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## Solid recovered fuels — Determination of calorific value

*Combustibles solides de récupération — Détermination du pouvoir  
calorifique*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 300, *Solid recovered fuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 343, *Solid Recovered Fuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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](http://www.iso.org/members.html).

## Introduction

This document is based on EN 15400 [1].

The result obtained is the gross calorific value of the sample analysed at constant volume with all the water of the combustion products as liquid water. In practice, solid recovered fuels are burned at a constant (atmospheric) pressure and the water is either not condensed (removed as vapour with the flue gases) or condensed. Under both 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; Formulas are given for calculating both values.

General principles and procedures for the calibrations and the solid recovered fuels experiments are presented in the main part of this document, whereas those pertaining to the use of a particular type of calorimetric instrument are specified in [Annexes A](#) to [C](#). [Annex D](#) contains the formulae to calculate the removed ash contributors. [Annex E](#) contains checklists for performing calibration and fuel experiments using specified types of calorimeters. [Annex F](#) gives examples to illustrate some of the calculations.

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# Solid recovered fuels — Determination of calorific value

**WARNING** — Strict adherence to all of the provisions specified in this document should ensure against explosive rupture of the combustion vessel, or a blow-out, provided that the vessel is of standard design and construction and in good mechanical condition.

## 1 Scope

This document specifies a method for the determination of gross calorific value of solid recovered fuels at constant volume and at the reference temperature 25 °C in a combustion vessel calorimeter calibrated by combustion of certified benzoic acid.

## 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 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

ISO 16993, *Solid biofuels — Conversion of analytical results from one basis to another*

ISO 21637, *Solid recovered fuels — Vocabulary*

ISO 21644, *Solid recovered fuels — Methods for the determination of biomass content*

ISO 21646,<sup>1)</sup> *Solid recovered fuels — Sample preparation*

ISO 21660-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

EN 15358, *Solid recovered fuels — Quality management systems — Particular requirements for their application to the production of solid recovered fuels*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://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 recovered fuel burned in oxygen in a calorimetric combustion vessel 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 combustion vessel reaction, and of solid ash, all at the reference temperature (3.4).

1) Under preparation. Stage at the time of publication: ISO/DIS 21646:2021.

### 3.2

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

absolute value of the specific energy of combustion, in Joules, for unit mass of a solid recovered fuel 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* (3.4)

### 3.3

#### **net calorific value at constant pressure**

absolute value of the specific heat (enthalpy) of combustion, in Joules, for unit mass of a solid recovered fuel burned in oxygen at constant pressure under such conditions that all the water of the reaction products remains as water vapour (at 0,1 MPa), the other products being as for the gross calorific value, all at the *reference temperature* (3.4)

### 3.4

#### **reference temperature**

international reference temperature for thermo-chemistry of 25 °C is adopted as the reference temperature for calorific values (see 9.7)

Note 1 to entry: The temperature dependence of the calorific value of solid recovered fuels is small [less than 1 J/(g · K)].

### 3.5

#### **effective heat capacity of the calorimeter**

amount of energy required to cause unit change in temperature of the calorimeter

### 3.6

#### **corrected temperature rise**

change in calorimeter temperature caused solely by the processes taking place within the combustion vessel calorimeter

Note 1 to entry: It is the total observed temperature rise corrected for heat exchange, stirring power etc. (see 9.6).

Note 2 to entry: The change in temperature can be expressed in terms of other units: resistance of a platinum or thermistor thermometer, frequency of a quartz crystal resonator etc., provided that a functional relationship is established between this quantity and a change in temperature. The *effective heat capacity of the calorimeter* (3.5) can be expressed in units of energy per such an arbitrary unit. Criteria for the required linearity and closeness in conditions between calibrations and fuel experiments are given in 9.3.

Note 3 to entry: A list of the symbols used and their definitions is given in Annex G.

Note 4 to entry: Annex J explains additional relevant terms that could be of interest, more specifically in association to Annex D. The terms can provide some clarification in certain cases.

### 3.7

#### **removed ash contributors**

##### **rac**

coarse inert material (i.e. metals, glass, stones, tiles, etc.) removed from the sample before preparation, in order to avoid damage to the preparation equipment

Note 1 to entry: The removed ash contributors (rac), after sample pre-drying, are taken into account for the calculation of the ash, carbon, hydrogen, nitrogen and sulfur content in the analysed sample.

Note 2 to entry: See Annex D for further information.

## 4 Principle

### 4.1 Gross calorific value

A weighed portion of the analysis sample of a solid recovered fuel is burned in high-pressure oxygen in a combustion vessel calorimeter under specified conditions. The effective heat capacity of the



calorimeter is determined in calibration experiments by the 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 vessel initially to give a saturated vapour phase prior to combustion (see 9.2.1 and 10.2.2), 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 combustion vessel reaction and gaseous sulfur dioxide, i.e. the required reaction product of sulfur in the solid recovered fuel. The corresponding energy effect between aqueous and gaseous hydrochloric acid can be negligible for solid recovered fuels of mainly biomass origin.

The corresponding energy effect between aqueous and gaseous hydrochloric acid depends on the sample characteristics, e.g. the content of inorganic and organic chlorine, mineral composition and the actual pH-value in combustion vessel liquid. At the present time no values are available for this chlorine correction. Attention should be paid to the extremely high chlorine content in the test sample because e.g. PVC fractions can affect the calorific value significantly.

Automatic equipment may be used if the method is validated by parallel measurements. This automatic equipment shall fulfil all the requirements regarding sample size, heating procedure, temperature, atmosphere, and weighing accuracy. Deviations from this paragraph shall be reported and justified.

NOTE [Annex H](#) shows a flow chart for a routine determination of calorific value.

## 4.2 Net calorific value

The net calorific value at constant volume and the net calorific value at constant pressure of the solid recovered fuel are 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 [Annex H](#) shows a flow chart for a routine determination of calorific value.

## 5 Reagents

**5.1 Oxygen**, at a pressure high enough to fill the combustion vessel to 3 MPa, pure with an assay of at least 99,5 % volume fraction, and free from combustible matter.

NOTE Oxygen made by the electrolytic process can contain up to 4 % volume fraction 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 (see NOTE 1 of 9.2.1).

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

**5.4 Standard volumetric solutions and indicators**, only for use if analysis of final combustion vessel solutions is required.

**5.4.1 Barium hydroxide solution**,  $c[\text{Ba}(\text{OH})_2] = 0,05 \text{ mol/l}$ .

**5.4.2 Sodium carbonate solution**,  $c(\text{Na}_2\text{CO}_3) = 0,05 \text{ mol/l}$ .

**5.4.3 Sodium hydroxide solution**,  $c(\text{NaOH}) = 0,1 \text{ mol/l}$ .

**5.4.4 Hydrochloric acid solution**,  $c(\text{HCl}) = 0,1 \text{ mol/l}$ .

**5.4.5 Screened methyl orange indicator**, 1 g/l solution: dissolve 0,25 g of methyl orange and 0,15 g of xylene cyanole FF in 50 ml of ethanol with a volume fraction of 95 % and dilute to 250 ml with water.

**5.4.6 Phenolphthalein**, 10 g/l solution: dissolve 2,5 g of phenolphthalein in 250 ml ethanol with a volume fraction of 95 %.

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

NOTE 1 Benzoic acid is the sole substance recommended for calibration of an oxygen-combustion vessel 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.

NOTE 2 [Annex K](#) shows an environmental checklist (see [Table K.1](#)) that indicates possible environmental aspects according to the analysis of solid recovered fuels to take into account.

NOTE 3 The benzoic acid is burned in the form of pellets. It is usually used without drying or any treatment other than pelletizing; the sample certificate provides information. It does not absorb moisture from the atmosphere at relative humidities below 90 %.

The benzoic acid shall be used as close to certification conditions as is feasible; significant departures from these conditions shall be accounted for in accordance with the directions in the certificate. The energy of combustion of the benzoic acid, as defined by the certificate for the conditions utilised, shall be adopted in calculating the effective heat capacity of the calorimeter (see [10.2](#)).

## 6 Laboratory conditions

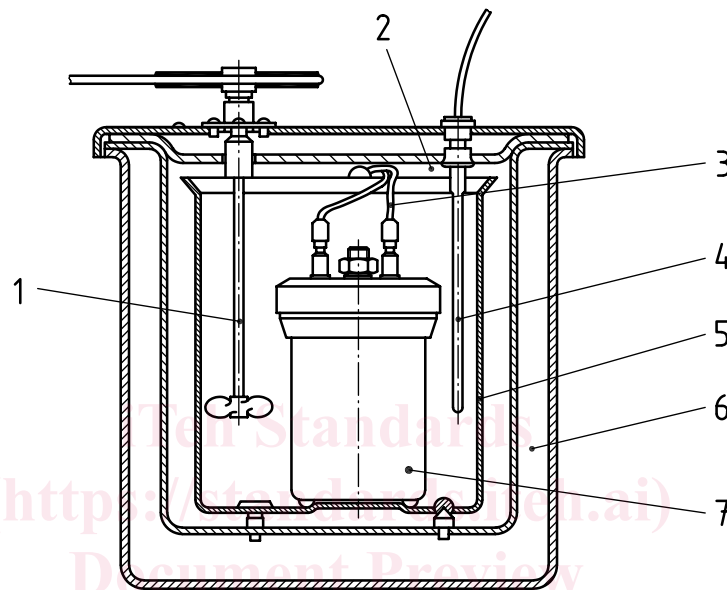
The laboratory for the determination of calorific value shall meet the following conditions.

- a) A laboratory for the determination of calorific value should not carry out other test items simultaneously in the same room.
- b) The room temperature should remain relatively stable, the change of room temperature should not exceed 1 °C per measurement, and the room temperature should be in the range of (15-30) °C.
- c) There should be no strong air convection in the room, so there should be no strong heat source, cold source and fan, etc.
- d) The experiment should avoid sunlight, otherwise the calorimeter should be placed in a place free from direct sunlight.

## 7 Apparatus

### 7.1 General

**7.1.1 Calorimeter** (see [Figure 1](#)), consists of the assembled combustion vessel ([7.1.2](#)), the calorimeter can ([7.1.3](#)) (with or without a lid), the calorimeter stirrer ([7.1.4](#)), 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 ([7.1.5](#)). 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.



#### Key

- |                                     |   |
|-------------------------------------|---|
| 1 stirrer ( <a href="#">7.1.4</a> ) | 5 calorimeter can ( <a href="#">7.1.3</a> ) |
| 2 thermostat lid                    | 6 thermostat ( <a href="#">7.1.5</a> )      |
| 3 ignition leads                    | 7 combustion vessel                         |
| 4 thermometer                       |   |

**Figure 1 — Classical-type combustion vessel calorimeter with thermostat**

In aneroid systems (systems without a fluid) the calorimeter can, stirrer and water are replaced by a metal block. The combustion vessel itself constitutes the calorimeter in some aneroid systems.

In combustion vessel 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 combustion vessel volume, oxygen pressure, vessel liquid, reference temperature of the measurements and repeatability of the results. A print-out of some specified parameters from the individual measurements is essential. Details are given in [Annex C](#).

As the room conditions (temperature fluctuation, ventilation etc.) can have an influence on the precision of the determination, the manufacturer's instructions for the placing of the instrument shall always be followed.

Equipment, adequate for determinations of calorific value in accordance with this document, is specified in [7.2](#) to [7.4](#).

**7.1.2 Combustion vessel**, 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 solid recovered fuels. A suitable internal volume of the combustion vessel would be 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. Manufacturers' instructions regarding the safe handling and use of the vessel shall be observed. Take into account any local regulations regarding the safe handling and use of the vessel. If more than one combustion vessel of the same design is used, it is imperative to use each vessel as a complete unit. Swapping of parts can lead to a serious accident.**

**7.1.3 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 combustion vessel while the water is being stirred. A lid generally helps reduce evaporation of calorimeter water, but unless it is in good thermal contact with the can it lags behind in temperature during combustion, giving rise to undefined heat exchange with the thermostat and a prolonged main period.

**7.1.4 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 (7.1.5) to minimise transmission of heat to or from the system; this is of particular importance if the stirrer shaft is in direct contact with the stirrer motor. If a lid is used for the calorimeter can (7.1.3), this section of the shaft should be above the lid.

**NOTE** The rate of stirring for a stirred-water type calorimeter is determined large enough to make sure that hot spots do not develop during the rapid part of the change in temperature of the calorimeter. A rate of stirring such that the length of the main period can be limited to 10 min or less is usually adequate (see Annexes A and B).

**7.1.5 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. A passive constant temperature ("static") thermostat shall have a heat capacity large enough to restrict the change in temperature of its water. Criteria for satisfactory behaviour of this type of water jacket are given in Annex B.

**NOTE 1** For an insulated metal static jacket, satisfactory properties are usually ensured by making a wide annular jacket with a capacity for water of at least 12,5 l.

**NOTE 2** Calorimeters surrounded by insulating material, creating a thermal barrier, are regarded as static-jacket calorimeters.

If the thermostat (water jacket) is required to follow closely the temperature of the calorimeter, it should be of low mass and preferably have immersion heaters. Energy shall be supplied at a rate sufficient to maintain the temperature of the water in the thermostat to within 0,1 K of that of the calorimeter water after the charge has been fired. If in a steady state at 25 °C, the calculated mean drift in temperature of the calorimeter shall not exceed 0,000 5 K/min (see A.3.2).

**7.1.6 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. Because of environmental aspects mercury (thermometers) should be the last

option due to disposal concerns according the Minamata treaty. 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 which conform to ISO 651<sup>[2]</sup>, ISO 652<sup>[3]</sup>, ISO 1770<sup>[4]</sup> or ISO 1771<sup>[5]</sup> satisfy the requirements, but only to be used if no other options are available. A viewer with magnification about 5× 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 (see 9.4). If this is not available, the thermometer shall be tapped manually before reading the temperature.

## 7.2 Auxiliary equipment

**7.2.1 Crucible**, of silica, nickel-chromium, platinum or similar unreactive 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.

If smears of unburned carbon occur, a small low-mass platinum or nickel-chromium crucible, for example 0,25 mm thick, 15 mm in diameter and 7 mm deep, may be used.

## 7.2.2 Ancillary pressure equipment

**7.2.2.1 Pressure regulator**, to control the filling of the combustion vessel with oxygen.

**7.2.2.2 Pressure gauge** (e.g. 0 MPa to 5 MPa), to indicate the pressure in the combustion vessel with a resolution of 0,05 MPa.

**7.2.2.3 Relief valve or bursting disk**, operating at 3,5 MPa, and installed in the filling line, to prevent overfilling the combustion vessel.

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

## 7.2.3 Ignition circuit

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

Where the firing is done manually, the firing switch shall be of the spring-loaded, usually open type, located in such a manner that any undue risk to the operator is avoided (see warning in 9.4).

## 7.3 Balances

**7.3.1 Balance for weighing the sample, fuse**, with a resolution of at least 0,1 mg; 0,01 mg is preferable and is recommended if the sample mass is of the order of 0,5 g or less (see 9.2.1).

**7.3.2 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, see 9.3).

**7.4 Thermostat** (optional), for equilibrating the calorimeter water before each experiment to a predetermined initial temperature, within about  $\pm 0,3$  K.



## 8 Preparation of test sample

The solid recovered fuel sample used for the determination of calorific value shall be the general analysis sample (ground to pass a test sieve with an aperture of 1,0 mm) prepared according to the procedure given in ISO 21646.

The preparation of test sample for determining calorific value of biomass/non-biomass part of SRF shall be carried out in accordance with ISO 21644.

Due to the low density of solid recovered fuels they shall be tested in a pellet form. Press a pellet with a mass of  $(1 \pm 0,1)$  g with a suitable force to produce a compact test piece. Alternatively, the test may be carried out in powder form, closed in a combustion bag or capsule.

NOTE 1 For sample materials containing high content of plastics or rubber, the mass of the sample is reduced to a mass in the range from 0,4 g to 0,8 g.

NOTE 2 For sample materials containing a mass fraction of ash  $\geq 30$  % on dry basis, a combustion aid is used (see 9.2.2).

For sample materials, the mass of the sample may be reduced depending on the calorimeter specification.

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.

The moisture content of the analysis sample shall be determined in accordance with ISO 21660-3.

## 9 Calorimetric procedure

### 9.1 General

The calorimetric determination consists of two separate experiments, combustion of the calibration reference (benzoic acid) and combustion of the solid recovered fuels, both under same specified conditions. The calorimetric procedure for the two types of experiment is essentially the same. In fact, the overall similarity is a requirement for proper cancellation of systematic deviations caused, for example, by uncontrolled heat leaks not accounted for in the evaluation of the corrected temperature rise  $\theta$ .

The experiment consists of quantitatively carrying out a combustion reaction (in high-pressure oxygen in the combustion vessel) to defined products of combustion and of measuring the change in temperature caused by the total combustion vessel process.

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 as outlined in [Figure 2](#). 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 (see [Annex A](#)). 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 if combustion takes place. The fore and after periods then have to be longer (see [Annex B](#)).

The power of stirring shall be maintained constant throughout an experiment which calls 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.