



Guidance on sampling liquid materials in accordance with BSI PAS110:2014

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1. Scope

The Publicly Available Specification for digestate derived from anaerobically digested source-segregated biodegradable materials (BSI PAS 110:2014) requires that each final sample sent for testing is representative of the batch (or portion of production) from which it was obtained.

This document has been developed to provide specific guidance for the development of standard operating procedure(s) (SOPs) for sampling liquid samples in respect of BSI PAS110:2014. The development and appropriate use of SOPs for sampling will enable operators to uphold clause 7.1.9 of the latest version of the Biofertiliser Certification Scheme (BCS) Scheme Rules.

For separated fibre, as per section 10.4 of BSI PAS110:2014, the British Standard 12579 provides general procedure(s) for obtaining representative samples from quantities of (solid) soil improvers and growing media – sampling either bulk or packaged material. As such, sampling of separated fibre should follow the REAL's '*Guidance on sampling composted materials in accordance with BSI PAS100:2018*'

(https://www.qualitycompost.org.uk/upload/files/f40_REAL_CCS_sampling_guidance_December_2020.pdf)

NOTE: The incorporation of health and safety requirements for sampling SOPs are beyond the scope of this guidance.

2. Objective

The objective of sampling is to produce a sample of sufficient amount for laboratory testing that is representative of the sampled batch or portion of production. This applies where the sample is an original sample or a retest sample following corrective action. The sample for laboratory testing is produced following these steps:

1. taking incremental samples from a portion of production,
2. mixing incremental samples to create a combined sample that represents the portion of production

As per clause 4.3 of BSI PAS110:2014, each person whose duties affect digestate quality (and therefore with this the assessment of digestate quality) shall be appropriately trained.

The sampling operation should be carried out over a sufficiently short period of time and within a maximum of one day. Necessary steps should be taken to avoid altering sample characteristics during sampling or handling of samples prior to sending to the laboratory.

3. Timing

Section 10 of BSI PAS110:2014 indicates that the sample should be taken when the digested material is ready for use.

Sampling early in the week (i.e. Monday to Wednesday) is recommended to ensure testing can be started at the laboratory within the required timeframe.

4. Frequency

The sampling frequency should be defined by the quality management system (QMS) as outlined in section 10.6 of BSI PAS110:2014. Minimum testing frequencies are specified in Table 4 of BSI PAS110:2014.

If laboratory tests show that a sample fails any of the minimum quality parameters specified in Table 3 of BSI PAS 110:2014, this would trigger an investigation into the causes of the failure, identification of corrective actions and a change in sampling frequency. The change in sampling frequency will be dependent on the type of the failure; representative samples may need to be taken from one or more batches to gain assurance that the quality of digestate is not compromised. Suggested actions to be taken in the event of a failure are presented outwith of this guidance.

5. Equipment and consumables

All equipment and consumables used must be clean and dry to prevent altering the characteristics of the digestate sample. In respect of microbial pathogen testing, the use of fresh sampling consumables should be maximised to avoid cross-contamination (e.g. fresh sample bottles).

Typical equipment required for sampling are a plastic jug and bucket which should be thoroughly cleaned prior to use.

On each sampling occasion, use fresh bottles provided by the chosen approved laboratory, in which to place the final homogenous sample.

A cool box with ice packs should be used to ensure the integrity of the sample is maintained, after the sample has been removed from storage, until testing.

6. Size and number of increments

Ensure that the sampled material consists of only one output type i.e. separated liquor or whole digestate. The sampled material should ideally represent a discrete batch; however, it is recognised that in a continuous process this may be a portion of production - further guidance can be found in clause 10.6 of BSI PAS110:2014.

Take incremental samples using suitable sampling equipment and consumables (section 5). Ensure that the volume of each incremental sample is **at least 1 litre** to ensure that the resulting combined sample will be of sufficient volume for the laboratory.

The number of incremental samples to be taken is based on portion of production (or batch) sizes as outlined (Table 1).

Table 1. Number of incremental samples according to volume of the sampled portion of production

Volume of sampled portion / batch (m ³)	Number of incremental samples
< 1,000	10
1,000 – 3,000	15
3,000 – 5,000	20
> 5,000	25

Using this approach, a minimum of 10 litres of sample will be available from which a combined sample can be prepared (see section 8) for sending to an approved laboratory.

In the case of re-tests, a small volume of sample will be sufficient in which case the combined sample should be subsampled after mixing (see section 8). Approved laboratories should be consulted on the quantity of sample required for specific tests.

7. Sampling approaches

a) Sampling from closed storage tanks

Before incremental samples are taken from the storage tank, thoroughly mix the portion of production to be sampled. After mixing, no 'float' or 'sink' layers should exist in the tank.

In some cases, the complete mixing of flat layers can be omitted if they persist when the digested material is discharged for use. In such cases, the producer should ensure that the material below the float layer is thoroughly mixed before any incremental sample is taken and that samples do not contain components of the float layer.

b) Sampling from open topped storage tanks without sampling nozzles

When taking incremental samples, use sample scoops with a telescopic rod, sealed plunging siphons or pumps as appropriate.

The incremental samples should be taken from different positions / time periods (as appropriate).

c) Sampling from open topped storage tanks with sampling nozzle(s)

Before taking incremental samples from a sampling nozzle, remove material between the tank and the nozzle 'dead zone'. Drain out at least double the dead area volume before taking an incremental sample.

Take incremental samples by opening and closing the valve at regular time intervals. Catch each incremental sample in a container suitable for checking the sample volume (e.g. plastic measuring jug). Each one can then be poured into a larger container (e.g. plastic bucket).

d) Sampling from pipes to/from an external circulation pump

Some digested material storage tanks are used in conjunction with an external pump that circulates the material via pipes running to and from the storage tank and pump. If any of the pipes have a sealing valve / sampling nozzle, incremental samples may be taken at this location.

Use the pump and any other necessary equipment to thoroughly mix the portion of production immediately prior to taking any incremental sample. No float or sink layers should exist when samples are being taken.

Before taking any incremental sample from the sealing valve / sampling nozzle in the pipe, remove at least double the volume of the digested material in the pipe between the tank and the valve. The period of time required to flush the pipe will depend on the pipe's length and diameter, the available 'head' (i.e. the pressure exerted by the bulk of the sludge stored above the sample line) and, if a pump is fitted, the pump rate.

Take incremental samples by opening and closing the valve at regular intervals. Catch each individual sample in a container suitable for checking the sample volume (e.g. plastic measuring jug). Each one can then be poured into a larger container (e.g. plastic bucket).

e) Sampling when discharging or dispatching digested material for use.

Incremental sample can be taken when digested material is ready to use, such as when being discharged from the storage tank or after it has been discharged into the transport container(s).

Prior to discharge, ensure that the digested material is thoroughly mixed. Catch each incremental sample in a container suitable for checking the sample volume (e.g. plastic measuring jug). Each one can then be poured into a larger container (e.g. plastic bucket).

If taking incremental samples from a transport container thoroughly mix the digested material in the container and take incremental samples immediately afterward.

8. Preparation of combined sample

Regardless of the sampling approach used, the collected incremental samples must be mixed to create a single combined sample before sending to the laboratory.

Combine the incremental samples in a suitable container (e.g. large plastic open-topped tub or drum). The combined sample should comprise at least 10 litres and be thoroughly mixed. From this, take sufficient samples and volumes to fill the containers provided by the approved laboratory.

An additional sample taken from the combined sample can be maintained as an archive sample for potential use in the case of test failures (see section 13.5 of BSI PAS110:2014). The sample should be stored under cool conditions (e.g. in a dedicated fridge or freezer on site).

9. Sampling records

The following information should be recorded at each sampling occasion in line with section 10.7 of BSI PAS 110:2014. Records should be kept on site and be available for inspection during audits:

- sampling date;
- sample type (i.e. separated liquor or whole digestate);
- code of the batch or portion of production from which the sample was taken;
- digestion facility name;
- information that identifies the composting process;
- name of the person who carried out the sampling;

A copy of the completed digestate analysis request form (see section 10) maintained on site serves as a record of sampling. The only exception to this is the requirement to maintain a record of the person responsible for sampling.

10. Sample dispatch

Samples shall be dispatched for testing within one working day of taking the sample.

If a sample is to be tested for pathogens, use a service that will deliver it to the laboratory within 24 hours – the exception to this being extreme geographical locations where a 48 hour service should be used with samples dispatched on the same day as sampling. For samples not scheduled for pathogens, use a service that will deliver it to the laboratory within 48 hours.

The sample sent for laboratory testing must be packaged in such a way that sample characteristics are unaltered on arrival at the laboratory. The sample container must be sealed to prevent contamination, spillage or changes in sample characteristics. Samples should be transported under cool conditions i.e. using a cool box and ice packs.

A sample label bearing the required sample information should be attached to the laboratory sample packaging/containers. **The final sample sent to the laboratory must be accompanied by a completed current version of the Digestate Analysis Request form which can be downloaded via the following link**

<https://www.biofertiliser.org.uk/certification/laboratory-tests>

Ensure that all the details are filled in appropriately without mistakes or ambiguous characters in any text. If there are omissions and/or mistakes on this form it may delay the reporting of results.

A copy of this form should be generated and maintained onsite (see section 9).

11. Sample receipt at approved laboratories

The testing laboratory shall send a confirmation to the producer to acknowledge the receipt of sample(s). In absence of a sample receipt from the testing laboratory, producers should seek confirmation that the sample has been received at the laboratory and has been accepted for testing.

In the event of late reception outside of the recommended timeframes, the laboratory may accept the sample for testing but should seek confirmation from the operator.

On receipt of the test sample the laboratory will carry out checks on the sample and accompanying documentation to identify any potential issues before accepting the sample for testing for certification purposes. The laboratory shall inform the producer should they find any issues with the sample.

Glossary of terms

Batch – material processed under the same conditions. In the case of continuous anaerobic digestion processes, portion of production is considered a more appropriate term.

Combined sample – prepared by mixing multiple incremental samples of equal size for sending to the laboratory for testing

Consumable – material used once or a limited number of times for sampling

Incremental sample – a discrete sample taken from a storage tank, associated pipework or transport container.

Portion of production – as per BSI PAS110:2014, this should be defined by the producer based on the specific process in question