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## **Soil, sludge and treated biowaste — Determination of impurities and stones**

*Boden, Schlamm und behandelte biologische Abfälle — Bestimmung von Fremdstoffen und Steinen*

*Sol, boue et déchets biologiques traités — Détermination des matières étrangères et pierres*

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## Foreword

This document TC BT 151 WI has been prepared by Technical Committee CEN/TC BT 151 "Horizontal", the secretariat of which is held by DS.

This document is a working document.

The following TC's have been involved in the preparation of the standard:

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

[table to be filled and amended by the standards writer]

Material	Validated	Document
Treated Biowaste	<input type="checkbox"/>	[reference]
Sludge	<input type="checkbox"/>	
Soil	<input type="checkbox"/>	

## Introduction

This standard is developed in the European project 'HORIZONTAL'. It is the result of a desk study "Horizontal European standard for determination of AOX in sewage sludge and comparable matrices" which aimed at evaluating the latest developments in assessing AOX in sludge, soil, treated biowaste and neighbouring fields. After an evaluation study, in which e.g. the ruggedness of the method was studied, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN. The standard is part of a modular horizontal approach in which the standard belongs to the analytical step.

Until now test methods determining properties of materials were often prepared in Technical Committees (TCs) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for every material or product. Consequently this has resulted in duplication of work. To avoid such duplication of work for parts of a testing procedure references to parts of test methods from other TCs were introduced. However the following problems are often encountered while using references in this way: 1) The referenced parts are often not edited in a way that they could easily be referred to, 2) the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with

certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). A beneficial feature of this approach is that "modules" can be replaced by better ones without jeopardizing the standard "chain".

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The modules that relates to this standard are specified in section XX Normative references.

An overview of modules and the manner, in which modules are selected will be worked out later, at which time proper reference in this standard will be provided.

### **Safety warning**

Care shall be taken when handling samples, since they may contain sharp fragments, chemical contaminants or possible pathogenic organisms. When using bleach, care must be taken to avoid inhaling fumes containing Cl<sub>2</sub>.

## **1 Scope**

A method to determine the physical impurities > 2 mm and stones > 5 mm in soils, sludges and treated biowaste is described. Pieces of wood or bark are not considered as impurities.

NOTE Although the title of the method and body of the text states "soil sludges and treated biowaste" this does not mean that the method shall not be suitable for other forms of waste.

## **2 Normative references**

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 12579:2000, *Soil improvers and growing media – Sampling*.

EN 13040:1999, *Soil improvers and growing media – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*

ISO 3310-1:2000, *Test sieves – Technical requirements and testing Test sieves of metal wire cloth*

ISO 3310-1/Corr 1:2004, *Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth – Technical Corrigendum 1*

ISO 3310-2, 1999, *Test sieves – Technical requirements and testing Test sieves of perforated metal plate*

CR 13456:1999, *Soil improvers and growing media – Labelling, specifications and product schedules*

## **3 Terms and definitions**

For the purpose of this European Standard, the following terms and definitions given in CR 13456, EN 12579 and EN 13040 apply. Until definitions of soil, sludges and treated biowaste are supplied by HORIZONTAL, the following definitions are offered as a guideline, and a guideline only, for the reader.

**3.1****Soil**

1 The unconsolidated mineral or organic material on the immediate surface of the earth that serves as a natural medium for the growth of land plants

2 The unconsolidated mineral or organic matter on the surface of the earth that has been subjected to and shows effects of genetic/(biotic) and environmental factors of: climate (including water and temperature effects), and macro- and micro-organisms, conditioned by relief, acting on parent material over a period of time (SSSA, 2001).

**3.2****Sludges**

Solid material resulting from the treatments of urban waste water, consisting of household effluents and water from rainfall runoff

**3.3****Treated biowaste**

Solid particulate material of biological origin treated in such a way that the material is sanitised, stabilised and confers beneficial effects when added to soil and/or used in conjunction to plants

**3.4****Stone**

Unattached pieces of rock 2 mm in diameter or larger that are strongly cemented or more resistant to rupture (SSSA, 2001). Rock being hard consolidated mineral matter

**3.5****Glass**

Material consisting mostly of presumably man-made hard not crystallized minerals

**3.6****Metal**

Material consisting mostly of metals

**3.7****Plastic**

Material consisting mostly of presumably man-made synthetics

**3.8****Other materials**

Any unexpected material not accounted for in the method. It will at least be recorded in weigh but shall be labelled qualitatively when possible e.g. "mainly leather fragments"

Bark and wood are considered acceptable natural constituents of the samples.

Limestone, including added limestone, is counted as stone.

NOTE Definitions may be revised at a later date.

**4 Principle**

After drying, the test material is dry sieved, then, if necessary, either water-washed and/or bleach-washed and wet sieved on a 2 mm sieve (as necessary). The fraction > 2 mm are again dried when necessary and the fractions of stones > 5 mm and differentiated impurities > 2 mm are determined by weight or, for plastics, by weight and area. The test is carried out in duplicate.

NOTE: The purpose of measuring both, the weight and the area of plastics, is to characterise two aspects of the contamination with plastics. Weight characterises the sheer bulk of plastics present. Area characterises the visual presence of plastics. The latter anticipates rejection by consumers of materials with high areas of plastic even when the actual amount on weight basis would be low.

## 5 Reagents

### 5.1 Bleach

The strongest commercially available bleach is used, i.e. 9.6 % chlorine (48° in other units). This is a mixture of NaOCl, sometimes written as NaClO, and NaCl and NaOH. The acceptable range is 7.2 – 9.6 % (or 36° to 48°). Bleach is to be used in specific cases only.

### 5.2 Water

normal drinking water quality tap water or purer.

## 6 Apparatus

### 6.1 Sample tray

constructed of material thermally stable up to 150 °C, surface approximately 1250 cm<sup>2</sup>.

### 6.2 Drying oven

ventilated, fan assisted, capable of holding sample trays at 80 °C ± 3 °C.

### 6.3 Analytical balance

2 kg maximum with an accuracy of 0.01 g.

### 6.4 Container

a plastic container of 10 litre capacity

### 6.5 Extractor hood

any fumes containing Cl<sub>2</sub> must be safely removed by using an extractor hood or fume cupboard using forced ventilation and a suitable fume filter such as an active carbon filter.

### 6.6 Skin protection

normal laboratory wear and synthetic gloves to avoid skin contact with bleach whenever bleach is used.

### 6.7 Eye protection

like plastic laboratory glasses or a face shield to protect the eyes from bleach droplets when bleach is used.

### 6.8 Glass rod

a 40-60 cm rod for stirring the solution in the container which can resist bleach and temperatures up to 100 degrees Celsius.

### 6.9 Sieves

diameter 200 mm or 300 mm, with 2 mm, 5 mm and 40 mm apertures, ISO 3310-1:2000 or ISO 3310-2, 1999.

### 6.10 Beaker

300 ml

**6.11 Tweezers/forceps.**

**6.12 Camera and graph paper,**

**6.13 Temperature measuring device capable of measuring up to 100 °C**

## **7 Procedure**

### **7.1 Sample preparation**

#### **7.1.1 Large objects**

Prepare the test sample in accordance with EN 13040, clause 8.1, 8.2. Where 80 % w/w or more of the sample passes a 40 mm sieve, the procedure can be continued. If not the method is not appropriate as the material contains too many large objects.

#### **7.1.2 Amount of laboratory sample**

Determine the amount of sample to be analysed depending on the coarseness of the sample. For a sample with particles up to 100 mm in size, 7.5 l are taken. For a sample with particles up to 40 mm in size, 3 l is taken. For a sample with particles up to 25 mm in size, 1.5 l is taken. For fine materials of 0 mm -12 mm, 1 l is taken. The appropriate amount is then put in the sample tray (6.1).

#### **7.1.3 Drying**

Dry the materials for at least 16 hours at 80 °C ± 3 °C until constant weight in the drying oven (6.2).

#### **7.1.4 Weighing**

Determine the total dry weight with the balance (6.3).

#### **7.1.5 Dry sieving**

Using the beaker (6.10), transfer portions of 100 ml of the sample on to the 2 mm sieve (6.9). Record the weight of all material < 2 mm and discard.

NOTE Discarding the material < 2 mm will facilitate the choice for sieve analysis without washing (7.1.6) and will substantially reduce the amount of bleach necessary if bleach is used (7.2).

#### **7.1.6 Choice for sieve analysis without washing**

If the different impurities can be easily discriminated by eye, the sample may be subjected to dry sieve analysis (7.3) without prior washing. If the different impurities are coated with any matter which hinders visual discrimination, the sample shall be water- or bleach-washed (7.2). As an indicative criterion, laboratory staff should be able to analyse a sample within 30 minutes.

### **7.2 Choice for water and/or bleach washing**

For samples with visibly low organic matter content, stirring the material for 5 minutes with water instead of bleach is allowed. An organic matter content of < 15.0 w% of the dry sample serves as an indicative criterion. If there is any doubt about the proper discrimination and classification of impurities, bleach washing shall be performed.

NOTE For some samples it may be necessary to carry out the bleach treatment following a water wash.

**7.2.1 Water wash.**

**7.2.1.1 First washing**

Put portions of up to 1500 ml of the weighed dry material (7.1.5) in a 10 litres container (6.4). Cover the sample with 2 litres water (5.2) and stir with a glass rod (6.8) for 5 minutes. Finally pour the sample on a sieve (6.9) with 2 mm meshes and wash through with water.

**7.2.1.2 Second washing**

Put the fraction > 2 mm (7.2.1.1) back into the container (6.4) and repeat first step (7.2.1.1).

**7.2.2 Bleach wash.**

**7.2.2.1 First washing**

Put portions of up to 1500 ml of the weighed dry material (7.1.5) in a 10 litres container (6.4). Put the container under an extractor hood (6.5) to safely and continuously remove chlorine and carbon dioxide gasses formed and use skin protection (6.6) and eye protection (6.7). Add bleach until the sample is submerged under 5 mm bleach with a maximum of two litres bleach (5.1) and stir with a glass rod (6.8). The chemical reaction is exothermic, very quick and produces large quantities of fumes. To minimise possible overflows do not stir until the temperature is below 80 degrees Celsius (6.13). Prevent the formation of a gaseous cake on the liquid during the first 15 minutes by breaking the cake gently with the glass rod (6.8). The material must be in contact with the bleach for two hours. Finally pour the sample on a sieve (6.9) with 2 mm meshes and wash through with water.

**7.2.2.2 Second washing**

Put the fraction > 2 mm (7.2.2.1) back into the container (6.4) and add bleach and repeat the first step (7.2.2.1) but leave the material in contact with the bleach for four hours. Finally pour the sample on a sieve (6.9) with 2 mm meshes and wash through with water.

NOTE All the above work to be carried out under fume extraction.

**7.2.3 Drying**

Dry the materials > 2 mm (7.2.1.2 or 7.2.2.2) for at least 16 hours until constant weight in the drying oven (6.2).

**7.3 Sieve analysis.**

**7.3.1 Sieving and sorting stone > 5 mm**

Using the beaker (6.10), transfer portions of 100 ml of the dried sample (7.1.6 or 7.2.3) onto the 5 mm sieve (6.9) and hand shake. Spread the > 5 mm fractions on a flat surface and gather the stone particles > 5 mm with help of the tweezers/forceps (6.11). Continue this procedure until the entire sample (7.2.3) has been sieved and sorted. Determine the total weight of the fraction stones > 5 mm using the balance (6.3). Recombine the fractions > 5 mm and < 5 mm but without the stones > 5 mm. Record the recombined weight.

**7.3.2 Sieving > 2 mm**

Using the beaker (6.10), transfer portions of up to 100 ml of the recombined sample (7.3.1) on to the 2 mm sieve (6.9) until all the sample has been sieved. Discard all material < 2 mm.

NOTE The sieving > 2 mm (7.3.2) is optional. It is recommended for samples which after water or bleach washing have many fragments < 2 mm which hinder sorting.



## 7.4 Sorting > 2 mm

Spread the recombined fractions > 2 mm (7.3.1 or 7.3.2) one by one on a flat surface and search out all visual recognisable impurities using the tweezers/forceps (6.11). Sort out the following materials: glass, metals, plastics, and the impurity class "other material". Determine the weight of the individual type of impurities using the balance (6.3). Determine the total surface area of the fraction of plastics using graph paper or a camera (6.12). The plastic films are spread and pasted on a sheet of graph paper of 1 mm<sup>2</sup> mesh. The sheet is photocopied or photographed and the copy is enlarged to facilitate counting the squares. The area covered by the plastic films is counted.

Image analysis is an alternative method in which plastics are spread and pasted as flat as possible on a contrasting surface such as a sheet of bright blue paper of known dimensions.



**Figure 1 — Selected plastics on a blue sheet, note the rumpling and discolouration**

A photograph with a digital camera is taken with > 0.9 Mb per picture and more than 75 % of the image area filled by the contrasting sheet of known dimensions. The image is processed with a simple program e.g. Image-pro. First the parts around the sheet with the contrasting colour are clipped of. From the resulting area of known dimensions, the part showing the contrasting colour of the sheet is then estimated in percent of the total area. The area of the plastics is then calculated as (known area of background paper).

NOTE 1 percentage filled by background paper colour)/100

NOTE 2 The fractioning of the dried samples into 100 ml portions is to prevent clogging of the sieve as well as to ensure proper recognition of impurities.

NOTE 3 Lime particles > 5 mm will be counted as stone particles as they often cannot be discriminated from stone particles, which might even contain carbonate themselves.

NOTE 4 Wood or bark particles > 2 mm will not be counted as they are thought to be a customer accepted natural ingredient in soils, sludges and treated biowaste.

**Table 1 — Data to be recorded after water or bleach washing and sieving of samples**

		Weight in g	Surface in cm <sup>2</sup>
> 5 mm	Stones	Y	-
> 2 mm	Glass	Y	-
> 2 mm	Metals	Y	-

> 2 mm	Plastics	Y	Y
> 2 mm	Other materials	Y*	-
*Where possible other materials should be identified and their weights recorded			

### 7.5 Treatment of waste fumes and liquids

When using bleach (7.2.2), fumes are to be force ventilated away through an appropriate filter such as an active carbon filter. This will result in a filter containing Cl<sub>2</sub> which is to be replaced at specified intervals and handled as chemical waste. Any excess bleach must be poured off through an appropriate filter such as an active carbon filter. When bleach-washing the samples (7.2.2) all liquid residues shall also be disposed of through this filter.

### 7.6 Schedule for the Procedure

A schedule presenting the procedure described in paragraphs 7.1 - 7.5.

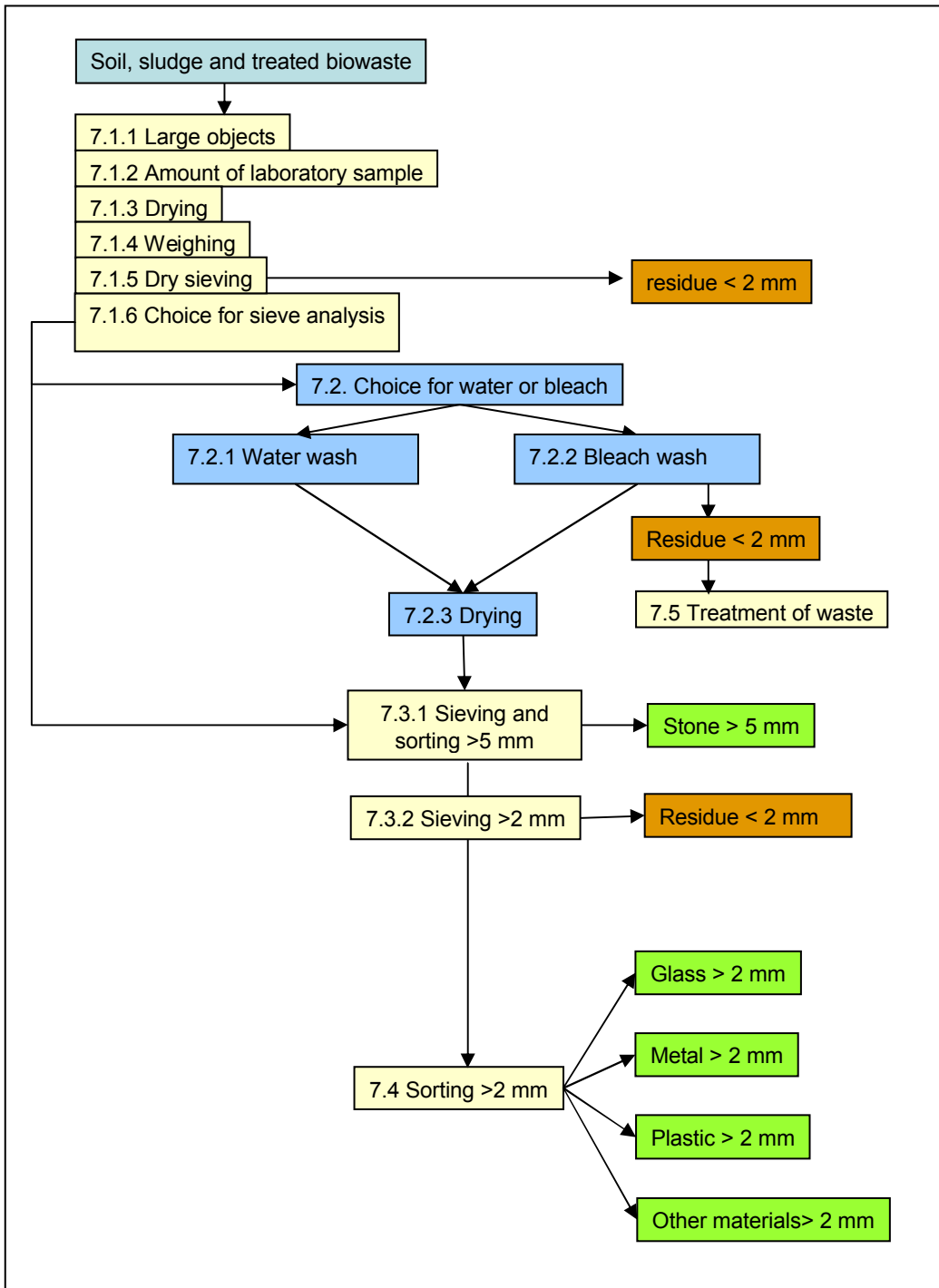


Figure 2 — Schedule of the procedure described in paragraphs 7.1 - 7.5 with appropriate numbering

## 8 Calculations and expression of results

The mass of the impurities and the area of plastics are expressed on the total dry weight before sieving. The average results are calculated of the duplicates.

$$I_{S > 5 \text{ mm}} = \frac{W_{S > 5 \text{ mm}}}{T} \times 100 \% \quad (1)$$

$$I_{G > 2 \text{ mm}} = \frac{W_{G > 2 \text{ mm}}}{T} \times 100 \% \quad (2)$$

$$I_{M > 2 \text{ mm}} = \frac{W_{M > 2 \text{ mm}}}{T} \times 100 \% \quad (3)$$

$$I_{P > 2 \text{ mm}} = \frac{W_{P > 2 \text{ mm}}}{T} \times 100 \% \quad (4)$$

$$I_{O > 2 \text{ mm}} = \frac{W_{O > 2 \text{ mm}}}{T} \times 100\% \quad (5)$$

$$I_{P > 2 \text{ mm}} = \frac{A_{P > 2 \text{ mm}}}{T} \quad (6)$$

NOTE Expressed in  $\text{cm}^2 \cdot \text{g}^{-1}$

Where

$I$  is the impurity fraction (% w/w or  $\text{cm}^2 \cdot \text{g}^{-1}$ )

$W$  is weight of impurity type (g)

$A$  is the area of impurity type ( $\text{cm}^2$ )

$T$  is the total dry weight (g)

$S$  is stones

$G$  is glass

$M$  is metal

$P$  is plastic

$O$  is "other material"

## 9 Precision data

The performance characteristics of the method (Annex A) data have been evaluated. Table 2 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Table A.2 in Annex A by taking the median value and rounding the numbers.

**Table 2 — Typical values and observed ranges of the repeatability and reproducibility limits**

The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. Conversely, the repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample. They are reported in % as the bulk density of the samples may differ with more than one order of magnitude e.g. 1800 kg.m<sup>-3</sup> for sludge and 180 kg.m<sup>-3</sup> for peat.

Results of the validation of the determination of impurities and stones in soil, sludge and treated biowaste	Concentration level > x %	Typical value %	Observed range %
Stones > 5 mm , repeatability limit, r%	1 %WW	85	60-110
Stones > 5 mm, reproducibility limit, R%	1 %WW	110	80-160
Glass > 2 mm, repeatability limit, r%	1 %WW	8	8-100
Glass > 2 mm, reproducibility limit, R%	1 %WW	17	16 -100
Metal > 2 mm, repeatability limit, r%	0.3 %WW	31	#
Metal > 2 mm, reproducibility limit, R%	0.3 %WW	36	#
Others > 2 mm, repeatability limit, r%	0.2 %WW	85	60-110
Others > 2 mm, reproducibility limit, R%	0.2 %WW	130	100-150
Plastics > 2 mm, repeatability limit, r%	0.2 %WW	42	40-150
Plastics > 2 mm, reproducibility limit, R%	0.2 %WW	120	120-180

# Very low metal contents in most samples studied.

NOTE 1. The above results refer to the difference that may be found between two test results performed on two laboratory samples obtained under the same conditions. In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the above typical reproducibility values and observed reproducibility ranges should be divided by  $\sqrt{2}$  to obtain the corresponding typical dispersion limit and its observed range. In the example of Glass > 2 mm in Compost E the result and its dispersion limit is  $0.98 \pm 0.12$  ( $2 * sR = 12.4 \%$  of 0.98). This means that with a 95 % statistical confidence, the values reasonably attributable to the measured parameter are larger than 0.86 %WW and lower than 1.10 %WW.

NOTE 2. The repeatability limit (r) and the reproducibility limit (R) as given in Table A.2 (Annex A) and in this table are indicative values of the attainable precision if the determination of impurities and stones is performed in accordance with this standard [CSS99049].

NOTE 3. A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the the determination of impurities and stones in soil, sludge and treated biowaste.

NOTE 4 In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table A.2 (Annex A) and this table.

## 10 Test report

The test report shall include the following information:

- a) A reference to this Standard.
- b) A complete identification of the sample.
- c) The results of the different fractions expressed as % on dry matter basis on 2 decimal places.
- d) A description of the procedure used i.e. dry sieve, water and or bleach treatment.
- e) Any details not specified in the Standard, or which are optional, as well as any other factor, which may have affected the results.

## **Annex A (informative)**

### **Repeatability and reproducibility data**

#### **A.1 Performance characteristics**

##### **A.1.1 Objective of the interlaboratory comparison**

In a European wide interlaboratory comparison study according to ISO 5725-2, the performance characteristics of the standard “Determination of impurities and stones in soil, sludge and treated biowaste” were established.

##### **A.1.2 Materials used in the interlaboratory comparison study**

The interlaboratory comparison of determination of impurities and stones in soil, sludge and treated biowaste was carried out with 11-13 European laboratories on 6 materials. The materials selected for the interlaboratory comparison were chosen to represent soil, sludge and biowaste as broad as possible, because the standard will find general application across different types of soil and soil related materials. (detailed information can be found in the final report on the Interlaboratory comparison study mentioned in the Bibliography).

In the interlaboratory comparison study the following starting points were used:

The laboratory samples were all taken from one large batch of the different materials according to the normal practice. The normal size reduction and the normal repeated mixing were carried out as needed to obtain representative laboratory samples from the large batch sample (ref JRC).

Note : the samples provided for the validation should not be confused with reference samples provided for certification purposes, as the performance results obtained have to be directly applicable to daily practice (less rigorous sample preparation than for a reference material).

The experimental plan was designed by project HORIZONTAL on the basis of each laboratory being given two laboratory samples of each material to be tested. This is in accordance with ISO 5725-2.

The materials examined cover all the grain size classes to which the the determination of impurities and stones in soil, sludge and treated biowaste applies: very fine grained materials (like sludge: 0  $\mu\text{m}$  to about 125  $\mu\text{m}$ ) and fine-grained materials (soil and compost: 0 mm to 4 mm).

Table A.1 provides a list of the types of materials chosen for testing and the selected components.

Table A.2 — Material types tested and components analysed in the interlaboratory comparison of the method for the determination of impurities and stones in soil, sludge and treated biowaste.

Grain size class	Code and material type tested	Components analysed
0 µm to about 125 µm	A: Sandy soil with some organic matter	1)
0 µm to about 4 mm	B: Sewage sludge	1)
0 µm to about 4 mm	C: Horticultural green compost	1)
0 µm to about 4 mm	D: Half municipal/half green waste compost***	1)
0 µm to about 4 mm	E: Horticultural green compost	1)
0 µm to about 4 mm	F: Half municipal/half green waste compost***	1)
1) Weigh of stones >5 mm, glass >2 mm, metal >2 mm, plastic >2 mm others >2 mm and area of plastics >2 mm		

### A.1.3 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation ( $s_r$ ) and the reproducibility standard deviation ( $s_R$ ) were obtained (Table A.2).

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The repeatability limit was calculated using the relationship :  $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$  with the critical range factor  $f = 2$ .

For instance, the repeatability limit around a measurement result of 1.0 % ww Glass > 2 mm is  $\pm 0.084$  % ww Glass > 2 mm (i.e  $\pm 8$  % of 1).



NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value  $f = 2$  used in the factor  $f \cdot \sqrt{2}$  corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value  $f = 2$  corresponds to the usual value  $k = 2$  of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). However it may be necessary to use a larger value for  $f$  in situation as described in clause 9.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The reproducibility limit was calculated using the relationship:  $R = f \cdot \sqrt{2} \cdot s_R$  with the critical range factor  $f = 2$ .

For instance, the reproducibility limit around a measurement result 1.0 % ww Glass > 2 mm is  $\pm 0.168$  % ww Glass > 2 mm (i.e  $\pm 17$  % of 1)..

NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value  $f = 2$  used in the factor  $f \cdot \sqrt{2}$  corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value  $f = 2$  corresponds to the usual value  $k = 2$  of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the dispersion limit is equal to  $k \cdot s_R$  with the usual value  $k = 2$ , resulting in a dispersion limit lower than the reproducibility limit (i.e. a ratio of  $\sqrt{2}$ ). However it may be necessary to use a larger value  $f \cdot \sqrt{2}$  (or  $k$ ) in situation as described in clause 9 .

In case of relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Tables A.2 (this means that the value chosen for the critical range factor  $f$  is larger than 2 as well as for the coverage factor  $k$  for dispersion). This is because the extreme results may have been obtained in accordance with the present standard and/or be caused by the variability within, or in between, the laboratory samples.

Table A.3 — Results of the interlaboratory comparison studies of the determination of impurities and stones (dry sieving) in soil, sludge and treated biowaste. All concentrations in %WW.

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data
Sludge A	Stones > 5mm	1.68	39.7%	57.5%	1.87	2.71	13	0	29
Sludge A	Glass > 2mm	0.01	785%	785%	0.17	0.17	11	2	25
Sludge A	Metal > 2mm	0.01	783%	783%	0.17	0.17	11	0	25
Sludge A	Plastic > 2mm	0.01	799%	799%	0.16	0.16	11	2	25
Sludge A	Other > 2mm	0.11	22.7%	36.2%	0.07	0.11	8	5	16
Compost C	Stones > 5 mm	0.75	60.0%	64.0%	1.25	1.34	13	0	25
Compost C	Glass > 2 mm	0.04	170%	214%	0.21	0.26	13	0	25
Compost C	Metals > 2 mm	0.03	216%	237%	0.19	0.21	7	0	13
Compost C	Plastics > 2 mm	0.05	40.8%	57.8%	0.06	0.09	4	9	8
Compost C	Other > 2 mm	0.03	105%	167%	0.09	0.14	11	2	21
Soil B	Stoness > 5 mm	2.83	21.3%	28.3%	1.68	2.24	13	0	25
Soil B	Glass > 2 mm	0.04	37.9%	37.9%	0.04	0.04	11	2	21
Soil B	Metals > 2 mm	0.04	187%	206%	0.19	0.21	10	3	20
Soil B	Plastics > 2 mm	0.09	54.3%	65.2%	0.13	0.16	13	0	25
Soil B	Other > 2 mm	0.15	38.1%	54.8%	0.16	0.24	6	7	12
Compost D	Stones > 5 mm	0.22	82.3%	82.3%	0.50	0.50	13	0	25
Compost D	Glass > 2 mm	0.11	45.2%	45.2%	0.14	0.14	11	2	21
Compost D	Metals > 2 mm	0.01	74.8%	74.8%	0.02	0.02	4	9	8
Compost D	Plastics > 2 mm	0.16	32.8%	43.5%	0.15	0.19	11	2	21
Compost D	Others > 2 mm	0.02	52.5%	393%	0.03	0.25	7	6	13
Compost E	Stones> 5 mm	0.46	31.4%	37.9%	0.41	0.49	11	2	21
Compost E	Glass > 2 mm	0.98	2.9%	6.2%	0.08	0.17	13	0	25
Compost E	Metal > 2 mm	0.29	11.2%	12.8%	0.09	0.10	13	0	25
Compost E	Plastic > 2 mm	0.21	14.8%	43.7%	0.09	0.25	13	0	25
Compost E	Other > 2 mm	0.08	90.5%	90.5%	0.20	0.20	11	2	21

Abbreviations: sr Repeatability standard deviation;SR Reproducibility standard deviation; r Repeatability limit (comparing two measurements); R Reproducibility limit (comparing two measurements); p Number of labs.

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