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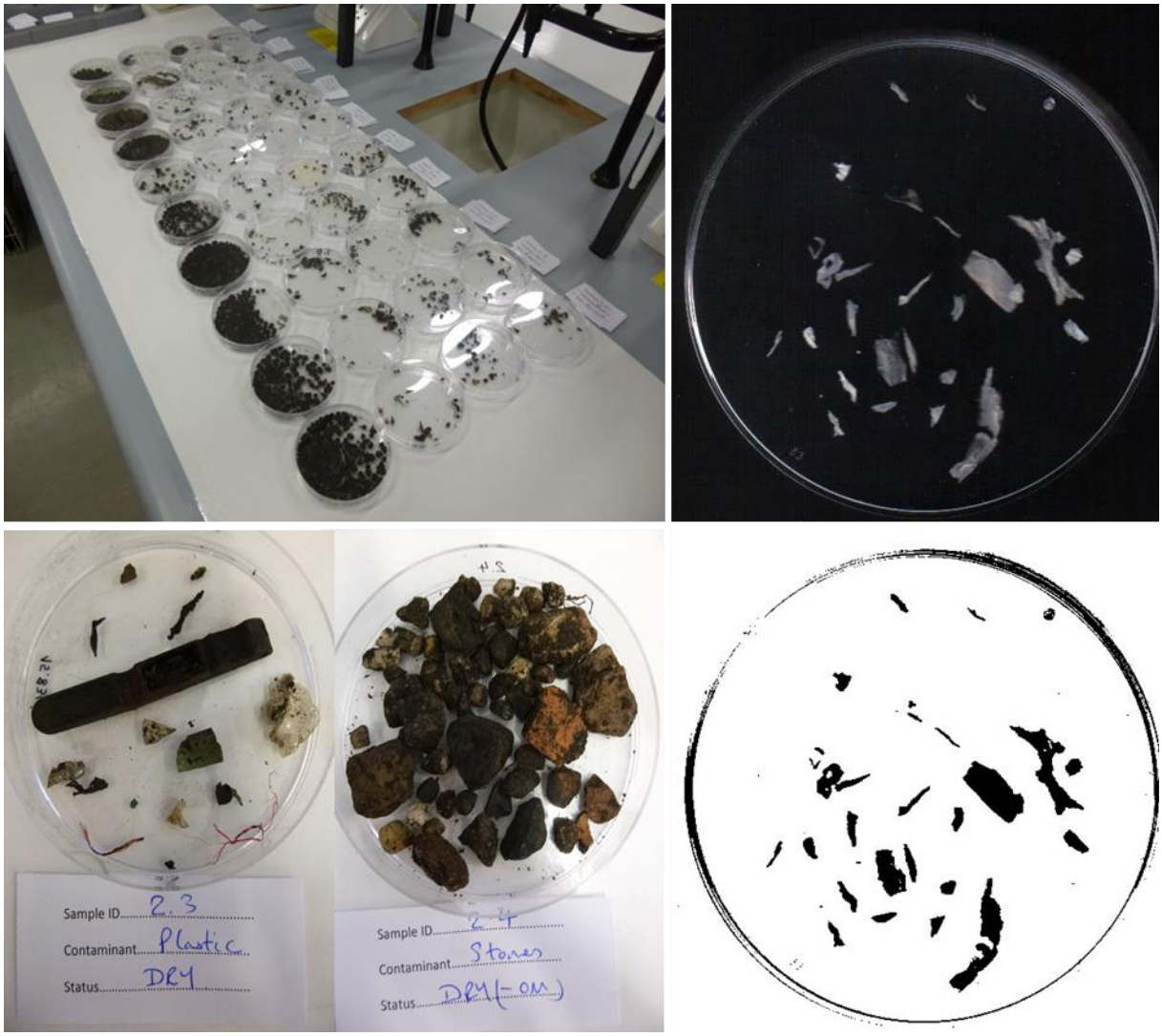

Sampling and testing of physical contaminants in PAS composts and digestates

Project code: OIN008-001
Research date: Nov 2014 – Mar 2015 **Date:** 31st March 2016

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Front cover photography: Physical contaminants removed from PAS compost (top left) and digestate (top right), and weight (bottom left) and area (bottom right) based measurement (photos courtesy of Thomas Aspray)

Executive summary

It is WRAP's aim to support the composting and anaerobic digestion (AD) industries to maintain end-user market confidence by meeting their respective scheme BSI PAS100:2011 and BSI PAS110:2014 standards and producing 'quality' products. A recent quality review of Renewable Energy Assurance Ltd (REAL) appointed laboratories responsible for testing compost and digestate products for scheme members has identified data inconsistencies related to physical contaminants (WRAP project reference OMK009-003). As such the primary aim of this work was to investigate and, where necessary, improve the robustness of the physical contaminant testing and sampling methodologies specified in the PAS schemes. This is to ensure that representative samples are taken and 'true' values are reported, so markets can have absolute confidence in the schemes and the quality products. A second aim of the work was to evaluate a surface area (rather than weight) based method for physical contaminant analysis. The reason for this is in response to the lowering of PAS100/PAS110 limits for physical contaminants for certain agricultural markets.

The specific objectives of the project were to:

1. Determine whether the current sampling methods (specified by the Biofertiliser Certification Scheme (BCS) and Compost Certification Scheme (CCS)) are sufficiently robust to deliver truly representative results.

2. Understand the inter-laboratory variability in the analysis of physical contaminants. Make recommendations (as required) to improve robustness of the methods which can be implemented directly by BCS and CCS.

3. Understand the intra-laboratory variability in physical contaminant analysis. Make recommendations (as required) to improve robustness of physical contaminant analysis for both BCS and CCS.

4. Understand whether the German approach to film plastics (in which their presence is quantified on an area basis, as well as a weight basis) could be implemented in the UK, and at what cost.

From telephone interviews held with compost and digestate producers it was found that compost producers used different sampling approaches which in some cases deviated from CCS guidelines. 17 out of 20 compost sites took the minimum 12 incremental samples and combined these to generate a composite sample for laboratory analysis. In a number of cases incremental samples were added directly to sample bags, without explicitly mentioning mixing prior to sending to the laboratory. For digestate, nine out of 17 AD sites took one to two isolated sample(s) to represent a 'portion of production' and eight sites took three or more samples. Such a deviation from the BCS sampling guidance may not be sufficient for obtaining a representative sample of digestate for physical contaminant testing and therefore requires further investigation.

The cleaning of samples for the inter-laboratory trial (Objective 2) highlighted limitations to the BCS guidelines for physical contaminant analysis method. It is recommended that improvements are made to this before the effect of deviating from the BCS sampling method can be properly assessed. Specifically, this work highlights the low sensitivity of the JAS-497/001 (which has been superseded by JAS497/002) weight method in only reporting to two decimal places, as well as inconsistency in the ability of a 2 mm sieve to catch film plastic fragments with at least one dimension >2 mm. The investigation also found that

liquor digestate screened to <2mm (which is exempt from physical contaminant analysis as per PAS110:2014) contained >2 mm physical contaminants in the tested separated liquor.

To assess variability in physical contaminant testing (Objective 2) and provide recommendations for improvements to both PAS100 and PAS110 methods an inter-laboratory trial was carried out. Compost and digestate samples were cleaned prior to spiking with known quantities of physical contaminants and sending to REAL appointed laboratories. This was backed up by laboratory visits to help identify potential intra-laboratory variability (Objective 3).

The results of the compost inter-laboratory trial highlighted variability between the laboratories in terms of their ability to find all spiked contaminants, as well as, the identification/classification of physical contaminants particularly associated with the 'stones' and 'other' categories. In terms of classification, there was uncertainty around what could be included in the 'other' category with clear evidence of natural non-compost material being included such as quartz and graphite. A number of recommendations are proposed to improve the robustness of the CCS laboratory test method. Discussions with laboratories found that there was variability in compost sample drying practice including method non-conformance by one laboratory.

The results of the digestate inter-laboratory trial showed the two laboratories involved performed well at isolating spiked plastics from whole digestate and separated liquor. Results showed greater variability with the separated fibre samples, including under reporting of spiked metal fragments by one laboratory. There was also inter and intra-laboratory variability in laboratory balance readability with weighing to either three or four decimal places.

The final aspect of the project involved the consideration of a surface area (rather than weight) based method for physical contaminant analysis with a focus on plastic fragments (Objective 4). Initially, a review of the development and application of this approach in Europe (with emphasis on Germany) was carried out. The German area method was taken and developed (supported by in-house evaluation on compost derived plastics at Heriot-Watt University) into a protocol for the REAL appointed laboratories to evaluate using the freely available image analysis software ImageJ. Two of the four laboratories appointed by REAL for PAS100 engaged with this aspect of the project. Both laboratories confirmed that an area based method could be implemented with costs of £7 and £35, respectively, as a bolt on to current weight based physical contaminant tests. Further in-house work confirmed that the same methodology would work for whole and separated liquor digestates. Stakeholder engagement found there was clear interest from a number of digestate producers whereas compost producers had very mixed responses.

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Glossary

ABP	Animal by-products
AD	Anaerobic digestion
BCS	Biofertiliser Certification Scheme
BGK	German Compost Quality Assurance Organisation (Bundesgütegemeinschaft Kompost)
CCS	Compost Certification Scheme
CFW	Commercial food waste
CLO	Compost like output
DM	Dry matter
DW	Dry weight
FM	Fresh matter
GW	Green waste
HWU	Heriot-Watt University
IVC	In-vessel composting
LDPE	Low density polyethylene
MC	Moisture content
OM	Organic matter
PAS	Publically Available Specification
PSD	Particle size distribution
QMS	Quality Meat Scotland
RBP	Residual biogas potential
REA	Renewable Energy Association
REAL	Renewable Energy Assurance Limited
REML	Restricted maximum likelihood
SOP	Standard operating procedure

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1.0 Introduction

Background

In the UK, 'quality' compost and digestate products are deemed as those which meet their respective BSI PAS100:2011 and BSI PAS110:2014 specifications whilst adhering to quality protocol or position statement guidance. Ensuring compliance of compost and digestate producers with PAS100 and PAS110 lies ultimately with the Compost Certification Scheme (CCS) and Biofertiliser Certification Scheme (BCS), respectively. Laboratories that undertake the analysis of composts and digestates for the CCS and BCS schemes are appointed by REAL.

Preliminary assessment of test results from the REAL appointed laboratories have indicated variability between laboratories, especially for physical contaminant (glass, metal, plastics and other 'non-stone' man-made fragment) analysis by the recent WRAP project 'Developing a laboratory proficiency framework for the UK compost and digestate certification schemes' (OMK009-003). A need for more rigorous internal quality control measures within the laboratories has been identified and recommendations made. The robustness of the data on physical contaminants also depends on the sampling and testing methodologies themselves.

This project sought to investigate both aspects in order to verify fitness for purpose and, if necessary, make recommendations for change so that end users can have full confidence in the results and that market confidence can be assured.

A second aspect of the project was to evaluate a surface area (rather than weight) based method for physical contaminant analysis. The reason for this is in response to the lowering of PAS100/PAS110 limits for physical contaminants for agricultural markets. Specifically, the Quality Meat Scotland's (QMS) Cattle and Sheep Standards require the following reductions to the plastic sub-limits:

- 'Compost: The quantity of physical contaminants does not exceed half that permitted by PAS100:2011'.
- 'Digestate: The quantities of physical contaminants do not exceed 8% of those permitted by PAS110:2014'.

Compliance with these reduced limits, in addition to all other requirements in PAS100/ PAS110 enables certified products to be used on QMS member farms.

Objectives

The objectives of this project were to:

1. Determine whether the current sampling methods (specified by BCS and CCS) are sufficiently robust to deliver truly representative results, particularly for anaerobic digestion (AD) sites where the sampling method needs to accommodate different engineering/design approaches as well as the range of digestate types covered by the PAS (whole, separated fibre and separated liquor). If necessary, recommend how the sampling methodologies could be revised to improve clarity and robustness.

2. Understand the inter-laboratory variability in physical contaminant results through the testing of key product types with known levels of physical contaminants. If necessary make recommendations to improve robustness of the methods which can be directly implemented by BCS and CCS.

3. Work with the appointed laboratories to understand the intra-laboratory variability in physical contaminant analysis. Make recommendations (as required) to improve robustness of physical contaminant analysis for both BCS and CCS.

4. Understand whether the German approach to film plastics (in which their presence is quantified on an area basis, as well as a weight basis) could be implemented in the UK, and at what cost.

2.0 Materials and Methods

2.1 Approaches to understand sampling in practice and product variability

2.1.1 Producer onsite and telephone discussions

Two question sets were prepared for compost and digestate producers based on the CCS and BCS sampling guidance respectively (Appendices 1 and 2). These were used to guide discussions during site visits made to six compost sites and six AD sites during December 2014 - February 2015 and subsequent telephone discussions with a wider number of producers. In total 14 discussions were held with PAS100 compost producers (representing 20 sites) and 17 with PAS110 AD sites.

2.1.2 Product sampling and variability

Samples were collected from six sites (3 x compost and 3 x AD) during December 2014-January 2015 to represent different key product types; 0-10 mm compost, 0-25 mm compost, 0-40 mm compost, whole digestate, separated liquor and separated fibre (Table 1). Twenty spot samples (>1.2 kg) were taken of finished compost products (after scraping away at least 2 inches of surface material). Samples of whole digestate and separated liquor were collected from the sample valve situated on a transfer pipe, and then decanted into a 1 litre sample bottle. The digestate fibre samples were collected from the discharge point of the separator. For each sample a bucket of at least 1 litre of fibre was collected and placed directly into a plastic bag. The digestate fibre was not produced to PAS110, and is currently applied to land under an exemption. Samples were subsequently stored at 4 °C until further use.

Table 1. Product types (and grade) used in this project.

Site	Principal feedstock(s)	Process	Product type	Sampling location	No. of discrete samples
1	GW, BMW, woodchip	In-vessel compost	0-10 mm compost	Finished product pile	20
2	GW	Open windrow compost	0-25 mm compost	Finished product pile	20
3	GW	Open windrow compost	0-40 mm compost	Finished product pile	20
4	CFW	Mesophilic anaerobic digestion	Separated liquor (2 mm sieved)	Valve on final dispatch pipe exiting storage tank	15
5	CFW	Mesophilic anaerobic digestion	Whole digestate (5 mm sieved)	Valve on pipe between separator and storage tank	15
6	CFW:Maize	Mesophilic anaerobic digestion	Separated fibre	Separator	15

GW – green waste; CFW – commercial food waste; BMW – biodegradable municipal waste

All physical contaminant fragments were removed from 1.2 kg (fresh weight) compost samples by hand and classified into glass (>2 mm), plastic (>2 mm), metal (>2 mm), other (>2 mm) and stones (>4 mm). Physical contaminants were removed from fresh digestate

samples by hand and classified into glass (>2 mm), plastic (>2 mm), metal (>2 mm), 'other' (>2 mm) and stones (>5 mm). Further details on the procedures for compost and digestate can be found in Appendix 3. Examples of physical contaminants removed can be found in Appendices 5-8. The cleaning of samples was primarily to prepare material for the inter-laboratory trial. However, this exercise also afforded the opportunity to evaluate the variability of physical contaminants in the different product types.

2.2 Intra- and inter-laboratory variability

2.2.1 Sample spiking

Cleaned compost samples (prepared as described in section 2.1.2) were then spiked with a known weight and number of physical contaminants (Appendices 4-6). Whole digestate and separated liquor samples were spiked with plastic only (Appendix 7). Separated fibre samples were spiked with known physical contaminants (Appendix 8). A summary of the sample spiking regime is presented (Table 2).

Table 2. Commercial laboratory testing regime and contaminant levels prepared in individual compost and digestate subsamples.

ID	Product Type	Contaminant Loading	No. of labs	No. of replicates	Sample no.	Plastics (% w/w)	Total PCs (% w/w) or kg/t*
1	Compost 0-10 mm	Low	3	3	9	~0.02	~0.12
		High	3	3	9	~0.01	~0.26
2	Compost 0-25 mm	Low	3	3	9	~0.04	~0.12
		High	3	3	9	~0.04	~0.26
3	Compost 0-40 mm	Low	3	3	9	~0.12	~0.12
		High	3	3	9	~0.1	~0.26
				Subtotal	54		
4	Whole digestate	Low	2	3	6	n/a	~0.017
		High	2	3	6	n/a	~0.22
5	Separated liquor	Low	2	3	6	n/a	~0.017
		High	2	3	6	n/a	~0.22
6	Separated fibre	Low	2	3	6	n/a	~0.017
		High	2	3	6	n/a	~0.22
				Subtotal	36		
Total no. of commercial laboratory sample tests					90		

*Composts on a dry weight and digestates on a fresh weight basis. A total nitrogen content of 5 kg/t (on a fresh weight basis) was assumed for digestates.

The physical contaminants added to each sample were weighed (after drying as appropriate) and photographed. Each laboratory received the same number of each fragment type for each product type and loading rate (unless otherwise stated). Real aged physical contaminants (isolated from compost and digestate samples) were used as far as was practically possible. Pristine fragments were used where additional material was needed (for example to achieve high loadings) or where real aged physical contaminants were considered too variable in appearance for the inter-laboratory trial.

2.2.2 Commercial laboratory analysis

Spiked compost samples were prepared and were sent to three REAL appointed laboratories for analysis as per the PAS100 method. Spiked digestate samples were subsequently prepared and sent to two laboratories for analysis as per the PAS110 method, one REAL appointed laboratory and one laboratory going through the process of registration. The only variation to both methods was that the laboratories were requested to process all the material supplied. In the case of compost this meant that any sample used for moisture content determination was added back to the sample from which it was taken. For digestates, the laboratories were instructed to report raw weight data rather than report contaminants on a % DM (dry matter) basis so that a greater number of potential sources of variability could be identified. The simple calculation for reporting of a % DM basis was assumed not to be a major source of variability. Samples were sent to the laboratories in batches between December 2014 and February 2015.

Clear sealable bags were supplied to the commercial laboratories labelled with sample number and physical contaminant type e.g. 1.1 glass. The laboratories were asked to place extracted fragments in these bags and return them by post to HWU. The purpose of this was to confirm whether introduced fragments had been placed in their correct categories and allow correction of data if materials had not been completely cleaned before spiking. The latter was considered particularly important at the time for composts and the separated fibre. Specifically, composts were cleaned fresh rather than dry to ensure sample integrity and for the separated fibre we were unable to use bleach (to breakdown organics) for the same reason making a 100% effective cleaning process hard to achieve.

2.2.3 Commercial laboratory visits

To aid interpretation of the commercial laboratory compost testing results, and to explore potential intra-laboratory variability, two of the REAL appointed laboratories undertaking analysis were visited to discuss the current methods. Laboratory visits were undertaken in early February 2015.

2.2.4 Statistical analysis

Linear regression analysis was carried out for each of the contaminants separately, allowing different intercepts and slopes for each of the laboratories and each of the separate physical contaminant categories (glass, plastic, metal, other and stone). The statistical analysis was performed using the software 'R'.

2.3 Area method evaluation

2.3.1 Desk based research on methodologies used in Europe

Using the website of the BGK (Bundesgütegemeinschaft Kompost e.V.), the contact details of a number of laboratories that are certified to determine the surface area parameters were identified. Experts at the ECN and BGK were contacted and the current situation and future developments relating to physical contaminant testing in Germany and Europe were discussed. Relevant literature was identified using the search term "Flächensumme Kompost" as well as on the publications site of the BGK (kompost.de).

2.3.2 In-house evaluation of area based method on physical contaminants separated by composts and digestates

Film plastics separated from composts were tested at HWU initially using the US (TMECC, 2002) and German area methods (Kehres and Thelen-Jüngling, 2006). From these initial tests a draft protocol for area based quantification of plastic contaminants was developed (Appendix 9). Plastics isolated from digestate samples were later assessed in the same manner except placed on the base of clear plastic Petri dishes rather than attached to A4 sheet paper.

2.3.3 Evaluation of area based method by REAL appointed laboratories

The four laboratories appointed by REAL for PAS100 testing were contacted and provided with a draft protocol for area based quantification of plastic contaminants (Appendix 9) in January 2015. The protocol included instructions for installing and using ImageJ, a freely available image analysis software package, to enable the laboratories to trial the method fully should they wish. The laboratories were asked to comment on the practicalities of implementation and the potential cost of analysis for customers.

3.0 Results

3.1 Sampling and product variability

3.1.1 Compost sampling requirements and producer practice

The CCS sampling guidelines (based on BS EN 12579:2000) are designed to ensure that a representative sample is taken from an identified portion of production (preferably a single batch) for testing against the suite of PAS100 criteria. As part of this project, we assessed the compliance of compost producers with the CCS sampling guidelines in informal and confidential discussions both in site visits and by telephone. We also gathered information on producers' knowledge of the PAS100 physical contaminant testing procedure and interest in an area based method for physical contaminant analysis where time permitted.

All compost producers (n=14) reported that samples for PAS100 testing were taken from single batches. In 13 cases this was through incremental sampling of a pile whereas one producer reported sampling from a flow of material during the course of a whole day. A mixture of spade, trowel and hand sampling (the latter for the site sampling material in flow) was carried out.

Cleaning of sampling utensils was carried out by the majority of compost producers but not all. Where utensil cleaning was carried out before sampling, this varied from use of cold water only, water & soap, water & disinfectant to boiling water. Utensils were usually kept for the sole purpose of PAS100 sampling.

When sampling from piles, producers' selected random sample points rather than sampling from specific pre-defined locations. Several mentioned scraping away the surface layer of material due to concerns about its contamination by microbial pathogens from the surrounding environment. Increment samples were placed on plastic sheeting, matting, into buckets or directly onto hard standing (concrete) pad by six producers prior to mixing. The other eight producers placed increment samples directly into the sample container (usually plastic bulk 'rubble' bag) – an approach defended by one site because it would avoid faecal contamination from higher than normal bird activity adjacent to a landfill.

The CCS sampling methodology states:

12.3 Each sample shall be representative of the compost batch from which it is obtained.

NOTE BS EN 12579 provides guidance about how to obtain a representative sample of compost from a batch. The maximum batch size from which the representative sample is derived should be appropriate to the system, test results history for the compost grade and the intended customer's supply requirements. The statistically valid number of sub-samples to take from the batch and then be thoroughly mixed is given by formula: $n_{sp} = 0.5 (V^{1/2})$, where V is the volume of the batch sampled. A minimum of 12 and a maximum of 30 sub-samples apply. Thus for a batch sized 250 m^3 or 500 m^3, 12 sub-samples should be taken. For batches sized 1 000 m^3 and 2 000 m^3, 16 and 22 sub-samples should be taken respectively. For a batch sized 3 600 m^3 or more, 30 sub-samples should be taken. To minimize any changes in compost properties, any archived samples should be kept in a dark, dry place where the temperature is less than 10 °C but not less than 1 °C.

In the context of batch size and incremental sample number, batch sizes were found to range from 280 – 3000 m^3. As such, to follow the sampling guidance, all sites should take a minimum of 12 incremental samples. In practice, the reported number of incremental samples was less than the minimum 12 for three sites. For most of the sites the specific number of incremental samples taken was unknown. The number of incremental samples taken appeared to be governed by the sampling utensil and the sample container size (which acted as a gauge of the quantity of material required by the laboratory).

As previously stated, the majority of producers (n=8) placed incremental samples directly into sample containers. In several cases there was no mention of mixing incremental samples with this approach. For the six other producers, mixing (typically using a spade) was carried out prior to placing the laboratory sample in a bag. Coning and quartering (for the preparation of replicate laboratory and archive samples) was mentioned by one producer.

Compost samples were placed by producers into 'bulk' bags or occasionally plastic boxes usually sourced locally rather than provided by the laboratories. The laboratories were responsible for providing couriers. In all cases, samples were collected the same day or the next day after sampling. All samples were scheduled for overnight delivery to the laboratories (where feasible).

Archive samples were taken by ten producers. Two producers taking archive samples stored these until the laboratory test results were reported. In one case this sample was stored in a fridge. The other eight producers taking archive samples, kept these for a minimum of six months as per the sampling guidance instructions. These archive samples were stored in portacabin offices, barns and garages.

On completion of the set questions around sampling, compost producers were asked whether they were interested in the idea of an area based method applicable to the plastics fraction. Thirteen compost producers felt they understood the current weight based physical contaminant testing method and had no concerns about this method. Regarding the idea of a potential area based method, interest varied widely. Some thought it was a good idea, whereas, others felt additional testing was not needed. Several producers were more concerned with the topic of end use criteria for agricultural markets and limits for microbial pathogens (*E. coli*), stability and stones.

3.1.2 Variability of physical contaminants in different compost products

The cleaning of samples for the inter-laboratory trial, afforded the opportunity to assess the composition and variability of physical contaminants in different product types. Specifically, physical contaminants were removed, classified and weighed from 20 spot samples taken from finished product piles. Averages of the physical contaminants found are presented

below (Table 3) with individual sample results, statistical analysis and histograms in Appendix 10 (where samples had <0.01, these were treated as zero for the statistical analysis).

Table 3. Physical contaminants isolated from 20 spot samples from site 1 (0-10 mm), site 2 (0-25 mm) and site 3 (0-40 mm) compost

Site	Physical contaminants (% g/g DM)					
	Glass (>2 mm)	Metal (>2 mm)	Plastic (> 2 mm)	Other (> 2 mm)	Total (>2 mm)	Stone (> 4mm)
1	0.14 (0.08)	0	0.03 (0.02)	0.04 (0.03)	0.21 (0.09)	2.16 (0.99)
2	0.03 (0.05)	0	0.10 (0.09)	0.23 (0.22)	0.37 (0.26)	12.32 (4.10)
3	0.08 (0.17)	0.06 (0.26)	0.06 (0.07)	0.22 (0.26)	0.41 (0.45)	12.33 (6.56)
PAS limits			**0.12**		**0.25**	**8 or 10***

*depending on product type. PAS limits are % g/g 'air dried'
Values are means (n = 20) with standard deviations in parentheses

Taking into consideration feedstock, the in-vessel compost (site 1) had higher glass contamination than the two composts processing only green waste. By contrast the two green waste only composts (site 2 and 3) had considerably higher levels of plastic, other and stones. All three composts had metal (silver foil) in a small number of samples, however, usually only one or two small fragments, the weight of which was below the reporting threshold for sites 1 and 2. Two samples from site 3 had pieces of metal contamination above the reporting limit. 'Other' contamination was typically cardboard/paper in the in-vessel compost. In one of the green waste composts (site 2) a wider range of other man-made physical contaminants was found including rubber, string, polystyrene and textiles. Unknown 'man-made' physical contaminants were found and categorized as 'other'.

3.1.3 Digestate producer sampling practice

All 22 AD sites listed on the BCS website[1] at the time of the study were contacted, resulting in discussions with 17 of the sites. All information below is pertinent to those 17 sites.

All sites which were happy to discuss their AD sampling procedure operate wet, continuous digestion systems. Feedstock types included solid and liquid food waste from a variety of sectors, vegetable waste, animal slurry and maize. The 17 sites were either producing PAS110 whole digestate or PAS110 separated liquor. As only one site is currently producing PAS110 separated fibre, we have minimised comments specifically on sampling of this material to preserve anonymity.

Process for removing physical contaminants
All sites receiving packaged/bagged waste have a front-end de-packaging system. For solid food waste containing ABPs, all sites use a macerator or screw press to reduce feedstock particle size to 12mm or less (a requirement of the Animal By-product Regulations). The majority of sites also employ post-digestion screening to remove physical contaminants for both whole and liquor products. For sites receiving packaged food waste, the post-digestion screening systems (generally <2mm for liquor and 0.5-5mm for whole digestate) are predominantly in place for the removal of physical contaminants. At sites with a proportion

[1] *www.biofertiliser.org.uk/members*

of more fibrous feedstock such as vegetable wastes and maize generally use a screen (<1mm) or screw press at the end of the process for the production of separated liquor and fibre. Some sites interviewed screen digestate samples in their own laboratory using a sieve to assess physical contaminant levels, to check their screening equipment is working correctly.

For all of the 17 sites that were involved in the discussions, the following was confirmed:

- A site-specific sampling procedure is either included in their standard operating procedure, quality management system documents or is a separate document.
- RBP and samples for the other PAS110 tests are usually taken from the same sampling point, or the RBP sample is taken earlier in the process.
- Samples for *Salmonella* spp. and *Escherichia coli* are either taken earlier in the process or at the same sampling point as the PAS110 samples.

The sampling point location varied between sites. No sites obtained samples from a storage lagoon or an off-site storage tank. For all of the sites that were involved in the discussions, the following points were observed:

- Where the site has a post-digestion screen or screw press (12 sites), PAS110 samples are taken after screening, either directly post-screening from a pipe or buffer tank, or from a storage tank.
- When the site does not have any post-digestion screening, PAS110 samples are either taken from the storage tank or the dispatch pipe (5 sites).
- When samples are taken (either from a dedicated sample valve or a valve on the point of dispatch pipe) from a buffer or storage tank, to ensure mixing of the sample either 1) the mixing system was switched on for at least an hour prior to sampling, 2) the tank has continuous mixing, or 3) there was no specific mixing system, instead a continuous flow of digestate into and out of the tank (9 sites)
- When samples are taken from a transfer pipe, after separation or at the point of dispatch there is digestate regularly passing through the pipe (9 sites).
- At least double the volume of digestate within the sampling point pipe and valve is released prior to obtaining the digestate sample (17 sites).
- For sites obtaining one or two digestate samples from a transfer pipe or point of dispatch, either a bucket or jug is used to obtain the digestate sample(s), which is then decanted into a sample bottle (6 sites) or a sample bottle is held directly under the sampling point (3 sites).
- When sites were obtaining three or more incremental samples (8 sites), these were from dedicated sampling points on storage tanks with mixing systems or a transfer pipe.
- Sampling equipment is always cleaned before and after use and stored inside in a clean area such as a cupboard or on-site laboratory.

At nine sites one or two samples were taken and combined to form the sample sent for testing. At eight sites individual samples were taken at intervals (generally 2-10 minutes between individual samples) and combined to form a bulk sample, which was well mixed and then a portion used to fill the sample bottles. In general, staff felt that the sample of digestate obtained for the PAS110 tests was representative of the digestate being dispatched. At all sites where BCS auditing had occurred the site operator highlighted that the auditors had been happy with the on-site sampling procedure.

Sample storage and dispatch
The sample bottles, cool boxes, freezer blocks and a courier service are generally provided by the commercial laboratory. Regarding sample transport, a courier is booked prior to sampling and the samples are collected on the same day as sampling (or rarely the next day) and transported to the laboratory. Some sites place the still warm digestate sample bottles directly into the cool box (or a cardboard box when a cool box is not available) and arrange for collection the same day. Other sites place samples in a fridge for a few hours or overnight to cool down, with collection arranged for later the same day or the next morning, when samples are placed in the cool box.

3.1.4 Digestate product variability

As with the compost samples, cleaning of digestate samples for the inter-laboratory trial afforded the opportunity to assess the composition of physical contaminants in different product types as well as the variability of individual batches. Specifically, variability of the three digestate products was assessed by determining the physical contaminants in 15 samples. A summary of the 'total' results can be found in Table 4 with further analysis in Appendix 11.

The whole digestate and separated liquor samples contained plastics and 'other' contamination (> 2mm), the latter with the appearance of 'man-made' textile fibres. Two separated liquor samples contained 'glass' and, one of these also contained a single 'stone' (> 5 mm) fragment. In the separated fibre samples, 'glass', 'metal' (silver foil), 'plastic' and 'other' contaminants were found. *Note – as the separated fibre was cleaned without the use of bleach, the reported contamination levels (Table 5 and Appendix 11) should be considered as indicative rather than exhaustive.*

The reporting to two decimal places only (as per PAS110:2014) meant that a large number of whole and separated liquor samples were below the reporting threshold despite containing physical contaminants. The separated liquor (sieved to 2 mm on site) contained a higher level (by weight) of contaminants than the whole digestate in a few samples.

Table 4. Physical contaminants isolated from 15 subsamples in the three 'product' types.

Sample number	Total (>2 mm) physical contaminants (kg/t FM)		
	Site 4 – separated liquor	Site 5 – whole digestate	Site 6 – separated fibre
1	<0.01	0.01	0.16
2	0.01	<0.01	0.39
3	<0.01	<0.01	0.20
4	<0.01	<0.01	0.39
5	<0.01	0.01	0.26
6	<0.01	0.02	0.57
7	0.02	<0.01	0.63
8	0.03	0.04	0.30
9	0.03	<0.01	0.40
10	0.04	0.01	0.20
11	0.02	<0.01	0.22
12	0.03	<0.01	0.33
13	0.01	<0.01	0.19
14	0.12	0.01	0.33
15	0.05	<0.01	0.28
PAS110:2014 Limit*	**0.04-0.36**		

*Limit based on sample total nitrogen content

3.2 Inter-laboratory physical contaminant analysis variability

3.2.1 Commercial laboratory analysis of composts spiked with physical contaminants

To determine inter-laboratory variability of the PAS100 physical contaminant test (which incorporates the determination of particle size distribution (PSD)), three laboratories currently appointed by REAL were sent compost samples each spiked with a known weight and number of physical contaminants (as described in section 2.2.1). On completion of analysis, the laboratories were requested to send isolated physical contaminants back to HWU for further analysis (as detailed in section 2.2.2), which all three laboratories supported.

Contaminant identification and classification
Linear regression analysis of the reported physical contaminant weights were compared with the known spikings (Appendix 12). The results show that overall the laboratories were fairly consistent in analysis of spiked glass, stone and plastic fragments with correlation coefficients of 0.94, 0.91 and 0.83 respectively. For stones in particular, laboratory A over reported low loaded samples (site 1) which, on inspection at HWU were found to contain compost and wood materials. Higher loaded samples (sites 2 and 3) were underreported due to missed fragments. The laboratories generally struggled to find 'other' fragments illustrated by the negative correlation coefficient. All spiked metal fragments were clearly not picked up readily by one laboratory. Based on the raw results it appears overall that laboratory A was less accurate than laboratories B and C.

Looking further at the returned contaminants, we found there was discrepancy in the classification in low loaded samples for site 1. Two of the laboratories classified fragments of glass as quartz and reported these under 'other' contamination.

Finally, in the returned contaminants we found a large number of stones <2 mm, despite the PAS100 method stating that only stones >4 mm should be reported. Laboratory B weighed and reported stones retained on the 2 mm sieve. Although these were excluded from the contaminant % calculation, this could be a potential source of confusion for both analysts and end users. 1-3 stones <4 mm were found in returned samples from laboratory A and may have been created due to >4 mm stones breaking during transit to HWU. Laboratory C however returned a large number of <2 mm stones (more than 40 in two samples). Although the results certificates from this laboratory do not report weights for stones retained on the 2 mm sieve, the reported weight of stones in these samples (compared to spiked weights) suggest they may have been included with >4 mm stones by mistake.

3.2.2 Commercial laboratory analysis of digestates spiked with physical contaminants

To assess potential inter-laboratory variability of the PAS110 physical contaminant test, two laboratories were sent digestate samples that were each spiked with a known weight and number of physical contaminants (as described in section 2.2.1). The laboratories were asked to report on physical contaminants by weight only to aid interpretation of results. Balance precision was not stipulated to the laboratories to reflect this omission in the JAS-497/001 Determination of Physical Contaminants and Stones in Digestate method which was used in this project (superseded by JAS497/002). On completion of analysis, the laboratories were requested to send isolated physical contaminants back to HWU for further analysis (as discussed in section 2.2.2.), which both laboratories supported.

Linear regression analysis of the reported physical contaminant weights were compared with the known spiking (Appendix 13). Although the analysis method is essentially the same for the three digestate types, the nature of the separated fibre, with significantly higher OM content, meant these samples took longer for the laboratories to report and (more importantly from a laboratory performance perspective) harder to find physical contaminants. As stated in the materials and method section, the separated fibre samples were also difficult to clean prior to spiking as we were unable to use bleach in order to ensure sample integrity. Therefore, considering the whole digestate and separate liquor results in isolation it was found that both laboratories had positive correlation coefficients close to 1. Interestingly, returned plastic contaminants from 4 out of 12 liquor and 6 out of 12 whole digestate samples contained fragments which were > 2 mm but were not part of the spikings. These fragments (all film plastics) are assumed to have passed through the 2 mm sieve during sample cleaning at HWU but were caught in a second pass in the commercial laboratory. Effectively, leading to the under reporting of plastic contamination in these samples.

One laboratory initially reported results for whole digestate and separated liquor on a % FM basis (as per PAS110:2014), the laboratory was asked again to report raw contaminant weight only which they subsequently did. Reporting the raw contaminant weights facilitated in identifying potential sources of variability than would have been the case with reporting on a % DM or % FM basis. For the whole digestate and separated liquor both laboratories weighed contaminants to four decimal places. One laboratory initially reported separated fibres results on a % DM basis (as per PAS110:2009). The laboratory was again asked to report on a raw contaminant weight basis which they subsequently did. For the separated fibre one laboratory reported to four decimal places and the other to three. As such, balance readability is one aspect of inter laboratory variability of method JAS-497/001.

3.3 Intra-laboratory physical contaminant analysis variability

3.3.1 Intra-laboratory variability and physical contaminant analysis of composts

Given the high level of human input and decision making in the PAS100 physical contaminant method, there is potential for a high degree of intra-laboratory variability where more than one analyst processes samples. At all three laboratories involved in this project, it was confirmed that more than one analyst could process compost samples on any given day to cover staff absences or to deal with high sample numbers as might be expected. All three laboratories reported that analysts did not carry out physical contaminant analysis in isolation and so, at least in terms of characterisation of unusual physical contaminants, advice was taken from colleagues to reach a consensus on classification.

For the samples analysed in this project specifically, we know that one analyst processed samples at laboratory A, three analysts processed samples at laboratory B and one analyst processed samples at laboratory C. Given the number of samples analysed it was not possible to perform statistically robust analysis at the individual analyst level for laboratory B.

3.3.2 Intra-laboratory variability and physical contaminant analysis of digestates

Similar to compost analysis, at the REAL appointed laboratory offering PAS110 analysis, there is more than one analyst trained in the method to cover for staff absences. Physical contaminants in the whole digestate and separated liquor samples tested were mainly plastics, which were relatively straightforward to identify and classify.

As in section 3.2.2 a number of potential sources of inter-laboratory variability may manifest as sources of intra-laboratory variability without tightening of the JAS-497/001 method.

These include the use of bleach, the washing of sieves and the weighing of fragments. In fact, in the case of fragment weighing, intra-laboratory variability was shown in the reporting of raw weight data, where one analyst reported weights to three decimal places and a second analyst to four decimal places.

3.4 Evaluation of an area based method for physical contaminant analysis

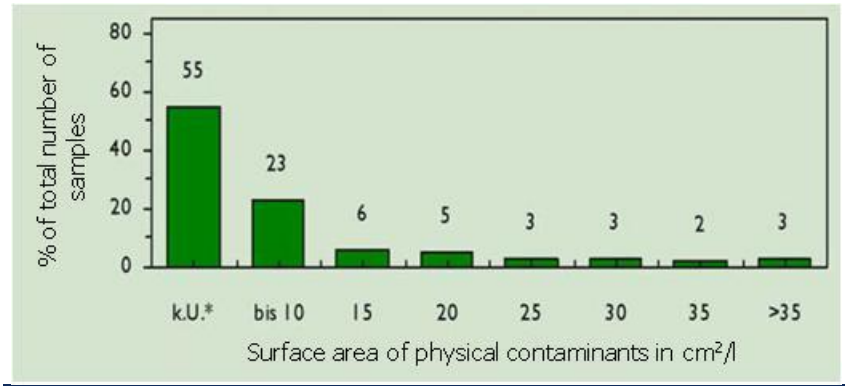
3.4.1 Application of area methods in Europe for compost and digestate

Background

The German Compost Quality Assurance Organisation (Bundesgütegemeinschaft Kompost BGK) is recognised by the German Institute for Quality Assurance and Certification (RAL), as being the organisation to facilitate the monitoring and controlling of the quality of compost and digestate in Germany. The German RAL quality scheme requires that plastic contaminants in composts and digestates do not exceed a specified maximum level. It was found that a weight fraction limit for plastic contaminants is not enough to ensure that the visual appearance of composts and digestates is acceptable. Low density, high surface area film plastics have the potential to dominate the visual appearance once the composts or digestates are applied to the field.

The visual effect of plastic contaminants within a compost or digestate sample can be quantified by measuring the contaminant's combined surface area. A study was carried out in Germany in 2006, analysing the surface area of the physical contaminants of 1,116 compost and digestate samples, of which 504 samples had physical contaminant levels which exceeded 0.1 % DM (see Fig 1) (Thelen-Jüngling, 2006).

Figure 1 Surface area parameters measured in compost and digestate samples in Germany in the first half of 2006 (captions translated, Thelen-Jüngling, 2006)



(KU = samples where the weight of physical contaminants (non-organic materials such as glass, plastics, biodegradable plastics, metals, rubber, bone fragments, paper and composite materials – excluding stones, volcanic and clay granules) on a DM basis was below 0.1%, and so the surface area parameter determination was not required).

The study found that only 8-9 % of the total number of samples exhibited contaminant levels with a surface area of more than 25 cm^2/l fresh sample (Thelen-Jüngling, 2006), while the majority of samples were below this level.

Although this information could clearly guide the development of a surface area limit for plastics, an independent study would be necessary prior to UK implementation. Currently there is no large dataset on the material type distribution and prevalence of light weight, large surface area contaminants such as film plastics in the UK industry.

German trends
Composts or digestates with up to 0.5% DM of physical contaminants (definition as above) with a surface area of less than 25 cm^2/l fresh material are currently deemed compliant (Thelen-Jüngling, 2006) with the German RAL quality scheme. Measuring the surface area parameter is currently compulsory only for those composts or digestates that contain more than 0.1% DM physical contaminants.

The conditional requirement to measure the surface area was introduced in Germany in 2006 following a two-year trial period. The surface area limit for physical contaminants of a maximum of 25cm^2/l fresh material took effect on 01/07/2007 (BGK, 2008).

This conditional requirement may be changed in the near future, so that measuring the surface area parameter would be a requirement for all composts and digestates that test positive for plastic contamination. This new rule would not affect digestates and composts that test negative for plastic contamination (Thelen-Jüngling, 2014).

Hence the new rules would take into account the fact that even composts or digestates that have a very low % DM plastic content may still fail the surface area test of 25 cm^2/l fresh material as there is only little more than 29 mg of thin film (LDPE, 12.5 µm thickness, density 0.94 g/cm^3) required to exceed this limit. For comparison, the 0.5% DM level implies a physical contaminant level of the order of 1g, which could be sufficient to surpass the area limit by a factor of 10 to 100 if the contaminants were purely made from thin plastic foil or thin film.

Method considerations and costing
In Germany there has not been a great incentive for automating determination of the surface area parameter, which costs around €20 per test (Thelen-Jüngling, 2014). Even a high throughput laboratory which undertakes 600-700 compost and digestate surface area tests per year removes contaminants manually and places them on a scanner (Marciniszyn, 2014). Photoshop is used to identify and remove the background features of the scanner lid. Shadows of larger plastic pieces are removed. Where film plastic is transparent, the "contrast" function in Photoshop is used to determine the borders of the fragment, and the "fill" option is used to fill in the area within the borders. Contrast is changed to make even faintly absorbing plastic parts appear black. Finally the blackened area is determined using Photoshop.

Marciniszyn (2014) found that even typically transparent film plastics will change its level of transparency once it has undergone the composting or digestion process as well as the plastic sample preparation process (sieving and drying).

Method reliability
The BGK surface parameter method for the optical determination of the area of physical contaminants performed very well in the most recent ring test in 2013 in Germany, Switzerland and Austria (Schaaf, 2013). 27 out of the 28 participating laboratories achieved results below the tolerated error margin of ±10 % for the determined area values (Marciniszyn, 2014).

Ongoing European developments
There are plans to introduce the surface area method in Sweden in 2015 (Barth, 2014), however using a different maximum allowed surface area and a different reference entity (units). The difference in the method and units is mainly due to the predominance of anaerobic digestion plants in Sweden compared to a smaller number of composting plants.

3.4.2 In-house evaluation of the area based method

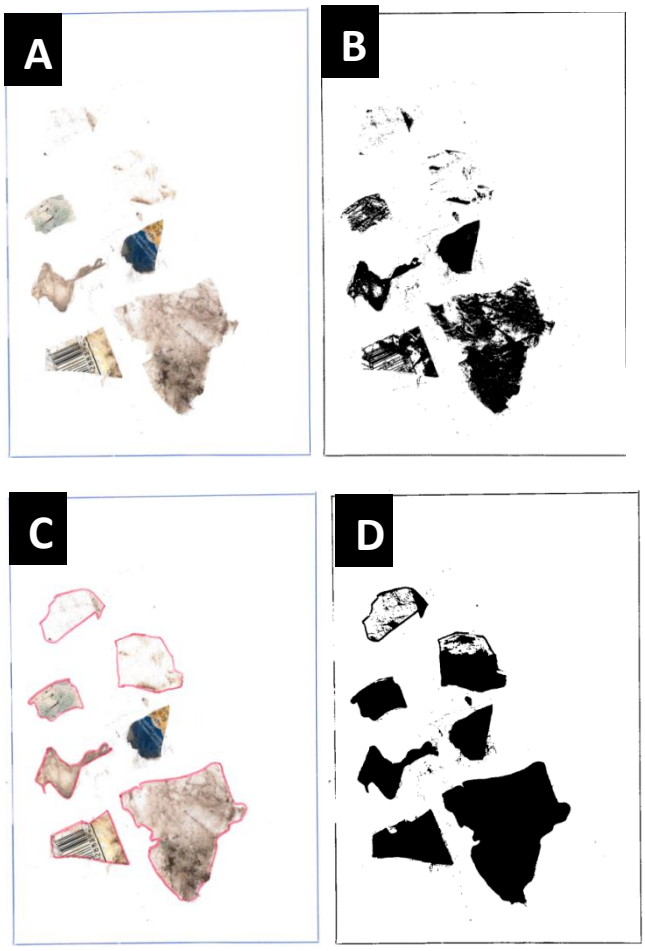
Evaluation on plastics isolated from compost products

As indicated above the German area method does not employ any physical method to enhance detection, relying solely on software (e.g. 'Photoshop') based manipulation of an image taken using visible light only. However, an area method using graphite coating is documented in the US test methods for examination of composting and compost (TMECC, 2002). This method was trialled to enhance detection of transparent film plastics (Figure 2); however, the mess involved in handling graphite powder (particularly in laboratories with electrical equipment) outweighed the potential advantage of this approach.

Figure 2 Enhancing the detection of transparent film plastic by graphite coating (right) alongside uncoated transparent film (left)

As such we found that tracing the outline of problematic fragments either by hand prior to scanning (as illustrated in Figure 3) or within the ImageJ software itself (more suitable for small fragments) using the polygon selection tool was effective.

Figure 3 Enhancing the detection of transparent film plastic by hand outline tracing (C) with conversion to binary image format (D). Untraced scanned image (A) and binary image (B) also shown.



Evaluation of plastics from digestates

Using the area based method in combination with the PAS110 weight method JAS-497/001, fragments of film plastic isolated from separated liquor samples were compared (Table 5). If we assume a sample fresh weight of 1000 g and minimum 0.01 g plastic is required to register the presence of contamination based on weighing and reporting to two decimal places, samples 4.1-4.7 which contained between 2-5 fragments of film plastics, were not detectable using the weight method. Using an analytical balance with readability to 0.001 g would enable some but not all of these samples to register the presence of plastic contamination (depending on actual sample fresh weight). By comparison, on a measuring only basis, the area based method was able to quantify the plastic in all samples.

There was a weak positive correlation (R^2 = 0.45) between weight and area measurements of fragments from samples 4.8-4.15. The main reason for this is likely due to differences in film types (varying thickness and plastic density).

In terms of image processing, the auto selection function was utilised as much as possible to outline fragments in ImageJ for measurement. However, some samples were not straightforward to process, such as those where scratches on reused Petri dishes masked fragments when converting the image to binary format. In this case we used a manual outlining tool in the ImageJ software.

Table 5. Separated liquor sample physical contaminant type, weight and area analysis.

Sample	Contaminants	Weight (g)	Area (cm^2)	Area analysis image processing
4.1	Film plastic only	<0.01	0.11	Binary & auto selection
4.2	Film plastic only	<0.01	0.25	Manual outlining
4.3	Film plastic only	<0.01	0.19	Binary & auto selection
4.4	Film plastic only	<0.01	0.15	Manual outlining
4.5	Film plastic only	<0.01	0.16	Binary & auto selection
4.6	Film plastic only	<0.01	0.08	Binary & auto selection
4.7	Film plastic only	<0.01	1.70	Binary & auto selection
4.8	Film plastic only	0.01	3.74	Binary & auto selection
4.9	Film plastic only	0.02	6.19	Binary & auto selection
4.10	Film plastic only	0.03	13.85	Binary & auto selection
4.11	Film plastic only	0.02	5.29	Binary & auto selection
4.12	Film plastic only	0.01	4.85	Binary & auto selection
4.13	Film plastic only	0.01	12.34	Binary & auto selection
4.14	Film plastic only	0.05	15.78	Binary & auto selection
4.15	Film plastic only	0.03	7.13	Binary & auto selection

Plastics isolated from whole digestate samples were assessed in the same way as those from separated liquor samples, except with weights reported to three decimal places (Table 6). All samples contained plastic fragments; however, two of these contained plastic below the weighing threshold.

The samples above the weight threshold showed a weak positive correlation with the area method - samples 5.6 and 5.8 illustrate nicely how samples with very similar weights can have a large (10 fold) difference in fragment area depending on whether or not rigid plastics are present.

Table 6. Whole digestate sample physical contaminant type, weight and area analysis.

Sample	Contaminants	Weight (g)	Area (cm^2)	Area analysis image processing
5.1	Film & rigid plastic	0.006	2.585	Manual outlining
5.2	Film & rigid plastic	0.002	0.138	Manual outlining
5.3	Film & rigid plastic	0.003	0.519	Binary & auto selection
5.4	Film plastic only	0.001	0.497	Manual outlining
5.5	Film plastic only	0.006	0.829	Manual outlining
5.6	Rigid plastic	0.019	0.469	Manual outlining
5.7	Film & rigid plastic	0.002	0.170	Binary & auto selection
5.8	Film plastic only	0.020	4.090	Manual outlining
5.9	Film plastic only	0.001	0.433	Binary & auto selection
5.10	Film plastic only	0.007	0.305	Binary & auto selection
5.11	Film plastic only	<0.001	0.291	Manual outlining
5.12	Film plastic only	<0.001	0.051	Binary & auto selection
5.13	Film plastic only	0.001	0.465	Binary & auto selection
5.14	Film & rigid plastic	0.008	2.139	Manual outlining
5.15	Film plastic only	0.001	0.451	Manual outlining

3.4.3 Consideration of area based method by REAL appointed laboratories

All four laboratories appointed by REAL were asked to consider the draft area based method. Two laboratories would not consider the method unless it was implemented. The other two laboratories were happy to consider the method, making comments on practicalities and costings during visits to discuss current weight based methods. Both laboratories were of the opinion that the method could be implemented for the analysis of (film) plastics in compost (or digestate). The cost of an area measurement (assuming it was carried out at the same time as weight based measures) was estimated at £7 and £35, respectively.

One laboratory reported that they routinely carry out the PAS100 physical contaminant test on other materials, which may have significantly higher quantities of physical contaminants e.g. potential feedstocks or compost like outputs (CLO). Customers wanting an area based method on these materials may have to pay a higher price.

4.0 Discussion

This section discusses the results of the research in relation to the four main objectives, which were to provide information and data regarding:

1. Determination of whether the current sampling methods (specified by the Biofertiliser and Compost Certification Schemes) are sufficiently robust to deliver truly representative results. Particularly for AD sites where the sampling method needs to accommodate different engineering/design approaches as well as the range of digestate types covered by the PAS (whole, separated fibre and separated liquor). If necessary, the sampling methodologies should be revised to improve clarity and robustness and made available so that the revised version can be implemented by the scheme.
2. Understanding the inter-laboratory variability in physical contaminant results through the testing of key product types with known levels of physical contaminants. Make recommendations (as required) to improve robustness of the methods which can be directly implemented by BCS and CCS.
3. Working with the appointed laboratories to understand the intra-laboratory variability in physical contaminant analysis. Make recommendations (as required) to improve robustness of physical contaminant analysis for both BCS and CCS.
4. Understanding whether the German approach to film plastics (in which their presence is quantified on an area basis, as well as a weight basis) could be implemented in the UK, and at what cost.

Objective 1 – robustness of sampling methodologies

Compost sampling and product variability

Based on the information supplied by 14 PAS100 compost producers, it is clear there is variability in sampling approach and deviation from the CCS sampling guidance. However, all compost producers spoken to appear to be trying to produce a sample for testing that is representative of a product batch through the taking and combining of incremental samples. As highlighted by several producers, there is no point in cheating the system as this would affect customer confidence of their product and the wider industry. That said compost producers are generally neglecting to follow the CCS sampling guidance in terms of the specific number of incremental samples needed based on the specific batch size. Many producers are also not mixing the combined incremental samples (especially when placed

directly in sample containers), perhaps assuming that this will be appropriately carried out by the laboratory.

As shown by the individual samples used in the inter-laboratory trial (presented in Appendix 10), physical contaminants are heterogeneous in compost. Therefore, with virtually all compost producers taking the minimum 12 incremental samples to prepare the composite sample the reported deviations from the sampling guidance are not considered to be critical in the context of physical contaminants; however they may have implications for other PAS100 parameters. Specifically, a number of compost producers reported issues with microbiological testing results, particularly *Escherichia coli*, yet these producers did not seem to consider that their deviations in sampling practice may be a significant factor. In support of this, several producers reported having changed practice in recent months/years to deal with issues around microbiological testing. These included, for example, updating procedures to include the use of pre-sterilised sampling containers, wearing of disposable gloves etc.

Regarding archiving samples, ten compost producers took archive samples. Two of these producers stored samples until results were reported whereas; the others stored them for a minimum six month period as per the sampling guidance. Storage conditions were, however, in the main not optimal for sample integrity (e.g. portacabin office) as are expected to be above the maximum 10 °C stated in the sampling guidance.

RECOMMENDATIONS

Although beyond the scope of this project, a number of recommendations are suggested to be taken forward to improve sampling of compost for PAS100 analysis generally.

- The CCS sampling guidance should be revised to better convey the importance of equipment cleanliness and procedure in collecting samples for microbiological testing which can be found in BS EN12579:2000. We have noted that updated sampling guidance (BS EN12579:2013) contains additional points on microbiological testing.
- The CCS sampling guidance on archive samples should be revised to reflect that found in the document 'actions you are required to take in the event of any test failure'.

Digestate sampling and product variability
The current BCS sampling method guidelines state that:

Individual samples must be taken and combined to derive a final sample for testing that is representative of the sampled portion of production. If a portion of production cannot easily be distinguished from other portions of production, the producer may take traceable samples at defined production time intervals. This may be the case in a continuous flow anaerobic digestion process.

Through discussions with 17 PAS110 AD sites, there is clear variability in some aspects of the sampling approach, with nine sites taking one or two samples which were either directly collected in the sample bottle, or transferred directly from a bucket into a sample bottle, and eight sites obtaining three or more individual samples and combining these to form a final sample. All sites which have been audited stated that the BCS auditor was happy with their sampling method.

Good industry practice was recognised at one site where every tanker was tested onsite for physical contaminants prior to being released from site. The test was a simplified version of the PAS110 test but fit for purpose in quantifying the physical contaminants present.

There was in some cases concern that any time delay in sealing the sample (such as obtaining samples over a series of minutes or hours and combining them) could result in contamination and hence influence the microbiology, or residual biogas potential (RBP), results. Moreover, operators generally felt that the sample taken was representative of the final product, due to the continuous flow nature of the process and internal mixing systems.

Sites receiving packaged and municipal food waste highlighted that the removal of physical contaminants was an integral part of their AD process, and that achieving very low or zero levels of physical contaminants in the final product was vital to maintaining customer confidence of their product and the wider industry. Views were divided as to whether the addition of a surface area method for film plastics to the PAS110 suite of tests would be of benefit. Those in favour highlighted that end users would have additional information which is important for spreading and visibility of film plastics. These producers were open to the idea of paying a small additional cost for surface area measurement. Those against felt that the current physical contaminant method was sufficient (with current PAS110 physical contaminant limits), and some expressed concern that the price for digestate testing would increase if the surface area method was introduced.

Another aspect which did vary between sites was whether warm digestate samples were placed directly in the cool box and collected the same day, or whether samples were placed in a fridge and only placed in a cool box and collected when cool. Although this should not influence results for physical contaminant analysis it may be important for other parameters.

Finally, regarding the requirement for physical contaminant testing of separated liquor samples; although the PAS110:2014 states that physical contaminant test is not required on materials sieved to 2 mm, we found physical contaminants (>2 mm) in separated liquor sieved on site to 2 mm.

RECOMMENDATIONS

Based on this work, one recommendation for digestate producers would be to:

- Consider tightening their product quality control procedure through onsite testing of individual loads destined for market in terms of physical contaminants. This was being effectively achieved by one producer through the use of a simplified physical contaminant test. The approach is more likely to protect against dispatch of unsuitable quality of digestate in the event of potential plant (e.g. screen) failure.

Through this work, one recommendation is suggested for future revisions of PAS110.

- Reconsider the exemption of screened (<2 mm) separated liquors from physical contaminant testing. Instead, all digestate products should be tested for physical contaminants regardless of onsite screen aperture size. As shown in this project, a 2 mm screened separated liquor was found to contain physical contaminants. Although within the current PAS110:2014 limit (assuming reasonable kg/tonne N content), with some markets driving for significantly lower physical contaminant limits this may not be the case in the future. In addition, exemption from testing makes no allowance for potential failure or deterioration of onsite screening equipment.

FURTHER WORK

In order to gain further insight into whether spot digestate sampling (widely used by the industry) is truly representative, two options for further research are proposed:

1. Desk based review of sampling practice in other European countries with similar schemes to PAS110
2. Site work to assess sample variability both by taking digestate samples as per the BCS guidelines and by individual spot sampling (at various time intervals). In addition, this work should consider multiple AD sites with different systems. Note this work will initially require the improvement to the physical contaminant analysis method to provide robust data to be able to draw conclusions.

Objective 2 –Inter-laboratory variability

Physical contaminant analysis of compost

As anticipated, a key aspect on inter-laboratory variability was in the identification and classification of physical contaminants due to individual judgements required in the method. This was particularly the case for 'other' physical contaminants (those clearly not stones, glass, metal or plastic). Obviously the guidance cannot be exhaustive in terms of the type of physical contaminants that may be present but the wording could be developed both for 'other' and 'stones' classification to improve consistency.

In the inter-laboratory trial at least two laboratories classified a few 'glass' fragments under 'other' identifying them as quartz. If these were indeed quartz fragments (hard to tell on naked eye inspection alone) it would appear they should have been included with 'stones' as consolidated materials (assuming they were >4 mm). The classification of fragments (rightly or wrongly) under 'glass' can therefore have a huge impact on whether a sample fails the 'total' physical contaminant category. From discussions with the laboratories there also seemed to be confusion around whether or not 'natural' physical contaminants such as bone and shells should be included in 'other'.

The other key issue in terms of variability between laboratories relates to the drying process. Prior to conducting the inter-laboratory trial, one laboratory confirmed that they took samples to dryness at 105°C (rather than air-drying at 40°C to less than 15% moisture). Although this temperature is used in methods on the continent, those methods are physical contaminant only tests i.e. are not carried out in conjunction with particle size distribution (PSD). This can be problematic particularly when drying sticky compost (such as some in-vessel composts) which can form stone-like organic particles. These stone-like organic particles may affect the PSD component of the PAS100 method (Blok & Wever, 2006). In addition, drying at 105°C may be a problem for some plastics (such as PVC) which have melting temperatures below this.

The other two laboratories stated that they follow the protocol by drying at 40°C. This typically involved drying for a set period of time so, depending on the wetness and composition of the sample, the moisture content could vary. With this in mind, an attempt was made to calculate the final moisture content of samples processed by these two laboratories. Specifically, the 'compost retained' weight reported on the PSD table was considered alongside known weights and moisture contents of compost samples supplied to the laboratories, however, this approach was found not to be robust. One of the laboratories however did report moisture content data of the air dried material. Samples processed by this laboratory varied in moisture content from 0-5%, with variability within a single product type around 2-3%.

Finally, one laboratory commented that occasionally it was not possible to dry compost to <15% moisture even after a week using a 40°C drying process. Although the laboratory in question followed the published method they felt it would be better at a higher temperature (e.g. 105°C).

RECOMMENDATION

A number of recommendations are suggested to improve consistency in analysis of physical contaminants in compost:

- Results should be reported on DM rather than 'air dried' basis. A combination of anecdotal evidence and data show that there is variability in drying practice by the laboratories which for a test reporting based on 'air dried' compost is a source of variability. Reporting based on DM would bring the method in line with those in Europe (Hogg et al., 2002). NOTE – this recommendation does not suggest that the material itself should be completely dried as this could affect the PSD analysis particularly 'sticky' composts such as some in-vessel composts.
- Amend the guidance on physical contaminant classification found in AfOR MT PC&S version 2 by incorporating the following underlined wording:
    - *extraneous, hard mineral matter greater than 4mm in any dimension NOTE Does not include glass, plastic or metal, but does include pebbles and pieces of aggregate, concrete, tile, rubble, pottery and any other consolidated mineral particles (including graphite and quartz) greater than 4 mm in any dimension.*
    - *paper, cardboard/fibreboard, rubber, polystyrene, textiles/fabric, string/rope and any man-made materials other than glass, metal and plastic, which are > 2 mm in any dimension. 'Unknown' is an appropriate identification for fragments which are clearly **man-made** physical contaminants but not glass, metal or plastic.*
- The physical contaminant analysis report template should be updated to include common fragment type codes for the 'other' category description and create consistency between laboratories.
- The physical contaminant analysis report template should be updated to blank out the table 'cell' for 2 mm stones
- As well as improving the wording of the guidance on physical contaminant classification it may be appropriate to offer training for analysts given the high level of human decision making in the method – this approach would be consistent with other industries such as asbestos analysis in soil. Given issues with misclassification and identification of physical contaminants it would give confidence to compost producers if analysts held basic training common to all laboratories. This could be in the form of half day training, ideally with analysts from the different laboratories, where samples of typical and more unusual physical contaminants can be looked at and best practice shared. The analysts could also then be tested with spiked samples. Analysts completing the training and returning satisfactory results for the spiked samples could be awarded a certificate of competence.

FURTHER WORK

This project highlights a couple of aspects of further work.

- Effect of 'air drying' and taking to dryness on PSD. Discussion with one of the appointed laboratories on this topic also supported the idea that compost PSD could be affected (particularly in-vessel composts) by drying.

- Impact of drying at higher temperatures on physical contaminant integrity – does drying at 105 °C affect plastic integrity? There are both 'pull and push' factors for this with one laboratory already drying at this temperature and a second laboratory with a preference for this temperature due to problems drying some samples at 40 °C. The prime consideration is the effect of drying temperature on integrity of physical contaminants.
- Incorporating techniques to the PAS100 method to facilitate isolation and identification/classification of fragments.

Physical contaminant analysis of digestates

The test method referenced in this project is version JAS-497/001 which was produced in 2010 when physical contaminants were evaluated under PAS110:2009 on a DM basis. This test method was updated in 2014 (JAS497/002) in line with PAS110:2014 in which physical contaminants are reported on a FM basis. To avoid inconsistency, and to aid interpretation of inter-laboratory trial data, the laboratories were asked to report physical contaminants on a fresh weight only basis. The balance readability limit was not stipulated to the laboratories to reflect the omission of this information in the JAS-497/001 method.

The results of the inter-laboratory trial showed inconsistency in raw contaminant weight reporting. Assuming JAS-497/001 is a single method for all three digestate types, this is a source of both inter and intra-laboratory variability.

Informed by our own evaluation of the method and discussion with the two laboratories, three further (potential) sources of inter-laboratory variability were identified. The first of these was around the use of bleach to remove organic material as this is currently an optional component of the method both for whole digestate/separated liquor and separated fibre. For example;

8.1.9 If, after the drying of the whole digestate material, it is hard to separate the material to determine the contaminants then the material may have to undergo further preparation using the bleach washing process

In practice, speaking to laboratories after analysis of the spiked samples was complete; both used bleach for the separated fibre samples whereas no bleach was used for the whole digestate and separated liquor. Despite this, the use of bleach for the different sample types should be qualified to limit variability in the future. In support of this, we found from our own experience with the method, that retention of flexible physical contaminants such as film plastics could alter depending on whether or not bleach was used.

The second potential source of inter-laboratory variability was in the washing of sieves, the method states:

8.1.7 In order to do the washing the sieves are taken off the bucket/container and then the 2 mm sieve is rinsed with a fine mist of water (e.g. using tap attachments such as hoselock) or with tap water at very low velocity.

We do not have data at this time to support this, however we believe sieve washing should be considered in future revisions of the method.

The final source of variability was in the behaviour of film plastics with at least one dimension > 2mm. We have found that fragments >2mm may pass through the 2mm sieve and subsequently become trapped on a 2 mm in a second pass.

There was also evidence to support the need for a revision in the reporting limit/units of the method. Specifically, many whole digestate and separated liquor samples were below the reporting limit which was reported to two decimal places on a kg/t basis as per PAS110:2014, yet they clearly contained physical contaminants. With at least one market now demanding limits of around 8 % of the current levels, this would make the method unsuitable in its current form.

RECOMMENDATIONS

The JAS-497/001 (now JAS-497/002) method appears to be a good straightforward method for the evaluation of digestates (especially whole and separated liquors) with apparent low inter-laboratory variability however; a number of amendments are suggested to ensure consistency.

- The BCS should consider whether or not bleach needs to be used routinely (for the different digestate types). This decision should be taken with involvement of several commercial laboratories. If it is decided that bleach should be used routinely on digestate samples, then the method should state the type and concentration of bleach to be used (currently lacking).
- The number of decimal places for weighing should be stipulated in the method. Evidence from this project suggests weighing/reporting to four decimal places would be appropriate.
- The method needs improvement to ensure that fragments >2 mm are consistently captured and quantified

Objective 3 – Intra-laboratory variability

Physical contaminant analysis of composts

Following discussions with the laboratories it was identified that there was typically one analyst responsible for processing the majority of compost samples, with 2-3 other staff covering absence in busy periods. As such the method is susceptible to intra-laboratory, as well as, inter-laboratory variability in terms of subjective identification and classification of physical contaminants. The method is also susceptible to intra-laboratory variability in terms of reporting results on an 'air dry' rather than dry matter basis with individual samples of the same product type varying in initial moisture content and speed of drying.

RECOMMENDATIONS

- Train analysts (as previously discussed)
- Audit laboratories – an activity currently organised by REAL

Physical contaminant analysis of digestate

As previously stated, reporting weight basis results to different decimal places varied for one of the laboratories and created intra-laboratory variability.

RECOMMENDATIONS

- Update the method to include details on weighing accuracy
- The subsequent auditing of laboratories and their standard operating procedures

Objective 4 – area based methods

Application of area based method for compost

Discussion with two commercial laboratories and our own in-house evaluation confirm that an area based method could be applied to evaluate plastics in PAS100 compost. This was not fully explored in this project, but the method could also be used to evaluate other physical contaminant categories.

As outlined in the introduction, the driver for this aspect of the project was the lowering of the PAS100 limits to 50% of current levels for certain markets. Our evaluation, suggested that even at the reduced level the weight based method could still be considered sufficiently robust using a two decimal place balance. Speaking to compost producers, feelings were mixed about the idea of an area method. Those against it were more concerned about microbial pathogens or market specific (agricultural) end use criteria.

RECOMMENDATIONS

Compost producer opinion on the area based method was sought as part of this project; however, it was only a very minor aspect and as such no clear conclusion on the true interest can be drawn. As such we would recommend the CCS undertake a poll of a larger representation of PAS100 compost producers before deciding whether or not to further consider the area based method.

Application of area based method for digestate

Concerns were raised at the start of this project that the small size and large number of fragments may be an issue for laboratories to quantify, however, our experience was that the method was very simple to perform and potentially straightforward to incorporate the area based measurement alongside weight determination. In addition, improvements to depackaging processes together with finer onsite screening means that weight/number of physical contaminants is decreasing in end products and as such many samples tested in this project were below the method reporting limit.

The area method is clearly sensitive and robust based on analysis of the whole and separated liquor digestates tested here, detecting to the individual fragment level. In addition, reporting on weight only basis does not differentiate between many film plastic fragments and a few rigid plastic fragments. As such the reporting of physical contaminants by area alongside weight would provide useful information for both digestate producers and end users.

Physical contamination in separated fibre is highly variable depending on feedstock (e.g. commercial food waste only or crop biomass (e.g. maize) and food waste). Therefore it is less clear cut at this stage what recommendations to make based on the assessment of one source of fibre as to the use of the area based method.

RECOMMENDATIONS

Given the discussed benefits of the area method for whole digestate and separated liquor samples (the current main digestate products), and interest from the industry we would recommend the method is taken forward for further evaluation.

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Appendix 1 – Compost producer question set

The following general questions will help us to put the results into context.

Operational system

- ☐ Windrow, IVC, other (technology used?)
- ☐ Feedstock types and quantities
- ☐ Source of feedstocks
- ☐ Cleaning pre-shredding or composting e.g. handpicking

Composting volume:

- ☐ Product types – principal/additional (growing medium component, soil improver, mulch, topdressing, grades of each) and quantities?
- ☐ Current separating equipment
- ☐ Easy/ borderline/ hard to achieve PAS100 for each product?

Q. Compost product	Glass	Metal	Plastic	Other	Stones
1.					
2.					
3.					

Q. How long is the PAS100 composting process as defined at your site i.e. the period until sampling? (in days/weeks)
or
Q. For how long (on average) and how is the digestate fibre stored prior to sampling? (in days/weeks). Are there season effects?

Sampling questions

The original text from the Guidelines is included as green text, to understand the background to each question.

3. Equipment

Q. What equipment do you use for sampling?

- ☐ Bucket/spade/trowel/mixing sheet/bags/etc
- ☐ Is equipment washed and dried before each product sampling
- ☐ Other ……

4. Stage at which to take samples

Q. At what stage in the process is the product sampled?
Q. How often is the product sampled (times a year?)?
Q. Typical batch size?

5. Size and number of incremental (individual) samples

Q. What is the size and number of incremental (individual) samples per batch? If not incremental, how do you sample? (from a moving stream of product?)

6. Sampling procedure

Q. Please describe your sampling procedure

- Where are the samples taken from (top of the windrow, deep down etc)?

7. Sample preparation

Q. Describe how you prepare the sample (combining incremental samples) and create the laboratory and archive sample.

8. Storage of archived sample

Q. What container do you store the archive sample in? Where do you store the archived sample?

9. Laboratory sample

Q. How do you package and send the laboratory sample or is this another person's responsibility?
Typically, how long is a laboratory sample stored before dispatch? What is the minimum and maximum?

Final open questions:

Q. Any thoughts on how the CCS sampling procedure could be made clearer?

Q. Any thoughts on how the CCS sampling procedure could be improved?

Q. What is your level of knowledge/understanding of the PAS100 physical contaminants analysis method? Would you like to know more?

Q. Have you experienced / are you aware of any issues with the PAS100 physical contaminants method?

Appendix 2 – Digestate producer question set

Site questions

Operational system

- ☐ Single stage
- ☐ Two-stage
- ☐ Mesophilic
- ☐ Thermophilic
- ☐ Wet
- ☐ Dry
- ☐ Continuous
- ☐ Batch ('Plug flow')
- ☐ Other……

Digestate storage system and volume:

- ☐ Feedstock types and quantities?
- ☐ Digestate types (whole /liquor /fibre) and quantities?
- ☐ Current separating equipment for pre- and post-digestion?
- ☐ Easy/ hard to achieve PAS110 for this parameter?

		Main types of physical contaminants and proportion (%)			
Digestate type	**Quantity of digestate produced**	**Plastic (specify if film, hard plastic, bags etc)**	**Glass**	**Stones**	**Other**
Whole					
Liquor					
Fibre					

Which physical contaminant limits are easy/borderline/hard to achieve for each output material?

Sampling questions for digestate fibre

The questions designed for compost sites will be used (based in BS EN 12579 procedures), with the following additional questions.

Q. How long is the digestate fibre stored prior to sampling? (in days/weeks). Is there a maturation stage with/ without turning?

Sampling questions for whole and liquor digestates

The numbers correlate to the Biofertiliser Scheme Annex C information.

A 2.4 Stage at which to take samples

Q. How do you choose where to sample from?
Q. Is the sampling point defined in your quality management system?
Q. At what stage of the process is the digestate sampled? (Earlier in the process for RBP?)
Q. Do you have a definite pattern/programme of sampling?
Q. On average, how often is the digestate sampled?
Q. Does the sampling time depend on the operation cycle (eg full treatment duration/minimum storage period)? Y/N. If Y explain…….
Q. Where is the digestate sampled from?
Q. How is the digestate sample removed? Large pipe/ tap/ storage tank from above / other

A 2.5 Size and number of individual samples
Q. Volume of each individual sample?
Q. Are the samples bulked together and then a subsample taken?

A 2.3 Equipment

Q. What equipment do you use for sampling?

- ☐ Sample scoop with/without telescopic rod.
- ☐ Sealed plunging siphon
- ☐ Pump
- ☐ Plastic measuring jug.
- ☐ Mixing container type and size
- ☐ Storage container type and size
    - Is the storage container clean, dry, gas and liquid tight, and able to resist the pressure exerted by the gasses?
- ☐ Bucket
- ☐ Other ……

Note: Before asking the next set of questions, the nature of sampling will be ascertained.
Storage tanks: Closed: 2.6.1 / Open: 2.6.2&3

A 2.6.1 Taking individual samples from closed storage tanks
Q. Is the digestate always mixed prior to sampling? Y/N.
If Y, how is mixing achieved?
If Y, are you able to tell if the layers have been combined?
If not always, under what circumstances?

Q. When using the sealing valve / sampling nozzle, do you take an amount of digestate out of the supply pipe prior to obtaining the sample for analysis?
If yes, how much? E.g. same volume as in supply pipe / double / 3x / 4x

Q. Do you leave a time interval between obtaining each consecutive sample?
If Y how long?

A 2.6.2 & 3 Taking individual samples from open topped storage tanks with/without sampling nozzles
Q. Can you show me your H&S procedure for obtaining samples from open top storage tanks?
When did you last receive training on this?

Q. Is the digestate mixed prior to sampling? Y/N.
If Y, how is mixing achieved?
If Y, are you able to tell if the layers have been combined?

Q. When using the sealing valve / sampling nozzle, do you take an amount of digestate out of the supply pipe prior to obtaining the sample for analysis?
If yes, how much? E.g. same volume as in supply pipe / double / 3x / 4x

Q. Do you leave a time interval between obtaining each consecutive sample?
If Y how long?

A 2.6.4 Taking individual samples from pipes to/ from an external circulation pump

Same Qs as for 2.6.2

A 2.6.5 Taking individual samples when discharging or dispatching digested materials for use

Same Qs as for 2.6.2

A 2.7 Deriving a final sample for testing

Please can you describe to me the steps you go through to obtain the final sample that you send to the laboratory?

Q. What size sample bottles do you use, and how many are needed each time you take samples?
Q. What is the total volume of your mixed individual samples (in litres)?
Q. Do sink and float layers appear in this combined sample?
If Y, is this mixed prior to taking out the final sample?
Q. Are the lab and archive samples prepared at the same time in the same way?
If not, what happens differently?

A 2.8 Final sample container

Q. Type of sample container

A 2.9 Final sample labelling

Q. How are samples labelled? Words/ code?

A 2.10 Final sample storage prior to collection

Q. How are the final sample containers (containing the digestate) stored before collection by courier?

- ☐ In the dark/ light
- ☐ In a fridge
- ☐ 2-8°C
- ☐ Other…….

- ☐ Is storage temperature and length of storage time recorded?
- ☐ Average (or range) length of time stored prior to collection in hours / days

A 2.11 Time between obtaining the final sample and sending it to the lab

Q. After sampling

- ☐ Are the samples stored as above for the whole time until collection? If no, how else are they stored?
- ☐ When are the samples collected? Same day/ next day/ other…….
  What is the minimum and maximum?

A 2.12 Final sample transport

Q. How do you package and send the final samples to the lab?

- ☐ Insulated cool box
  - Plastic/ polystyrene/ other…….
  - Does the cool box contain a frozen block?
- ☐ Small refrigerator

- ☐ Refrigerated van
- ☐ Unrefrigerated van - courier
- ☐ Other

Q. Are the transport containers, van storage racks and/or boxes
- ☐ used exclusively for this purpose?
- ☐ kept clean and dry when not in use?
- ☐ regularly disinfected? – how often?

Q. Prior to use, are the transport containers:
- ☐ kept indoors in a clean area
- ☐ kept indoors in a general area
- ☐ kept covered outdoors
- ☐ kept uncovered outdoors

Final open questions:

Q. Any thoughts on how the BCS sampling procedure could be made clearer or improved?
Q. What is your level of knowledge/understanding of the PAS110 physical contaminants analysis method? (only use if staff unsure):
Q. Have you experienced / are you aware of any issues with the PAS110 physical contaminants analysis method?
Q. WRAP have been informed by digestate customers that film plastics are a major concern, and since they are so light there is a possibility that the current PAS test won't detect them accurately. An option would be to have an area method for film plastics, but this hasn't been tested in the UK yet. Any thoughts?
Q. In Germany they use an additional method which looks specifically at the surface area of plastic films. Do you feel this would be a useful inclusion to PAS110? (method described briefly if required).

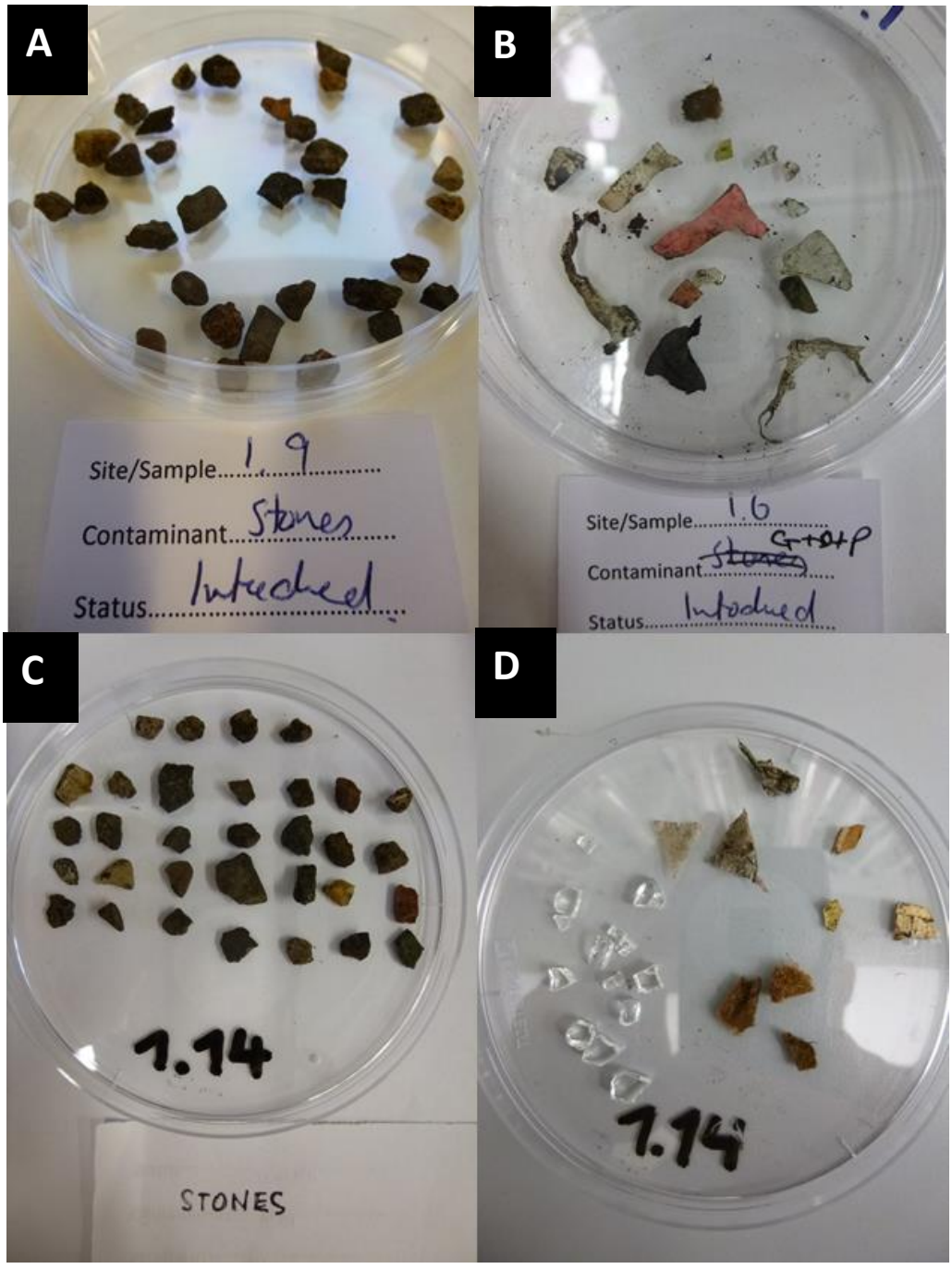
Appendix 3 – Initial characterisation of compost and digestate samples

The fragments were weighed before and after drying, and after removal of loosely bound organic material. The fragment types were photographed after removal of loosely bound organic material. Moisture content of the compost was determined by gravimetric method (drying at 105 °C) before and after removal of fragments to allow for change in moisture content during manipulating. The clean compost samples were weighed to 1 kg fresh weight, bagged and stored at 4 °C until spiking (section 2.2.1).

Physical contaminants were removed from ~800 ml of whole digestate and separated liquor following the PAS110 method JAS-497/001 (Determination of Physical Contaminants and Stones' in Digestate. NRM Laboratories, 2012) after removal of two 100 g subsamples for dry matter (DM) determination. A consistent volume of water (40 ml) was used to swill out the samples bottles and rinse the sieves. For both whole digestate and separated liquor, undiluted 'thin' domestic bleach was used (as per the JAS-497/001method) to facilitate detection of physical contaminants and ensure a consistent approach between samples. The separated physical contaminants (exclusively plastics in both products) were placed onto pre-weighted clear plastic Petri dishes enabling direct determination of both weighing and area. The resulting clean digestate samples were returned to their original bottles, weighed and stored at 4°C until spiking (section 2.2.1).

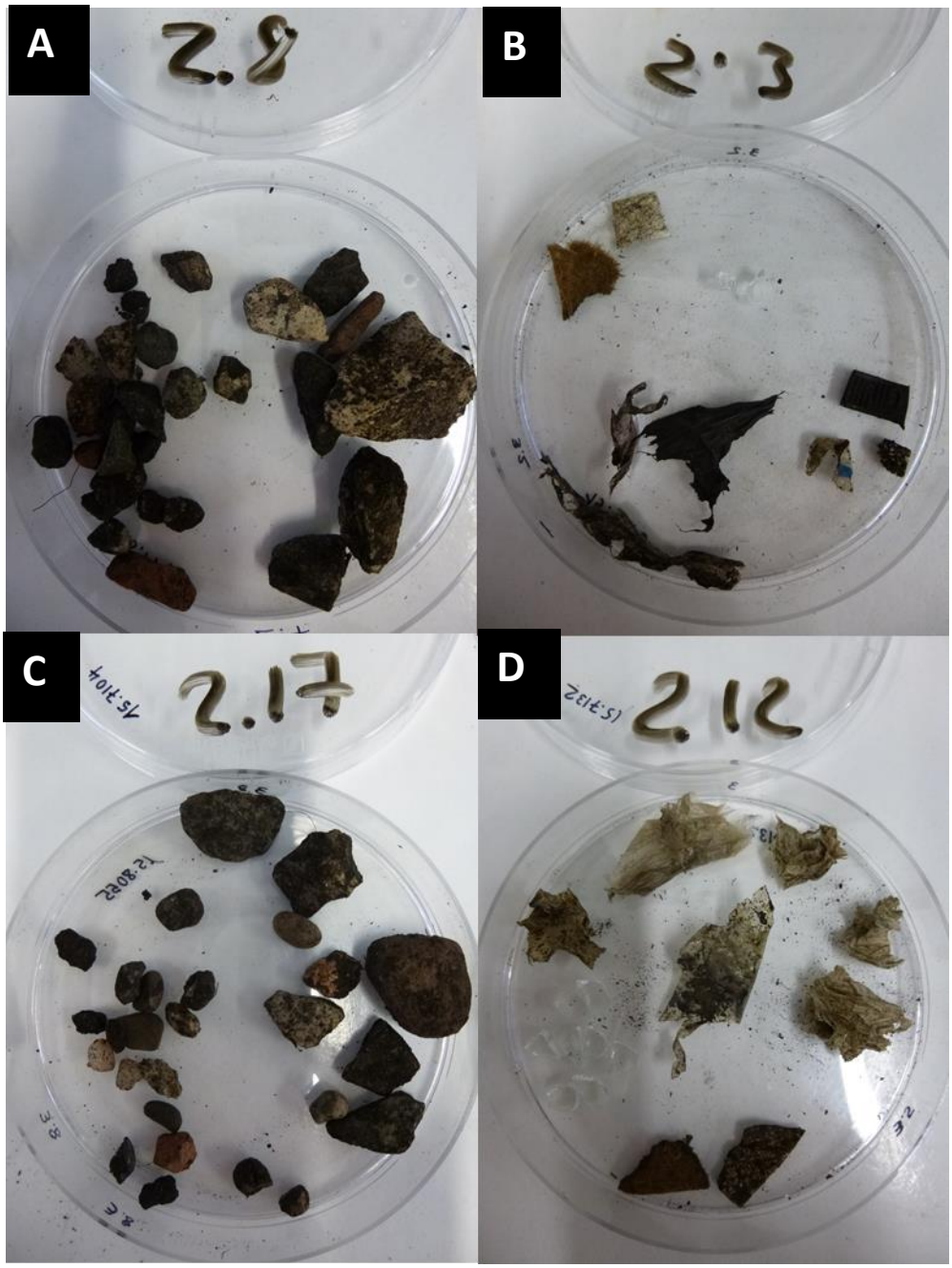
Physical contaminants were removed from separated fibre samples by hand picking after spreading onto trays. This approach was used to ensure integrity of the material for the inter-laboratory trial which would otherwise have been compromised by drying and/or bleach treatment. The physical contaminants were classified into glass (>2mm), plastic (>2mm), metal (>2mm), 'other' (>2mm) and stones (>5mm). The contaminant fragments were weighed after drying (40°C) and removal of loosely bound organic material. Two 100g subsamples of the cleaned fibre samples were taken for dry matter (DM) determination. Cleaned fibre samples were returned to their original bags, weighed and stored at 4°C until spiking (section 2.2.1).

Appendix 4 – Spiking of compost site 1 samples



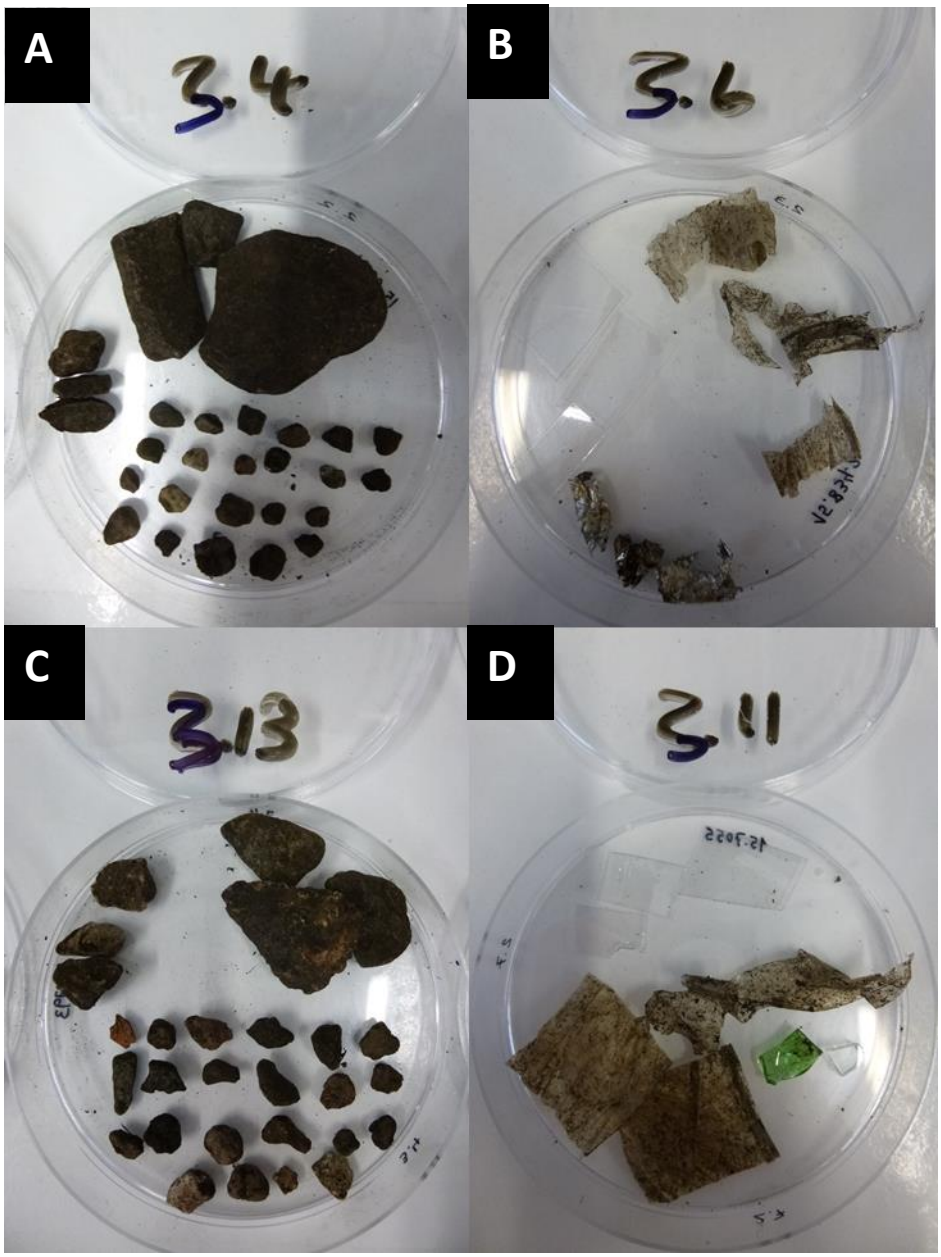
Spiking in site 1 compost samples. A+C) 32x stones both low and high loading, B) 6-7x glass, 6x plastic, 3x other – low loading, D) 14x glass, 6x plastic, 3x other – high loading

Appendix 5 – Spiking of compost site 2 samples



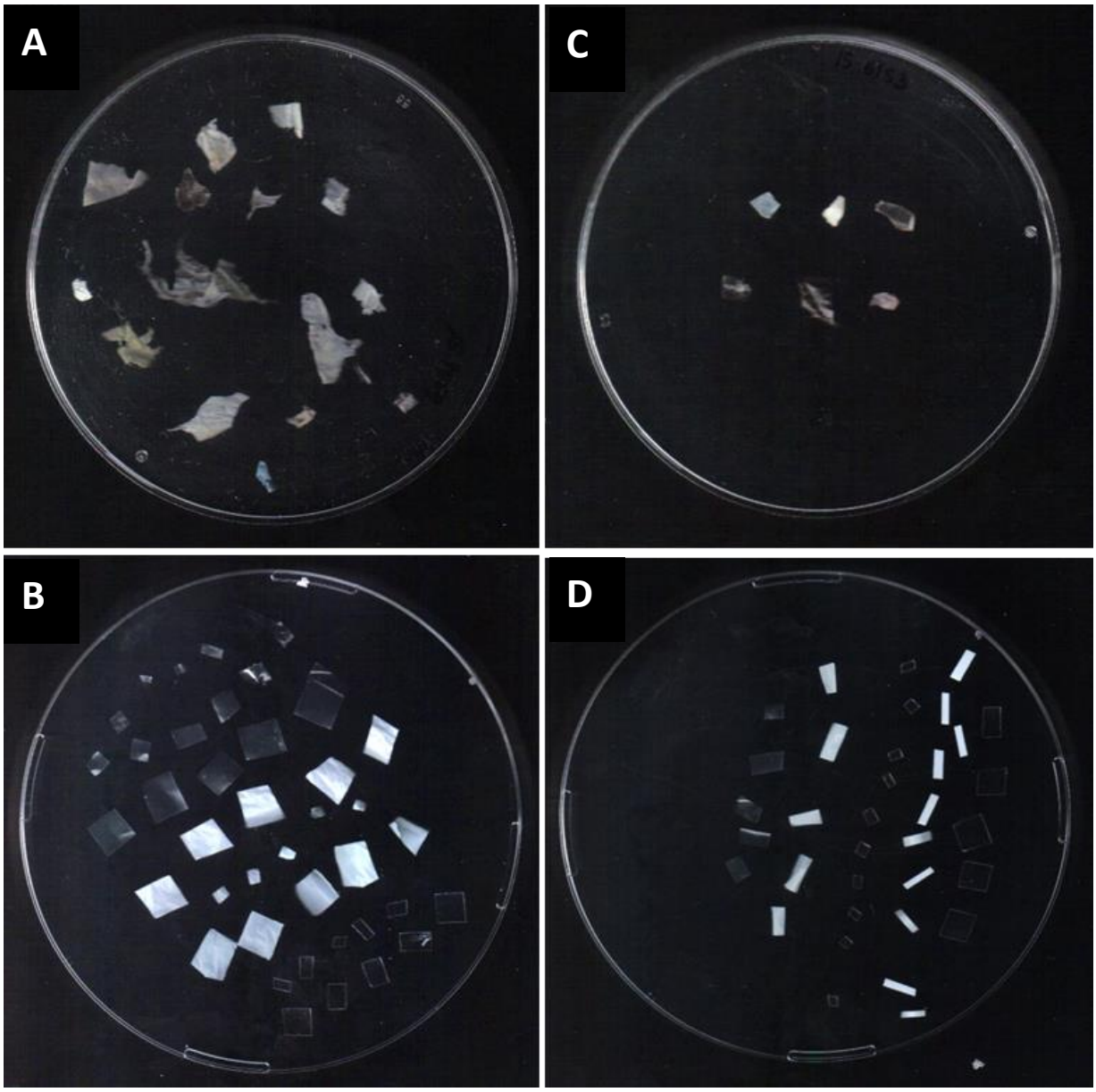
Spiking in site 2 samples. A+C) 27x stones both low and high loading, B) 2x glass, 6x plastic, 2x other – low loading, D) 8x glass, 6x plastic, 2x other – high loading

Appendix 6 – Spiking of compost site 3 samples



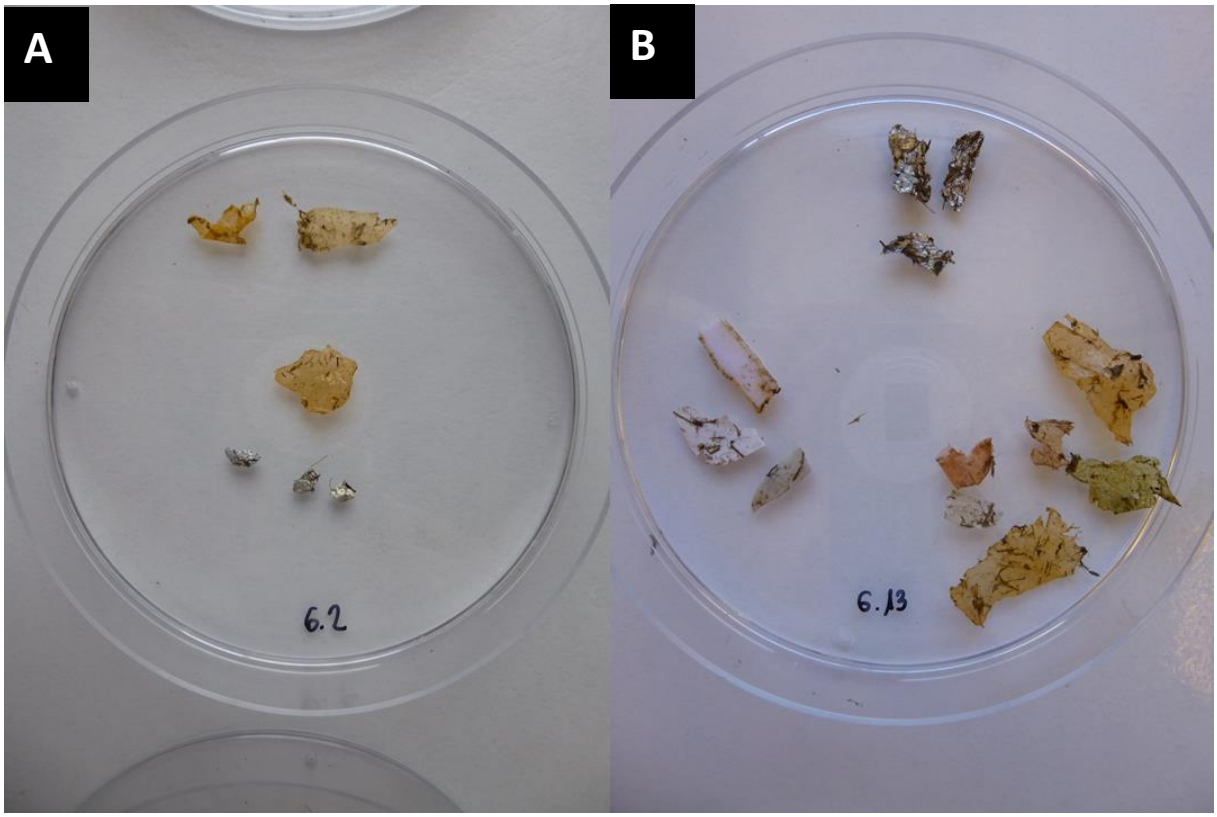
Spiked in site 3 compost samples. A+C) 28x stones both low and high loading, B) 6x plastic and 3 x metal foil – low loading, D) 2x glass and 6x plastic – high loading.

Appendix 7 – Spiking of whole digestate and separated liquor samples



Spiked in site 4 samples A) 15 film plastic – 90 mm plate B) 30 film and 10 rigid plastic – 150 mm plate.
Spiked in site 5 samples C) 3 film and 3 rigid plastic fragments – 90 mm plate, D) 10 film and 25 rigid plastic fragments – 150 mm plate

Appendix 8 – Spiking of separated fibre samples



Spiked in site 6 samples. A) 3x film plastic and 3x silver foil – low loading, B) 6x film plastic, 3x rigid plastic and 3x silver foil – high loading

Appendix 9 – Film plastic surface area determination by digital processing

Materials/Equipment:

- Scanner (flat bed, minimum 8-bit grey scale)
- A4 sheet of white paper
- Single sided colourless and transparent adhesive tape (e.g. Sellotape®)
- Image analysis software. *Instructions in red below are based on using the publicly available software ImageJ which can be freely downloaded to fully evaluate the surface area method (http://imagej.nih.gov/ij) if commercial software is not readily available. Alternative software such as Paintshop Pro or SigmaScan may be required for commercial use.*

Procedure:

1. Unfold/ uncrumple (so far as is practicable) film plastic fragments (previously extracted using the PAS100 or PAS110 method) from a single sample of compost/digestate and lay on one side of a single sheet of A4 paper, leaving space between each fragment.
2. Stick fragments to the A4 sheet using pieces of single sided adhesive tape.
3. Scan the A4 sheet (with stuck fragments) and save as a .tiff or .tif file
4. Open the saved scanned .tiff or .tif file in ImageJ using the File>Open menus
5. Adjust the scale using Analyse>Set Scale menus. Adjust 'unit of length' to cm. Adjust 'distance in pixel' to no. pixels corresponding to A4 sheet width. Adjust 'known distance' to actual A4 sheet width (in cm).
6. Adjust the image using Process>Binary menus. Select 'Make Binary'
7. Open ROI manager using Analyse>Tools menus. Select 'ROI manager'
8. Tick box 'labels' in ROI manager
9. Select 'Wand' from ImageJ main toolbar to pick fragments
10. Highlight one fragment using the wand tool. Once highlighted press 'Add' in ROI manager
11. Continue to add fragments to ROI manager as in 10
12. Once all fragments have been added select 'Measure' in ROI manager
13. Areas (cm^2) of individual fragment can then be summed and reported per sample weight (or volume) as required

The area determination of irregular shaped fragments should be verified by scanning and measuring the area of a regular object of known area

Appendix 10 – Physical contaminants in individual original compost samples

Statistical analysis of data derived from sub-samples

The full data per site and product for the incremental sub-samples are shown along with statistical analysis and histograms. Note that if the data are not showing a 'normal' distribution in the histogram for a contaminant then the statistics shown may not be relevant.

The data may be skewed (so not a normal distribution) by, for example, a larger piece of metal or glass, a piece of hard plastic amongst film plastic, etc.

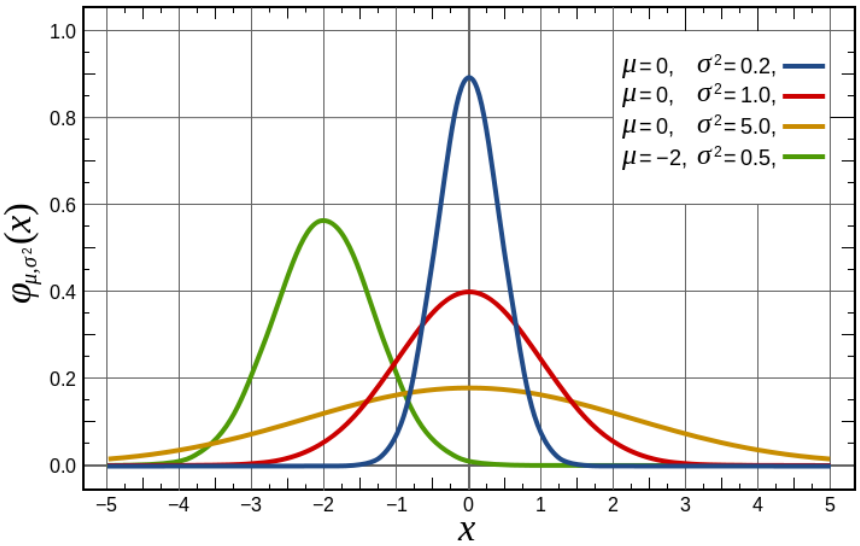


Figure X http://en.wikipedia.org/wiki/Normal_distribution

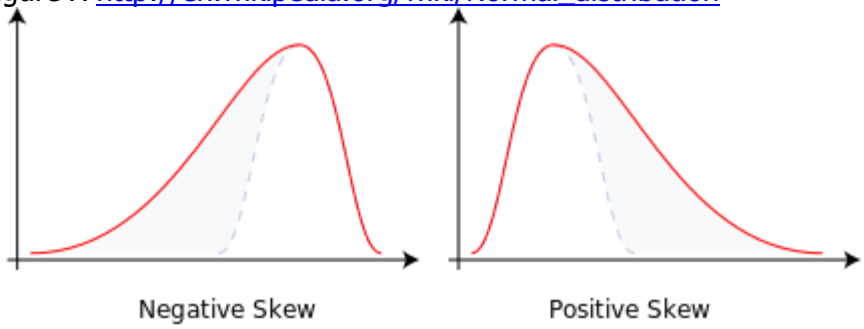


Figure Y http://en.wikipedia.org/wiki/Skewness

The PAS100 upper limit for the total glass, metal, plastic and any 'other' non-stone fragments > 2mm is 0.25%, of which 0.12% is plastic, mass/mass on 'air dry' sample. Stones > 4mm in grades other than 'mulch' shall be < 8% mass/mass 'air dry' sample.

Site 1 analysis

From the averaged data of the sub-samples of the 0-10mm compost taken from Site 1, the compost would have passed PAS100 for non-stone physical contaminants and stones. No metal was found in 16 out of 20 samples. The data tend to be positively skewed. The totals of the non-stone contaminants per sub-sample are primarily affected by the glass found therein, with six of the 20 sub-samples being over the PAS100 upper limit.

How many sub-samples should be taken from a batch? If there is a great deal of variability in sub-samples, more sub-samples should be taken; otherwise there is a greater chance of a final portion that is sent to the laboratory having a higher level of contamination. For Site 1, there is a 1 in 20 chance that the total contaminants will exceed the upper limit in a portion purely due to the variability when 20 sub-samples are taken. If only 12 sub-samples are taken there is a 1 in 16 chance; 6 sub-samples, 1 in 7.

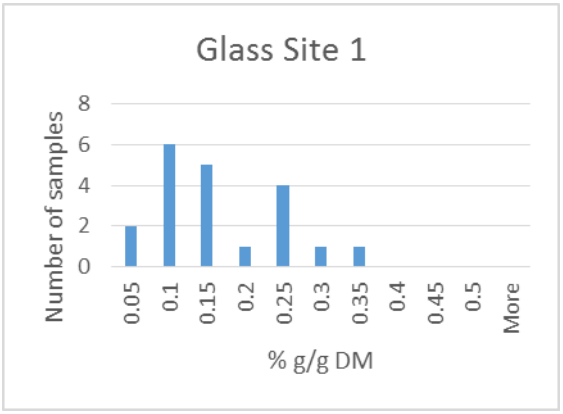
Table 1. Physical contaminant characterisation of 20 discrete compost samples from site 1 (0-10 mm product). Grey shaded cells indicate those over current PAS100 limits

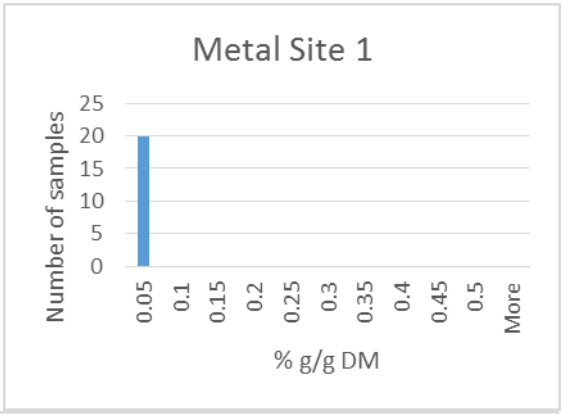
Sample	Physical contaminants (% g/g DM)					
	Glass (>2 mm)	Metal (>2 mm)	Plastic (> 2 mm)	Other (> 2 mm)	Total (>2 mm)	Stone (> 4mm)
1.1	0.10	0	0.01	0.06	0.17	2.99
1.2	0.11	0	0.03	0.03	0.17	1.33
1.3	0.09	0	0.03	0.02	0.14	2.43
1.4	0.10	0	0.04	0.04	0.18	2.05
1.5	0.04	0	0.01	0.01	0.06	0.58
1.6	0.06	<0.01	0.02	0.03	0.11	1.02
1.7	0.31	<0.01	0.02	0.04	0.37	3.35
1.8	0.08	0	0.04	0.04	0.16	1.32
1.9	0.11	0	0.01	0.12	0.24	1.48
1.10	0.13	0	0.02	0.02	0.17	1.65
1.11	0.05	0	0.02	0.01	0.08	1.23
1.12	0.08	0	0.03	0.04	0.15	1.59
1.13	0.30	0	0.04	0.04	0.38	2.28
1.14	0.22	0	0.03	0.02	0.27	1.77
1.15	0.17	0	0.02	0.07	0.26	2.71
1.16	0.21	<0.01	0.02	0.02	0.25	3.91
1.17	0.22	0	0.06	0.02	0.30	4.32
1.18	0.22	<0.01	0.07	0.06	0.35	2.21
1.19	0.15	0	0.01	0.02	0.18	1.90
1.20	0.11	0	0.03	0.03	0.17	3.16

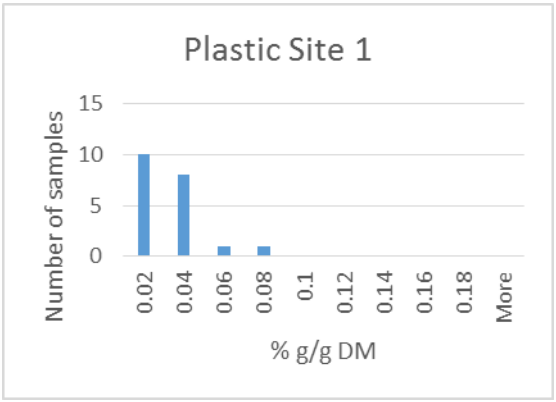
Site 1 Statistics

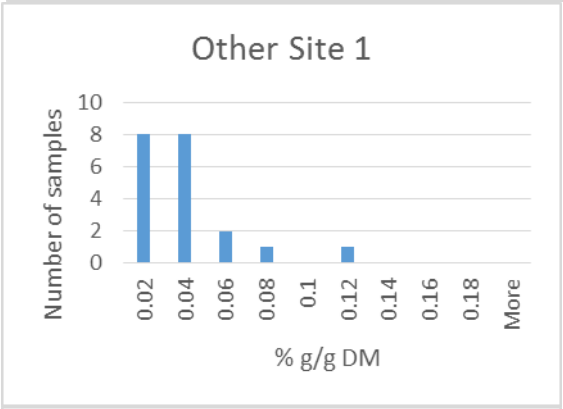
	Glass	Metal	Plastic	Other	Total	Stone
	(>2 mm)	(>2 mm)	(> 2 mm)	(> 2 mm)	(>2 mm)	(> 4mm)
PAS Limits=			**0.12**		**0.25**	**8**
Count=	20	20	20	20	20	20
Mean=	**0.143**	**0.000**	**0.028**	**0.037**	**0.208**	**2.164**
Median=	0.110	0.000	0.025	0.030	0.175	1.975
Max=	0.310	0.000	0.070	0.120	0.380	4.320
Min=	0.040	0.000	0.010	0.010	0.060	0.580
StDev s=	0.079	0.000	0.016	0.026	0.092	0.988
Variance s2=	0.006	0.000	0.000	0.001	0.0084	0.977
Stderr=	0.018	0.000	0.004	0.006	0.020	0.221
look up t	2.1	2.1	2.1	2.1	2.1	2.1
for d.f. =	19	19	19	19	19	19
95% C.I.(20)=	0.037	0.000	0.008	0.012	0.043	0.464
Mean+C.I. (20)=	0.180	0.000	0.036	0.049	0.251	2.628
95% C.I.(12)=	0.050	0.000	0.010	0.016	0.058	0.622
Mean+C.I. (12)=	0.193	0.000	0.038	0.053	0.266	2.786
C.V. s/mean=	55.5	n.a.	57.5	69.1	44.0	45.7

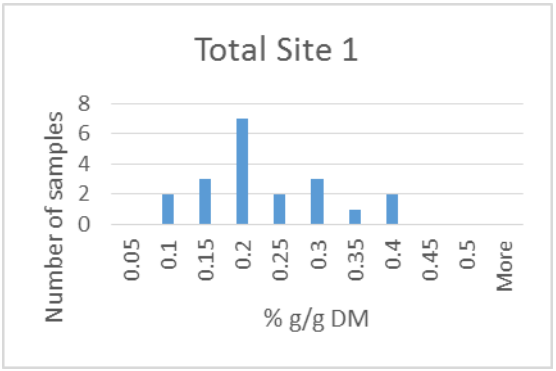
Histograms for Site 1

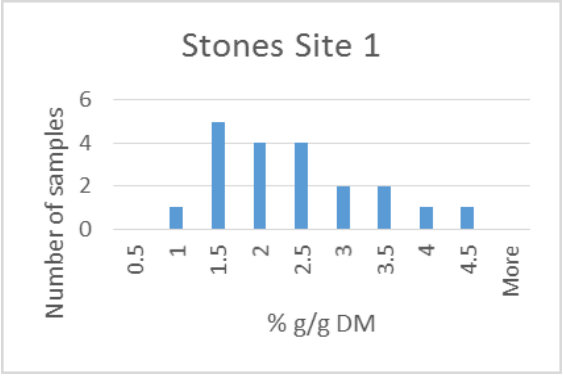












Site 2 Analysis

The 0-25mm compost from Site 2 would have failed PAS100 for both non-stone contaminants and stones. There was less glass at Site 2 than Site 1 and no metal. However, plastic was close to the upper limit and the 'other' category of contaminants resulted in a total of the non-stone contaminants exceeding the upper limit. Six of the 20 sub-samples were above the plastic upper limit due to film and/or rigid plastics.

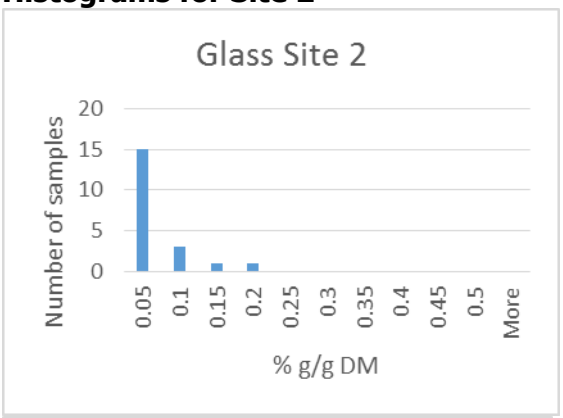
Table 2. Physical contaminant characterisation of 20 discrete compost samples from site 2 (0-25 mm product). Grey shaded cells indicate those over current PAS100 limits

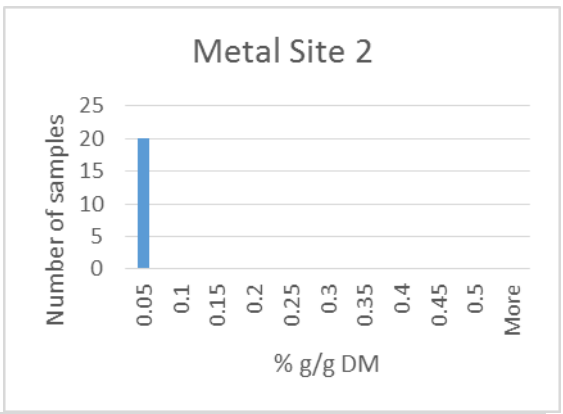
Sample	Physical contaminants (% g/g DM)					
	Glass (>2 mm)	Metal (>2 mm)	Plastic (> 2 mm)	Other (> 2 mm)	Total (>2 mm)	Stone (> 4mm)
2.1	0	0	0.04	1.02	1.06	7.18
2.2	0	0	0.08	0.41	0.49	12.23
2.3	0.07	0	0.29	0.13	0.49	9.55
2.4	0	0	0.03	0.24	0.27	7.57
2.5	0	0	0.21	0.3	0.51	9.67
2.6	0.01	0	0.05	0.03	0.09	12.83
2.7	0	0	0.31	0.26	0.57	11.24
2.8	0.08	0	0.12	0.09	0.29	13.16
2.9	0.01	0	0	0.08	0.09	14.28
2.10	0	0	0.03	0.05	0.08	14.22
2.11	0.18	0	0.23	0.35	0.76	11.36
2.12	0.02	0	0.08	0.21	0.31	12.49
2.13	0.02	0	0.06	0.19	0.27	16.68
2.14	0.01	0	0.09	0.26	0.36	8.55
2.15	0.06	0	0.02	0.02	0.1	23.17
2.16	0.12	0	0.18	0.42	0.72	12.64
2.17	0.02	0	0.05	0.12	0.19	10.33
2.18	0	0	0.08	0.21	0.29	8.32
2.19	0.02	0	0.06	0.17	0.25	10.28
2.20	0.03	0	0.02	0.12	0.17	20.69

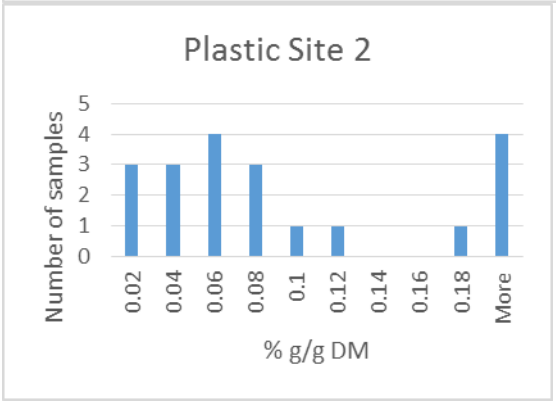
Site 2 Statistics

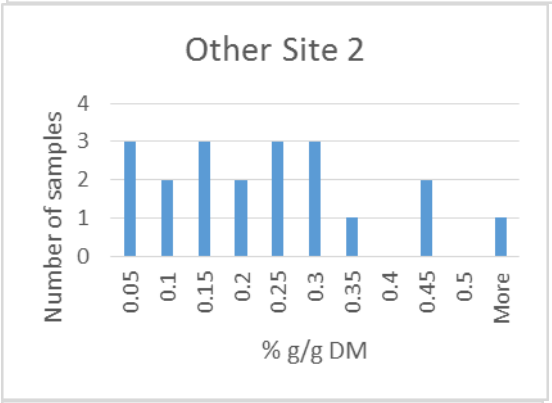
	Glass	Metal	Plastic	Other	Total	Stone
	(>2 mm)	(>2 mm)	(> 2 mm)	(> 2 mm)	(>2 mm)	(> 4mm)
PAS Limits=			**0.12**		**0.25**	**8**
Count=	20	20	20	20	20	20
Mean=	**0.033**	**0.000**	**0.102**	**0.234**	**0.368**	**12.322**
Median=	0.015	0.000	0.070	0.200	0.290	11.795
Max=	0.180	0.000	0.310	1.020	1.060	23.170
Min=	0.000	0.000	0.000	0.020	0.080	7.180
StDev s=	0.048	0.000	0.092	0.219	0.259	4.100
Variance s2=	0.002	0.000	0.009	0.048	0.0672	16.813
Stderr=	0.011	0.000	0.021	0.049	0.058	0.917
look up t	2.1	2.1	2.1	2.1	2.1	2.1
for d.f. =	19	19	19	19	19	19
95% C.I.(20)=	0.022	0.000	0.043	0.103	0.122	1.925
Mean+C.I. (20)=	0.055	0.000	0.145	0.337	0.490	14.247
95% C.I.(12)=	0.030	0.000	0.058	0.138	0.163	2.580
Mean+C.I. (12)=	0.063	0.000	0.160	0.372	0.531	14.902
C.V. s/mean=	147.0	n.a.	90.9	93.6	70.4	33.3

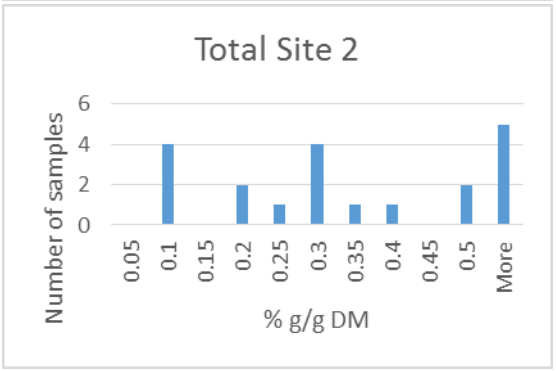
Histograms for Site 2

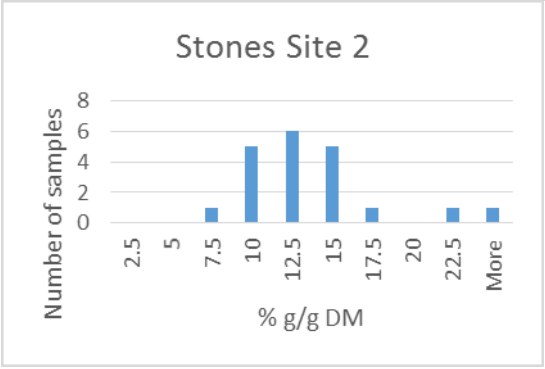












Site 3 Analysis

The 0-40mm compost from Site 3 would have failed PAS100 for both non-stone contaminants and stones. The glass content was skewed by samples 3.5 and 3.6 with one and three larger fragments, respectively. The metal content was skewed by one very large piece in sample 3.5. Four samples failed the plastic either due to large pieces of film and/or rigid plastic. Combined with 'other' category of contaminants, the total non-stone content was more than double the upper limit and 15 of the 20 sub-samples were above the stone limit.

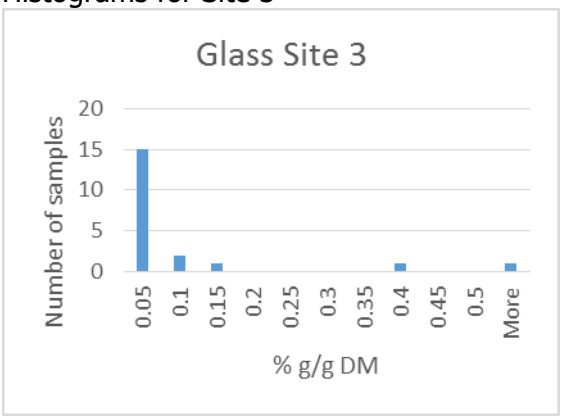
Table 3. Physical contaminant characterisation of 20 discrete compost samples from site 3 (0-40 mm product). Grey shaded cells indicate those over current PAS100 limits

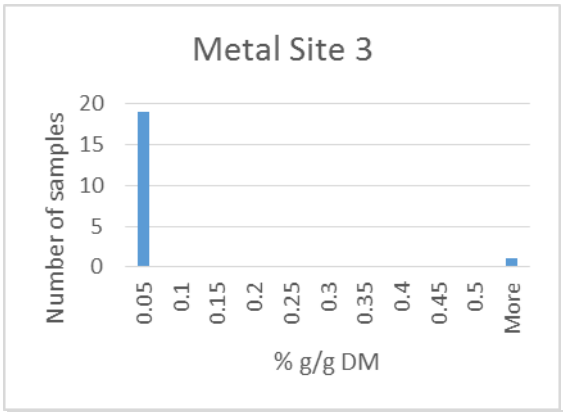
Sample	Physical contaminants (% g/g DM)					
	Glass (>2 mm)	Metal (>2 mm)	Plastic (> 2 mm)	Other (> 2 mm)	Total (>2 mm)	Stone (> 4mm)
3.1	0	0.03	0.05	0.44	0.52	6.71
3.2	0	0	0.1	0.13	0.23	4.33
3.3	0.01	0	0.01	0.22	0.24	7.59
3.4	0	0	0.05	0.17	0.22	15.91
3.5	0.36	1.15	0.23	0.01	1.75	13.59
3.6	0.71	0	0.01	0.35	1.07	13.47
3.7	0	0	0.15	0.09	0.24	12.4
3.8	0.02	0	0.02	0.34	0.38	6.73
3.9	0.03	0	0.1	0.03	0.16	12.88
3.10	0.03	0	0	0	0.03	9.81
3.11	0	0	0.02	0.02	0.04	16.71
3.12	0.1	0	0.01	0.01	0.12	13.05
3.13	0	0	0.1	0.09	0.19	8.73
3.14	0	0	0.15	1.15	1.3	12.6
3.15	0.03	0	0.01	0.3	0.34	12.55
3.16	0.02	0	0	0.04	0.06	34.13
3.17	0	0	0.01	0.21	0.22	9.72
3.18	0.01	0	0.03	0.26	0.3	21.12
3.19	0.14	0	0.02	0.12	0.28	5.98
3.20	0.08	0	0.18	0.33	0.59	8.53

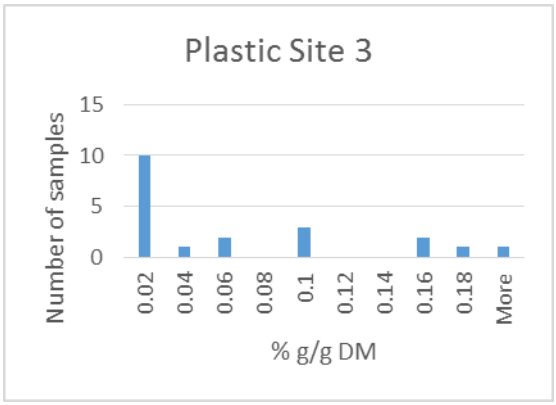
Site 3 Statistics

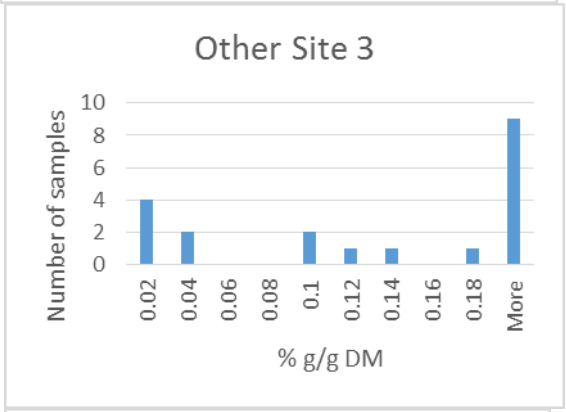
	Glass	Metal	Plastic	Other	Total	Stone
	(>2 mm)	(>2 mm)	(> 2 mm)	(> 2 mm)	(>2 mm)	(> 4mm)
PAS Limits=			**0.12**		**0.25**	**8**
Count=	20	20	20	20	20	20
Mean=	**0.077**	**0.059**	**0.063**	**0.216**	**0.414**	**12.327**
Median=	0.015	0.000	0.025	0.150	0.240	12.475
Max=	0.710	1.150	0.230	1.150	1.750	34.130
Min=	0.000	0.000	0.000	0.000	0.030	4.330
StDev s=	0.171	0.257	0.069	0.258	0.451	6.564
Variance s2=	0.029	0.066	0.005	0.067	0.2033	43.086
Stderr=	0.038	0.057	0.015	0.058	0.101	1.468
look up t	2.1	2.1	2.1	2.1	2.1	2.1
for d.f. =	19	19	19	19	19	19
95% C.I.(20)=	0.080	0.121	0.032	0.121	0.212	3.082
Mean+C.I. (20)=	0.157	0.180	0.095	0.337	0.626	15.409
95% C.I.(12)=	0.108	0.162	0.043	0.162	0.284	4.131
Mean+C.I. (12)=	0.185	0.221	0.106	0.378	0.698	16.458
C.V. s/mean=	221.9	435.4	110.0	119.7	108.9	53.2

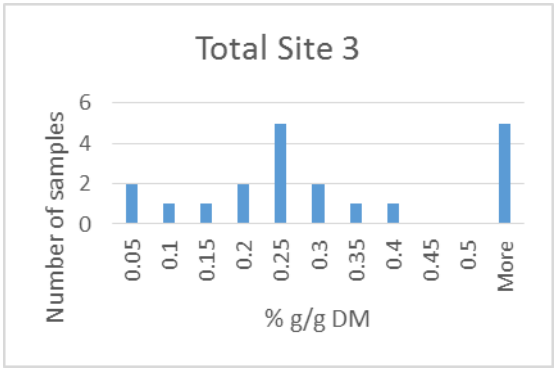
Histograms for Site 3

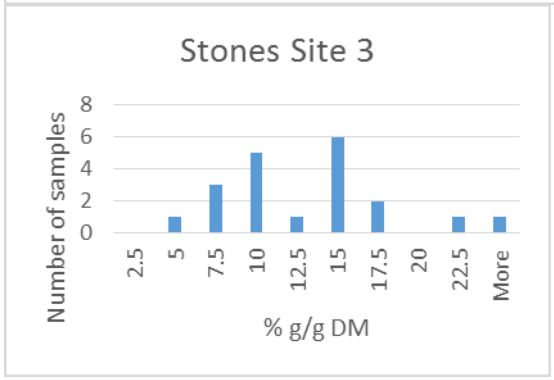












Appendix 11 – Physical contaminants in individual original digestate samples

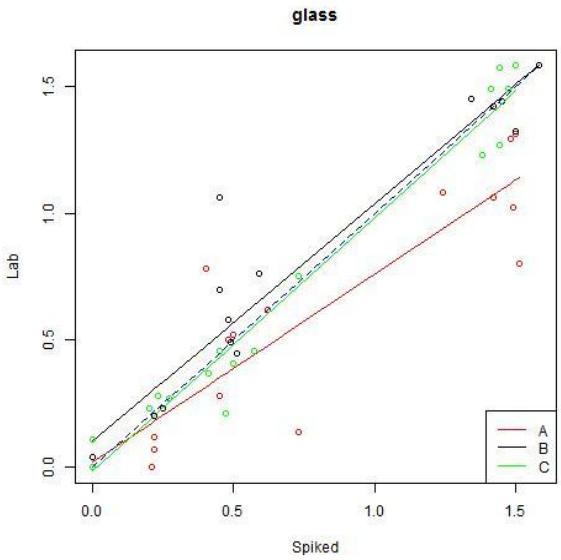
Sample number	Total (>2 mm) physical contaminants (kg/t FM)		
	Site 4 – separated liquor	Site 5 – whole digestate	Site 6 – separated fibre
1	<0.01	0.01	0.16
2	0.01	<0.01	0.39
3	<0.01	<0.01	0.2
4	<0.01	<0.01	0.39
5	<0.01	0.01	0.26
6	<0.01	0.02	0.57
7	0.02	<0.01	0.63
8	0.03	0.04	0.3
9	0.03	<0.01	0.4
10	0.04	0.01	0.2
11	0.02	<0.01	0.22
12	0.03	<0.01	0.33
13	0.01	<0.01	0.19
14	0.12	0.01	0.33
15	0.05	<0.01	0.28
PAS110:2014 Limit*	**0.04-0.36**		

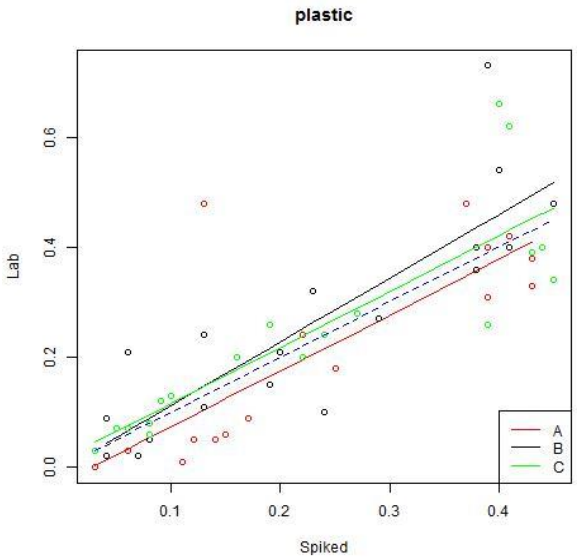
*Limit based on sample total nitrogen content

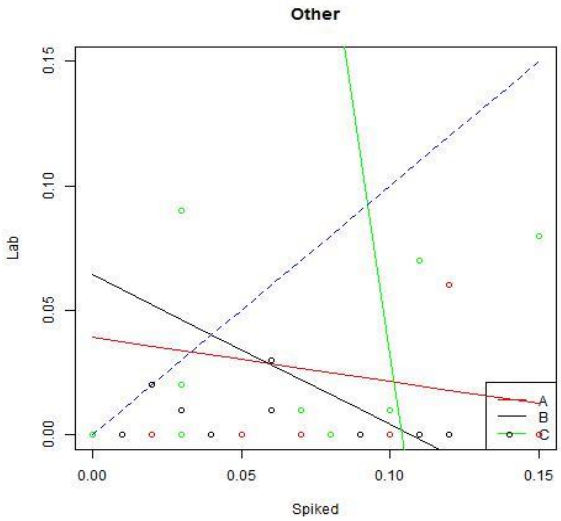
Digestate sample statistics

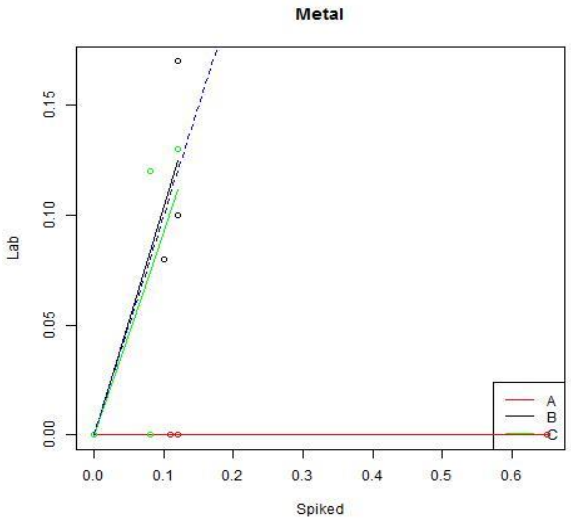
	Site 4 – separated liquor	Site 5 – whole digestate	Site 6 – separated fibre
Count=	15	15	15
Mean=	**0.024**	**0.007**	**0.323**
Median=	0.020	0.000	0.300
Max=	0.120	0.040	0.630
Min=	0.000	0.000	0.160
StDev s=	0.031	0.011	0.137
Variance s2=	0.001	0.000	0.019
Stderr=	0.008	0.003	0.035
look up t	2.14	2.14	2.14
for d.f. =	14	14	14
95% C.I.(15)=	0.017	0.006	0.076
Mean+C.I. (15)=	0.041	0.013	0.399
C.V. s/mean=	129.7	166.9	42.3

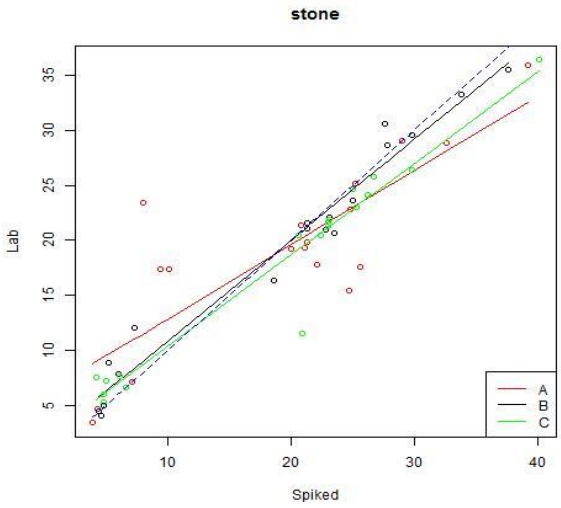
Appendix 12 – Compost inter-laboratory trial results



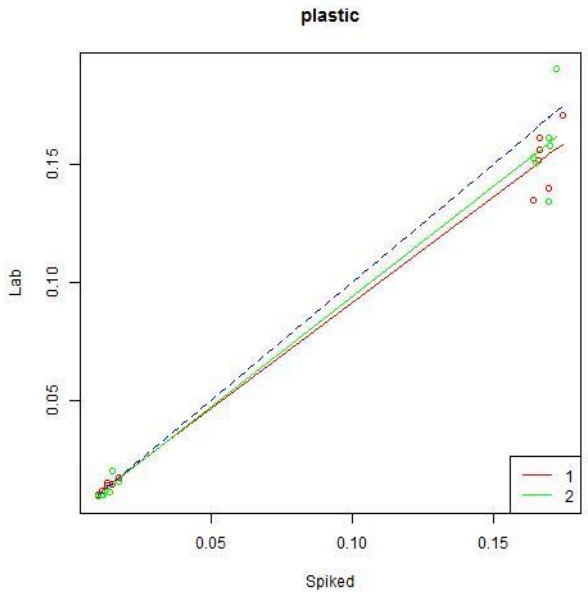


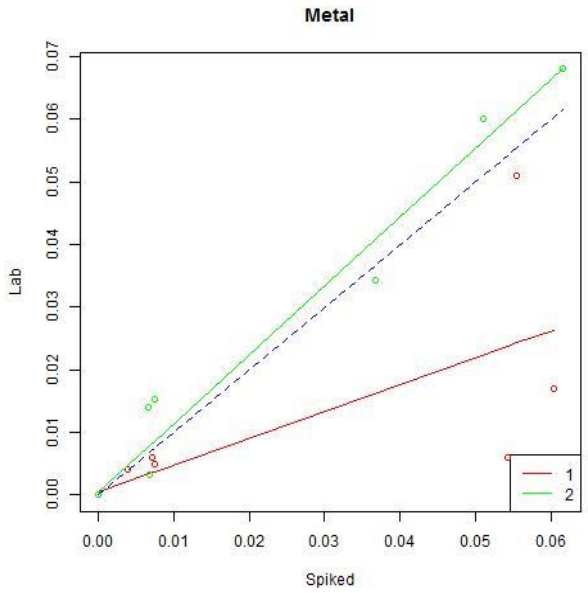






Appendix 13 – Digestate inter-laboratory trial results





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