PD CEN/TR 15404:2010

BSI Standards Publication

Solid recovered fuels — Methods for the determination of ash melting behaviour by using characteristic temperatures

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National foreword

This Published Document is the UK implementation of CEN/TR 15404:2010. It supersedes DD CEN/TS 15404:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

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

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

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Date	Text affected

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English Version

Solid recovered fuels - Methods for the determination of ash melting behaviour by using characteristic temperatures

Combustibles solides de récupération - Méthode de détermination de la fusibilité des cendres

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Schmelzverhaltens der Asche bei Anwendung charakteristischer Temperaturen

This Technical Report was approved by CEN on 12 June 2010. It has been drawn up by the Technical Committee CEN/TC 343.

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Foreword

This document (CEN/TR 15404:2010) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

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

This document supersedes CEN/TS 15404:2006.

CEN/TS 15404:2006 was not to be kept as a Technical Specification and respectively not converted into a European Standard due to lack of acceptable precision data (see [1], [2]).

This document differs from CEN/TS 15404:2006 as follows:

a) symbol of shrinking temperature changed from ST to SST;

b) charts and key of Figure 1 corrected;

c) clause "Precision" deleted;

d) clause on evaluation of results added;

e) interlaboratory test results supplemented as graphs in an informative annex;

f) whole document editorially revised.

Introduction

Ash melting is a complex process where also shrinkage, sintering and swelling can occur.

The test methods described in this Technical Report provide information about fusion and melting behaviour of the composite inorganic constituents of the fuel ash at high temperatures.

The test methods available are empirical in most cases. The ashes used for the tests are homogeneous material, prepared from the fuel, and the determination is performed at a controlled rate of heating in a controlled atmosphere. In contrast, under full-scale conditions, the complex processes of combustion and fusion involve heterogeneous mixtures of particles, variable heating rates and gas compositions.

The methods described in this document should be used dependent of the following aspects and parameters, respectively:

— repeatability;

— reproducibility;

— reliability;

— time efforts (rapid test methods);

— cost effectiveness;

— possibilities for automatic testing.

The aim of this document consists in providing a common and successful practice for describing the ash melting behaviour.

The terms ash fusibility and ash softening are synonyms to ash melting.

1 Scope

This Technical Report describes exemplarily methods for the determination of shrinking, deformation, hemisphere and flow temperature for characterising the ash melting behaviour of all solid recovered fuels.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

prEN 15357:2008, *Solid recovered fuels — Terminology, definitions and descriptions*

prEN 15403, *Solid recovered fuels — Determination of ash content*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN 15357:2008 and the following apply.

3.1
shrinking temperature
SST
temperature at which shrinking of the test piece occurs, i.e. when the area of the test piece falls below 95 % of the original test piece area at 550 °C

NOTE Shrinking can be due to liberation of carbon dioxide, volatile alkali compounds, and/or sintering and partial melting.

3.2
deformation temperature
DT
temperature at which the first signs of roundings of the edges due to melting of the test piece occur

3.3
hemisphere temperature
HT
temperature at which the test piece forms approximately a hemisphere, i.e. when the height becomes equal to half the base diameter

3.4
flow temperature
FT
temperature at which the ash is spread out over the supporting tile in a layer, the height of which as half of the height of the test piece at the hemisphere temperature

NOTE Half of the height of the test piece is defined due to frequently occurring bubbling effects. This is especially important for automatic image evaluation. This definition is different to other standards.

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4 Principle

A test piece made from the prepared ash is heated up with constant rate whereas the deformation is continuously observed. The temperatures at which characteristic changes of the shape occur are recorded.

5 Reagents

5.1 Water, demineralised.

5.2 Dextrin, 100g/l solution: 10 g of dextrin are dissolved in 100 ml water.

5.3 Ethanol, with a purity of greater than 95 %.

5.4 Carbon dioxide

5.5 Gas mixture, of carbon dioxide (5.4) and carbon monoxide: A volume fraction of 55 % to 65 % carbon monoxide is mixed with a volume fraction of 35 % to 45 % carbon dioxide (5.4).

5.6 Gold wire, with a diameter of 0,5 mm or greater, or a **gold plate**, with a thickness of 0,5 mm to 1,0 mm, a purity of at least 99,99 % and a certified melting point (e.g. 1 064 °C).

5.7 Nickel wire, with a diameter of 0,5 mm or greater, or a **nickel plate,** with a thickness of 0,5 mm to 1,0 mm, a purity of at least 99,9 % and a certified melting point (e.g. 1 455 °C).

NOTE Nickel is used for reducing atmosphere.

5.8 Palladium wire, with a diameter of 0,5 mm or greater, or a **palladium plate**, with a thickness of 0,5 mm to 1,0 mm, a purity of at least 99,9 % and a certified melting point (e.g. 1 554 °C).

6 Apparatus and auxiliary means

6.1 Furnace, electrically heated, capable to:

a) reach the maximum temperature (≥ 1 500 °C) at which the properties of the ash shall be determined;

b) provide and maintain an adequate zone of uniform temperature which to heat the test piece(s) in;

c) provide means for heating the test piece(s) at an uniform rate from 550 °C upwards;

d) maintain the required test atmosphere around the test piece(s);

e) provide means for observing the change of shape of the test piece(s) during heating.

6.2 Dish, consisting of inert material, such as porcelain, silica, platinum, with a depth from 10 mm to 20 mm and of such a size that the sample loading does not exceed 0,1 g/cm^2 bottom area.

6.3 Pyrometer, consisting of a platinum/platinum-rhodium thermocouple.

6.4 Mould, of brass, stainless steel or other suitable material for preparing the test piece.

6.5 Spring pressure hand press for producing the test piece, capable of providing a spring pressure of about 1,5 N/mm^2.

6.6 Support for the test piece, consisting of such an inert material that it neither is distorted nor absorbs the ash during the determination.

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NOTE Supports of sintered alumina or fine-textured mullite are generally satisfactory but difficulties can arise with individual ashes, in which case a non-absorbent interface such as platinum foil can be used between the original support and the test piece.

6.7 Flowmeters, two, for measuring the components of the reducing gases.

NOTE If using oxidising gas, it is not necessary to measure the flow rate.

6.8 Grinding device, such as agate mortar and pestle.

6.9 Test sieve, of aperture 0,075 mm and diameter of at least 100 mm complete with lid and receiver, in accordance with ISO 3310-1.

6.10 Optical instrument, such as a camera or video equipment, for observing the profile of the test piece throughout the determination.

7 Test conditions

7.1 Test atmosphere

Oxidising or reducing atmosphere is used depending on the application. Air or carbon dioxide is applied for an oxidising atmosphere. For a reduced atmosphere, the following mixtures shall be passing the test piece at a minimum linear rate of flow from 100 mm/min to 250 mm/min calculated at ambient temperature:

— 55 % volume fraction to 65 % volume fraction carbon monoxide with 35 % volume fraction to 45 % volume fraction carbon dioxide, and

— 45 % volume fraction to 55 % volume fraction hydrogen with 45 % volume fraction to 55 % volume fraction carbon dioxide.

NOTE The flow rate is not very critical, provided that it is sufficient to prevent any leakage of air into the furnace in case of reducing atmosphere. However, the same flow rate level is also recommended for oxidising atmosphere. For open-type furnaces with a larger diameter, a flow rate of about 400 mm/min could be needed for reducing atmosphere. In all cases it should also be referred to manufacturer instructions. The flow rate for rotameter adjustment can be calculated by multiplying the flow rate, expressed in millimetres per minute, with the inside cross-section area of the furnace tube converting into litres per minute.

WARNING — When using reduced atmosphere as given above, the gases emerging from the furnace will contain a proportion of carbon monoxide; therefore it is essential to ensure that these gases are vented to the outside atmosphere, preferably by means of a hood or an efficient fan system. If hydrogen is used in the reducing atmosphere, care shall be taken to prevent an explosion occurring by purging with carbon dioxide both prior to the introduction of the hydrogen and after the hydrogen supply is shut off.

7.2 Shape of test piece

The test piece shall have sharp edges to facilitate observation.

The mass of the test piece shall be such as to ensure equalisation of the temperature within the test piece. Hence, dimensions that are too large shall be avoided. Several test piece shapes are used, e.g. cylinders, pyramids, cubes, truncated pyramids, with dimensions (diameter or height) in the vicinity of 3 mm to 5 mm. Figure 1 illustrates a cylindrical shape test piece at the various characteristic temperatures.

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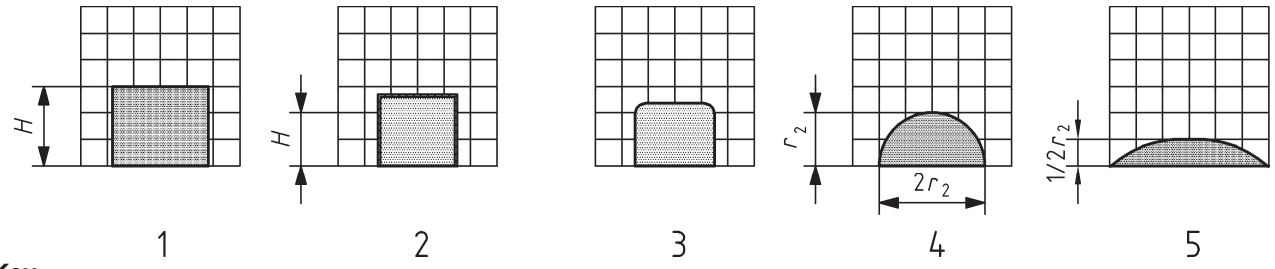

Key

1 original sample
2 sample at shrinkage temperature
3 sample at deformation temperature
4 sample at hemisphere temperature
5 sample at flow temperature

Figure 1 — Phases in the ash melting process (original shape = shape and size at 550 °C)

8 Methodology

The ash shall be prepared in accordance with prEN 15403 (see also prEN 15442 and prEN 15443). A complete incineration of the sample is of paramount importance. The ash is grinded until the maximum particle size is less than 0,075 mm. None of the ash may be withdrawn during grinding. A sufficient quantity of the prepared ash with a particle size less than 0,075 mm is moistened with water (5.1), dextrin (5.2) or ethanol (5.3), made into a paste and pressed into a mould. Then allow the test piece to dry.

9 Procedure

The test piece is transferred on the support (6.6) into the furnace (6.1) and the composition and flow rate of the atmosphere adjusted. The temperature is raised at a uniform rate within the range of 3 K/min to 10 K/min. A picture is taken at least every 10 K. Then the temperature is raised to a point below the expected deformation temperature such that the temperature interval between the point and the expected deformation temperature exceeds 150 °C.

If possible, shrinking, deformation, hemisphere and flow temperatures are determined.

NOTE 1 There are several standard methods, e.g. DIN 51730, to determine the ash melting behaviour of solid fuels and there are minor differences in the methodology and the interpretation of characteristic temperatures that should be noted, especially the shape of the test piece and the definition of the characteristic temperatures.

NOTE 2 Using a computerised image evaluation, the deformation temperature is reached if the shape factor F has changed by 1,5 %. In order to determine the shape factor, the circumference of a perfect semicircle with the same area as the shadow of the test piece is calculated. This idle circumference b is then put in relation to the actual measured circumference a of the test piece. This relation gives the shape factor $F = b/a$. A schematic of the images obtained by the computerized evaluation is shown in Figure 2.

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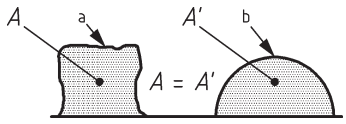

Key

A shadow area of the test piece (actual image)
A′ area of the test piece after computerized image evaluation (equivalent to the shadow area)
a actual measured circumference
b idle circumference

Figure 2 — Schematic of the images obtained by the computerised evaluation

In the case of some ashes, difficulties can be encountered owing to such effects as blistering, distortion, shrinking, swelling, non-wetting of the support caused by high surface tension and bursting of internal gas bubbles. In such cases, these phenomena should be recorded and, if necessary, the experiment be repeated using a different type of support.

10 Evaluation of available results

The characteristic temperatures, i.e. shrinking, deformation, hemisphere and flow temperatures, are expressed in degrees Celsius and rounded to the nearest 5 °C or 10 °C if determined. For selected solid recovered fuels, examples of ash fusibility temperatures determined by using graphic and photographic methods are shown in Figure 3 to Figure 8.

A real-time graphical representation of the test piece during the ash fusion test is shown in Figure 3. The curves are based on a combination of the photographic method used in this document and a graphical one specified in a French Standard (NF M03-048, see Bibliography) to generate a visual impression of the fusion process. In this case, changes of specimen height with respect to their initial height at 550 °C were obtained photographically and plotted against the corresponding temperatures. Distinct positions on these curves represent the shrinking, the deformation, the hemisphere, and the flow temperatures of the ashes.

The ash fusibility temperatures derived from the curves in Figure 3 as well as the fusibility temperature of a reference hard coal are faced in Figure 4.

Results of an interlaboratory test undertaken by three laboratories to determine the ash melting behaviour of selected solid recovered fuels in accordance with this Technical Report are shown by diagrams in Annex A.

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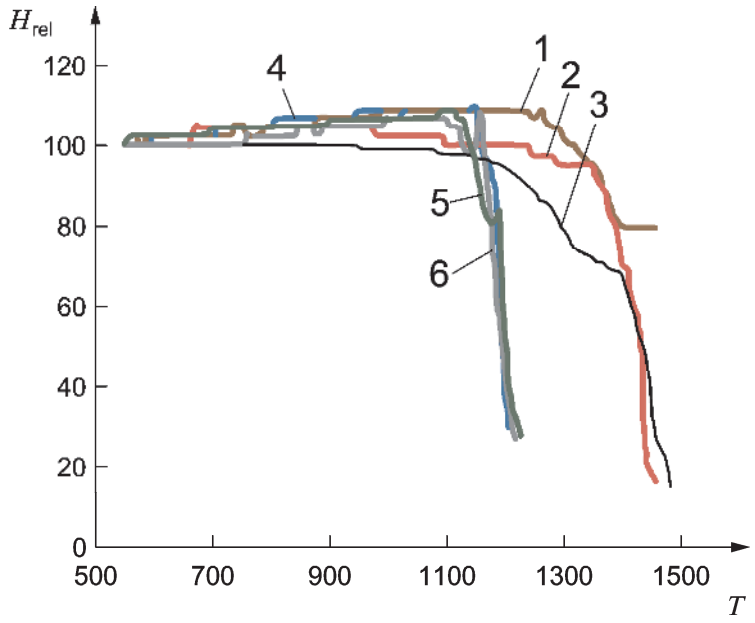
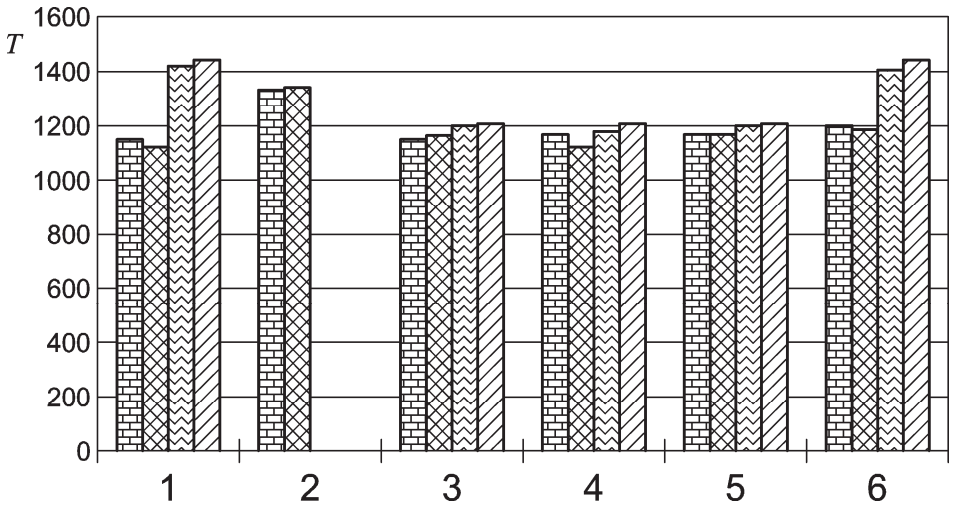


Key

H_{rel} relative height of the sample related to the height of the original sample in %
T temperature in °C

1 demolition wood
2 tyre
3 hard coal
4 municipal solid waste
5 dried sludge
6 paper and plastic fluff

Figure 3 — Graphically obtained ash fusibility curves for different types of solid recovered fuels under oxidising atmosphere



Key

T temperature in °C

shrinking temperature, ST
deformation temperature, DT
hemisphere temperature, HT
flow temperature, FT

1 shredded tyre
2 demolition wood
3 dried sludge
4 paper and plastic fluff
5 municipal solid waste
6 hard coal

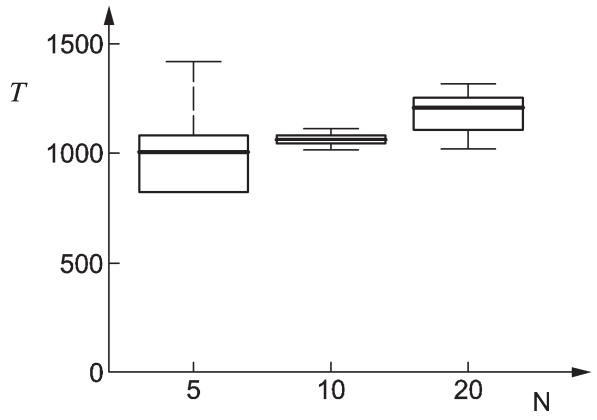
Figure 4 — Characteristic temperatures for solid recovered fuels

Annex A
(informative)

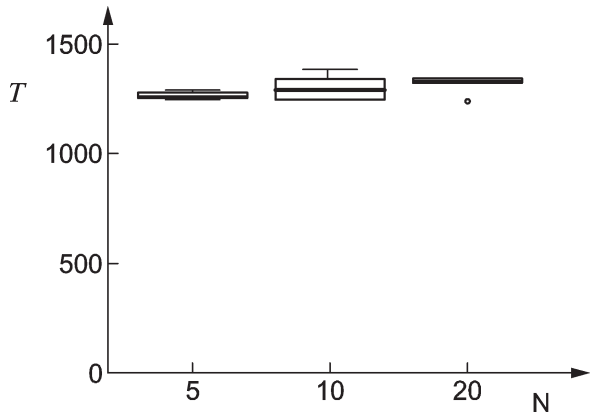
Interlaboratory test results

A statistic evaluation of interlaboratory test results was not performed due to minor number of individual values and a too great dispersion of them.

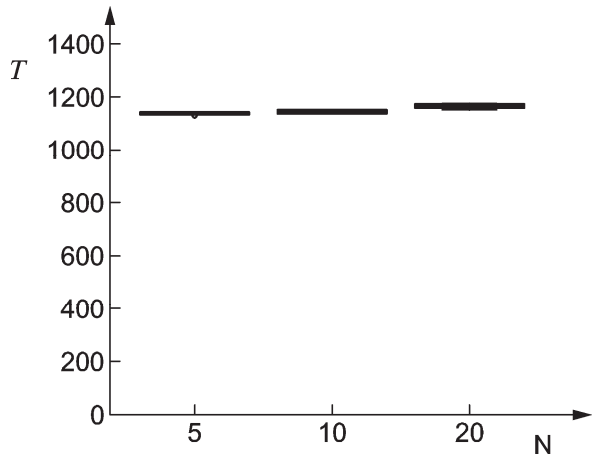
The deviations of the test results between the individual laboratories for each sample type and characteristic temperature are shown in Figures A.1 to A.4.



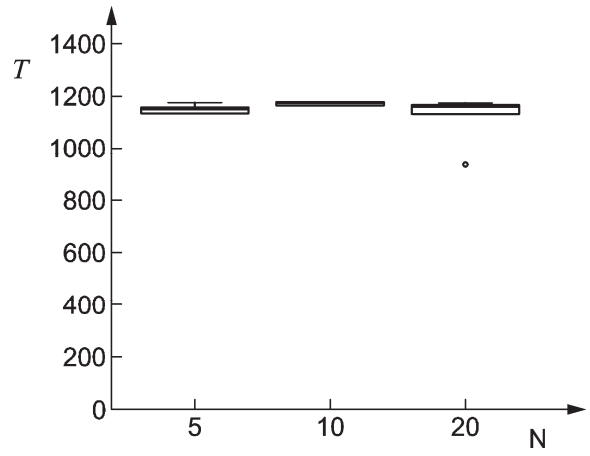
a) Shredded tyre



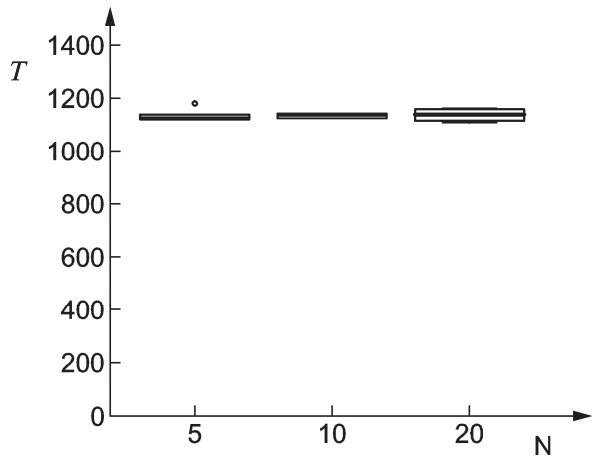
b) Demolition wood



c) Dried sludge



d) High calorific fraction of municipal solid waste

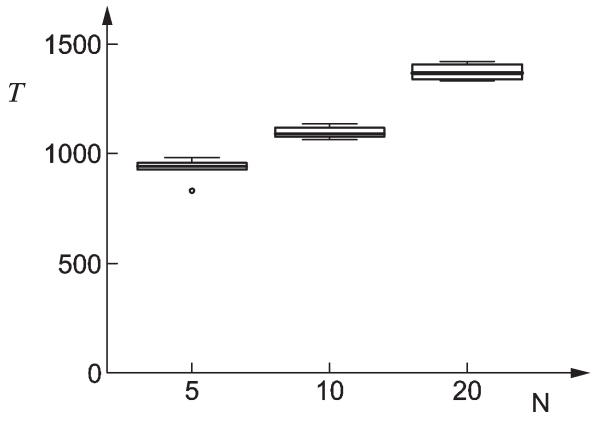


e) Paper and plastic fluff

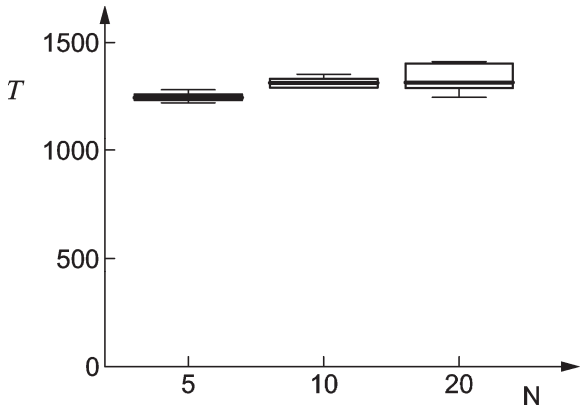
Key

T temperature in °C
N number of the individual laboratory

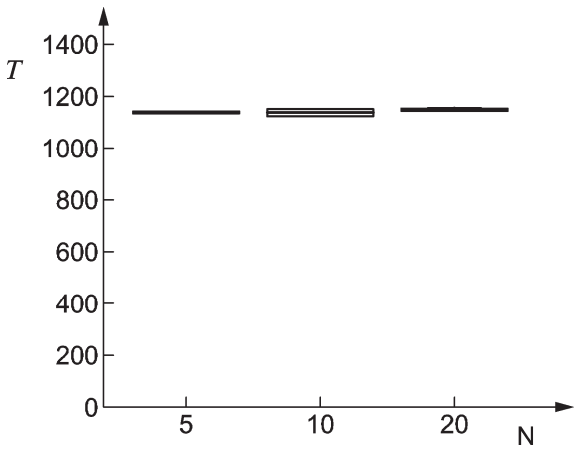
Figure A.1 — Deviations of shrinking temperature between the individual laboratories



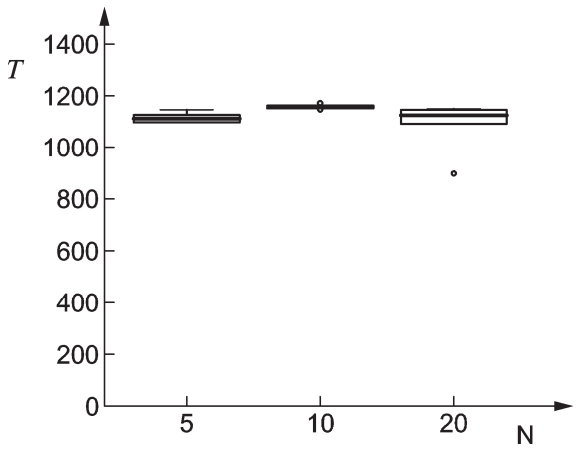
a) Shredded tyre



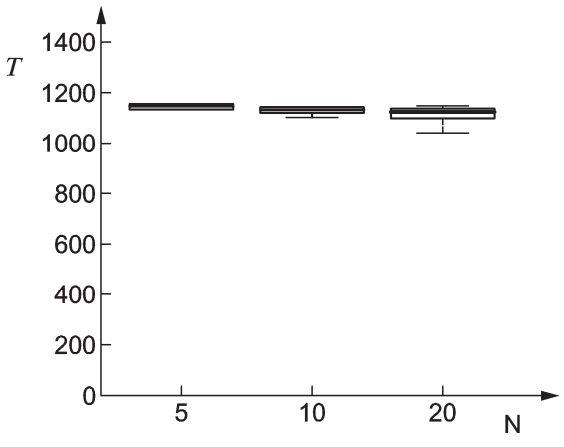
b) Demolition wood



c) Dried sludge



d) High calorific fraction of municipal solid waste

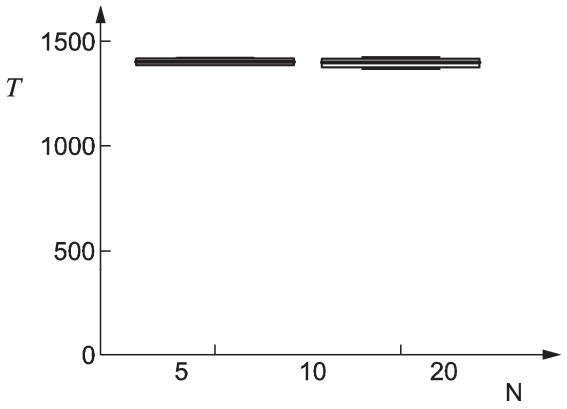


e) Paper and plastic fluff

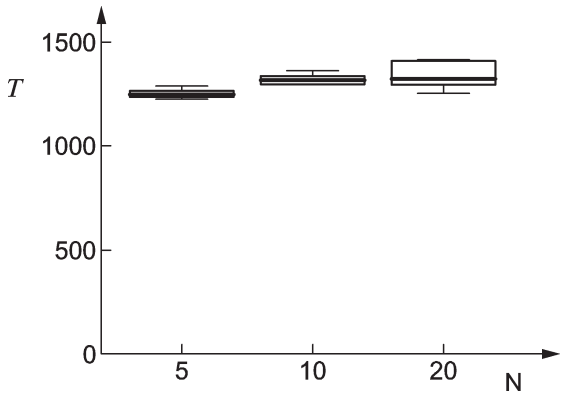
Key

T temperature in °C
N number of the individual laboratory

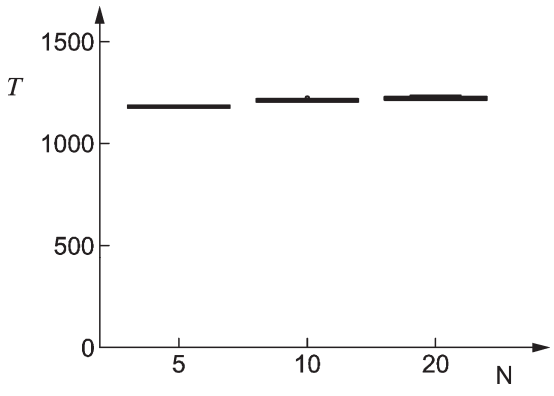
Figure A.2 — Deviations of deformation temperature between the individual laboratories



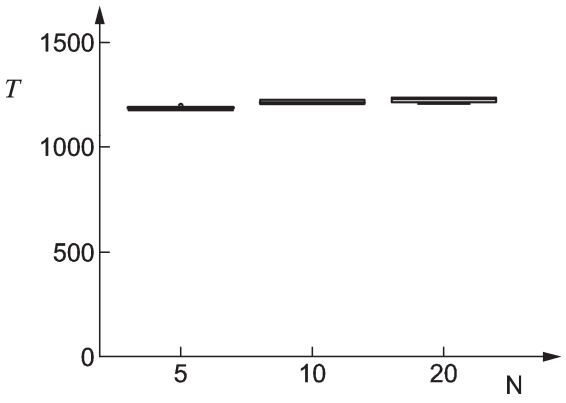
a) Shredded tyre



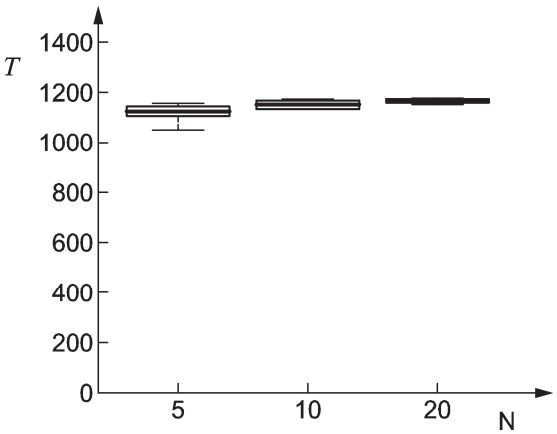
b) Demolition wood



c) Dried sludge



d) High calorific fraction of municipal solid waste

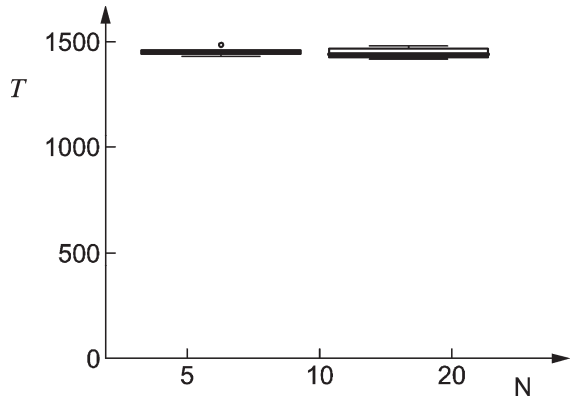


e) Paper and plastic fluff

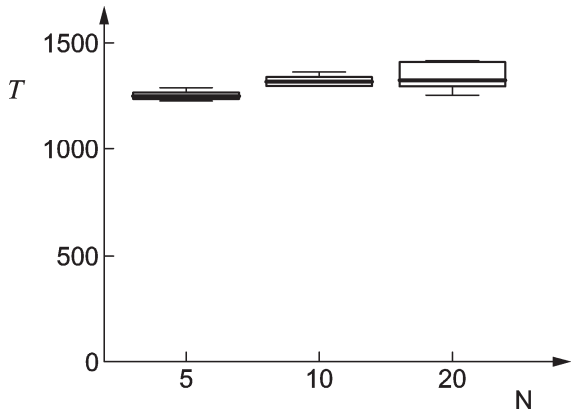
Key

T temperature in °C
N number of the individual laboratory

Figure A.3 — Deviations of hemisphere temperature between the individual laboratories



a) Shredded tyre



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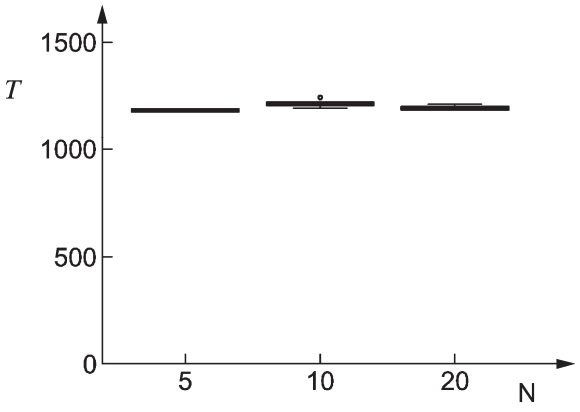

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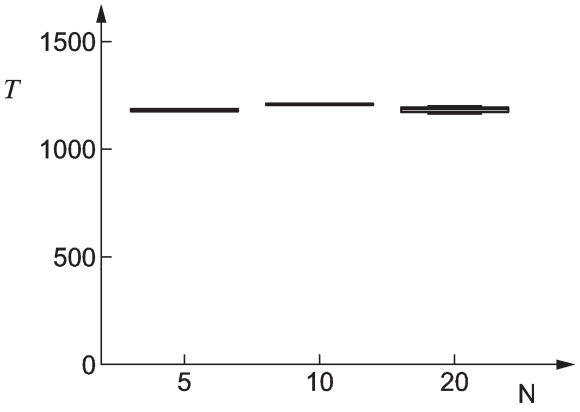

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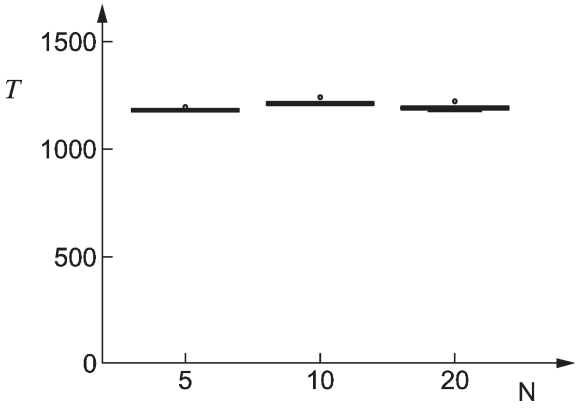

e) Paper and plastic fluff

Key

T temperature in °C
N number of the individual laboratory

Figure A.4 — Deviations of flow temperature between the individual laboratories

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[7] NF M03-048, *Solid mineral fuels — Determination of ash fusibility*

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