Soil, sludge and treated biowaste - Determination of impurities and stones

Contents

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_2.

Foreword

This document TC xxx WI zzz has been prepared by Technical Committee CEN/TC xxx "", the secretariat of which is held by yyy.

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
Waste	☐	[reference]
Sludge	☐	
Soil	☐	

1. Scope

A method to determine the physical impurities > 2mm 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 method 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 method only when incorporated in it by amendment or revision. For undated references the latest edition of the publications referred to apply.

ISO 3310-1:2000	Test sieves of metal wire cloth.
ISO 3310-2, 1999	Test sieves of perforated metal plate.
ISO 5725:1994	Precision of test methods - determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.
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.
EN 14175-2: 2003	Safety and performance requirements of fume cupboards.

3. 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.

4. Definitions

For the purpose of this standard the definitions given in CR 13456, EN 12579 and EN13040 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.

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).

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

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.

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 (WordReference.com English Dictionary).

Glass. Material consisting mostly of presumably man-made hard not crystallized minerals.
Metal. Material consisting mostly of metals.
Plastic. Material consisting mostly of presumably man-made synthetics.
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.

5. Reagents

5.1 **Bleach,** The strongest commercially available bleach is used, i.e. 9.6% chlorine (48 º 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 36º to 48º). 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 ^{o}C, surface approximately 1250 cm^{2}.

6.2 **Drying oven,** ventilated, fan assisted, capable of holding sample trays at 80 ±3 ^{o}C.

6.3 **Analytical balance, 2kg 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_2 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 40mm 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** ^{o}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 100mm 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 0-12 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 80ºC ±3 **ºC** 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 mm2 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) * ((percentage filled by background paper colour)/100).

NOTE 1 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 2 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 3 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 cm2
> 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_2 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 A schedule presenting the procedure described in paragraphs 7.1-7.5.

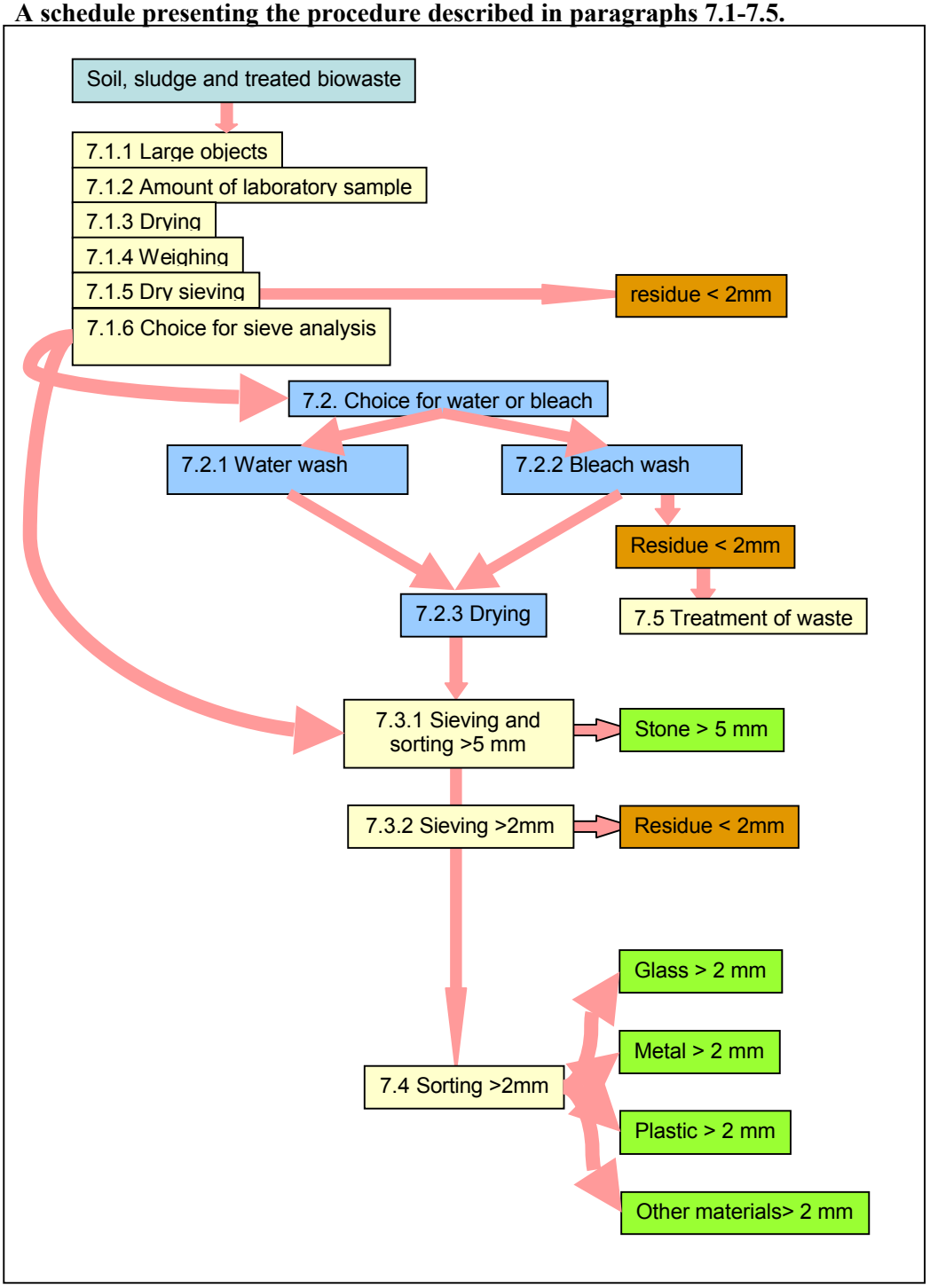

Figure 2 Schedule of the procedure described in paragraphs 7.1-7.5 with appropriate numbering

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.

$$I_{S>5\,mm} = \frac{W_{S>5\,mm}}{T} \times 100\ \% \qquad I_{G>2\,mm} = \frac{W_{G>2\,mm}}{T} \times 100\%$$

$$I_{M>2\,mm} = \frac{W_{M>2\,mm}}{T} \times 100\% \qquad I_{P>2\,mm} = \frac{W_{P>2\,mm}}{T} \times 100\%$$

$$I_{O>2\,mm} = \frac{W_{O>2\,mm}}{T} \times 100\%$$

(7.3, 7.4)

$$I_{P>2\,mm} = \frac{A_{P>2\,mm}}{T}$$ *NOTE Expressed in $cm^2.g^{-1}$*

(6.12)

Where

I is the impurity fraction (% w/w or cm^2/g)
W is weight of impurity type (g)
A is the area of impurity type (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

Area of plastics in cm^2, starting with 1 cm^2. From 0-10 cm2 +/- 0.5 cm2. From 10 cm2 and larger with 5% accuracy. No further data on precision have been defined yet.

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.
d) Any details not specified in the Standard, or which are optional, as well as any other factor, which may have affected the results.

Literature

The Glossary of Soil Science Terms. Soil Science Society of America, 2001. Also www.soils.org/sssagloss/
English Dictionary. WordReference.com 26-06-2006.