Impurities and stone content

Comparison of three methods by five laboratories

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Acknowledgement

This work has been carried out with financial support from the following EU Member States: UK, Germany, France, Italy, Spain, Nordic countries, Netherlands, Denmark, Austria, EU DG XI and JRC, Ispra.

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SUMMARY

EU directives require a reduction in landfill and where possible recycling of waste in the form of composted materials. Methods of testing are required to indicate the amount of impurities. This will make it possible to agree on guaranteed maximum amounts of impurities. A reliable and safe product will encourage the use and repeated use of treated bio waste by customers. Adverse experience, such as a hand cut on a glass shard, will lead to customer rejection, adverse publicity and possibly financial liability.

As no standard methods existed, in a previous desk study a draft standard based on the German method for compost testing was proposed. The main impurities to be characterised were the fraction of coarse stones >5 mm, 2 qualities of plastic >20 mm, stone >2 mm, glass>2 mm, metal>2 mm and 2 qualities of plastic > 2mm. The impurities were sieved and subsequently sorted by hand into plastics, metals, stones and glass. Removing part of the organic matter was still a topic of discussion. The reason for possibly removing organic matter from the samples was that the impurities present were often obscured by a coating of organic and very fine material. This increased the number of mistakes in classification of impurities and also increased the time necessary for sorting the impurities. At the other hand, the destruction of organic matter asked for additional labour, materials and increased the time necessary for the measurement.

Therefore the next stage, described in this report, was to test and compare three methods of impurity characterisation: dry sieving, bleach washing and pressure washing. The objective was to compare the three methods for accuracy, costs, labour demand and methodological flaws. This was done in an inter laboratory trial with five laboratories in four countries. Four samples were prepared in duplo and were sent to the laboratories involved.

The dry sieving method consisted of drying the fresh material and sieving it into the various fractions mentioned. The bleach washing method consisted of destruction of a large part of the organic matter present by bleach before drying and sieving. The pressure washing consisted of washing the material with a high pressure water jet before drying and sieving.

The results showed that dry sieving had to be rejected as the accuracy was low, costs intermediate and labour demand high. The other methods were more or less equal in accuracy. The bleach method being more labour demanding and having a duration of 3-5 days as compared to 2-3 days for the pressure washing. Unfortunately the pressure washing had methodological flaws (unequivocal description) which made it risky to use the method in various laboratories.

Therefore the bleach method was adopted and adapted as the basis for impurity testing, with a limited possibility to use a fast version of it for samples in which masking by organic matter is not a major problem.

1. INTRODUCTION

1.1.1 Background

EU directives require a reduction in landfill and where possible recycling of waste in the form of composted materials. Methods of testing are required to indicate the amount of impurities. This will make it possible to agree on guaranteed maximum amounts of impurities. A reliable and safe product will encourage the use and repeated use of treated bio waste by customers. Adverse experience, such as a hand cut on a glass shard, will lead to customer rejection, adverse publicity and possibly financial liability.

1.1.2 Previous work

In a previous desk study a selection was made from several methods from over the world. (Blok and Wever, 2005). A draft standard based on the German method for compost testing was proposed (Kehres and Pohle, 1998). The French BNSCAO method was proposed as an alternative (CEN/TC 223 N264, 2002). Decisions were made on the nature and the size of the impurities to be tested, the characterisation of plastics, the process temperature and the sample size in relation to particle size.

The main impurities to be characterised were decided to be stones, plastics, glass and metals. Stones include all hard mineral particles, natural or man made.

The fractions to be characterized were decided to be the fractions of coarse stones >5 mm, 2 qualities of plastic >20 mm, stone >2 mm, glass>2 mm, metal>2 mm and 2qualities of plastic > 2mm.

The plastics to be reported were divided into rigid plastics and soft plastics (films). The films were characterized by weight and by area. The area was included as the negative impression of a given weigh of plastic film depends on the area present rather than the weigh.

The maximum process temperature was set on 80 degrees Celsius. Most common plastics (poly styrene, poly ethylene, poly vinyl chloride and poly propylene) deform at temperatures between 80 and 100 degrees Celsius. A temperature lower than 80 degrees Celsius was thought unnecessary as during composting temperatures of up to 80 degrees Celsius may occur.

The sample size for samples with a nominal maximum size of aggregates surpassing 40 mm was extrapolated from an existing dataset. Thus a sample size of 7.5 l for the fraction 40-100 mm was proposed, based on a relation ship $y=1.10 * x$, y being the sample size in kg, x being the average of a nominal maximum aggregate size class (Blok and Wever, 2005).

1.1.3 Goal

The objective was to compare three methods for accuracy, costs, labour demand and methodological flaws.

The three methods of impurity characterisation were: dry sieving, bleach washing and pressure washing. The dry sieving method consisted of drying the fresh material and sieving it into the various fractions mentioned. The bleach washing method consisted of destruction of a large part of the organic matter present by bleach before drying and sieving. The pressure washing consisted of washing the material with a high pressure water jet before drying and sieving.

The main reason to compare these methods was the discussion on if and how organic matter had to be removed. Removing organic matter from the samples was thought necessary because the

impurities present were often obscured by a coating of organic and very fine material. This increased the number of mistakes in classification of impurities and also increased the time necessary for sorting the impurities. At the other hand, the destruction of organic matter asked for additional labour, materials and increased the time necessary for the measurement.

1.1.4 Method and sampling

The action advised was to organise an inter laboratory trial with five laboratories in various countries. Four samples were to be measured with the three methods chosen, in duplo, by each laboratory. The samples had to be prepared in one place to ensure optimal sub sampling from a well mixed lot.

2. METHODS

2.1 The laboratories involved

Table 1 introduces the laboratories and researchers involved in sampling and measuring.

Table 1 Names and addresses of the laboratories involved

Laboratory	Adres	Main researcher	Country	Involved in
Applied Plant Science, Rooting media laboratory	Kruisbroekweg 5, 2671 KT Naaldwijk	A. van Winkel	The Netherlands	Sampling
Cemagref	17, avenue de Cucille, CS 64427 35044 Rennes cedex	B. Morvan	France	Comparison
INFU mbH - Geschäftsbereich PlanCoTec	Karlsbrunnenstr. 11, D-37249 Neu Eichenberg	E. Marciniszyn	Germany	Comparison
ARPAV - Dipartimento Provinciale di Treviso Servizio Osservatorio Suolo e Rifiuti Osservatorio Regionale per il Compostaggio	Via Baciocchi 9 31033 Castelfranco Veneto (Treviso)	L. Paradisi	Italy	Comparison
Dipartimento di Produzione Vegetale Università degli Studi di Milano	Via Celoria 2 - 20133 Milano, Italy	F. Adani	Italy	Comparison
Applied Plant Science, Rooting media laboratory	Kruisbroekweg 5, 2671 KT Naaldwijk	A. van Leeuwen	The Netherlands	Comparison
Applied Plant Science, Rooting media laboratory	Kruisbroekweg 5, 2671 KT Naaldwijk	C. Blok	The Netherlands	Reporting

2.2 The samples

Table 2 characterises the samples used in the inter laboratory trial and their origin.

Table 2 Sample codes, description and origin

Code	Description	Company	Place (origin)
A*	Construction site soil	Dura Vermeer	Hoek van Holland
B*	Horticultural green compost	van Vliet	Hoek van Holland
C*	Sewage sludge	Valk en de Groot	Poeldijk
D*	Municipal park compost (inc. reed)	Ster compost	Alphen a/d Rijn

* The material used was not fully processed to ensure a relatively high impurity content

The samples were collected on the company sites from well within large piles by experienced sample takers. It was decided to use material which had not been fully processed as the total amount of impurities in the fully processed materials were said to be often below 1% in weight. In an additional check it proved impossible to find recognizable plastics in the fully processed products.

All samples were spread out on a flat clean surface. They were then, one after another, repeatedly mixed to obtain a homogeneous sample by using a scoop and a rake. The sub sample bags were filled with an equivalent weight of 1.5 litres. On a cutting machine bits of plastic 2.5 at 2.5 cm have been cut. To the samples were added; sample A one piece, B two pieces, C three and sample D four pieces of plastic. This was added to samples which in itself already contained some plastic!

Thus 4 materials x 3 treatments x 2 repetitions x 5 laboratories = 120 samples were prepared and sent to the laboratories involved. Each laboratory received 24 samples.

2.3 Draft method dry sieving

See Appendix 1.

2.4 Draft method bleach washing

See Appendix 2.

2.5 Draft method pressure washing

See Appendix 3.

3. RESULTS AND DISCUSSION

3.1 Data

Full data may be found in Appendix 4. Table 3 shows the average total impurity content found with each method for every sample. On average the labs measured 11% impurities in sample A, 6% in samples B and C and 3% in sample D. As could be expected the amount of stones in the A sample (construction site soil) was higher than in the others. The green compost B contained somewhat more stones than anticipated as substrate clay pellets were present.

Table 3 Average total impurity content on %w/w basis per sample per method

Code	Description	Bleach washing	Dry sieving	Pressure washing
A*	Construction site soil	8.75	15.73	8.01
B*	Horticultural green compost	6.25	4.78	6.31
C*	Sewage sludge	6.85	5.44	6.39
D*	Municipal park compost (inc. reed)	4.31	2.56	3.34

Table 4 shows the average finds of the different impurities defined. Dry sieving renders a larger amount of stones > 5 mm, but less metal and glass than the other two methods.

Table 4 Average impurity content per impurity class in % w/w per method

Averages	Bleach washing	Dry sieving	Pressure washing
%plastic rigid >20mm (g)	0.00	0.00	0.01
%plastic rigid >2mm (g)	0.15	0.13	0.15
%plastic soft >20mm (g)	0.01	0.01	0.02
%plastic soft >2mm (g)	0.07	0.08	0.09
%stones >5mm (g)	3.54	4.16	3.21
%stones >2mm (g)	2.22	2.35	1.96
%glass >2mm (g)	0.47	0.38	0.47
%metal >2mm (g)	0.07	0.02	0.10
plastic rigid >20mm (cm2/g*100)	0.03	0.00	0.00
plastic rigid >2mm (cm2/g*100)	0.43	0.32	0.36
plastic soft >20mm (cm2/g*100)	0.00	0.13	0.17
soft >2mm (cm2/g*100)	2.57	1.49	1.77

Table 5 shows the standard deviation on the data reported on the different impurities defined. In general the deviations are large. Dry sieving has slightly higher standard deviations for the stone fractions.

Table 5 Standard deviation per impurity class in % w/w per method

Averages	Bleach washing	Dry sieving	Pressure washing
%plastic rigid >20mm (g)	0.00	0.00	0.08
%plastic rigid >2mm (g)	0.18	0.18	0.24
%plastic soft >20mm (g)	0.05	0.02	0.05
%plastic soft >2mm (g)	0.09	0.10	0.11
%stones >5mm (g)	2.12	5.62	1.84
%stones >2mm (g)	2.66	3.09	2.52
%glass >2mm (g)	0.60	0.53	0.84
%metal >2mm (g)	0.13	0.05	0.29
plastic rigid >20mm (cm2/g*100)	0.15	0.00	0.00
plastic rigid >2mm (cm2/g*100)	1.11	0.82	1.01
plastic soft >20mm (cm2/g*100)	0.00	0.75	0.97
soft >2mm (cm2/g*100)	7.42	4.17	5.45

3.2 Experiences

One of the laboratories was unable to conclude the tests in time. The surface area measurement proved problematic for the labs, only one result was reported within the time limits. The amount of rigid and soft plastics > 20 mm were low enough to show the influence of incidental finds of larger pieces. The same is true to some extend for the metals.

Labs confirmed that the sorting process with dry sieving was laborious and prone to mistakes by overestimating the stone fraction to the expense of metals, plastics and glass. To be able to sort the material, clearly some kind of washing is necessary.

The pressure washing proved difficult. Some of the labs did not have the right equipment and used devices driven by tap water pressure. It was reported that larger pieces of soft plastic were pushed through the sieves into the smaller fractions whereas they would have been counted part of the larger fraction in dry sieving. This however is not confirmed by the data! Several labs were uncertain on the method.

From the area of added plastic (A: 6 cm^2, B: 12 cm^2, C: 18 cm^2, D: 24 cm^2) between 50% and 75% was found again, supposing the amounts naturally present were not very high.

According to procedure, to every 500 gr dried material 1-2 litre sodium hypochlorid was added. The sandy A samples however weighed some 2 kg and had to be divided in four sub samles. It was suggested to change the description to 500 ml.

From the sandy sample A (construction site soil) and the sandy sample C (sewage sludge) little organic matter remained after the first bleaching and subsequent washing over the sieve.

As the wet combustion by hypochlorid is exothermic, a glass stirring rod is suggested instead of plastic which deformed. It may be better only to stir the solution when the temperature is (well) below 80 degrees Celsius.

Sample C contained a lot of reed fragments (Phragmites). This proved difficult when sieving and created a gaseous cake on the hypochlorid. The risk is that the reaction in the cake slows down although there is ample hypochlorid under the cake. Therefore stirring is important.

The sampling of the flexible plastics in the, (after treatment) dried material is labour intensive, especially in the green compost samples.

Figure 1 Gaseous exothermic reaction of sample C (municipal parc waste)

4. CONCLUSIONS

1. The dry sieving method overestimates the stone content to the expense of other classes and increases variation. Further more the sorting is strenuous and time consuming work. It is therefore turned down as a Horizontal method.

2. The bleach and pressure washing method result in statistically equal figures.

3. There is no general confidence in the applicability of the pressure washing method. This method needs further methodological development in the area of unequivocal description and the applicability to an indoors lab routine. Preferably a tap driven or height driven water supply is asked for. The method is therefore turned down as a Horizontal method.

4. The bleach washing is laborious and sometimes much more bleach than necessary is added. This is indicated by a bleach coloured liquid without gas formation.

5. In conclusion it is therefore proposed to adapt the bleach method to leave out some or all of the washing steps if the sample nature allows the unequivocal discrimination of the impurities. It is important that some washing of the sample is guaranteed at all times. Therefore continuous washing with tap water for 5 minutes is introduced as the bare minimum for washing the material. The new draft method is added as Appendix 5.

REFERENCES

Blok, C., Wever, G., 2005. Impurities and stone content. HORIZONTAL project Impurities, phase 1.

CEN/TC 223 N264, 2002

Kehres, B. and A. Pohle red., 1998. Methodenbuch zur analyse von compost. Bundesgütegemeinschaft Kompost e.V., Köln Germany.

APPENDIX A DRY SIEVING METHOD

Draft Standard

NOTE1 Where italics appear in the draft method this indicates an area that requires additional work and confirmation.

NOTE 2 Although the title of the method and body of the text states 'composted organic materials' it does not mean that the method may not be suitable for other forms of waste.

A method to determine the visual recognisable impurities in composted organic materials *based on dry sieving*

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Safety warning

Care should be taken when handling samples that may contain sharp fragments, chemical contaminants or possible pathogenic organisms.

1. Scope and field of application

A method to determine the visual recognisable impurities in composted organic materials, soil improvers and growing media. The sample shall be obtained in accordance with SOIL IMPROVERS AND GROWING MEDIA - SAMPLING (EN 12579). The procedures described herein are only applicable to processed organic waste, sludge and soil.

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 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 test, determination of dry matter content, moisture content and laboratory compacted bulk density

3. Principle

After drying the test material the fractions of coarse stones (>5 mm) and plastics (>20 mm) are determined. Subsequently the fractions of differentiated impurities(>2 mm) are determined..

4. Definitions

For the purpose of this standard the definitions given in PD CR 13456, EN 12579, EN13040 and PAS 100 apply.

5. Reagents

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4.1.1.1.1.1 6 Apparatus

6.1 **Sieves,** diameter 200 mm or 300 mm 2, 5 and 20 mm apertures, ISO 3310-1:2000 or ISO 3310-2, 1999

6.2 **Analytical balance,** with an accuracy of 0.01 g

6.3 **Drying oven,** ventilated, fan assisted, capable of holding sample trays *80 ±3 °C.*

6.4 **Sample tray,** constructed of material thermally stable up to 150 °C, surface approximately 1250 cm^2

6.5 **Beaker,** 300 ml

6.6 **Tweezers**

6.7 Surface area meter
The plastics are spread as flat as possible on a transparent carrier like a sheet of foil and entered in a surface area meter such as the LICOR LI-3100 C.

6.8 Camera
The plastics are spread as flat as possible on a contrasting surface such as a sheet of bright blue paper of known dimensions. 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. First the parts around the sheet with the contrasting colour is 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) * (100-(percentage filled by background paper colour)/100).*

7 Procedure

7.1 Sample preparation

7.1.1 Prepare the test sample in accordance with EN 13040:1999, clause 8.1, 8.2. Where 20% w/w or less of the laboratory sample has been retained the procedure can be continued. If not the method is not appropriate.

NOTE larger quantities may be required for very coarse samples.

7.1.2 Determine the test amount of test sample depending on the coarseness of the sample. For 0-100 mm 7.5 l is taken, for a sample with a fraction 0-40 mm 3 l is taken, for a sample with a fraction 0-25 mm 1.5 l is taken and for fine materials 0-12 mm 1 l is taken and put in the sample tray **(6.4)**.

NOTE the method is performed in duplicate.

7.1.3 Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3)**.

7.1.4 Determine the dry weight with the balance **(6.2)**.

7.2 Sieving

Using the beaker **(6.5),** transfer portions of 100 ml or less of the dried sample (7.1.2) onto the 20 mm sieve **(6.1)**. Spread the >20 mm fractions one by one on a flat surface and gather the plastic particles > 20 mm with help of the tweezers **(6.6)**. Continue this procedure until the entire sample (7.1.2) has been sieved. Determine the total weight of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using the balance **(6.2)**. *Determine the total surface area of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using a surface area meter **(6.7)** or a camera **(6.8)**.*

Recombine the fractions < 20 mm and > 20 mm without the plastics > 20 mm. Transfer portions of 100 ml or less on the 5 mm sieve **(6.1)**. Spread the >5 mm fractions one by one on a flat surface and gather the stones > 5 mm with help of the tweezers **(6.6)**. Determine the weight of stones using the balance **(6.2)**.

Recombine the fractions <5 mm and >5 mm without the stones >5 mm. Sieve portions of 100 ml or less on the 2 mm sieve **(6.1)**. Spread the fractions >2 mm one by one on a flat surface and search out all visual recognisable impurities using the tweezers **(6.6)**. Sort out the following materials: stones, glass, rigid plastic, plastic light (flexible or film), metal. Determine the weight of the individual type of impurities using the balance **(6.2)**.

Thus the table below may be filled.

Table 1 Data recorded in the dry sieving for impurities

		weight In g	*Surface In cm^2*
> 20 mm	*Plastics rigid*	*y*	*y*
> 20 mm	*Plastics light*	*y*	*y*
> 5 mm	*Stones*	*y*	-
> 2 mm	*Stones*	*y*	-
> 2 mm	*Glass*	*y*	-
> 2 mm	*Plastics rigid*	*y*	*y*
> 2 mm	*Plastics light*	*y*	*y*
> 2 mm	*Metals*	*y*	-

8 Calculations and expression of results

The mass of the impurities is expressed on the total dry weight (before sieving). The average results are calculated of the duplicates.

$$I_{P>20\ mm} = \frac{W_{p>20\ mm}}{T} \times 100\%$$

$$I_{R>20\ mm} = \frac{W_{R>20\ mm}}{T} \times 100\%$$

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

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

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

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

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

Where

I is the impurity part (%)
W is weight of impurity type
T is the total dry weight
S is stones
G is glass
P is rigid plastic
R is plastic light (flexible or film)
M is metal

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 data

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

APPENDIX B BLEACH METHOD

Draft Standard

NOTE1 Where italics appear in the draft method this indicates an area that requires additional work and confirmation.

NOTE 2 Although the title of the method and body of the text states 'composted organic materials' it does not mean that the method may not be suitable for other forms of waste.

A method to determine the visual recognisable impurities in composted organic materials based on bleach washing

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Safety warning

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1. Scope and field of application

A method to determine the visual recognisable impurities in composted organic materials, soil improvers and growing media. The sample shall be obtained in accordance with SOIL IMPROVERS AND GROWING MEDIA - SAMPLING (EN 12579). The procedures described herein are only applicable to processed organic waste, sludge and soil.

2. Normative references

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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 test, determination of dry matter content, moisture content and laboratory compacted bulk density

3. Principle

After drying, the test material is bleach washed on a 2 mm sieve. The fraction > 2 mm is dried and the fractions of coarse stones (>5 mm) and plastics (>20 mm) and differentiated impurities (> 2 mm) are determined.

4. Definitions

For the purpose of this standard the definitions given in PD CR 13456, EN 12579, EN13040 and PAS 100 apply.

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 9,6 – 7,2 % (or 48º to 36º).

4.1.1.1.1.1.2 6 Apparatus

6.1 **Sieves,** diameter 200 mm or 300 mm 2, 5 and 20 mm apertures, ISO 3310-1:2000 or ISO 3310-2, 1999.

6.2 **Analytical balance,** with an accuracy of 0.01 g.

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

6.4 **Sample tray,** constructed of material thermally stable up to 150 ^{o}C, surface approximately 1250 cm^2.

6.5 **Beaker,** 300 ml.

6.6 **Tweezers.**

6.7 **Camera and graph paper,** the plastic films are spread and pasted on a sheet of graph paper (1 mm^2 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. 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 is 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) * (100-(percentage filled by background paper colour)/100).

6.8 **Container,** a 10 litre container of plastic.

7 Procedure

7.2 Sample preparation

7.1.5 Prepare the test sample in accordance with EN 13040:1999, clause 8.1, 8.2. Where 20% w/w or less of the laboratory sample has been retained the procedure can be continued. If not the method is not appropriate.

NOTE larger quantities may be required for very coarse samples.

7.1.6 Determine the test amount of test sample depending on the coarseness of the sample. For 0-100 mm 7.5 l is taken, for a sample with a fraction 0-40 mm 3 l is taken, for a

sample with a fraction 0-25 mm 1.5 l is taken and for fine materials 0-12 mm 1 l is taken and put in the sample tray **(6.4)**.

NOTE the method is performed in duplicate.

7.1.7 Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3)**.

7.1.8 Determine the dry weight with the balance **(6.2)**.

7.2 **Sieving and destruction of organic matter by bleach (2, 4 and 12 hours),** put portions of 500 ml or less of the dried material in a 10 litres container **(6.8)**. Put the container under an extractor hood to safely and continuously remove chlorine gasses and carbon dioxide formed. Cover the compost with 1-2 litres bleach **(5.1)** and blend. The chemical reaction is very quick and produces gasses, foresee possible overflows. Prevent the formation of a gaseous cake on the liquid. Leave the material for two hours in the bleach. Than pour the compost on a sieve with 2 mm meshes and let it drain (or wash briefly). Put the fraction > 2mm back into the container and bleach a second time i.e. cover the compost with bleach (1-2 litres) and blend. Leave the material for four hours in the bleach. Pour the compost on a sieve with 2 mm meshes and let it drain (or wash briefly). Put the fraction > 2mm back into the container and bleach a third time i.e. cover the compost with bleach (1-2 litres) and blend. Leave the material for twelve hours in the bleach. Pour the compost on a sieve with 2 mm meshes and rinse with hot water one last time what is on the sieve. Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3).**

Using the beaker **(6.5),** transfer portions of 100 ml or less of the dried sample (7.1.2) onto the 20 mm sieve **(6.1)**. Spread the >20 mm fractions one by one on a flat surface and gather the plastic particles > 20 mm with help of the tweezers **(6.6)**. Continue this procedure until the entire sample (7.1.2) has been sieved. Determine the total weight of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using the balance **(6.2)**. Determine the total surface area of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using graph paper and a camera **(6.7)**.

Recombine the fractions < 20 mm and > 20 mm without the plastics > 20 mm. Transfer portions of 100 ml or less on the 5 mm sieve (6.1). Spread the >5 mm fractions one by one on a flat surface and gather the stones > 5 mm with help of the tweezers (6.6). Determine the weight of stones using the balance (6.2).

Recombine the fractions <5 mm and >5 mm without the stones >5 mm. Spread the fractions >2 mm one by one on a flat surface and search out all visual recognisable impurities using the tweezers **(6.6)**. Sort out the following materials: stones, glass, rigid plastic, plastic light (flexible or film), metal. Determine the weight of the individual type of impurities using the balance **(6.2).**

Thus the table below may be filled.

Table 2 Data recorded in the dry sieving for impurities

		weight	Surface
		In g	In cm^2
> 20 mm	Plastics rigid	y	y
> 20 mm	Plastics light	y	y
> 5 mm	Stones	y	-
> 2 mm	Stones	y	-
> 2 mm	Glass	y	-
> 2 mm	Plastics rigid	y	y
> 2 mm	Plastics light	y	y
> 2 mm	Metals	y	-

10 Calculations and expression of results

The mass of the impurities is expressed on the total dry weight (before sieving). The average results are calculated of the duplicates.

$$I_{P>20\ mm} = \frac{W_{p>20\ mm}}{T} \times 100\%$$

$$I_{R>20\ mm} = \frac{W_{R>20\ mm}}{T} \times 100\%$$

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

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

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

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

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

Where

I is the impurity part (%)
W is weight of impurity type
T is the total dry weight
S is stones
G is glass
P is rigid plastic
R is plastic light (flexible or film)
M is metal

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

APPENDIX C PRESSURE WASHING METHOD

Draft Standard

NOTE1 Where italics appear in the draft method this indicates an area that requires additional work and confirmation.

NOTE 2 Although the title of the method and body of the text states 'composted organic materials' it does not mean that the method may not be suitable for other forms of waste.

A method to determine the visual recognisable impurities in composted organic materials *based on high pressure washing*

Contents **Page**

Safety warning

Care should be taken when handling samples that may contain sharp fragments, chemical contaminants or possible pathogenic organisms.

1. Scope and field of application

A method to determine the visual recognisable impurities in composted organic materials, soil improvers and growing media. The sample shall be obtained in accordance with SOIL IMPROVERS AND GROWING MEDIA - SAMPLING (EN 12579). The procedures described herein are only applicable to processed organic waste, sludge and soil.

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 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 test, determination of dry matter content, moisture content and laboratory compacted bulk density

3. Principle

After drying, the test material is high pressure washed on a 2 mm sieve. The fraction > 2 mm is dried and the fractions of coarse stones (>5 mm) and plastics (>20 mm) and differentiated impurities (> 2 mm) are determined.

4. Definitions

For the purpose of this standard the definitions given in PD CR 13456, EN 12579, EN13040 and PAS 100 apply.

5. Reagents

-

4.1.1.1.1.3 6 Apparatus

7.1 **Sieves,** diameter 200 mm or 300 mm 2 *(2 of these),* 5 and 20 mm apertures, ISO 3310-1:2000 or ISO 3310-2, 1999

7.2 **Analytical balance,** with an accuracy of 0.01 g

7.3 **Drying oven,** ventilated, fan assisted, capable of holding sample trays *80 ±3 °C.*

7.4 **Sample tray,** constructed of material thermally stable up to 150 °C, surface approximately 1250 cm^2

7.5 **Beaker,** 300 ml

7.6 **Tweezers**

7.7 Surface area meter
The plastics are spread as flat as possible on a transparent carrier like a sheet of foil and entered in a surface area meter such as the LICOR LI-3100 C.

7.8 Camera
The plastics are spread as flat as possible on a contrasting surface such as a sheet of bright blue paper of known dimensions. 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. First the parts around the sheet with the contrasting colour is 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) * (100-(percentage filled by background paper colour)/100).*

7.9 High pressure cleaner
A light commercially available high pressure cleaner such as Karcher HD 640 S is used with a working pressure of 30-200 bar (approx. 3-20 MPa) and a flow of 250-750 l/hour or preferably 10 MPa and 500 litre per hour. The water jet is not to be fully concentrated but to spread out by a slit type nozzle. The working distance from the sieves is 20-80 cm.

8 Procedure

7.3 Sample preparation

7.1.9 Prepare the test sample in accordance with EN 13040:1999, clause 8.1, 8.2. Where 20% w/w or less of the laboratory sample has been retained the procedure can be continued. If not the method is not appropriate.

NOTE larger quantities may be required for very coarse samples.

7.1.10 Determine the test amount of test sample depending on the coarseness of the sample. For 0-100 mm 7.5 l is taken, for a sample with a fraction 0-40 mm 3 l is taken, for a

sample with a fraction 0-25 mm 1.5 l is taken and for fine materials 0-12 mm 1 l is taken and put in the sample tray **(6.4)**.

NOTE the method is performed in duplicate.

7.1.11 Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3)**.

7.1.12 Determine the dry weight with the balance **(6.2)**.

7.2 Sieving and high pressure washing

*Top the sieve (6.1) with another sieve **(6.1)** of 2 mm to prevent material from jumping out of the sieves. Pressure wash with a high pressure cleaner **(6.9).** Wash until all smaller parts are removed. Sieve in portions of max. 1 cm thickness on the lower 2 mm sieve **(6.1).** Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3**).*

*Bring the dried material > 2mm in portions of 100 ml or less onto the 20 mm sieve **(6.1)**. Spread the >20 mm fractions one by one on a flat surface and gather the plastic particles > 20 mm with help of the tweezers **(6.6)**. Determine the total weight of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using the balance **(6.2)**. Determine the total surface area of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using a surface area meter **(6.7)** or a camera **(6.8)**.*

*Recombine the fractions < 20 mm and > 20 mm without the plastics > 20 mm. Transfer portions of 100 ml or less on the 5 mm sieve **(6.1)**. Spread the >5 mm fractions one by one on a flat surface and gather the stones > 5 mm with help of the tweezers **(6.6)**. Determine the weight of stones using the balance **(6.2)**.*

*Recombine the fractions <5 mm and >5 mm without the stones >5 mm. Spread the fractions (which are >2 mm) one by one on a flat surface and search out all visual recognisable impurities using the tweezers **(6.6)**. Sort out the following materials: stones, glass, rigid plastic, plastic light (flexible or film), metal. Determine the weight of the individual type of impurities using the balance **(6.2).***

Thus the table below may be filled.

Table 3 Data recorded in the dry sieving for impurities

		weight	*Surface*
		In g	*In cm^2*
> 20 mm	*Plastics rigid*	*y*	*y*
> 20 mm	*Plastics light*	*y*	*y*
> 5 mm	*Stones*	*y*	*-*
> 2 mm	*Stones*	*y*	*-*
> 2 mm	*Glass*	*y*	*-*
> 2 mm	*Plastics rigid*	*y*	*y*
> 2 mm	*Plastics light*	*y*	*y*
> 2 mm	*Metals*	*y*	*-*

12 Calculations and expression of results

The mass of the impurities is expressed on the total dry weight (before sieving). The average results are calculated of the duplicates.

$$I_{P>20\ mm} = \frac{W_{p>20\ mm}}{T} \times 100\%$$

$$I_{R>20\ mm} = \frac{W_{R>20\ mm}}{T} \times 100\%$$

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

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

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

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

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

Where

I is the impurity part (%)
W is weight of impurity type
T is the total dry weight
S is stones
G is glass
P is rigid plastic
R is plastic light (flexible or film)
M is metal

13 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 data

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 1 decimal place

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

APPENDIX D LABRESULTS AND STATISTICS

Table 1 A Original weight data from all labs all samples all methods

LAB	method	sample	rep	Dry weight	plastic rigid >20mm (g)	plastic soft >20mm (g)	stones >5mm (g)	stones >2mm (g)	glass >2mm (g)	plastic rigid >2mm (g)	plastic soft >2mm (g)	metal >2mm (g)	plastic soft >20mm (cm2)	plastic rigid >20mm (cm2)	plastic soft >2mm (cm2)	plastic rigid >2mm (cm2)
L3	dry sieving	A	1	2135.20			295.26	138.85			0.00				0.18	
L3	dry sieving	A	2	2131.50			284.85	96.51			0.00				0.02	
L3	dry sieving	B	1	536.84			14.96	2.74	7.44	1.77	1.09				43.48	18.01
L3	dry sieving	B	2	567.68		0.61	18.99	2.39	3.78	2.95	0.43				41.95	17.00
L3	dry sieving	C	1	1829.50			80.55	100.87	0.20	2.38	0.84	0.36			15.31	13.39
L3	dry sieving	C	2	1857.20			78.02	107.48		3.13	0.34	0.21			7.29	8.51
L3	dry sieving	D	1	495.36			9.12	1.71	1.78	0.38	0.79		21.00		67.48	6.36
L3	dry sieving	D	2	500.52			10.73	2.36	1.01	0.42	0.89				87.34	6.31
L3	pressure washing	A	1	2139.20			98.64	23.88	0.07		0.00				0.84	
L3	pressure washing	A	2	2130.50			100.61	25.46			0.00				0.15	
L3	pressure washing	B	1	534.50			10.97	0.80	6.45		0.60				45.40	21.11
L3	pressure washing	B	2	539.10		0.41	20.56	1.50	9.89	3.54	0.35	8.85			27.17	19.93
L3	pressure washing	C	1	1848.10			38.13	58.38	2.78	0.20	0.15	0.00			9.06	1.59
L3	pressure washing	C	2	1833.20			38.29	49.88	0.36	1.94	0.05				0.07	8.70
L3	pressure washing	D	1	496.40			9.62	2.34	2.01	0.57	0.83		27.19		119.24	10.04
L3	pressure washing	D	2	490.10			7.96	2.32	0.46	0.63	0.76				91.00	6.90
L3	bleach	A	1	2122.70			104.55	24.98	0.79		0.00				0.14	
L3	bleach	A	2	2133.30			127.62	24.46	0.01	0.01	0.03				2.18	
L3	bleach	B	1	563.39			31.23	9.22	3.34	1.95	0.37	0.01			25.23	27.66
L3	bleach	B	2	550.79		1.37	31.04	9.07	8.97	0.99	0.28				24.04	6.80
L3	bleach	C	1	1842.10			38.89	48.97	0.73		0.05	0.03			1.40	
L3	bleach	C	2	1828.50			50.70	45.29	0.78	1.07	0.05	0.38			0.68	10.36
L3	bleach	D	1	491.90			3.79	6.25	2.02	0.62	1.43	0.24		3.53	128.76	11.11
L3	bleach	D	2	499.31			7.60	6.07	1.92	0.69	1.26	0.19			132.01	6.64

Table 1 B Original weight data from all labs all samples all methods

LAB	method	sample	rep	Dry weight	plastic rigid >20mm (g)	plastic soft >20mm (g)	stones >5mm (g)	stones >2mm (g)	glass >2mm (g)	plastic rigid >2mm (g)	plastic soft >2mm (g)	metal >2mm (g)	plastic soft >20mm (cm2)	plastic rigid >20mm (cm2)	plastic soft >2mm (cm2)	plastic rigid >2mm (cm2)
L1	dry sieving	A	1	2124.50	0.00	0.00	88.80	103.75	0.00	0.00	0.02	0.00				
L1	dry sieving	A	2	2125.30	0.00	0.01	240.99	284.13	0.00	0.00	0.02	0.21				
L1	dry sieving	B	1	533.20	0.00	0.00	1.23	1.67	7.04	1.74	0.44	0.14				
L1	dry sieving	B	2	537.50	0.00	0.13	11.51	13.04	10.01	1.92	0.62	0.34				
L1	dry sieving	C	1	1865.20	0.00	0.03	41.60	72.22	0.83	0.00	0.03	0.00				
L1	dry sieving	C	2	1828.20	0.00	0.08	44.37	95.68	1.49	0.57	0.13	1.35				
L1	dry sieving	D	1	494.38	0.00	0.05	5.75	6.81	0.48	0.56	0.31	0.23				
L1	dry sieving	D	2	500.30	0.00	0.05	7.21	10.03	1.09	1.70	1.30	0.41				
L1	pressure washing	A	1	2125.50	0.00	0.00	174.78	226.36	0.00	0.00	0.00	0.00				
L1	pressure washing	A	2	2123.00	0.00	0.03	69.59	99.17	2.21	0.00	0.04	0.95				
L1	pressure washing	B	1	584.10	0.00	0.00	18.18	21.02	0.20	0.48	1.17	0.01				
L1	pressure washing	B	2	542.80	2.43	0.63	18.03	24.46	13.39	5.50	1.50	0.84				
L1	pressure washing	C	1	1835.20	0.00	0.00	113.29	134.75	0.21	0.11	0.17	0.65				
L1	pressure washing	C	2	1846.70	0.00	0.06	67.19	117.71	2.27	1.26	0.09	0.99				
L1	pressure washing	D	1	501.10	0.00	0.00	13.47	15.90	0.37	0.23	1.47	0.05				
L1	pressure washing	D	2	502.40	0.00	0.00	5.05	9.27	0.64	1.66	0.94	0.72				
L1	bleach	A	1	501.50	0.00	0.00	33.98	44.14	0.00	0.00	0.00	0.00				
L1	bleach	A	2	2129.50	0.00	0.01	107.05	126.08	0.02	0.14	0.01	0.75				
L1	bleach	B	1	504.00	0.00	0.19	12.60	19.52	4.53	0.77	0.94	2.92				
L1	bleach	B	2	533.20	0.00	0.03	10.97	15.54	6.41	3.91	0.45	0.09				
L1	bleach	C	1	502.40	0.00	0.00	28.32	46.69	2.13	0.00	0.00	0.00				
L1	bleach	C	2	1857.10	0.00	0.06	41.56	83.10	0.92	1.97	0.06	2.77				
L1	bleach	D	1	499.30	0.00	0.00	11.40	17.73	0.51	0.57	1.07	0.28				
L1	bleach	D	2	509.90	0.00	0.05	6.11	6.59	6.95	1.93	0.63	0.26				

Table 1 C Original weight data from all labs all samples all methods

LAB	method	sample	rep	Dry weight	plastic rigid >20mm (g)	plastic soft >20mm (g)	stones >5mm (g)	stones >2mm (g)	glass >2mm (g)	plastic rigid >2mm (g)	plastic soft >2mm (g)	metal >2mm (g)	plastic soft >20mm (cm2)	plastic rigid >20mm (cm2)	plastic soft >2mm (cm2)	plastic rigid >2mm (cm2)
L2	dry sieving	A	1	1660.30	0.00	0.00	8.87		0.00	0.24	0.02	0.00				
L2	dry sieving	A	2	1490.73	0.00	0.00	2.24		0.00	0.08	0.01	0.00				
L2	dry sieving	B	1	967.40	0.00	0.00	7.45		11.04	0.50	0.54	0.00				
L2	dry sieving	B	2	562.00	0.00	0.00	25.31		3.93	0.73	0.27	0.00				
L2	dry sieving	C	1	1360.07	0.00	0.00	3.06		1.05	0.05	0.09	0.00				
L2	dry sieving	C	2	1590.12	0.00	0.00	4.20		2.02	0.75	0.00	0.00				
L2	dry sieving	D	1	409.25	0.00	0.00	1.88		2.44	0.09	0.45	0.00				
L2	dry sieving	D	2	505.51	0.00	0.00	2.94		0.78	0.35	0.79	0.00				
L2	pressure washing	A	1	1541.00	0.00	0.00	81.99		1.05	0.03	0.01	0.00				
L2	pressure washing	A	2	1632.00	0.00	0.00	102.56		0.44	0.00	0.00	0.00				
L2	pressure washing	B	1	573.29	0.00	0.00	1.67		9.15	1.69	0.81	0.50				
L2	pressure washing	B	2	573.31	0.00	0.00	6.50		17.29	0.93	1.11	0.00				
L2	pressure washing	C	1	1552.90	0.00	0.00	35.61		1.23	0.01	0.05	0.05				
L2	pressure washing	C	2	1589.45	0.00	0.00	62.68		0.49	0.13	0.13	0.22				
L2	pressure washing	D	1	501.10	0.00	0.00	13.47		0.37	0.23	1.47	0.05				
L2	pressure washing	D	2	501.10	0.00	0.00	13.47		0.37	1.23	1.47	1.05				
L2	bleach	A	1	1052.20	0.00	0.00	94.97		0.00	0.00	0.02	0.00				
L2	bleach	A	2	1023.70	0.00	0.00	36.90		0.01	0.00	0.03	0.00				
L2	bleach	B	1	513.96	0.00	0.00	10.19		1.95	1.19	1.19	0.76				
L2	bleach	B	2	508.90	0.00	0.00	3.82		0.51	2.19	0.71	1.43				
L2	bleach	C	1	1390.80	0.00	0.00	59.16		4.33	1.56	0.15	0.00				
L2	bleach	C	2	1331.00	0.00	0.00	45.86		3.89	0.99	0.10	0.00				
L2	bleach	D	1	489.72	0.00	0.00	14.34		10.86	1.37	0.31	0.55				
L2	bleach	D	2	506.93	0.00	0.00	9.76		4.22	0.19	0.21	1.22				

Table 1 D Original weight data from all labs all samples all methods

LAB	method	sample	rep	Dry weight	plastic rigid >20mm (g)	plastic soft >20mm (g)	stones >5mm (g)	stones >2mm (g)	glass >2mm (g)	plastic rigid >2mm (g)	plastic soft >2mm (g)	metal >2mm (g)	plastic soft >20mm (cm2)	plastic rigid >20mm (cm2)	plastic soft >2mm (cm2)	plastic rigid >2mm (cm2)
L4	dry sieving	A	1	1597.10	0.00	0.02	257.70	126.40	0.00	0.00	0.02	0.00				
L4	dry sieving	A	2	1600.50	0.00	0.02	398.10	66.90	0.00	0.00	0.02	0.00				
L4	dry sieving	B	1	371.10	0.00	0.03	8.40	2.49	3.87	2.76	1.11	0.27				
L4	dry sieving	B	2	406.00	0.00	0.03	15.69	0.92	5.45	1.55	0.20	1.01				
L4	dry sieving	C	1	1297.90	0.00	0.08	25.77	24.10	1.25	1.15	0.16	0.25				
L4	dry sieving	C	2	1369.10	0.00	0.05	29.30	29.33	0.95	0.09	0.05	0.52				
L4	dry sieving	D	1	391.70	0.00	0.08	7.70	1.10	1.70	0.38	0.92	0.00				
L4	dry sieving	D	2	375.30	0.00	0.06	5.30	1.00	0.12	0.46	1.22	0.00				
L4	pressure washing	A	1	1667.19	0.00	0.01	112.45	25.30	0.03	0.00	0.00	0.11				
L4	pressure washing	A	2	1738.04	0.00	0.02	68.48	25.13	1.94	0.00	0.00	0.30				
L4	pressure washing	B	1	403.39	0.00	1.11	12.53	3.54	1.06	1.89	0.22	0.28				
L4	pressure washing	B	2	505.32	0.00	0.03	16.53	2.81	12.04	3.06	0.16	2.04				
L4	pressure washing	C	1	1377.69	0.00	0.04	23.26	39.45	0.14	0.76	0.00	0.39				
L4	pressure washing	C	2	1434.11	0.00	0.05	38.53	44.00	0.17	0.97	0.00	1.11				
L4	pressure washing	D	1	396.46	0.00	0.05	6.40	0.96	1.25	0.46	1.05	0.03				
L4	pressure washing	D	2	387.30	0.00	0.05	2.95	1.80	1.34	0.53	0.90	0.13				
L4	bleach	A	1													
L4	bleach	A	2													
L4	bleach	B	1													
L4	bleach	B	2													
L4	bleach	C	1													
L4	bleach	C	2													
L4	bleach	D	1													
L4	bleach	D	2													

Table 2 Averaged impurities in % w/w, per method and per lab

	Bleach			Pressure washing				Dry sieving			
Averages	L1	L2	L3	L1	L2	L3	L4	L1	L2	L3	L4
%plastic rigid >20mm (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.00	0.00	0.00
%plastic rigid >2mm (g)	0.19	0.15	0.11	0.15	0.04	0.16	0.18	0.19	0.09	0.13	0.18
%plastic soft >20mm (g)	0.01	0.00	0.03	0.01	0.00	0.01	0.01	0.02	0.00	0.01	0.04
%plastic soft >2mm (g)	0.08	0.06	0.08	0.07	0.05	0.09	0.12	0.12	0.12	0.06	0.07
%stones >5mm (g)	3.46	3.49	3.66	3.14	0.94	5.74	6.83	3.93	3.08	2.86	2.98
%stones >2mm (g)	5.02	*	1.65	4.18	*	3.01	2.19	5.27	*	1.20	1.38
%glass >2mm (g)	0.51	0.52	0.39	0.45	0.35	0.33	0.38	0.37	0.62	0.46	0.43
%metal >2mm (g)	0.11	0.10	0.01	0.04	0.00	0.00	0.05	0.06	0.04	0.21	0.08
plastic rigid >20mm (cm2/g*100)	*	*	0.09	*	*	0.00	*	*	*	0.00	*
plastic rigid >2mm (cm2/g*100)	*	*	1.29	*	*	1.26	*	*	*	1.45	*
plastic soft >20mm (cm2/g*100)	*	*	0.00	*	*	0.53	*	*	*	0.68	*
soft >2mm (cm2/g*100)	*	*	7.71	*	*	5.98	*	*	*	7.08	*

Table 3 Standard deviation on impurities in % w/w, per method and per lab

	Bleach			Pressure washing				Dry sieving			
Standard deviation	L1	L2	L3	L1	L2	L3	L4	L1	L2	L3	L4
%plastic rigid >20mm (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.16	0.00	0.00	0.00
%plastic rigid >2mm (g)	0.25	0.15	0.12	0.17	0.04	0.18	0.26	0.35	0.12	0.22	0.23
%plastic soft >20mm (g)	0.01	0.00	0.09	0.01	0.00	0.04	0.01	0.04	0.00	0.03	0.10
%plastic soft >2mm (g)	0.09	0.08	0.12	0.09	0.06	0.08	0.14	0.13	0.13	0.07	0.11
%stones >5mm (g)	2.04	2.50	2.09	3.50	1.46	4.93	8.79	2.24	2.02	1.29	1.84
%stones >2mm (g)	2.82	*	0.60	4.08	*	2.80	2.68	2.79	*	1.14	1.08
%glass >2mm (g)	0.57	0.74	0.55	0.72	0.42	0.49	0.53	0.85	1.11	0.69	0.80
%metal >2mm (g)	0.20	0.12	0.02	0.03	0.00	0.01	0.09	0.06	0.07	0.58	0.13
plastic rigid >20mm (cm2/g*100)	*	*	0.25	*	*	0.00	*	*	*	0.00	*
plastic rigid >2mm (cm2/g*100)	*	*	1.67	*	*	1.28	*	*	*	1.63	*
plastic soft >20mm (cm2/g*100)	*	*	0.00	*	*	1.50	*	*	*	1.94	*
soft >2mm (cm2/g*100)	*	*	11.64	*	*	6.82	*	*	*	9.40	*

Table 1 Variate: %plastic_rigid_20mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.007053	0.002351	1.02	0.389
Methode	2	0.004916	0.002458	1.07	0.350
LAB.methode	5(1)	0.012180	0.002436	1.06	0.391
Monster	3	0.006263	0.002088	0.91	0.443
Methode.monster	6	0.012526	0.002088	0.91	0.495
Residual	68(7)	0.156575	0.002303		
Total	87(8)	0.198139			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.0000	0.0000	0.0560
L2		0.0000	0.0000	0.0000
L4		-0.0086	0.0000	0.0000
L3		0.0000	0.0000	0.0000

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		-0.0022	-0.0022	-0.0022	-0.0022
dry sieving		0.0000	0.0000	0.0000	0.0000
pressure washing		0.0000	0.0560	0.0000	0.0000

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.02764	0.02394	0.04788	0.02764	0.04788

Table 1 Variate: %plastic_soft_20mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.008624	0.002875	1.71	0.173
methode	2	0.001896	0.000948	0.56	0.572
LAB.methode	5(1)	0.004586	0.000917	0.55	0.741
monster	3	0.029347	0.009782	5.82	0.001
methode.monster	6	0.005920	0.000987	0.59	0.740
Residual	68(7)	0.114313	0.001681		
Total	87(8)	0.159083			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.0072	0.0063	0.0151
L2		0.0000	0.0000	0.0000
L4		0.0290	0.0081	0.0391
L3		0.0311	0.0134	0.0095

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		0.0042	0.0527	0.0046	0.0057
dry sieving		0.0004	0.0185	0.0019	0.0070
pressure washing		0.0004	0.0591	0.0012	0.0030

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.02362	0.02045	0.04091	0.02362	0.04091

Table 1Variate: %stones_5mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	78.216	26.072	3.83	0.014
methode	2	15.811	7.906	1.16	0.320
LAB.methode	5(1)	111.790	22.358	3.28	0.010
monster	3	444.667	148.222	21.75	<.001
methode.monster	6	123.435	20.572	3.02	0.011
Residual	68(7)	463.445	6.815		
Total	87(8)	1202.845			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		3.46	3.14	3.93
L2		3.49	0.94	3.08
L4		5.16	6.83	2.98
L3		3.66	5.74	2.86

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		6.30	3.48	3.81	2.18
dry sieving		10.55	2.49	2.23	1.38
pressure washing		5.39	2.51	3.07	1.88

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	1.504	1.302	2.605	1.504	2.605

Table 1 Variate: %stones_2mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	286.882	95.627	34.33	<.001
methode	2	2.439	1.219	0.44	0.647
LAB.methode	5(1)	19.615	3.923	1.41	0.232
monster	3	130.475	43.492	15.61	<.001
methode.monster	6	36.875	6.146	2.21	0.053
Residual	68(7)	189.412	2.785		
Total	87(8)	659.192			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		5.02	4.18	5.27
L2		0.00	0.00	0.00
L4		1.73	2.19	1.38
L3		1.65	3.01	1.20

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		2.72	1.56	3.03	1.10
dry sieving		5.17	0.57	3.05	0.59
pressure washing		2.57	1.25	3.19	0.83

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.961	0.833	1.665	0.961	1.665

Table 1 Variate: %glass_2mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.1353	0.0451	0.22	0.880
methode	2	0.1765	0.0882	0.44	0.647
LAB.methode	5(1)	0.2948	0.0590	0.29	0.916
monster	3	20.6984	6.8995	34.22	<.001
methode.monster	6	4.8167	0.8028	3.98	0.002
Residual	68(7)	13.7114	0.2016		
Total	87(8)	38.6873			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.505	0.453	0.368
L2		0.517	0.349	0.621
L4		0.445	0.377	0.430
L3		0.392	0.328	0.464

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		0.000	0.794	0.187	0.878
dry sieving		0.000	1.183	0.063	0.262
pressure washing		0.039	1.600	0.055	0.189

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.2587	0.2240	0.4480	0.2587	0.4480

Table 1 Variate: %plastic_rigid_2mm_g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.12815	0.04272	1.98	0.125
methode	2	0.01109	0.00555	0.26	0.774
LAB.methode	5(1)	0.05690	0.01138	0.53	0.755
monster	3	1.91795	0.63932	29.64	<.001
methode.monster	6	0.03291	0.00549	0.25	0.956
Residual	68(7)	1.46660	0.02157		
Total	87(8)	3.45137			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.186	0.146	0.193
L2		0.146	0.043	0.095
L4		0.199	0.180	0.181
L3		0.106	0.164	0.127

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		0.014	0.359	0.072	0.192
dry sieving		0.002	0.355	0.059	0.116
pressure washing		0.000	0.410	0.040	0.146

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.0846	0.0733	0.1465	0.0846	0.1465

Table 1ariate: %plastic soft 2mm g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.004452	0.001484	0.46	0.710
methode	2	0.005427	0.002714	0.85	0.434
LAB.methode	5(1)	0.039470	0.007894	2.46	0.042
monster	3	0.646577	0.215526	67.13	<.001
methode.monster	6	0.016256	0.002709	0.84	0.541
Residual	68(7)	0.218309	0.003210		
Total	87(8)	0.888386			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.0767	0.0664	0.1213
L2		0.0624	0.0473	0.1165
L4		0.0852	0.1159	0.0730
L3		0.0833	0.0850	0.0638

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		0.0040	0.1292	0.0073	0.1670
dry sieving		0.0007	0.1163	0.0120	0.1858
pressure washing		0.0003	0.1342	0.0046	0.2355

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.03264	0.02827	0.05653	0.03264	0.05653

Table 1 Variate: %metal 2mm g

Source of variation	d.f.(m.v.)	s.s.	m.s.	v.r.	F pr.
LAB	3	0.01141	0.00380	0.11	0.956
methode	2	0.09190	0.04595	1.29	0.283
LAB.methode	5(1)	0.18201	0.03640	1.02	0.414
monster	3	0.40411	0.13470	3.77	0.015
methode.monster	6	0.16938	0.02823	0.79	0.581
Residual	68(7)	2.42944	0.03573		
Total	87(8)	3.25300			

Table 2 Labs * method

LAB	methode	bleach	dry sieving	pressure washing
L1		0.111	0.038	0.055
L2		0.098	0.000	0.040
L4		0.081	0.047	0.081
L3		0.014	0.004	0.205

Table 3 Method * samples

methode	monster	A	B	C	D
bleach		0.008	0.173	0.030	0.093
dry sieving		0.001	0.051	0.020	0.016
pressure washing		0.009	0.295	0.027	0.052

Table 4 Least significant differences of means (5% level)

Table	LAB	methode	LAB methode	monster	methode monster
rep.	24	32	8	24	8
d.f.	68	68	68	68	68
l.s.d.	0.1089	0.0943	0.1886	0.1089	0.1886

APPENDIX E METHOD FOR IMPURITY MEASUREMENT

Draft Standard

NOTE1 Where italics appear in the draft method this indicates an area that requires additional work and confirmation.

NOTE 2 Although the title of the method and body of the text states 'composted organic materials' it does not mean that the method may not be suitable for other forms of waste.

A method to determine the visual recognisable impurities in composted organic materials based on bleach washing

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Safety warning

Care should be taken when handling samples that may contain sharp fragments, chemical contaminants or possible pathogenic organisms.

1. Scope and field of application

A method to determine the visual recognisable impurities in composted organic materials, soil improvers and growing media. The sample shall be obtained in accordance with SOIL IMPROVERS AND GROWING MEDIA - SAMPLING (EN 12579). The procedures described herein are only applicable to processed organic waste, sludge and soil.

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 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 test, determination of dry matter content, moisture content and laboratory compacted bulk density

3. Principle

After drying, the test material is bleach washed on a 2 mm sieve. The fraction > 2 mm is dried and the fractions of coarse stones (>5 mm) and plastics (>20 mm) and differentiated impurities (> 2 mm) are determined.

4. Definitions

For the purpose of this standard the definitions given in PD CR 13456, EN 12579, EN13040 and PAS 100 apply.

5. Reagents

5.1 **Bleach, t**he 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 9,6 – 7,2 % (or 48º to 36º).

5.2 **Water,** normal drinking water quality tap water or purer.

4.1.1.1.1.1.4 6 Apparatus

7.9 **Sieves,** diameter 200 mm or 300 mm 2, 5 and 20 mm apertures, ISO 3310-1:2000 or ISO 3310-2, 1999.

7.10 **Analytical balance,** with an accuracy of 0.01 g.

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

7.12 **Sample tray,** constructed of material thermally stable up to 150 ^{o}C, surface approximately 1250 cm^{2}.

7.13 **Beaker,** 300 ml.

7.14 **Tweezers.**

7.15 **Camera and graph paper,** the plastic films are spread and pasted on a sheet of graph paper (1 mm^{2} 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) * (100-(percentage filled by background paper colour)/100).

7.16 **Container,** a 10 litre container of plastic.

7.17 **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.

8 Procedure

7.4 Sample preparation

7.1.13 Prepare the test sample in accordance with EN 13040:1999, clause 8.1, 8.2. Where 20% w/w or less of the laboratory sample has been retained the procedure can be continued. If not the method is not appropriate.

NOTE larger quantities may be required for very coarse samples.

7.1.14 Determine the test amount of test sample depending on the coarseness of the sample. For 0-100 mm 7.5 l is taken, for a sample with a fraction 0-40 mm 3 l is taken, for a sample with a fraction 0-25 mm 1.5 l is taken and for fine materials 0-12 mm 1 l is taken and put in the sample tray **(6.4)**.

NOTE the method is performed in duplicate.

7.1.15 Dry the materials for at least 16 hours until constant weight in the drying oven **(6.3)**.

7.1.16 Determine the dry weight with the balance **(6.2)**.

7.3 **Sieving and destruction of organic matter by bleach (2, 4 and 12 hours),**

7.1.1 **First washing.** Put portions of 500 ml or less of the dried material in a 10 litres container **(6.8)**. Put the container under an extractor hood to safely and continuously remove chlorine and carbon dioxide gasses formed. Cover the sample with 1-2 litres bleach **(5.1)** and mix with a glass rod **(6.9)**. The chemical reaction is exothermic, very quick and produces gasses. Foresee possible overflows and do not stir until the temperature is below 80 degrees Celsius. Prevent the formation of a gaseous cake on the liquid. Leave the material for two hours in the bleach. Than pour the sample on a sieve with 2 mm meshes and wash briefly with water.

7.1.2 **Second washing.** Put the fraction > 2mm **(7.1.1)** back into the container **(6.8)** and bleach a second time i.e. cover the sample with 1-2 litres bleach **(5.1)** and mix with a glass rod **(6.9)**. Leave the material for four hours in the bleach. Than pour the sample on a sieve with 2 mm meshes and wash briefly with water.

7.1.3 **Third washing.** Put the fraction > 2mm **(7.1.2)** back into the container **(6.8)** and bleach

a third time i.e. cover the sample with 1-2 litres bleach **(5.1)** and mix with a glass rod **(6.9)**. Leave the material for twelve hours in the bleach. Pour the sample on a sieve with 2 mm meshes and rinse with water one last time what is on the sieve.

7.1.4 **Drying.** Dry the materials **(7.1.3)** for at least 16 hours until constant weight in the drying oven **(6.3).**

7.1.5 **The 20 mm sieve.** Using the beaker **(6.5),** transfer portions of 100 ml or less of the dried sample **(7.1.4)** onto the 20 mm sieve **(6.1)**. Spread the >20 mm fractions one by one on a flat surface and gather the plastic particles > 20 mm with help of the tweezers **(6.6)**. Continue this procedure until the entire sample **(7.1.4)** has been sieved. Determine the total weight of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using the balance **(6.2)**. Determine the total surface area of the fraction rigid plastic and the fraction plastic light (flexible or film) individually using graph paper and a camera **(6.7)**.

7.1.6 **The 5 mm sieve.** Recombine the fractions < 20 mm and > 20 mm without the plastics > 20 mm **(7.1.5)**. Using the beaker **(6.5),** transfer portions of 100 ml or less of the recombined sample on to the 5 mm sieve **(6.1).** Spread the >5 mm fractions one by one on a flat surface and gather the stones > 5 mm with help of the tweezers **(6.6).** Determine the weight of stones using the balance **(6.2).**

7.1.7 **The 2 mm sieve.** Recombine the fractions <5 mm and >5 mm without the stones >5 mm **(7.1.6)**. Using the beaker **(6.5),** transfer portions of 100 ml or less of the recombined sample on to the 2 mm sieve **(6.1).**Spread the fractions >2 mm one by one on a flat surface and search out all visual recognisable impurities using the tweezers **(6.6)**. Sort out the following materials: stones, glass, rigid plastic, plastic light (flexible or film), metal. Determine the weight of the individual type of impurities using the balance **(6.2).**

NOTE For samples with a visibly low organic matter content.(or < 15.0 w% of the dry sample), washing the material for 5 minutes with water instead of bleach and without waiting time is allowed. If there is any doubt about the proper discrimination and classification of impurities, bleach washing should still be performed.

Thus the table below may be filled.

Table 4 Data recorded in the dry sieving for impurities

		weight	Surface
		In g	In cm^2
> 20 mm	Plastics rigid	y	y
> 20 mm	Plastics light	y	y
> 5 mm	Stones	y	-
> 2 mm	Stones	y	-
> 2 mm	Glass	y	-
> 2 mm	Plastics rigid	y	y
> 2 mm	Plastics light	y	y
> 2 mm	Metals	y	-

14 Calculations and expression of results

The mass of the impurities is expressed on the total dry weight (before sieving). The average results are calculated of the duplicates.

$$I_{P>20\ mm} = \frac{W_{p>20\ mm}}{T} \times 100\%$$

$$I_{R>20\ mm} = \frac{W_{R>20\ mm}}{T} \times 100\%$$

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

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

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

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

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

Where

I is the impurity part (%)
W is weight of impurity type
T is the total dry weight
S is stones
G is glass
P is rigid plastic
R is plastic light (flexible or film)
M is metal

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