign} + Q_N}{\theta} \quad (C.2)$$

where

- ε is the effective heat capacity of the calorimeter as determined in the calibration, in Joules per Kelvin (J/K);
- θ is the corrected temperature rise, in Kelvin (K);
- m_{ba} is the mass of benzoic acid, in grams (g);
- $q_{V,ba}$ is the certified gross calorific value of benzoic acid at constant volume, in Joules per gram (J/g);

- Q_{fuse} is the contribution from oxidation of the fuse, in Joules (J);
 Q_{ign} is the contribution from oxidation of the ignition wire, in Joules (J);
 Q_{N} is the correction from formation of nitric acid (from liquid water and gaseous nitrogen and oxygen), in Joules (J).

C.2 Sulfur correction

$$Q_{\text{S}} = 3,14 \cdot m(\text{SO}_4) \quad (\text{C.3})$$

where

- Q_{S} is the correction for taking the sulfur from the aqueous sulfuric acid to gaseous sulfur dioxide, in Joules (J);
 $m(\text{SO}_4)$ is the (absolute) mass of sulfate in the bomb washing, i.e. the quantitative washing of the interior of the bomb, including electrode and crucible, after the combustion containing all the formed sulfuric acid, in milligrams (mg).

Alternatively, Q_{S}/m_1 can be calculated from the elemental analysis of the waste:

$$Q_{\text{S}} = 94,1 \cdot \omega(\text{S}) \quad (\text{C.4})$$

where

- $\omega(\text{S})$ is the mass fraction of sulfur in the waste, expressed in percent (%).

C.3 Nitrogen correction

$$Q_{\text{N}} = 0,97 \cdot m(\text{NO}_3) \quad (\text{C.5})$$

where

- Q_{N} is the contribution from formation of nitric acid (from liquid water and gaseous nitrogen and oxygen), in Joules (J);
 $m(\text{NO}_3)$ is the (absolute) mass of nitrate in the bomb washing, i.e. the quantitative washing of the interior of the bomb, including electrode and crucible, after the combustion containing all the formed nitric acid from the combustion process, in milligrams (mg).

C.4 Halogens correction

$$Q_{\text{X}} = f(\text{X}) \cdot m(\text{X}) \quad (\text{C.6})$$

where

- Q_{X} is the contribution from formation of halogen acids (from ions in water to gaseous compounds), in Joules (J);
 $f(\text{X})$ is the factor for each halogen (e.g. fluorine: 3,23; chlorine: 2,08);
 $m(\text{X})$ is the (absolute) mass of halogen (fluorine, chlorine, or bromine) in the bomb washing, i.e. the quantitative washing of the interior of the bomb, including electrode and crucible, after the combustion containing all the formed hydro –fluoric, -chloric, -bromic and – iodic acid, in milligrams (mg).

Alternatively, Q_X/m_1 can be calculated from the elemental analysis of the waste:

$$Q_X = f^*(X) \cdot \omega(X) \quad (C.7)$$

where

- $f^*(X)$ is the factor for each halogen (fluorine: 32,3; chlorine: 20,8);
- $\omega(X)$ is the mass fraction of each halogen in the waste, expressed in percent (%).

C.5 Influence of the thermo-chemical corrections

According to Clause 11 the gross calorific value is calculated according to Formula (C.8):

$$q_{V,gr} = \frac{\varepsilon_{(n)} \cdot \theta - m_2 \cdot q_{V,2}}{m_1} \quad (C.8)$$

Compared to the correct calculation given by the Formula (C.1) the Formula (C.8) ignores the contributions from fuse and wire and all the thermo-chemical corrections. In the examples below, the significance of the omitted contributions and corrections is illustrated.

EXAMPLE 1 Mixture of solvents with 2,6 % sulfur, 3,5 % chlorine, 1,2 % fluorine, < 0,1 % bromine.

The analysed waste is classified in relation to hydrogen content as:

5 % H; wood, plastic (PVC), cotton, paper, aromatic solvents, coal.

The gross calorific value was measured using a calorimeter with an effective heat capacity of 3 371 J/K. For the analysis 0,186 2 g of sample and 0,195 0 g of combustion aid (paraffin, gross calorific value 46 MJ/kg) were used. The temperature rise equalled 3,879 3 °C and in the bomb solutions, a total of 1,2 mg nitrate were found.

The following gross calorific value with no thermo-chemical corrections was obtained; 22,1 MJ/kg.

— Influence of thermo-chemical correction of sulfur, halogens and nitrogen

The total fuse correction (ignition wire and cotton fuse) was set to 209,2 J.

The content of sulfur, nitrogen (both from sample and atmospheric air), chlorine and fluorine results in the following correction; 0,36 MJ/kg. The total fuse correction (ignition wire and cotton fuse) is 1,12 MJ/kg. Hence with the thermo-chemical correction the gross calorific value is 19,01 MJ/kg.

— Calculation of the net calorific value

Regarding the net calorific value, the content of hydrogen (H) in the dry waste product produces a difference of 1,06 MJ/kg compared to the gross calorific value. By that the net calorific value equals 21,04 MJ/kg when using the gross calorific values with no thermo-chemical corrections and 17,95 MJ/kg when using the gross calorific values with the thermo-chemical corrections.

EXAMPLE 2 Mixture of solvents with 1,1 % sulfur, 0,28 % chlorine, < 0,1 % fluorine, < 0,1 % bromine.

The gross calorific value was measured using a calorimeter with an effective heat capacity of 3 342 J/K. For the analysis 0,219 20 g of sample and 0,264 6 g of combustion aid (paraffin) were used. The total fuse correction (ignition wire and cotton fuse) was set to 209,2 J. The temperature rise equalled 5,579 2 °C and in the bomb solutions, a total of 0,75 mg nitrate were found.

The following gross calorific value with no thermo-chemical corrections was obtained: 29,5 MJ/kg.

— Influence of thermo-chemical correction of sulfur, halogens and nitrogen

The content of sulfur, nitrogen (both from sample and atmospheric air), chlorine and fluorine results in the following correction: 0,10 MJ/kg. Hence with the thermo-chemical correction the gross calorific value is 28,08 MJ/kg.

— Calculation of the net calorific value

Regarding the net calorific value, the content of hydrogen (H) in the dry waste product produces a difference of 1,6 MJ/kg compared to the gross calorific value. By that the net calorific value equals 27,9 MJ/kg when using the gross calorific values with no thermo-chemical corrections and 27,2 MJ/kg when using the gross calorific values with the thermo-chemical corrections.

Annex D
(informative)

Typical hydrogen contents in waste products

Table D.1 — Hydrogen contents of waste products

Waste product	Typical hydrogen content % dry matter	Assigned hydrogen content %	
Coke	0	0	
Coal	5	5	
Wood	6,2		
Aromates	5,0 to 7,7		
Halogenated hydrocarbons	0 to 6		
Paper	6,2		
Cotton	6		
PVC	4,8		
Unknown solid waste	–		
Sludge	8		10
Gasoline	10		
Unknown liquid waste	–		
Alcohols	12,5 to 13,5	15	
Plastic (PE/PP etc.)	14,3		
Gasoil	14,8		

Assign hydrogen content according to best knowledge of the waste. Include a comment in report on the choice of assumed hydrogen content.

Annex E (informative)

Summary of general requirements and recommendations

Purpose of this summary (Table E.1) is to support the organisation of sampling and sample pretreatment processes. The information given should be helpful to prepare a sampling plan.

Requirements not mentioned in the normative part of this document are considered as recommendations.

Table E.1 — General requirements and recommendations

Matrix restrictions	Waste
Typical working range	above 1 MJ/kg
Sampling instruments	no restrictions
Bottle pretreatment	clean, dry and free of solvents
Bottle material	any appropriate sample container
Transport conditions	dark and cool
Preservation	cooling at about 4 °C
Storage conditions	at 4 °C in the dark not longer than 1 week
Required amount	about 50 g untreated sample
Test portion	about 1 g of the homogenised sample
Drying procedure	drying only applies if samples contain negligible amounts of volatiles at 40 °C except water
Sieving (particle size)	not applicable
Compatibility with	sample can be used for determination of hydrocarbons (GC and gravimetric), TOC, dry matter as well. Depending on the number of parameters the sample amount has to be increased.

Bibliography

- [1] EN 13965-1:2004, *Characterization of waste — Terminology — Part 1: Material related terms and definitions*
- [2] EN 14918, *Solid biofuels — Determination of calorific value*
- [3] EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*
- [4] EN 15170, *Characterization of sludges — Determination of calorific value*
- [5] EN 15400, *Solid recovered fuels — Determination of calorific value*
- [6] ISO 1928, *Solid mineral fuels — Determination of gross calorific value by the bomb calorimetric method and calculation of net calorific value*
- [7] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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