
Solid recovered fuels — Determination of content of volatile matter

*Combustibles solides de récupération — Détermination de la teneur
en composés volatils*

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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).

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).

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.

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.

Introduction

The volatile matter is determined as the loss in mass of the analysed sample, after moisture removal, when material is heated up under specific conditions of temperature, time and in a reduced atmosphere (anoxic conditions). The test is empirical and, in order to ensure reproducible results, it is essential that the rate of heating, the final temperature and the overall duration of the test are carefully controlled. It is also essential to exclude air from the solid recovered fuel during heating to prevent oxidation.

The moisture content of the sample is determined at the same time as the volatile matter so that the appropriate correction can be made. Mineral matter associated with the sample can also lose mass under the conditions of the test, the magnitude of the loss being dependent on both the nature and the quantity of the minerals present.

This document is primarily geared toward laboratories, producers, suppliers and purchasers of solid recovered fuels, but is also useful for the authorities and inspection organizations.

The method specified in this document is based on EN 15402^[3] as well as ISO 562.

For information about environmental aspect, see [Annex B](#).

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Solid recovered fuels — Determination of content of volatile matter

1 Scope

This document specifies the requirements and a method for the determination of volatile matter of solid recovered fuels.

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 21637, *Solid recovered fuels — Vocabulary*

ISO 21645, *Solid recovered fuels — Methods for sampling*

ISO 21646¹⁾, *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*

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

ash

ash content on dry basis

total ash

A

mass of inorganic residue remaining after combustion of a fuel under specified conditions, typically expressed as a percentage of the mass of dry matter in fuel

Note 1 to entry: Depending on the combustion efficiency the ash may contain combustibles.

Note 2 to entry: If a complete combustion is realized, ash contains only inorganic, non-combustible components.

[SOURCE: ISO 16559:2014, 4.13, modified — “Note 1 to entry” was removed and the following ones renumbered, and symbol “A” was italicized.]

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

3.2

removed ash contributor rac

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

Note 1 to entry: Removed ash contributors are included in the ash content calculations.

[SOURCE: ISO 21637:2020, 3.62, modified – Note 1 to entry was added.]

3.3

total organic matter

combustible part of solid recovered fuels, which consists of the sum of volatile matter and fixed carbon

Note 1 to entry: It is calculated as: $100 - \text{moisture content} - \text{ash content}$.

Note 2 to entry: It is the mass fraction of the matter lost by ignition, also known as “Loss Of Ignition” (LOI).

3.4

volatile matter

relative part of the analysed sample, after moisture removal, that is lost when material is heated up under specific conditions of temperature, time and in a reduced atmosphere (anoxic conditions)

3.5

fixed carbon

relative part of carbon contained in a material that can only be degraded in oxic conditions and high temperature

Note 1 to entry: It is calculated as: $100 - \text{moisture content} - \text{volatile matter content} - \text{ash content}$.

3.6

pre-drying

drying process to minimise moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimise biological activity

4 Principle

A test portion of the general analysis sample is heated out of contact with ambient air at $(900 \pm 10) ^\circ\text{C}$ for 7 min. The percentage of volatile matter is calculated from the loss in mass of the test portion after deducting the loss in mass due to moisture. The test is empirical and, in order to ensure reproducible results, it is essential that the rate of heating, the final temperature and the overall duration of the test are carefully controlled.

Automatic equipment (such as thermogravimetric analysers) may be used as long as the equipment is validated by parallel measurements to the reference method. The 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.

5 Apparatus

5.1 Furnace

The furnace shall be heated electrically and capable of maintaining a temperature of $(900 \pm 10) ^\circ\text{C}$. It may be of the stop-ended type or fitted at the back with a flue with a diameter of about 25 mm and a length of about 150 mm (see [Figure 1](#)).

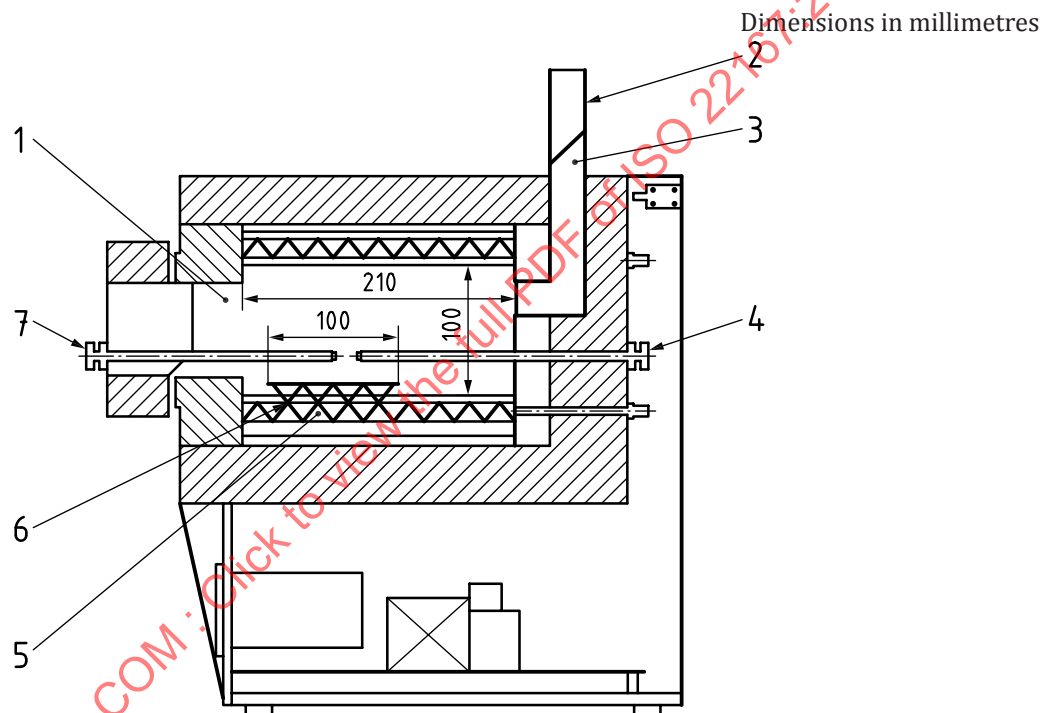
It is important for furnaces with flues that the furnace door seals well. The flue should not reach far out of the furnace and should be fitted with a butterfly valve to restrict airflow through the furnace.

The heat capacity of the furnace shall be such that, with an initial temperature of $(900 \pm 10) ^\circ\text{C}$, the temperature is regained within about 4 min after insertion of a cold stand and its crucibles. The temperature shall be measured with a thermocouple, as specified in 5.2.

NOTE Observing the temperature is very important in order to compensate for inherent deviations of the temperature measurement and lack of uniformity regarding the temperature distribution.

Usually the furnace will be designed specifically either for multiple determinations using a number of crucibles in one stand or for receiving one crucible and its stand. In the first case, the zone of uniform temperature shall be at least $160 \text{ mm} \times 100 \text{ mm}$; in the latter case, a zone of diameter about 40 mm is sufficient.

A position for the crucible stand shall be chosen within the zone of uniform temperature and this position shall be used for all determinations.



Key

- | | |
|-------------------------|-------------------------------|
| 1 chamber, 200 mm width | 5 heating system |
| 2 flue | 6 zone of uniform temperature |
| 3 valve | 7 check thermocouple |
| 4 thermocouple | |

Note No design tolerances given, because it is an example of design. Standard practices can be followed.

Figure 1 — Example of suitable furnace

5.2 Thermocouple

The thermocouple shall be an unsheathed wire with a thickness $\leq 1 \text{ mm}$. It shall be long enough to reach the centre of the underside of each crucible when placed into the zone of uniform temperature on being inserted through the front or rear of the furnace. The thermo junction shall be placed midway between the base of the crucible in its stand and the floor of the furnace. If the stand holds more than one crucible, the temperature under each crucible shall be checked in the same manner.

If desired, a sheathed thermocouple may be permanently installed in the furnace (5.1) (see Figure 1) with its thermo junction as close as possible to the centre of the zone of uniform temperature; in

this case furnace temperature readings shall be correlated at frequent intervals with those of the unsheathed thermocouple which is thus inserted only if necessary.

NOTE The temperature/electromotive force relationship of a thermo junction maintained at elevated temperatures gradually changes with time.

5.3 Crucible

The crucible shall be cylindrical, with a well-fitting lid, both of fused silica, porcelain or other suitable material. The crucible with lid shall have a mass from 10 g to 14 g and dimensions approximating to those shown in Figure 2. The lid shall be selected to match the crucible such that the horizontal clearance between them is not more than 0,5 mm. After selection it is good practice to label the lid and crucibles so it is easy to identify the matching lid and crucible.

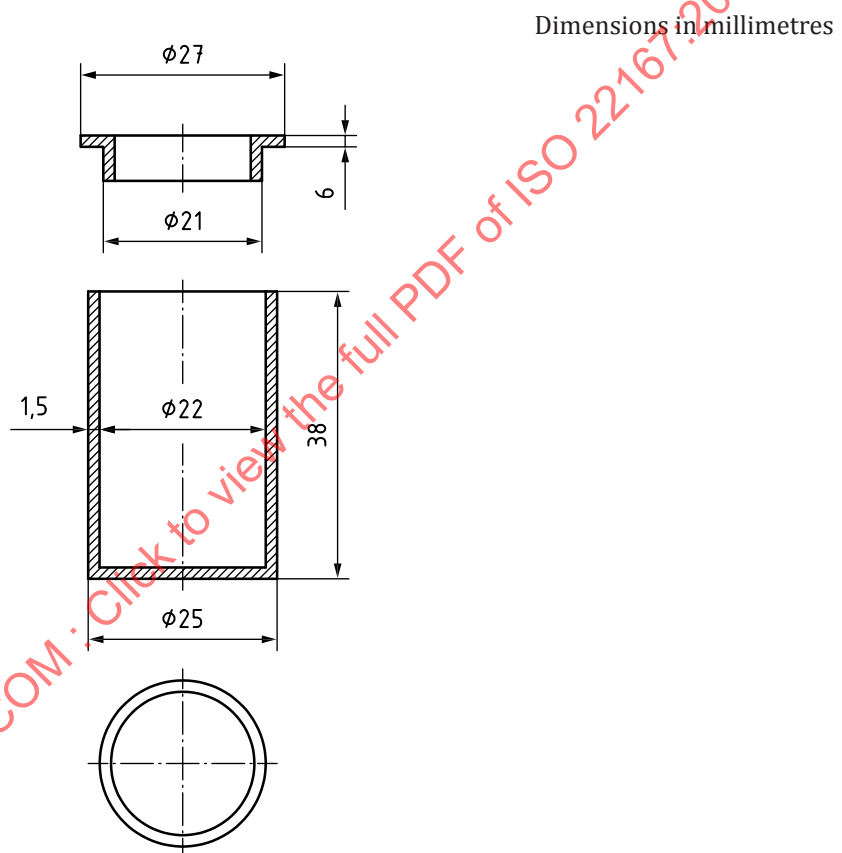


Figure 2 — Crucible and lid

If performing multiple determinations of expanding solid recovered fuels, it can be necessary to use taller crucibles; these may be up to 45 mm in height without affecting the determined volatile matter, provided that the specified rate of temperature recovery is maintained.

5.4 Crucible stand

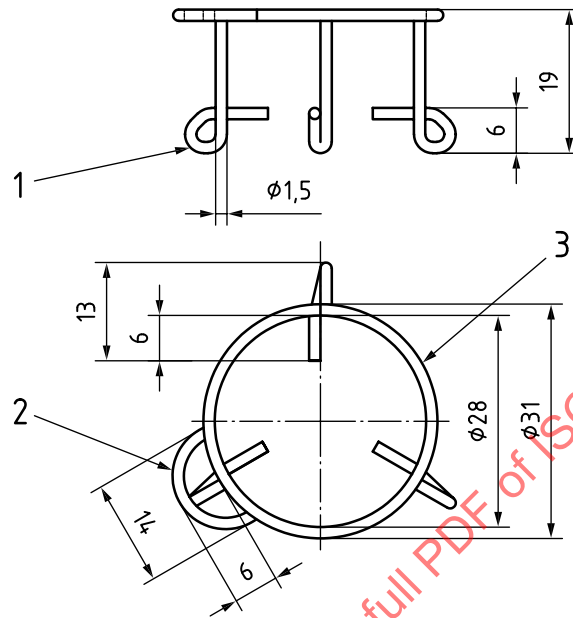
The crucible stand which the crucible (5.3) is placed on in the furnace (5.1), shall be such that the appropriate rate of heating is achievable. For example, it may consist of the following:

- a) for single determinations, a ring of heat-resistant steel wire as shown in Figure 3 a), with ceramic discs with a diameter of about 25 mm and a thickness of about 2 mm, resting on the inner projection of its legs

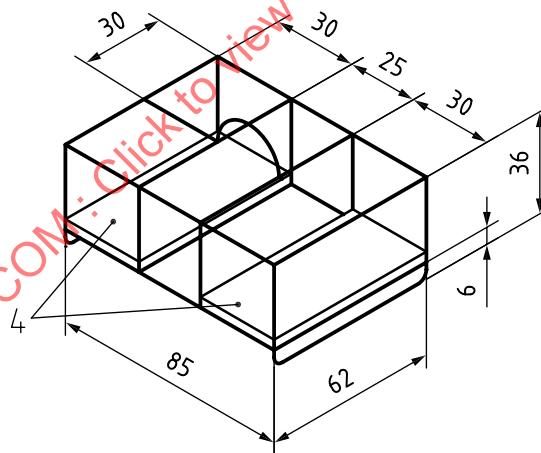
or

- b) for multiple determinations, a tray of heat-resistant steel wire as shown in [Figure 3 b\)](#), of appropriate size, with ceramic plates with a thickness of about 2 mm supporting the crucibles.

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determinations

Key

- 1 three legs spaced 120° apart
- 2 handle
- 3 ring
- 4 ceramic plates

Figure 3 — Crucible stand

5.5 Balance

The balance shall be readable to the nearest 0,1 mg.

6 Sampling and sample preparation

The general analysis sample shall be taken and prepared in accordance with ISO 21645 and ISO 21646. It shall be well mixed and in moisture equilibrium with the laboratory atmosphere.

A test portion of the general analysis sample shall be separated for the determination of moisture content at the same time to the determination of volatile matter. The moisture content shall be determined in accordance with ISO 21660-3.

7 Procedure

7.1 Use of automatic equipment

Automatic equipment may be used if the procedure based on such an equipment is validated with reference materials of an adequate type. This equipment shall fulfil all the requirements given in [Clauses 5](#) to [8](#) regarding sample size, atmosphere, and temperatures and weighing accuracy. Deviations from these specifications shall be reported and justified.

7.2 Number of determinations

A minimum of two determinations shall be carried out on the general analysis test sample.

7.3 Furnace temperature checking

Adjust the temperature of the zone in the furnace ([5.1](#)), containing either a stand with one crucible and lid [see [Figure 3 a](#)] or a stand with the requisite number of crucibles and lids [see [Figure 3 b](#)], to $(900 \pm 10) ^\circ\text{C}$ as indicated by the correctly located thermocouple ([5.2](#)). Check that this temperature is observed at the same height under each crucible ([5.3](#)).

Temperature checking should be made before starting determinations. However, if several analyses are performed per day, a daily temperature check is usually sufficient. The check of the temperature recovery criterion (see [5.1](#)) should be dealt with in a similar way.

7.4 Volatile matter determination

Fill either a stand with one empty crucible ([5.3](#)) and lid [see [Figure 3 a](#)] or a stand with the requisite number of empty crucibles and lids [see [Figure 3 b](#)] and insert it into the furnace ([5.1](#)). Maintain the temperature at $(900 \pm 10) ^\circ\text{C}$ for 7 min. Remove the crucible(s) from the furnace and allow to cool to room temperature on a thick metal plate.

As soon as they are cool, weigh each empty crucible and lid and weigh into each crucible, to the nearest 0,1 mg, $(1 \pm 0,1)$ g of the general analysis sample. Replace the lid and tap each crucible on a clean hard surface until the test portion forms a layer of even thickness on the bottom of the crucible.

NOTE 1 In practise, 2 g is also possible due to the density being too low.

Place the charged crucible(s) in a cold stand, transfer to the furnace, close the door and leave for (420 ± 5) s.

Remove and allow to cool to room temperature. When cool, weigh the crucible(s) to the nearest 0,1 mg in the same manner as for the empty crucible(s).

NOTE 2 The same treatment of the crucible before and after the determination minimises the effect of any film of water adsorbed on its surface, while the rapid cooling reduces absorption of moisture by the coal or coke residue.

NOTE 3 If multiple determinations are carried out, any vacant places in the stand are filled with empty crucibles.

8 Calculation

8.1 Case A – without removed ash contributors

The volatile matter, V_{ad} , in the sample as analysed, expressed as mass fraction in percent, is given by [Formula \(1\)](#):

$$V_{ad} = 100 \cdot \frac{(m_2 - m_3)}{(m_2 - m_1)} - M_{ad} \quad (1)$$

NOTE In this case, the “as analysed” sample is similar to the “as received” sample if there is no other preparation operation of the sample before analysis.

The volatile matter, $V_{ad,db}$, in the analysed sample, in dry basis, expressed as mass fraction in percent, is given by [Formula \(2\)](#):

$$V_{ad,db} = \left[100 \cdot \frac{(m_2 - m_3)}{(m_2 - m_1)} - M_{ad} \right] \cdot \frac{100}{(100 - M_{ad})} \quad (2)$$

where

m_1 is the mass of the empty crucibles ([5.3](#)) plus lid, in grams;

m_2 is the mass of the crucibles plus lid plus sample before heating, in grams;

m_3 is the mass of the crucibles plus lid plus sample after heating, in grams;

M_{ad} is the mass fraction of moisture in the general analysis sample as analysed, in percent

8.2 Case B – with removed ash contributors

Pre-drying of wet samples is carried out to minimise moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimise biological activity. If it is necessary to dry a sample by heating, it shall be dried in a furnace at a temperature not exceeding the temperature according ISO 21646.

If ash contributors (inert material) were removed before analysis (the removing operation shall be done after the first pre-drying step), then the removed ash contributors shall be taken into account in calculations:

The volatile matter, V_{ad} , in the sample as analysed (received pre-dried sample and the ash contributors have already been removed), expressed as mass fraction in percent, is given by [Formula \(3\)](#):

$$V_{ad} = \left[100 \cdot \frac{(m_2 - m_3)}{(m_2 - m_1)} - M_{ad} \right] \quad (3)$$

The volatile matter, $V_{ad,db}$, in the sample as analysed, on a dry basis, expressed as mass fraction in percent, is given by [Formula \(4\)](#):

$$V_{ad,db} = V_{ad} \left[\frac{(100)}{(100 - M_{ad})} \right] \quad (4)$$

The volatile matter, $V_{ar,wb}$, in the sample as received, taking into account removed ash contributors (after pre-drying process), expressed as mass fraction in percent, is given by [Formula \(5\)](#):

$$V_{ar,wb} = V_{ad,db} \left[\frac{(100 - M_T - A_{rac})}{(100 - M_{ad})} \right] \quad (5)$$

where

- $V_{ar,wb}$ is the volatile matter in the general received sample before first drying and removing ash contributors, in percent;
- m_1 is the mass of the empty crucibles ([5.3](#)) plus lid, in grams;
- m_2 is the mass of the crucibles plus lid plus sample before heating, in grams;
- m_3 is the mass of the crucibles plus lid plus sample after heating, in grams;
- M_{ad} is the mass fraction of moisture in the general analysis sample as analysed (received pre-dried sample, after removing ash contributors), in percent;
- M_T is the total mass fraction of water lost, after complete drying, from the as received sample (before removing ash contributors), in percent;
- A_{rac} is the mass fraction of removed ash contributors, in percent.

The total moisture content, M_T , of the “as received” sample, after complete drying (first drying and second drying) and taking into account removed ash contributors is given by [formula \(6\)](#):

$$M_T = M_P + M_{ad} \cdot \frac{100 - M_P + A_{rac}}{100} \quad (6)$$

where M_P is the mass fraction of water lost, after first drying (= pre-drying), from the as received sample (before removing ash contributors), in percent;

The mass fraction of removed ash contributors, A_{rac} , in percent given by [formula \(7\)](#):

$$A_{rac} = 100 \frac{m_{rac}}{m_{ar}} \quad (7)$$

where

- m_{ar} is the mass of a sample as received
- m_{rac} is the mass of the removed ash contributors from the received pre-dried sample

Report the result as the mean of duplicate determinations, rounded to the nearest 0,1 % mass fraction. The results of the determination described in this document shall be reported on dry basis.

9 Precision

9.1 Repeatability limit

The maximum difference to be expected between two independent single test results of one laboratory at a confidence level of 95 % will not exceed the repeatability limit in more than 5 % of cases when measuring the same measurand in the same medium, using the same facilities and fulfilling all requirements of the test method (interlaboratory testing).

Precision data derived from an interlaboratory test in Europe from 2008 are given in [Annex A](#).

9.2 Reproducibility limit

The maximum difference to be expected between two independent single test results of different laboratories at a confidence level of 95 % will not exceed the reproducibility limit in more than 5 % of cases when measuring the same measurand in the same medium, each laboratory using their own facilities and fulfilling all requirements of the test method (interlaboratory testing).

Precision data derived from an interlaboratory test in Europe from 2008 are given in [Annex A](#).

10 Test report

The test report shall include the following information:

- a) identification of the laboratory and the testing date;
- b) identification of the sample tested;
- c) a reference to this document, i.e. ISO 22167:2021;
- d) calculation case used;
- e) test results of the determination performed on dry basis (see [Clause 8](#));
- f) content of removed ash contributors, in % mass fraction, if applicable (see [8.2](#));
- g) any deviation from this document;
- h) any unusual features observed during the determination which may have affected the test result and details of any operations not included in this document or regarded as optional.

Annex A (informative)

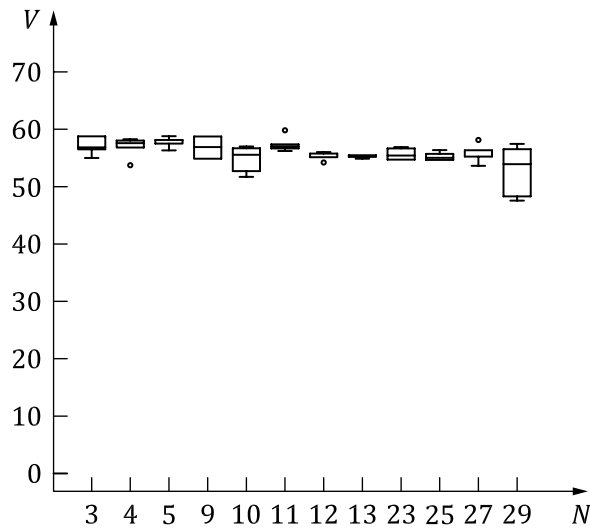
Interlaboratory test results

The statistic evaluation of the interlaboratory test results from an interlaboratory test in Europe from 2008 was carried out in accordance with ISO 5725-5. The precision data obtained are shown in [Table A.1](#).

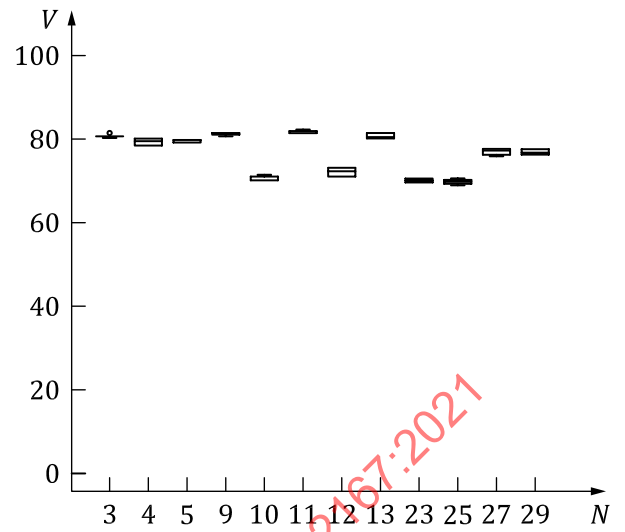
Table A.1 — Precision data

Designation	Shredded tyre	Demolition wood	Dried sludge	Municipal waste	Plastic/paper fluff
Number of laboratories participating	12	12	12	12	10
Total number of values (without outliers)	69	70	72	69	60
Mean value, in % mass fraction	56,10	76,40	25,90	71,21	66,23
Laboratory effect, in % mass fraction	1,16	1,52	0,18	0,51	0,86
Sample effect, in % mass fraction	0,50	0,09	0,01	0,17	0,19
Repeatability standard deviation, s_r , in % mass fraction	1,05	0,20	0,06	0,27	0,56
Repeatability limit, r : ($r = 2,8 \times s_r$) in % mass fraction	2,94	0,56	0,17	0,76	1,57
Reproducibility standard deviation, s_r , in % mass fraction	1,83	4,65	1,82	2,75	3,22
Reproducibility limit, R : ($R = 2,8 \times s_r$) in % mass fraction	5,12	13,02	5,10	7,70	9,02

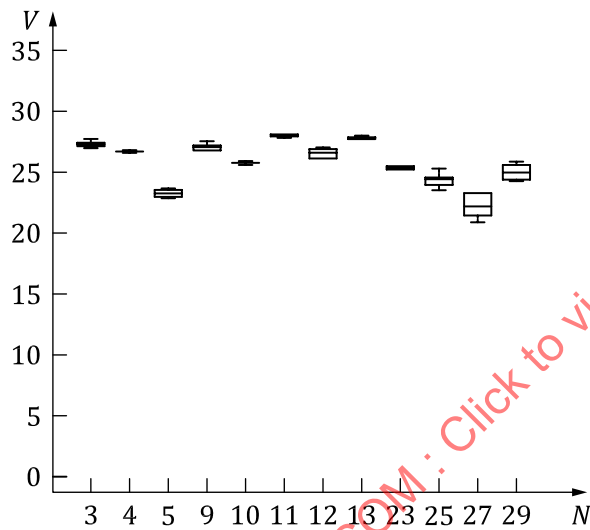
The deviations of the test results between the individual laboratories for each sample type are shown in [Figures A.1 a\) to e\)](#).



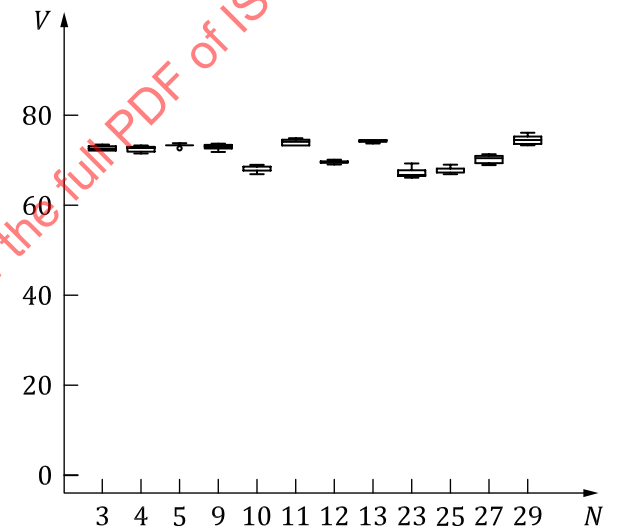
a) Shredded tyre



b) Demolition wood



c) Dried sludge



d) Municipal waste