Impurities and stone content

Validation of a method by eight laboratories

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Acknowledgement

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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. Negative aspects of a too high amount of impurities in compost which are to be avoided are the visual accumulation of impurities and the risk of injury on handling e.g. by glass shards.

The project Horizontal started in 2003 to unify several European tests regarding soils, bio-wastes and sludges. As part of the work package, impurities were discussed. After a desk study, a draft standard based on the German method for compost testing was proposed (Blok and Wever, 2005). The method was debated and finally it was decided to have three methods tested by five laboratories throughout Europe (Blok, 2006). This resulted in an adapted method (Blok, 2006b). This method was validated by eight laboratories and the results are presented in this report.

The main impurities to be characterised were the fraction of coarse stones >5 mm, glass>2 mm, metal>2 mm and plastic > 2mm expressed as weight and plastics > 2 mm expressed as area. The objective was to find the inner laboratory variance reported as repeatability and the inter laboratory variance reported as the reproducibility according to the ISO standard (ISO 5725-5). This was done in an inter laboratory trial with eight laboratories in five countries. Six samples were prepared in duplo and were sent to the laboratories involved.

Figure 1 Filling the sample bags prior to shipping

The results showed that the samples contained 0.5-5.0 % impurities by weigh. The standard variation between samples was 50-200% of the amount of impurities found (W/W). The outcomes varied a lot for samples with very low contents of specific impurities such as the classes “metal” or “others”. Although the amount of impurities per class was often below 0.01% of the total weigh, no minimum weigh of individual of impurities per impurity class was set. The water and bleach washing did not add any discriminating power in this test.

The repeatability is typically some 20% lower than the reproducibility. The reproducibility for the class “others > 2 mm” is much larger than the repeatability (about 140% and 350% respectively).

In conclusion the repeatability for stones > 5 mm, glass > 2 mm, others > 2 mm and plastics > 2 mm is put at 150%. For metals > 2mm it is put at 350%. The reproducibility for stones > 5 mm, glass > 2 mm and plastics > 2mm is put at 170%. For metals > 2mm and others > 2 mm it is put at 350%.The method is not very suitable for samples with very low numbers of specific impurities, especially if the incidental impurities are fairly large and thus cause outliers for the whole impurity class of that sample

1. INTRODUCTION

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. Negative aspects of a too high amount of impurities in compost which are to be avoided are the visual accumulation of impurities and the risk of injury on handling e.g. by glass shards. Figure 2 shows the visual effects of an impurity rich compost layer several months after the layer has been applied to a soil. Part of the organic material is decomposed and the impurities are washed clean by rains.

Figure 2 Glass, plastics and other impurities accumulating in a compost-treated plot.

1.2 Previous work within the Horizontal project

In a previous desk study a selection was made from several methods (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.

This preliminary method was debated and finally it was decided to have three methods tested by five laboratories throughout Europe (Blok, 2006).

The results of the tests were debated and concluded in an adapted method (Blok, 2006b).

This method was validated by eight laboratories and the results are presented in this report.

The main impurities to be characterised were 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 stones >5 mm, stone >2 mm, glass >2 mm, metal >2 mm and plastic >2 mm measured as weight and plastics >2 mm measured as 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.

1.3 Goal

The objective was to find the inner laboratory variance reported as repeatability and the inter laboratory variance reported as the reproducibility according to the CEN standard (CEN 15722).

1.4 Method and sampling

The action advised was to organise an inter laboratory trial with eight or more laboratories in various countries. Six samples were to be prepared in duplo and sent to the laboratories to test. The samples were prepared centrally to ensure uniform sub sampling. In the minutes of task group seven, the member states suggested that the samples should include one soil, one treated sludge (sludge cake) and 3-5 treated bio-wastes preferably green waste compost, half municipal/half green waste compost and municipal solid waste compost.

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
Wageningen-UR Greenhouse Horticulture, 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
SAS Laboratoire	270, Avenue de la pomme de pin, BP 10636, Ardon, 45166 Olivet CEDEX	G. Pousson	France	
Austrian Agency for Health and Food Safety Department for Fertilizer Control and Microscopy	Spargelfeldstrasse 191 A-1226 Vienna, Austria	F. Wernitznig E. Pfundner	Austria	Comparison
INFU mbH - Geschäftsbereich PlanCoTec	Karlsbrunnenstr. 11, D-37249 Neu Eichenberg	E. Marciniszyn	Germany	Comparison
Fachhochschule Weihenstephan, Forschungsanstalt für Gartenbau	85350 Freising	E. Meinken	Germany	
Finnish Food safety Authority Evira	Mustialankatu 3, 00790 Helsinki	A. Pelkonen	Finland	Comparison
Stichting RHP	Zuidweg 42, 2671 MN Naaldwijk	J. Verhagen	The Netherlands	
Provincie Limburg, Bureau Advies & Onderzoek	Postbus 5700, 6202 MA Maastricht	M. Severijnen J. Maurits	The Netherlands	Comparison
Wageningen-UR Greenhouse Horticulture, Rooting media laboratory	Kruisbroekweg 5, 2671 KT Naaldwijk	A. van Leeuwen	The Netherlands	Comparison
Wageningen-UR Greenhouse Horticulture, Rooting media laboratory	Kruisbroekweg 5, 2671 KT Naaldwijk	C. Blok	The Netherlands	Reporting

2.2 The samples

Member states had suggested including one soil, one treated sludge (sludge cake) and 3-5 treated bio-wastes preferably green waste compost, half municipal/half green waste compost and municipal solid waste compost. 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	Sandy soil with some organic matter		Naaldwijk
B*	Sewage sludge	Valk en de Groot	Poeldijk
C*	Horticultural green compost	Sortiva	Hoek v Holland
D*	Half municipal/half green waste compost***	Ster compost	Alphen a/d Rijn
E**	Horticultural green compost	Sortiva	Hoek v Holland
F**	Half municipal/half green waste compost***	Ster compost	Alphen a/d Rijn

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

** The materials received additional precise amounts of glass, metal and plastic (table 3)

*** The composition fluctuates with the season. This particular material contained two thirds of municipal waste versus on third of green waste

The samples were collected on the company sites from well within large piles by experienced sample takers. The material used was not fully processed (cleaned after composting) as previous experience had shown that the total amount of impurities in fully processed material was well below 1% in weight, making measurements rapidly highly variable.

The bulk density of each material was measured under standardized conditions (CEN 13041). Subsequently, all samples were, one by one, thoroughly homogenized using a drum mixer. The sub sample bags were filled with an equivalent weight of 1.5 litres, using the bulk densities previously measured.

Previous experience was that the variation within labs was high and depending on the amount of impurities present. It was therefore decided to repeat two samples (C and D) and to add precise and known amounts of glass, metal and plastic to them. Table 3 shows the nature of the added materials per 1.5 litre sample.

Table 3 Amounts of glass, metal and plastic added to two samples

Code	Glass added	Metal added	Plastic added*
E	4.95 gram +/- 0.1 gram	1.41 gram +/- 0.1 gram	30 cm^2 +/- 1.0 cm^2
F	4.95 gram +/- 0.1 gram	1.41 gram +/- 0.1 gram	30 cm^2 +/- 1.0 cm^2

* The weight of plastics was +/-1 gram, the total weigh of added impurities was 6.5 gram

Thus 6 materials x 2 repetitions x 11 laboratories = 131 samples were prepared and sent to the laboratories involved. Each laboratory received 12 samples. The labs were informed that all materials could be dry sieved but were asked to bleach wash sample F if they were equipped for that.

2.3 The method

See Appendix 1.

3. RESULTS AND DISCUSSION

3.1 General statistics

Full data may be found in Appendix 2-7. Table 4 shows the average total impurity content found for every sample. On average the labs measured 24.1 gram impurities in sample A, 77.7 gram in sample B, 5.8 gram in sample C and 3.9 gram in sample D. In samples E and F 6.5 gram extra impurities were the result of adding extra impurities to the materials C respectively D as reported in Table 3.

As could be expected the amount of stones in the A and B samples (soil respectively sewage sludge) was about 10 times higher than in the C and D samples which were composts.

The amount of glass was more or less equal in samples A, C and D and only twice as high in sample B. Sample B also contained about 10 times as much metal as the samples A, C and D.

The amount of other materials in sample B (sewage sludge) was about ten times higher than in the other samples presumably because many dirt objects are washed down into the sewer. It was composed of odd materials like shell fragments, wool, textile and ceramics. Furthermore some of the reported single elements like tar lumps were rather big (29 gram).

The amount of plastics in weigh was about ten times lower in A (soil) than in C and D (the composts). In B (sludge) the amount of plastic in weigh was twice that of the composts. The area of plastics was as expected unrelated to the weigh present. It shows virtually no foil present in sample A and most foil is in sample D which contains municipal compost.

Table 4 Average impurity content per impurity class and per sample in gram and also in cm^2, for the plastics only

Averages	A	B	C	D	E	F
stones >5mm (g)	21.56	54.90	3.85	1.10	3.24	0.96
glass >2mm (g)	0.37	1.17	0.53	0.52	5.21	5.47
metal >2mm (g)	0.26	2.75	0.29	0.17	1.69	1.62
others >2mm (g)	1.78	17.29	0.44	1.25	0.63	1.51
plastic >2mm (g)	0.08	1.62	0.70	0.83	1.14	1.23
Total of impurity weigh	24.05	77.74	5.81	3.87	11.91	10.79
Total sample weigh	1406	1887	538	504	543	512
plastic >2mm (cm^2)	1.50	24.55	23.73	44.98	48.01	60.12

Table 5 shows the standard deviation on the data reported on the different impurities defined. In general the deviations are large.

Table 5 Standard deviation per impurity class and per sample in gram, and also in cm^2 for plastics only

Averages	A	B	C	D	E	F
stones >5mm (g)	13.48	14.81	2.31	0.69	2.11	0.51
glass >2mm (g)	0.45	1.30	0.46	0.22	0.40	0.38
metal >2mm (g)	0.40	2.31	0.36	0.21	0.62	0.24
others >2mm (g)	3.02	30.04	0.40	1.41	0.56	1.81
plastic >2mm (g)	0.17	1.00	0.56	0.41	0.49	0.54
plastic >2mm (cm^2)	1.37	19.15	17.81	22.22	16.34	21.07

Table 6 shows the average total impurity content found for every sample. On average the labs measured 1.7 % impurities in sample A, 4.1 % in samples B, 1.0 % in sample C and 0.7 % in sample D. In samples E and F 1.3 % extra impurities were the result adding extra impurities to the materials C respectively D.

Table 6 Average impurity content per impurity class and per sample in % w/w of total impurities per sample and also in $cm^2.g^{-1}$, for the plastics only

Averages	A	B	C	D	E	F
stones >5mm (% W/W)	1.53	2.91	0.72	0.22	0.60	0.19
glass >2mm (% W/W)	0.023	0.062	0.085	0.097	0.963	1.069
metal >2mm (% W/W)	0.018	0.145	0.046	0.031	0.312	0.316
others >2mm (% W/W)	0.127	0.919	0.083	0.248	0.115	0.298
plastic >2mm (% W/W)	0.006	0.086	0.114	0.144	0.211	0.241
Totals of %	1.71	4.12	1.04	0.74	2.20	2.11
plastic >2mm ($cm^2.g^{-1}$)	0.001	0.013	0.044	0.089	0.088	0.118

Table 5 shows the standard deviation on the data reported on the different impurities defined. In general the deviations are large.

Table 7 Standard deviation per impurity class and per sample in % w/w of total impurities per sample, and also in $cm^2.g^{-1}$ for plastics only

Averages	A	B	C	D	E	F
stones >5mm (% W/W)	0.956	0.778	0.440	0.138	0.393	0.102
glass >2mm (% W/W)	0.031	0.069	0.087	0.048	0.088	0.079
metal >2mm (% W/W)	0.028	0.122	0.064	0.041	0.117	0.050
others >2mm (% W/W)	0.215	1.592	0.077	0.284	0.103	0.363
plastic >2mm (% W/W)	0.013	0.053	0.106	0.096	0.090	0.105
plastic >2mm ($cm^2.g^{-1}$)	0.001	0.010	0.034	0.045	0.031	0.041

Table 8 Standard deviation per impurity class and per sample in % of the specific impurity reported in %

Averages	A	B	C	D	E	F
stones >5mm (% W/W)	62%	27%	61%	63%	66%	54%
glass >2mm (% W/W)	135%	111%	102%	49%	9%	7%
metal >2mm (% W/W)	156%	84%	139%	132%	38%	16%
others >2mm (% W/W)	169%	173%	93%	115%	90%	122%
plastic >2mm (% W/W)	217%	62%	93%	67%	43%	44%
plastic >2mm ($cm^2.g^{-1}$)	100%	77%	77%	51%	35%	35%

In table 8 the standard deviation is expressed as a percentage of the absolute value of the impurity measured. Thus one can readily see which impurity shows uncommonly large deviations caused by incidental large objects. Sample B shows less variation in stones, but much higher variation in the class “others” caused by various objects such as tar lumps. It also becomes obvious that in sample A, some uncommonly large pieces of plastic have been found. Samples E and F resemble C respectively D for stones > 5 mm and others>2mm but are much smaller – because of the added fixed amounts- for glass, metal, plastics expressed in weigh and especially when the plastics are expressed in area.

3.2 Repeatability and reproducibility

Labs have suffered from incidental large objects which create a lot of variance between duplo's and between labs. Table 8 shows an overview of the repeatability and reproducibility found expressed in % of the mean. Typically repeatability is somewhat smaller than reproducibility as there are unavoidable (and avoidable) differences in execution of the procedure.

Table 9 Repeatability per impurity class and per sample in % of the specific impurity content

Averages	A	B	C	D	E	F
stones >5mm (% W/W)	124%	60%	168%	156%	88%	-
glass >2mm (% W/W)	-	106%	488%	118%	8%	-
metal >2mm (% W/W)	-	524%	604%	210%	32%	-
others >2mm (% W/W)	63%	107%	132%	139%	237%	-
plastic >2mm (% W/W)	-	153%	179%	92%	42%	-
plastic >2mm ($cm^2.g^{-1}$)	-	-	-	-	-	-

- No value is reported as either some labs did not report full data (lab 11 for column F), or the values reported were rejected as atypical for more than three labs.

Table 10 Reproducibility per impurity class and per sample in % of the specific impurity content

Averages	A	B	C	D	E	F
stones >5mm (% W/W)	166%	79%	179%	156%	106%	-
glass >2mm (% W/W)	-	106%	525%	118%	17%	-
metal >2mm (% W/W)	-	577%	664%	210%	33%	-
others >2mm (% W/W)	101%	153%	196%	1068%	237%	-
plastic >2mm (% W/W)	-	153%	185%	122%	110%	-
plastic >2mm ($cm^2.g^{-1}$)	-	-	-	-	-	-

- No value is reported as either some labs did not report full data (lab 11 for column F), or the values reported were rejected as atypical for more than three labs.

3.3 Experiences

Three of the laboratories were unable to start or at least conclude the tests in time. The surface area measurement proved problematic for one lab.

Labs confirmed that the sorting process with dry sieving was laborious but they favoured it over washing and bleach washing as more practicable. The amount of "other materials" in sample B, sludge, was reported qualitatively by most labs and caused some concern on the variation as caused by incidental large objects.

The area of added plastic found was 24 cm^2 for sample E and 15 cm^2 for sample F. As the area added was about 30 cm^2, this was 83% respectively 50%. The poor recovery in sample F may have been caused by obscuring by organic matter. It was also dependent on the laboratory staff. L2 and L3 were not able to reliably recover plastic in sample F. It is believed that washing or bleach washing would improve their performance.

The labs which bleached or washed sample F, L4 and L5 respectively, did not improve their recovery. It is therefore concluded that the preference of labs for dry sieving is justified if their staff is well trained.

4. CONCLUSIONS

1. The repeatability for stones > 5 mm, glass > 2 mm, others > 2 mm and plastics > 2mm is put at 150%. For metals > 2mm it is put at 350%. The repeatability will improve when an under limit for the impurity content per class is set. For stones > 5 mm, glass > 2 mm, others > 2 mm and plastics > 2mm an under limit of 0.15 % W/W is suggested. For metals > 2mm an under limit of 0.45% is suggested. With those under limits, the repeatability is put at 100%.

2. The reproducibility for stones > 5 mm, glass > 2 mm and plastics > 2mm is put at 170%. For metals > 2mm and others > 2 mm it is put at 350%. For stones > 5 mm, glass > 2 mm, others > 2 mm and plastics > 2mm an under limit of 0.15 % W/W is suggested. For metals > 2mm an under limit of 0.45% W/W is suggested. With those under limits, the reproducibility is put at 120%.

3. The repeatability is usually some 20% lower than the reproducibility.

4. The reproducibility for the class others > 2 mm is much larger than the repeatability (about 140% and 350% respectively). This indicates that some laboratories put more material in this class than others which seems to indicate a classification problem for some of the labs.

5. The method is not very suitable for samples with very low contents of specific impurities. In itself this is not a problem as such samples are acceptable for all purposes as foreseen in this standard. It does mean that the repeatability and reproducibility demands may only be met when the amounts of impurities are above an under limit. For practical help, safe under limits are suggested here, based on the data. For stones > 5 mm, glass > 2 mm, others > 2 mm and plastics > 2mm an under limit of 0.15 % W/W is suggested. For metals > 2mm an under limit of 0.45% is suggested. With those under limits, the repeatability and reproducibility may be set at 100% respectively 120%.

6. The method is not very suitable for samples with very low numbers of specific impurities, especially if the incidental impurities are fairly large and thus cause an outlyer for the whole impurity class of that sample. However, no requirement for a minimum number of individual of impurities per impurity class is set.

7. The water and bleach washing did not add any discriminating power in this test. It is maintained that washing or bleach washing may aid any untrained staff in discriminating between impurities obscured by either organic or mineral coatings.

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Appendix 1 The Method used (version per august 2006)

Soil, sludge and treated bio-waste - Determination of impurities and stones

Contents Page

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 bio-waste 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 bio-waste’ 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 bio-waste 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 bio-waste. 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.

4.1.1.1.1.1.1 **6 Apparatus**

6.1 **Sample tray,** constructed of material thermally stable up to 150 ^oC, surface approximately 1250 cm^2.

6.2 **Drying oven,** ventilated, fan assisted, capable of holding sample trays at 80 ±3 ^oC.

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** ^oC

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.

7.1.1 **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)**.

7.1.2 **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.

7.1.3 **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.**

7.1.1 **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.

7.1.2 **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 bio-waste.

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.

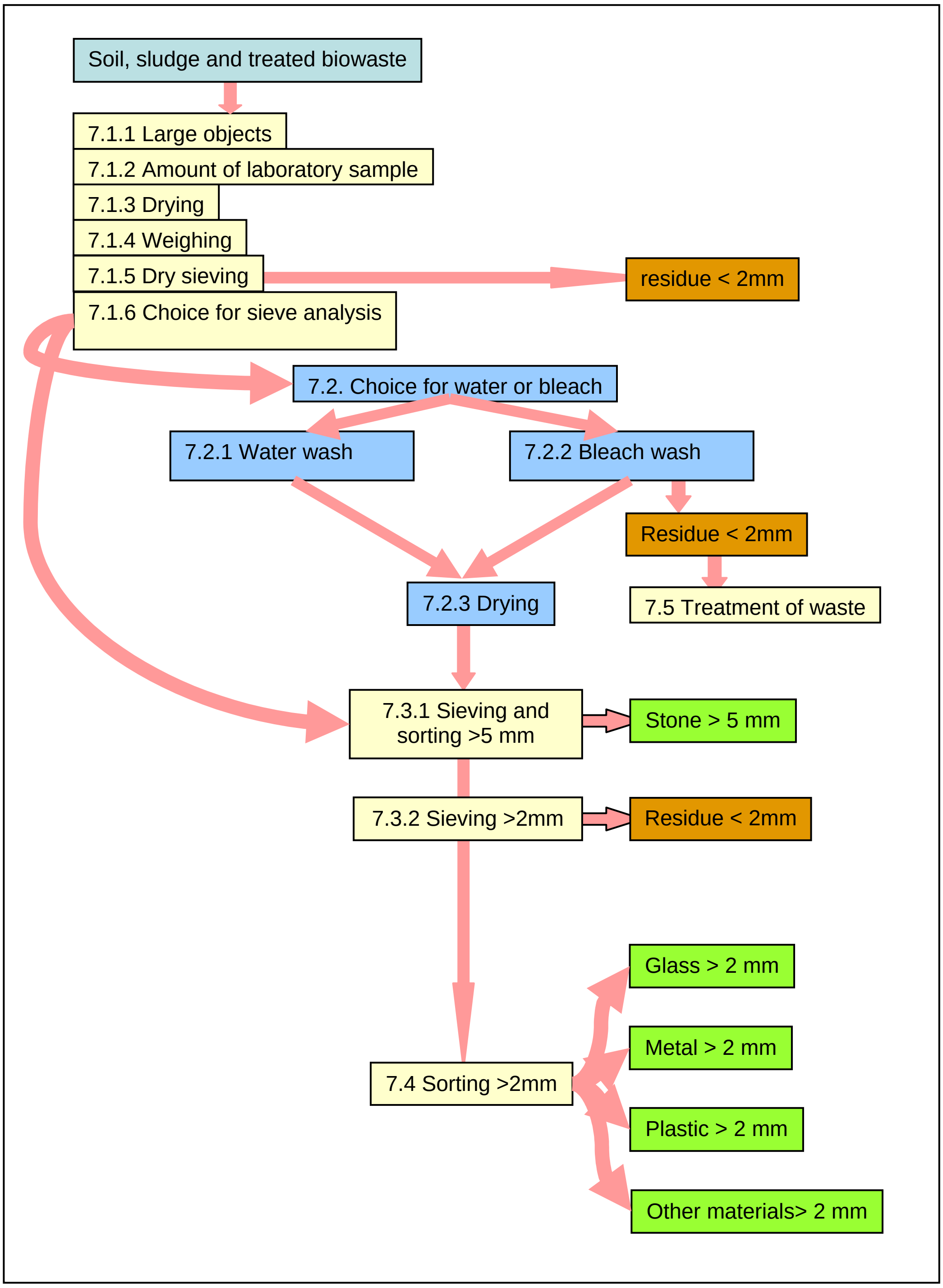

Figure 2 *Schedule of the procedure described in paragraphs 7.1-7.5 with appropriate numbering*

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.

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

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

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

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

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

(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"

8 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.

Appendix 2 Statistics of the fraction stones >5 mm in weigh

Table Weight of stones >5 mm in gram, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	14.9	16.9	37.6	50.4	8.2	4.3	1.8	1.0	2.7	2.5	1.1	0.9
L2	46.5	42.7	46.1	65.4	1.8	8.3	0.4	2.2	3.3	4.0	0.5	0.9
L3	6.8	27.1	44.9	38.6	2.4	1.1	1.3	1.2	2.8	1.1	0.3	1.6
L4	12.1	25.1	59.0	55.5	2.7	6.4	1.5	1.9	6.1	3.4	1.4	1.2
L5	7.6	17.2	77.1	49.4	4.2	3.6	0.9	1.1	2.4	2.9	0.8	0.3
L7	43.6	27.0	83.8	64.6	6.2	3.8	0.0	1.5	3.5	9.7	0.9	0.4
L8	25.2	2.4	50.3	27.8	3.2	2.6	0.6	0.3	2.8	1.8	0.4	1.1
L11	19.6	10.5	60.4	67.6	1.9	1.0	0.0	1.8	0.9	2.0	1.8	1.8
Average	22.0	21.1	57.4	52.4	3.8	3.9	0.8	1.4	3.1	3.4	0.9	1.0

Table Stones >5 mm in % W/W of the individual sample weigh, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	1.06	1.21	1.98	2.68	1.55	0.83	0.37	0.21	0.53	0.48	0.22	0.18
L2	3.31	3.01	2.43	3.46	0.32	1.57	0.08	0.44	0.59	0.75	0.10	0.18
L3	0.48	1.95	2.48	2.04	0.46	0.20	0.26	0.25	0.52	0.20	0.05	0.31
L4	0.86	1.78	3.14	2.92	0.52	1.20	0.30	0.37	1.15	0.63	0.27	0.24
L5	0.54	1.21	4.05	2.59	0.67	0.58	0.16	0.20	0.39	0.47	0.14	0.06
L7	3.09	1.94	4.43	3.43	1.17	0.72	0.00	0.30	0.65	1.80	0.17	0.08
L8	1.81	0.17	2.67	1.48	0.60	0.49	0.13	0.06	0.53	0.34	0.08	0.22
L11	1.39	0.74	3.18	3.59	0.36	0.19	0.00	0.36	0.16	0.38	0.36	0.36
Average	1.57	1.50	3.05	2.77	0.71	0.72	0.16	0.27	0.56	0.63	0.17	0.20

Table Inner and outer variance statistics of stones >5 mm in % W/W of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean	1.687		2.83		0.75		0.26		0.46			
Repeatability variance (S^2r)	0.559		0.36		0.20		0.02		0.02			
Repeatability standard (Sr)	0.748		0.60		0.45		0.14		0.15			
Between lab variance (S^2L)	0.447		0.28		0.03		0.00		0.01			
Reproducibility variance (S^2R)	1.006		0.64		0.23		0.02		0.03			
Reproducibility variance (SR)	1.003		0.80		0.48		0.14		0.18			
r	2.093	124%	1.68	59%	1.25	167%	0.4	154%	0.41	89%		
R	2.809	167%	2.24	79%	1.34	179%	0.4	154%	0.49	107%		

Appendix 3 Statistics of the fraction glass >2 mm in weigh

Table Weight of glass >2 mm in gram, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.24	0.00	0.47	0.43	0.00	0.00	0.00	0.14	5.15	4.96	5.43	5.34
L2	0.80	1.30	0.90	0.60	0.40	0.30	0.80	0.40	5.50	5.00	5.70	5.40
L3	0.24	0.73	0.95	0.92	0.40	0.05	0.59	0.60	5.07	4.75	5.26	5.98
L4	0.04	0.00	4.21	0.72	0.05	0.14	0.36	0.49	5.53	6.51	5.60	5.42
L5	0.02	0.59	1.30	0.20	0.90	0.80	0.50	0.80	5.00	5.20	5.70	5.40
L7	0.00	0.00	1.10	0.30	0.00	0.40	0.60	0.40	5.00	5.20	5.90	5.50
L8	0.00	1.08	0.58	4.61	1.59	0.45	0.23	0.60	5.32	5.05	5.38	5.29
L11	0.12	0.00	0.69	0.74	0.75	1.17	0.94	0.39	5.03	5.09	5.91	4.33
Average	0.21	0.53	1.28	1.07	0.58	0.47	0.57	0.48	5.20	5.22	5.61	5.33

Table Glass >2 mm in % W/W of the individual sample weigh, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.02	0.00	0.02	0.02	0.00	0.00	0.00	0.03	0.99	0.94	1.07	1.07
L2	0.06	0.09	0.05	0.03	0.07	0.06	0.15	0.08	0.98	0.94	1.08	1.07
L3	0.02	0.05	0.05	0.05	0.08	0.01	0.12	0.12	0.95	0.90	1.06	1.20
L4	0.00	0.00	0.22	0.04	0.01	0.03	0.07	0.10	1.03	1.22	1.09	1.07
L5	0.00	0.04	0.07	0.01	0.14	0.13	0.09	0.15	0.82	0.84	1.01	0.97
L7	0.00	0.00	0.06	0.02	0.00	0.08	0.12	0.08	0.93	0.96	1.14	1.08
L8	0.00	0.08	0.03	0.24	0.30	0.09	0.05	0.12	1.00	0.96	1.08	1.06
L11	0.01	0.00	0.04	0.04	0.14	0.23	0.19	0.08	0.97	0.97	1.18	0.86
Average	0.01	0.03	0.07	0.06	0.09	0.08	0.10	0.09	0.96	0.97	1.09	1.05

Table Inner and outer variance statistics of glass >2 mm in % W/W of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean			0.038		0.049		0.115		0.98			
Repeatability variance (S^2r)			0.000		0.007		0.002		0.00			
Repeatability standard (Sr)			0.014		0.086		0.049		0.03			
Between lab variance (S^2L)			0.000		0.001		-0.001		0.00			
Reproducibility variance (S^2R)			0.000		0.009		0.002		0.00			
Reproducibility variance (SR)			0.014		0.092		0.049		0.06			
r			0.04	105%	0.24	490%	0.136	118%	0.08	8%		
R			0.04	105%	0.258	527%	0.136	118%	0.17	17%		

Appendix 4 Statistics of the fraction metals >2 mm in weigh

Table Weight of metals >2 mm in gram, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.15	0.30	2.24	2.51	0.34	0.65	0.61	0.34	1.39	1.23	1.73	1.71
L2	0.50	1.50	5.20	5.50	1.30	0.40	0.20	0.30	1.70	1.80	1.90	1.90
L3	0.22	0.64	1.30	2.84	0.06	0.10	0.09	0.05	1.92	1.42	1.56	1.48
L4	0.12	0.44	2.57	7.17	0.35	0.41	0.30	0.53	3.89	1.79	1.54	1.65
L5	0.00	0.00	6.20	2.10	0.40	0.00	0.00	0.00	1.50	1.50	1.50	1.50
L7	0.00	0.00	0.10	0.40	0.00	0.00	0.00	0.00	1.60	1.50	1.40	1.40
L8	0.00	0.05	0.29	0.29	0.00	0.00	0.00	0.06	1.48	1.59	2.08	1.89
L11	0.00	0.00	4.54	0.73	0.00	0.00	0.00	0.00	1.24	1.42	1.41	1.21
Average	0.14	0.37	2.81	2.69	0.35	0.22	0.17	0.16	1.84	1.53	1.64	1.59

Table Metals >2 mm in % W/W of the individual sample weigh, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.011	0.022	0.118	0.134	0.065	0.124	0.123	0.069	0.267	0.232	0.342	0.342
L2	0.036	0.106	0.274	0.291	0.233	0.076	0.038	0.060	0.303	0.339	0.361	0.375
L3	0.020	0.050	0.070	0.150	0.010	0.020	0.020	0.010	0.360	0.270	0.310	0.300
L4	0.009	0.031	0.137	0.377	0.066	0.078	0.059	0.105	0.726	0.336	0.301	0.327
L5	0.000	0.000	0.330	0.110	0.060	0.000	0.000	0.000	0.250	0.240	0.270	0.270
L7	0.000	0.000	0.005	0.021	0.000	0.000	0.000	0.000	0.298	0.278	0.271	0.275
L8	0.000	0.004	0.015	0.015	0.000	0.000	0.000	0.012	0.278	0.301	0.418	0.379
L11	0.000	0.000	0.240	0.039	0.000	0.000	0.000	0.000	0.238	0.271	0.282	0.240
Average	0.009	0.026	0.149	0.142	0.054	0.037	0.030	0.032	0.340	0.283	0.319	0.314

Table Inner and outer variance statistics of metals >2 mm in % W/W of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean			0.037		0.032		0.011		0.298			
Repeatability variance (S^2r)			0.005		0.005		0.000		0.001			
Repeatability standard (Sr)			0.069		0.069		0.008		0.034			
Between lab variance (S^2L)			0.001		0.001		0.000		0.000			
Reproducibility variance (S^2R)			0.006		0.006		0.000		0.001			
Reproducibility variance (SR)			0.076		0.075		0.008		0.035			
r			0.192	519%	0.192	600%	0.022	200%	0.095	32%		
R			0.212	573%	0.211	659%	0.022	200%	0.097	33%		

Appendix 5 Statistics of the fraction others >2 mm in weigh

Table Weight of others >2 mm in gram, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	12.65	2.40	2.20	4.77	0.07	0.03	0.07	0.04	0.05	0.06	0.13	0.10
L2	1.21	1.91	4.39	3.80	0.65	0.35	0.91	1.00	1.06	0.23	0.41	0.85
L3	0.00	0.00	32.13	0.87	0.68	0.29	0.09	0.07	0.16	0.35	0.09	0.02
L4	0.00	0.00	0.00	0.00	0.08	0.12	0.00	0.00	0.04	0.29	0.00	0.00
L5	0.80	0.40	5.10	5.60	0.30	0.70	1.80	1.10	1.10	0.90	1.30	1.40
L7	0.50	1.10	1.40	0.90	0.20	0.00	0.40	1.50	0.70	0.50	1.10	1.10
L8	1.66	1.71	13.77	19.71	0.63	0.51	2.20	2.40	1.10	0.21	4.89	4.41
L11	2.29	1.91	87.80	94.20	0.97	1.48	4.14	4.31	1.61	1.72	4.32	4.10
Average	2.39	1.18	18.35	16.23	0.45	0.44	1.20	1.30	0.73	0.53	1.53	1.50

Table Others >2 mm in % W/W of the individual sample weigh, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.902	0.172	0.116	0.254	0.013	0.006	0.014	0.008	0.010	0.011	0.026	0.020
L2	0.086	0.135	0.231	0.201	0.116	0.066	0.175	0.201	0.189	0.043	0.078	0.168
L3	0.000	0.000	1.770	0.050	0.130	0.060	0.020	0.010	0.030	0.070	0.020	0.000
L4	0.000	0.000	0.000	0.000	0.015	0.023	0.000	0.000	0.007	0.054	0.000	0.000
L5	0.060	0.030	0.270	0.290	0.050	0.110	0.330	0.200	0.180	0.140	0.230	0.250
L7	0.035	0.079	0.074	0.048	0.038	0.000	0.080	0.304	0.130	0.093	0.213	0.216
L8	0.119	0.121	0.730	1.047	0.120	0.097	0.449	0.489	0.207	0.040	0.983	0.885
L11	0.162	0.136	4.633	4.996	0.187	0.289	0.830	0.865	0.309	0.328	0.864	0.813
Average	0.171	0.084	0.978	0.861	0.084	0.081	0.237	0.260	0.133	0.097	0.302	0.294

Table Inner and outer variance statistics of others >2 mm in % W/W of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean	0.109		0.154		0.067		0.024		0.095			
Repeatability variance (S^2r)	0.001		0.003		0.001		0.000		0.007			
Repeatability standard (Sr)	0.025		0.059		0.032		0.012		0.081			
Between lab variance (S^2L)	0.001		0.004		0.001		0.008		-0.002			
Reproducibility variance (S^2R)	0.002		0.007		0.002		0.008		0.007			
Reproducibility variance (SR)	0.040		0.084		0.047		0.091		0.081			
r	0.069	63%	0.164	106%	0.088	131%	0.033	138%	0.226	238%		
R	0.111	102%	0.236	153%	0.131	196%	0.254	1058%	0.226	238%		

Appendix 6 Statistics of the fraction plastics >2 mm in weigh

Table Weight of plastics >2 mm in gram, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.02	0.09	3.06	1.50	0.00	0.00	0.00	0.00	0.59	0.60	0.57	0.58
L2	0.01	0.02	1.41	0.61	1.78	1.88	0.89	1.53	1.96	2.05	1.88	2.11
L3	0.04	0.02	0.96	2.10	0.08	0.23	0.86	0.98	1.16	0.86	1.02	0.99
L4	0.03	0.05	2.39	2.71	0.84	0.77	1.77	0.74	1.90	1.02	2.52	1.32
L5	0.50	0.50	1.10	1.20	0.70	0.30	0.60	0.50	0.90	1.10	1.10	1.00
L7	0.00	0.00	1.80	1.50	0.30	0.50	1.10	0.80	1.40	1.50	1.40	1.50
L8	0.00	0.00	0.30	0.00	0.35	1.19	0.35	0.47	0.66	0.88	0.90	1.11
L11	0.00	0.00	1.56	3.78	0.43	0.48	0.37	0.68	0.68	1.02	0.79	0.90
Average	0.08	0.09	1.57	1.68	0.64	0.76	0.85	0.81	1.16	1.13	1.27	1.19

Table Plastics >2 mm in % W/W of the individual sample weigh, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.001	0.006	0.161	0.080	0.000	0.000	0.000	0.000	0.113	0.113	0.113	0.116
L2	0.001	0.001	0.074	0.032	0.319	0.356	0.171	0.308	0.349	0.386	0.357	0.417
L3	0.000	0.000	0.050	0.110	0.020	0.040	0.170	0.200	0.220	0.160	0.210	0.200
L4	0.002	0.004	0.127	0.142	0.159	0.146	0.351	0.147	0.355	0.192	0.492	0.262
L5	0.040	0.040	0.060	0.060	0.110	0.050	0.110	0.090	0.150	0.180	0.200	0.180
L7	0.000	0.000	0.095	0.080	0.057	0.095	0.219	0.162	0.261	0.278	0.271	0.294
L8	0.000	0.000	0.016	0.000	0.066	0.227	0.071	0.096	0.124	0.167	0.181	0.223
L11	0.000	0.000	0.082	0.200	0.083	0.094	0.074	0.137	0.131	0.195	0.158	0.179
Average	0.006	0.006	0.083	0.088	0.102	0.126	0.146	0.142	0.213	0.209	0.248	0.234

Table Inner and outer variance statistics of plastics >2 mm in % W/W of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean			0.094		0.092		0.160		0.224			
Repeatability variance (S^2r)			0.003		0.003		0.003		0.001			
Repeatability standard (Sr)			0.051		0.059		0.052		0.033			
Between lab variance (S^2L)			0.000		0.000		0.002		0.007			
Reproducibility variance (S^2R)			0.003		0.004		0.005		0.008			
Reproducibility variance (SR)			0.051		0.061		0.069		0.088			
r			0.143	152%	0.165	179%	0.147	92%	0.094	42%		
R			0.143	152%	0.17	185%	0.194	121%	0.245	109%		

Appendix 7 Statistics of the fraction plastics >2 mm in area

Table Area of plastics >2 mm in cm^2 per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.71	0.19	15.60	17.10	0.00	0.00	0.00	0.00	29.67	30.01	28.54	29.15
L2	1.42	2.82	33.43	10.30	40.70	70.10	53.81	62.69	69.03	64.59	55.27	66.52
L3	4.90	2.80	28.40	23.30	5.30	9.90	58.80	62.90	38.80	46.90	64.60	50.40
L4	0.50	1.00	15.00	64.00	33.00	16.00	81.00	35.00	63.00	42.00	86.00	57.00
L5	2.00	2.00	15.00	61.00	18.00	19.00	25.00	23.00	41.00	40.00	55.00	37.00
L7	1.90	0.70	35.40	24.00	24.70	25.60	44.90	63.90	59.50	80.80	99.90	88.40
L8	0.00	0.00	1.23	0.00	10.77	11.66	17.21	11.52	33.70	33.11	60.55	63.36
L11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Average	1.63	1.36	20.58	28.53	22.08	25.38	46.79	43.17	47.81	48.20	64.27	55.98

Table Plastics >2 mm in % W/W of the individual sample area, per lab and per individual sample

lab	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
L1	0.0005	0.0001	0.0082	0.0091					0.0569	0.0566	0.0564	0.0583
L2	0.0010	0.0020	0.0176	0.0055	0.0729	0.1327	0.1032	0.1260	0.1229	0.1216	0.1050	0.1314
L3	0.0040	0.0020	0.0160	0.0120	0.0100	0.0190	0.1190	0.1280	0.0730	0.0890	0.1300	0.1010
L4	0.0004	0.0007	0.0080	0.0336	0.0624	0.0303	0.1605	0.0695	0.1176	0.0789	0.1681	0.1130
L5	0.0010	0.0010	0.0080	0.0300	0.0300	0.0300	0.0500	0.0400	0.0700	0.0600	0.1000	0.0700
L7	0.0013	0.0005	0.0187	0.0127	0.0468	0.0487	0.0896	0.1295	0.1109	0.1499	0.1933	0.1734
L8	0.0000	0.0000	0.0007	0.0000	0.0205	0.0222	0.0351	0.0235	0.0634	0.0627	0.1218	0.1272
L11	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Average	0.0012	0.0009	0.0110	0.0147	0.0404	0.0471	0.0929	0.0861	0.0878	0.0884	0.1249	0.1106

Table Inner and outer variance statistics of plastics >2 mm in % area/area of the individual sample weigh, per lab and per sample (duplo)

lab	A	In %	B	In %	C	In %	D	In %	E	In %	F	In %
Mean												
Repeatability variance (S^2r)												
Repeatability standard (Sr)												
Between lab variance (S^2L)												
Reproducibility variance (S^2R)												
Reproducibility variance (SR)												
r												
R												