Final Technical Report

# Developing a laboratory proficiency framework for the UK compost and digestate certification schemes

Developing a framework for a laboratory proficiency testing (PT) scheme to ensure that laboratory performance is consistent and adequate across the range of tests required.

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# Executive summary

The Publicly Available Specifications for composted materials (BSI PAS100:2011) and anaerobically digested materials (BSI PAS110:2014) are a crucial component of market confidence in compost and digestate quality, consistency and safety. Integral to this requirement is that there is confidence in the quality of the laboratory data being used to assess material quality. Renewable Energy Assurance Ltd (REAL) operates the Compost and Biofertiliser Certification Schemes which accredit materials to the two specifications. Within these schemes REAL appoint a number of laboratories to test compost and digestate.

This work reviewed the use of test methods and quality control (QC) procedures used by the current PAS scheme laboratories. It also researched the relevance of existing proficiency testing schemes in providing additional control on data quality.

Internal laboratory quality assurance and control measures should always provide the first port of call in controlling data quality, while proficiency testing schemes should be used to supplement these. Scheme participation is no guarantee of consistency or accuracy unless poor performance is monitored and acted upon, but should be thought of as more of a final check that results are consistent with other laboratories.

The small number of current appointed laboratories could severely limit the value of a UK PAS-specific proficiency testing scheme(s) (PTS). Setting up a new scheme will be expensive and potentially not financially viable for the size of the current PAS testing business (currently 180 composters and 15 AD operators, each submitting circa three samples per year across four laboratories). In addition, with only a small number of participating laboratories there would be large uncertainties in test result interpretation and performance evaluation, as the most commonly used z-score approach relies on a sufficient number of participating laboratories for the mean of all tests to be close to the 'real value' and avoid bias from outlier or extreme results.

Rather than immediately deploy a PTS for UK compost and digestate testing laboratories, a number of steps are proposed to better ensure the reliability and accuracy of data. These are ranked in terms of the most immediate recommendations for action.

**Internal Quality Control**

1. Ensuring that standard internal quality control procedures are in place in all appointed laboratories, and that quality control data are monitored. A number of publications are available detailing the necessary actions and procedures to maintain control for data reporting.

2. All appointed laboratories should complete (or fill gaps relating to) validation of test methods for the relevant PAS matrix (compost or digestate).

3. Laboratories should produce a dried bulk internal reference or control sample that should be included in all test batches.
    - Aliquots of a suitable digestate for the PAS110 RBP test could be frozen to provide a suitable control.
    - Microbiological parameters could continue to be covered separately under the VETQAS PTS.

4. Control charts should be set up to record the results of the internal control sample to provide a continuing check on analytical performance. These charts can be used to

identify random step changes or longer term data trends that may be due to equipment maintenance or failure. Regular monitoring of these charts will ensure prompt remedial action.

**PAS laboratory organised PTS**

5. Where laboratories have appropriate internal QC measures in place, there would be value in undertaking a ring test to assess between-laboratory variability. A dried bulk control sample could be prepared by an appointed laboratory or independent third party organisation for distribution amongst the appointed laboratories to provide an internally run 'mini' PTS. If testing is repeated over time, REAL or an appointed organisation could produce Youden control charts to identify systematic bias between laboratories assuming the laboratories are prepared to share their test data. The cost for producing a bulk control sample is estimated as relatively minimal (1-2 days effort) and this approach controls costs incurred by the laboratories for testing. The evaluation of test data could be undertaken by REAL for ca. 2 days effort per distribution. This approach would keep costs to a minimum and avoid the costs of purchasing standard reference materials which are commonly provided in only 50-100g quantities. Consequently, it is recommend that a single ring test is completed regardless of recommendations for participation in an externally run PTS.

**External PTS**

6. The appointed laboratories should be encouraged to join an existing scheme with the benefit of a data set from a large number of participants; even if a few parameters are not covered. This is because the scoring is less affected by outlier data, and costs for participation (if these are to be borne by the laboratories) are likely to be minimised. Participation in a PTS will provide the opportunity to assess how PAS laboratories perform against each other – which monitoring of internal QC arrangements does not provide.

   Two PTS have particular synergy with PAS 100 and 110, and cover the majority of the parameters required:
   a. The first is the French run BIPEA scheme (PTS 45 Organic fertilising materials / PTS 38 - Activated sludge & sediments) which, with the exception of pathogens and the digestate stability tests, covers the majority of parameters required by both PAS 100 and 110. All current UK appointed laboratories already participate in the VETQAS scheme for pathogens and it is recommend that this continues.
   b. The second most likely match for the PAS 100 scheme is the US CAP programme which already distributes dried compost samples outside of the US and includes pathogen parameters. A similar scheme (MAP) provides more limited coverage of PAS 110 parameters and is solely for manure samples. Participation in the CAP programme alone would still provide value for laboratories under PAS110, especially for fibre digestate testing. The value of such participation will only, however, be realised if performance data is reviewed and anomalies or bias acted upon.

   International schemes operate between two and four test sample distributions per annum; it is recommended that laboratories should participate in a minimum of two distributions. The value of such participation will only be realised if performance data is reviewed and anomalies or bias acted upon.

No scheme has been identified that includes the volatile fatty acid (VFAs) and residual biogas potential (RBP) tests required by PAS 110.

7. A potential solution to accommodate the lack of any currently available PTS for VFAs and the RBP test for PAS 110 would be to ask the appointed laboratories to undertake a ring-test. With the permission of current AD operators and receipt of a larger sample, laboratories could alternate preparation and distribution of the test sample or this could be undertaken by an independent third party. Again, results could be shared if agreeable to all participating laboratories. Whilst the small participating cohort would restrict the use of performance rating using a z-score approach, simple tools such as Youden graphs would allow a laboratory to see if their results are consistently higher, lower or similar to the other participating laboratories, which could be a prompt for improved method control or adjustment. REAL could ask for this data to be returned on (for example) an annual basis. This could be for a minimum of two samples per annum. Freezing aliquots of such a sample would allow for more regular testing.

# Contents



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# Glossary

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| Accuracy | Degree of agreement of the observed value with the true value of the quantity of interest. Both random and systematic errors can contribute to a reduction in accuracy. |
| Analytical method | The set of instructions to be followed by the analyst in performing analysis. |
| ABP | Animal by-products. |
| Batch of analysis | A term used to refer to a series of determinations which have been made on a single occasion or under broadly similar experimental conditions. A batch may be chosen to correspond to analysis for which a given calibration applies, or it may be more convenient to consider a batch as a number analysis performed on a given day. The analyst should give careful consideration to what he regards as batch of analysis because this definition will imply the division between within-batch (i.e. short term) random error and between-batch (longer term) random error. It also can determine the frequency with which many routine quality control measures are carried out. |
| Between-batch standard deviation | A measure of the random error of analytical results which applies from one batch of analysis to another. |
| Bias (Systematic error) | Consistent difference between the mean of many (in theory, an infinite number) of measurements and the true value. Note that in most tests intended to measure bias, the term is defined by the experimental design adopted. |
| BSi | BSi (formerly British Standards Institute) are recognized as shaping many of the most used and implemented standards worldwide, such as the Quality and Environment Management series (ISO 9001 and ISO 14001) that have been adopted by over a million organizations all over the world. |
| Collaborative trial | A term used to describe an interlaboratory test intended to establish "repeatability" and "reproducibility" values for a method. |
| Compost | Compost means solid particulate resulting from controlled biological decomposition of biodegradable materials under managed conditions that are predominantly aerobic and that allow the development of thermophilic temperatures as a result of biologically produced heat. |
| Control chart (Shewhart) | A graphical technique used to monitor the operation of a process and to detect departure from a state of statistical control. |
| Control limit | A line drawn on a control chart beyond which it is unlikely that a point will lie unless the method has not functioned correctly. A control line is usually loosely associated with the probability of finding a point beyond it – e.g. "warning" limits at a 2 standard deviations from a centre line should contain 95% of points. |
| Determinand | The scope of the test measurement e.g. pH or microbial respiration rate. |
| Digestate | Digestate means whole digestate resulting from the controlled biological decomposition of biodegradable materials under managed conditions that are anaerobic, and any subsequently separated fibre or liquor fractions |

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| ICPMS | Inductively Coupled Plasma Mass Spectrometry. |
| ISO | The International Organization for Standardization is the world's largest developer of voluntary International Standards. |
| Method Validation | Method validation seeks to quantify the likely accuracy of results by assessing the magnitude of both systematic and random effects. NS30 (WRc 1989). Typically this is undertaken on a selection of representative 'samples' which would comprise: (i) a blank sample (to facilitate estimation of the method limit of detection); (ii) two standard solutions or samples at the lower and upper range of interest,(iii) a sample and a spiked sample (where possible) to facilitate estimation of recovery and precision on real samples. Typically an experimental design of 11 batches analysed on a separate occasion in duplicate for each sample is required (DWi, 2012). |
| MCERTS | Environment Agency's Monitoring Certification Scheme. The scheme provides a framework within which environmental measurements can be made in accordance with the Agency's quality requirements. |
| NCTC | National Collection of Type Cultures. |
| Parameter | The scope of the test measurement e.g. pH or microbial respiration rate. |
| PAS100 | British Standards Institution Publicly Available Specification 100 - Specification for Compost. |
| PAS110 | British Standards Institution Publicly Available Specification 110 - Specification for Digestate. |
| Precision (Random error) | The degree of agreement existing between repeated measurements made under specified conditions. |
| Potentially Toxic Elements (PTE) | Elements of concern because of their potential for long-term accumulation. Potentially toxic elements including zinc, mercury, chromium, cadmium, copper, nickel and lead enter the soil naturally as a result of soil forming processes such as the weathering of minerals. However, they are also added to the soil in the application of organic materials, sewage sludge, composts and inorganic fertilisers as well as though atmospheric deposition and run-off.<br><br>Many metals are essential in very small quantities; deficiencies of these can result in a reduction in crop yield or in adverse health effects in animals (copper, zinc). However, in high concentrations, the same elements may damage soil fertility, while the accumulation of others (e.g. cadmium and lead) in the food chain can damage human health. |
| Proficiency testing scheme (PTS) | Proficiency testing is an inter-laboratory system for the regular testing of the accuracy that the participant laboratories can achieve. In its usual form, the organisers of the scheme distribute portions of a homogeneous material to each the participants, who analyse the material under typical conditions and report the result to the organisers. The organisers compile the results and inform the participants of the outcome, usually in the form of a score relating to the accuracy of the result. |
| Quality Management Systems | Quality Management Systems (QMS) such as ISO17025 and Good Laboratory Practice provide a valuable role in controlling the quality of the data generated by the laboratory. A QMS defines the processes being undertaken in the laboratories, the control and monitoring systems used and documentation management. QMS can help to drive |

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| | continuous process improvement with the ultimate goal of ensuring the quality of data issued by the laboratory and hence increasing customer satisfaction and confidence. |
| RBP | Residual Biogas Potential. |
| REAL | Renewable Energy Assurance Ltd. |
| Recovery | The extent to which a known, added quantity of determinand can be measured by an analytical system. It is calculated from the difference between results obtained from a spiked (see below) and an un-spiked aliquot of sample and is usually expressed as a percentage. |
| Reference material | A material of which one or more properties have been characterised to the extent that it can be used as a check on the accuracy of a test method. "Standard Reference Materials" (SRM) or "Certified Reference Materials" are those which are prepared, characterised and distributed by a standards organisation. "In-house Reference Material" is the term used to describe a material which has been prepared and characterised by the laboratory which uses it. The range of measurement and validation procedures available to standards organisations usually means that the certified concentration of an SRM is an unbiased estimate of its true concentration although available sample weights are commonly too small to be useful in waste testing. In-house materials, which have no certified value, are usually used mainly for comparative purposes. |
| Repeatability | The variation in measurements taken by a single person or instrument on the same item and under the same conditions. |
| Reproducibility | The ability of an entire experiment or study to be reproduced, either by the testing laboratory or by another laboratory working independently. |
| Spike | A known quantity of determinand which is added to a sample, usually for the purpose of estimating the systematic error of an analytical system by calculating a recovery. |
| STA | Seal of Testing Assurance Program ('STA'), similar to PAS100, operating in North America and implemented by the US Composting Council in 2000 ensuring compost producers or marketers are following a quality protocol. |
| Total standard deviation | The standard deviation of individual analytical results selected at random from any batch of analysis. Total standard deviation comprises components of both within-batch and between-batch random error. It is a measure of all elements of random error which might affect results. |
| VFA | Volatile Fatty Acid. |
| Within-batch standard deviation | A measure of the precision of analytical results which have been obtained in a single batch of analysis. It provides an indication of the short-term random error analysis. |
| Youden graph | A graph used to present the results of different laboratories' analysis. Results obtained by each laboratory for one sample are plotted against those for a second sample. The graph gives a visual indication of whether systematic or random errors are predominant. |

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| z-scores | The standard score or z score is the number of standard deviations that a test result is above the mean. Thus, a positive z score represents a result above the mean, while a negative standard score represents a result below the mean. The z score is calculated by subtracting the sample mean from the value and dividing the result by the sample standard deviation. |

# 1.0 Background

The Publicly Available Specifications for composted materials (BSI PAS100:2011) and anaerobically digested materials (BSI PAS110:2010) are a crucial component of market confidence in compost and digestate quality, consistency and safety. Integral to the PAS schemes is that there is confidence in the quality of the test data being used to assess the material quality so that it can be can be relied upon. A recent survey (WRc 2012) of the Organics Recycling Industry showed that half of the surveyed composting sites produced material certified to the PAS100 specification. The robustness of PAS100 and PAS110 and their acceptance in the market are imperative if the specifications are to continue to be widely used and their credibility maintained.

Certification to PAS100 and PAS110 is overseen by schemes owned and managed by Renewable Energy Assurance Ltd (REAL), and audited by independent third party certification bodies. Both PAS certifications require routine sampling and testing of the materials produced for a specified list of parameters within the PAS documents (see Table 3 in Section 14 of PAS 100, and Table 1 in Section 11 of PAS 110). A number of laboratories are appointed by REAL to test compost and digestate for members of the schemes, but a preliminary 'ring test' of the compost testing laboratories has suggested that there is inconsistency between the laboratories for the different tests and that the level of inconsistency varies from test to test.

This report addresses a number of objectives set by WRAP at the beginning of the project which include:

- To review any existing inter-laboratory quality controls currently implemented by the appointed PAS100 and PAS110 laboratories in the UK (Section 2).
- To review relevant laboratory proficiency schemes worldwide which use an approach that could be applied to compost and digestate (Section 3).
- To provide recommendations for either the adoption of an existing laboratory proficiency scheme for the compost and digestate certification schemes, or, where an existing scheme is not considered appropriate, to provide the outline structure for a laboratory proficiency scheme bespoke to PAS100 and PAS110.

Section 4 discusses the benefits of general quality control measures and Proficiency Testing Scheme (PTS) and Section 5 identifies a number of activities that could be considered to ensure that laboratory performance is consistent and adequate across the range of tests required for the two PAS materials.

# 2.0 Review Quality Assurance – Quality Control (QA QC) procedures at existing PAS appointed laboratories

## 2.1 Approach

Quality Management Systems (QMS) such as ISO17025 and Good Laboratory Practice provide a valuable role in controlling the quality of the data generated by the laboratory. QMS can help to drive continuous process improvement with the ultimate goal of ensuring the quality of data issued by the laboratory and hence increasing customer satisfaction and confidence.

Whilst participation in an umbrella QMS scheme is recommended, basic quality control measures have a valuable part to play in ensuring the quality of data from a laboratory, as long as the data produced is acted upon and reviewed.

Following an initial introduction by REAL, WRc contacted all UK PAS100 and PAS110 appointed laboratories to review existing inter-laboratory quality control measures. An initial telephone was used to discuss the project requirements with each laboratory and information was collected using a standardised spreadsheet.

The data collected is summarised in tabular form in Appendix A – Adopted methods and quality control procedures within current PAS 100 / 110 laboratories, and provides background on the range of procedures / approaches being used by these laboratories for all parameters in the PAS 100 / 110 test schedule. The summarised data are presented using the same spreadsheet format sent to each facility. Information on a previous ring-test between the PAS100 laboratories was made available by REAL as part of this review. As of April 2014, one laboratory is seeking to be re-appointed, but their data is included for completeness.

## 2.2 Discussion

The test parameters where the inconsistency in data between appointed laboratories is greatest (i.e. there is high variability between samples and between laboratories) are *E Coli*, the plant response test, compost stability, physical contamination and nutrients in the PAS100 specification. At the time of writing[1] only a single laboratory and their sub-contractor are appointed for the PAS110 suite of tests so it is difficult to fully quantify any data reliability issues. Although, as the majority of test parameters are replicated between the two PAS specifications (albeit for different test matrices) the PAS 100 performance of this laboratory can be used to assess the likelihood of analytical bias. The exception is the single facility testing digestate for VFA (volatile fatty acids) and residual biogas potential (RBP).

Key points arising from the examination of laboratory approach include:

i. A number of laboratories provide sub-contract support for more than one appointed laboratory, particularly for the microbiological parameters, compost stability and weed, seed and propagule tests.

   This has significant implications for a Proficiency Testing Scheme (PTS) as it would mean that data comparisons can only be made between one or two laboratories. Using a standard measure of the 'derived mean of all laboratories' as a measure of the true value to look at inconsistencies between laboratories would therefore not be feasible. For most tests on these matrices it is not possible to identify the 'real or actual' value as would be available for a certified reference material.

ii. All appointed laboratories report that they use the methods specified in the PASs with the exception of:

   - Laboratory 1 for mercury analysis – where the laboratory uses the defined digestion procedure but determine mercury in the digest by ICPMS. Most modern laboratories would adopt this technique, if it was available in their laboratory, as it permits mercury analysis as part of a multi-element suite of analyses, against the single element procedure of cold vapour or fluorescence analysis as identified in the PAS method. Sensitivity and limit of detection are not compromised.
   - Laboratory 3 for dry matter analysis – where the laboratory dry the sample at 105°C for 24 hours, or at 60°C for 48 hours if mercury analysis is required. Unless the laboratory has evidence from historic tests it may be appropriate to

[1] *July 2014*

request that they demonstrate that their procedure does not give a significantly different answer to the PAS procedure.

iii. Laboratories have given a range of responses for whether the methods they use are validated using a recognised procedure e.g. NS30 (WRc, 1989) or DWi (DWi, 2012) or BS/ISO (ISO 16140); participation in a ring test was not considered to be sufficient to confirm method validation. Method validation is one of the key elements to understanding test data consistency and accuracy. Typically this is undertaken on a selection of representative 'samples' which would comprise: (i) a blank sample (to facilitate estimation of the method limit of detection); (ii) two standard solutions or samples at the lower and upper range of interest and (iii) a sample and a spiked sample (where possible) to facilitate estimation of recovery and precision on real samples. Guidance typically requires an experimental design of 11 batches of samples to be analysed in duplicate on separate occasions (WRc, 1989 & DWi 2012); ISO 16140 covers procedures suited to microbiological analysis.

iv. Not all laboratories use control samples (standards) and / or in-house reference materials when testing submitted compost or digestate samples.

v. Where control samples are used, control charts are not necessarily used to track and act on the results.

vi. Participation in external PTS is variable:

- All laboratories participate in VETQAS for pathogen parameters.
- With the exception of Laboratory 2, all laboratories participate in a PTS for PTEs, although in some cases these are for matrices other than compost or digestate PTEs.
- There is no PTS participation for stability, and weed, seed and propagule measurements other than the single AfOR organised ring-test. The results reported by the ring-test for these parameters were highly variable.
- There is no PTS participation for physical contaminants (potentially as a suitable scheme has not been identified). However, this parameter showed the highest variability in the ring-test.
- There is partial participation for dry matter (DM) and loss on ignition (LOI) (Aquacheck, WEPAL and MARSEP) in a range of relevant matrices (not necessarily compost or digestate) by the PAS appointed laboratories. No laboratories participate in PTS for particle size distribution, total organic carbon and calculation of the carbon:nitrogen ratio (Appendix A, Table F1).
- There is partial participation for the General Chemistry parameters (Appendix A, Table G1) and no participation in PTS for the extractable parameters (Appendix G, Tables H1 and I1). However, these are not covered in the PTS currently used i.e. Aquacheck, MARSEP).

vii. The most variable results in the AfOR Ring Test were noted for non-chemical test parameters i.e. tests where judgement is required on the part of the laboratory technician (e.g. identifying physical contaminants) or environmental conditions and differences in test equipment that could cause significant differences in the end result of the test (e.g. use of growth cabinets versus a winter time window sill). Whilst the use of control or reference samples is not as easy to implement, this can still be achieved.

viii. Sample preparation[2] is a key element for undertaking physical contaminants tests as it is very difficult to fully homogenise and keep 'suspended' a small number of contaminants when sub-sampling from a larger composite sample. Homogeneity is equally important for all PAS test parameters but easier to achieve by simple mixing. Whilst the methodology has been updated (AfOR MT PC&S Issue 1, Revision 2. Issued: 05/12/2012) to require a minimum volume of sample, differences in the previous ring test may be variable simply because the laboratories didn't receive identical samples.

# 3.0 Review world-wide proficiency testing (PT) schemes

## 3.1 Approach

Proficiency testing is an inter-laboratory system for the regular testing of the accuracy that the participant laboratories can achieve. An extensive web review of PTSs that include compost, digestate, and other 'similar' materials, such as the MCERTS scheme for soil testing has been completed. In addition information on schemes for other matrices was also captured where the basic approach could be relevant to PAS. The primary focus of the search was for the UK, Europe, the USA and Canada and other English speaking schemes to ensure maximum information could be captured in a limited timeframe, although schemes from France, Germany and the Netherlands were also reviewed in detail.

Information was collected on:

- parameter coverage;
- matrices currently included and whether there is scope for bespoke additions;
- charging approaches operated;
- frequency of testing;
- numbers of current participants and minimum numbers required for a viable scheme; and
- methods of data evaluation and performance criteria e.g. z scores.

An information summary of all schemes identified is provided in Appendix B. In agreement with WRAP and REAL, further information was then collated for four schemes that at face value provide good synergy with the PAS100 / 110 requirements. A summary for these four schemes is provided in Table 1.

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[2] The Test Method for the Examination of Composting and Compost (TMECC) provides detailed protocols for the composting industry to verify the physical, chemical, and biological condition of composting feedstocks, material in process and compost products at the point of sale.

US EPA - Chapter 9 Sampling Procedures and Analytical Methods for sludge and compost.

**Table 1** An information summary of PTS of relevance to PAS100 /110

| **PAS100/110** | **Colorado State University / Minnesota Department of Agriculture** | **BIPEA** | **Landers (multiple)** | **WEPAL** |
|---|---|---|---|---|
| | **US** | **France** | **Germany** | **Netherland** |
| | **CAP / MAP** | **PTS 45 Organic fertilising materials / PTS 38 - Activated sludge & sediments** | **LÜRV-A Bioabfall** | **MARSEP (MAnure and Refuse Sample Exchange Programme)** |
| **PT scheme description** | Colorado State University organises a proficiency testing scheme which includes the collection of up to nine reference compost materials from across the United States. These correspond to the diversity of feed-stocks seen in the US.<br><br>The Minnesota Department of Agriculture organise similar tests for manure. They use the results to certify labs with acceptable performance for manure testing. MAP procedure follows A3769 "Recommended methods of manure analysis".<br><br>*This scheme is the closest match to the determinand list contained in PAS100 / 110.* | BIPEA – International Bureau for Analytical Studies) is an international, non-profit association which organises proficiency testing programs for a range of environmental matrices.<br>In the 1990s, ring tests in food and 'contaminant' fields were created which have since been expanded. In 2009, BIPEA added inter-laboratory comparisons in cosmetics. After 40 years of existence, BIPEA has over 1200 participating laboratories worldwide and offers more than 60 regular proficiency testing schemes.<br><br>*BIPEA offer bespoke PTS and the current schemes cover a large number of the parameters in PAS100 /110.* | The Landers in Germany organise annual ring tests. Compost samples are collected from a commercial retailer and a product (brand and packaging) is specified by the organiser of the test. Sample preparation is specified in the test method.<br><br>*This scheme is organised amongst German laboratories to ensure compliance with general characterisation of compost in Germany.* | Wageningen Evaluating Programs for Analytical Laboratories (WEPAL) is a world-leading organiser of proficiency testing programmes in the field of plants, soil, sediments and organic waste testing. They have a 50 year track record and currently have over 500 participants in countries across the world.<br><br>*Dried samples are ground to < 0.5 mm. Not all parameters are covered. Relevant only for PTEs, though other parameters may be added in the future* |
| **Samples / Round** | 3 / 2 | 5 (2 compost) / 5 (3 sludge) | 1 (made of 7 samples) | 4 (2 compost and 2 sewage sludge) |
| **Matrices** | Compost / Manure | Compost / Sludge | Compost | Compost / Sewage sludge |
| **No. of participants** | 18 (Colorado) / 70 (Minnesota) | 70 (PTS45) / 40 (PTS 38) | 84 | 75 |
| **Method of data evaluation** | 95% confidence limit (CL) of the median and evaluation of intra-laboratory precision established based on the consensus sample relative standard deviation (RSD) | Z scores | Z scores | Z scores |
| **Starting date** | 1997 | 1983 / 1998 | 2001 | 1994 |
| **Approx Cost (per annum)** | £855 (+£102 shipping to UK) / £235 (+£130 shipping to UK) # | £550 / £802# | £123-287# | £581# |

# Approximate quote provided by PTS in US dollars or Euros and converted using following exchange rates 1 USD = 0.59 GBP, 1 EUR = 0.80 GBP (@25 June 2014)

| PAS100 / 110 | CAP / MAP | PT Scheme 45 / 38 | LÜRV-A | MARSEP (compost & S sludge) |
|---|---|---|---|---|
| **Pathogens** | | | | |
| ***E. coli*** | Yes / No | No / No | No | No |
| ***Salmonella*** spp. | Yes / No | No / No | Yes | No |
| **Potential toxic elements** | | | | |
| Cadmium | Yes / No | Yes / Yes | Yes | Yes |
| Chromium | Yes / No | Yes / Yes | Yes | Yes |
| Copper | Yes / Yes | Yes / Yes | Yes | Yes |
| Lead | Yes / No | Yes / Yes | Yes | Yes |
| Mercury | Yes / No | Yes / Yes | Yes | Yes |
| Nickel | Yes / No | Yes / Yes | Yes | Yes |
| Zinc | Yes / Yes | Yes / Yes | Yes | Yes |
| **Stability** | | | | |
| Microbial respiration rate | Yes* / NA | Yes / NA | No | No |
| Loss on ignition | Yes / No | Yes / Yes | No | No |
| **Characterisation - weed seeds and propagules** | | | | |
| Tomato plant generation | Yes / NA | Yes / NA | Yes | No |
| Tomato plant growth | Yes / NA | Yes / NA | Yes | No |
| Tomato plant abnormalities | Yes / NA | Yes / NA | Yes | No |
| **Contaminant** | | | | |
| Non-stone inerts >2mm | Yes / No | Yes / No | Yes | No |
| Stones >5mm | Yes / No | Yes / No | Yes | No |
| Stones >5mm (mulch) | Yes / NA | Yes / NA | Yes | No |
| Particle size distribution | Yes / NA | Yes / NA | No | No |
| **Recommended** | | | | |
| Dry matter or moisture | Yes / Yes | Yes / Yes | No | No |
| TOC | Yes / NA | Yes / NA | No | No |
| C:N ratio | Yes / NA | Yes / NA | No | No |
| Electrical conductivity | Yes / Yes | Yes / No | No | No |
| Bulk density | No / No | Yes / No | Yes | No |
| pH | Yes / Yes | Yes / Yes | Yes | No |
| Total nitrogen | Yes / Yes | Yes / Yes | Yes | No |

| PAS100 / 110 | CAP / MAP | PT Scheme 45 / 38 | LÜRV-A | MARSEP (compost & S sludge) |
|---|---|---|---|---|
| Total phosphorus (P)[1] | Yes / Yes | No / Yes | Yes | No |
| Total potassium (K)[1] | Yes / Yes | No / Yes | Yes | No |
| Calcium[1] | Yes / NA | No / NA | Yes | No |
| Magnesium[1] | Yes / NA | No / NA | Yes | No |
| Sulphur[1] | Yes / NA | No / NA | No | No |
| Boron[1] | Yes / NA | No / NA | No | No |
| Copper[1] | Yes / NA | No / NA | No | No |
| Iron[1] | Yes / NA | No / NA | No | No |
| Manganese[1] | Yes / NA | No / NA | No | No |
| Zinc[1] | Yes / NA | No / NA | No | No |
| Sodium[1] | Yes / NA | No / NA | No | No |
| Ammonia[2] | Yes / NA | Yes / NA | No | No |
| Phosphorus[2] | Yes / NA | Yes / NA | No | No |
| Potassium[2] | Yes / NA | Yes / NA | No | No |
| Calcium[2] | Yes / NA | Yes / NA | No | No |
| Magnesium[2] | Yes / NA | Yes / NA | No | No |
| Sulphur[2] | Yes / NA | Yes / NA | No | No |
| Boron[2] | Yes / NA | Yes / NA | No | No |
| Chloride[2] | Yes / No | Yes / Yes | No | No |
| Copper[2] | Yes / NA | Yes / NA | No | No |
| Iron[2] | Yes / NA | Yes / NA | No | No |
| Manganese[2] | Yes / NA | Yes / NA | No | No |
| Zinc[2] | Yes / NA | Yes / NA | No | No |
| Sodium[2] | Yes / Yes | Yes / Yes | No | No |

* A range of microbial respiration tests are offered; [1] BSEN 13651 - CaCl extraction; [2] BS EN 13652 - water soluble nutrients and elements. NA = Not Available

| PAS110 | MAP | PT Scheme 38 | LÜRV-A | MARSEP (compost & S sludge) |
|---|---|---|---|---|
| VFA | No | No | NA | No |
| Anaerobic digestion | No | No | NA | No |

## 3.2 Key findings

i. While there are a considerable number of PTS operating across the world many are relatively restricted in terms of the parameters covered, relevant sample matrices or importantly the number of participating organisations.
ii. Twenty PTS have been identified with at least one determinand or group of determinands similar to the PAS100 or PAS110 analytical suite, and for a comparable matrix.
iii. Four schemes have been identified with a better match of parameters and matrices for the PAS100 and 110 schemes than others (Table 1). All of them have reported that additional matrices and parameters could be added to their existing scheme:
   - US CAP and MAP (two schemes)
     - The Compost Analysis Proficiency (CAP) Programme has the greatest synergy of the identified PTS to PAS100 and have recently added microbial pathogens to their suite of parameters. Pathogens are however already covered by the UK VETQAS scheme which is used by current PAS100 and PAS 110 appointed laboratories. The CAP scheme only provides compost samples but if costs prevent participation in a second scheme the CAP PTS is considered to provide a relevant test for compost and digestate as there are sufficient matrix similarities.
     - The Manure Analysis Proficiency (MAP) programme covers a range of parameters required for PAS110 but only covers manure.
   - Dutch WEPAL (one scheme) – The MARSEP programme includes composts and manures but only covers PTEs that are relevant to PAS 100 or 110.
   - German LÜRV (one scheme) – LÜRV-A Bioabfall scheme for compost incudes tests for *Salmonella* spp., PTEs, contaminants, and growth tests but not the microbial respiration test.
   - French BIPEA (two schemes) – The 'PTS 45 Organic fertilising materials' scheme includes composts and the 'PTS 38 - Activated sludge & sediments' is for sludge. The scheme provides good parameter matching to the PAS 100 /110 requirements but does not include microbial tests – although it does encompass microbial respiration tests, plant response tests and (importantly) physical contaminants.
iv. No schemes have been identified for PAS 110 VFAs and RBP despite personal communications with ECN and LÜRV in Germany.
v. Most PTS will add additional matrices to existing schemes but the costs of adding an extra parameter or group of parameters for a small number of laboratories will be considerably higher than for those provided within the routine scheme.
vi. All PTS charge laboratories a fee for participation with prices varying from £200 to £1000 per sample distribution and this is additional to the internal costs incurred in testing the samples.

# 4.0 General considerations for QC and PTS

## 4.1 Routine Quality Control as part of good laboratory practice

It is well proven that the primary way of achieving a desired performance standard and ensuring it can be maintained and demonstrated aside from using appropriate analytical methods and instruments is the application and monitoring of *routine* quality control procedures.

Simple quality control measures e.g. ensuring sample homogeneity, representative sub-sampling, use of validated methodology for the appropriate test matrix, use of blanks, replicates and control samples and monitoring of this data using Shewhart control charts all help to prevent reporting of incorrect data. They also provide an audit trail to demonstrate the performance of the method (i.e. within pre-defined performance characteristics) and can help to identify the source of errors enabling remedial action to be undertaken. Use of control samples provides valuable data on overall method performance with respect to drift and step changes.

The first step in this approach is the production of an unambiguous description of the method, and ensuring that this method is appropriately followed. Test methods are prescribed (and in some cases, defined) in the PASs but there may be differences in interpretation. The physical contaminant test was examined as an example.

## 4.2 Physical Contaminant Test – Interpretation

The current appointed laboratories were specifically asked about (i) the quantities of material used in the physical contaminants' test and (ii) the number of replicates routinely undertaken (a volume of between 1.125 and 2.25l is specified in the PAS100 test method (AfOR MT PC&S). Although this represents only two aspects of the entire test procedure the responses indicated that there are variations in the way the test requirements have been implemented.

The specified method requires that three separate subsamples are tested. Prior to testing an 8 mm screening step is used to define the size of the subsamples: if 50% of the particle size is <8 mm a 2 litre sub-sample is required and if 50% is >8 mm a reduction in sample size to 1.25 l is permitted.

The three laboratories interpret this requirement in different ways, potentially leading to inconsistencies across the laboratories:

- Laboratory 1 sieves one litre of air dried material through an 8mm sieve as an initial step to determine the sample volume required for the test. If >50% of the material is <8 mm, two litres of the 'as received' sample will be taken for the test; if <50% passes through the sieve, 1.25l is taken. There is no routine replication.
- Laboratory 2 takes 1.5l of air dried compost and carries out three replicates and reports the average value.
- Laboratory 3 takes 1kg of fresh material which when dried equates to circa 500 to 800g of compost for testing, again with no replication.

Taking a representative sub-sample for testing from the primary laboratory sample can be particularly problematic as the physical contaminants are unlikely to be evenly distributed within the sample. In the case of stones and other inerts these will by their nature tend to settle to the bottom of a sample and be quite difficult to 're-suspend'.

No control or reference materials are currently used by any appointed laboratory for this parameter. However, as part of internal quality control procedures, tests should be undertaken to quantify within-laboratory precision and, where relevant, spiking recovery – even without participation in an external PTS. For example, a 'clean' in-house bulk reference compost could be spiked with a known weight of physical contaminants and the recovery calculated. This simple test could be undertaken in each batch tested and control charts produced. This could be an interesting activity to undertake using different laboratory personnel to see if the use of different employees leads to systematic bias. All laboratories should have some internal training exercise/procedure to demonstrate competency prior to undertaking routine analysis.

## 4.3 Participation in proficiency testing scheme(s) for PAS 100 and 110

While there are many benefits of participation in a PTS, they must never be seen as a substitute for routine laboratory internal quality control. Importantly, they do not provide the means to validate test methods.
A PTS can provide laboratories with an indication of problem areas but do not provide any diagnostics to help solve the problem; a successful performance for the analysis of one determinand does not indicate that a laboratory is equally competent for another.

Internal laboratory quality assurance and control measures should always provide the first port of call in controlling data quality. A PTS can be used to supplement standard quality control measures undertaken as part of good laboratory practice. PTS participation is no guarantee of consistency or accuracy unless poor performance is monitored and acted upon.

As long as internal quality control measures are in place, are monitored and acted upon, a PTS provides the final check that results are consistent with other laboratories. For this purpose it is therefore only necessary to participate in a PTS one or two times a year to keep the costs low.

PTSs are widely used in other sectors (for example, testing foodstuffs for mycotoxin contamination or soils for heavy metal contamination) and provide an important external and independent check on the quality of data provided by an individual laboratory as well as the consistency in data between laboratories. Specifically PTSs provide an on-going measure of performance and can be used to:

- identify the range of concentrations or values reported by a laboratory and for all participating test laboratories;
- confirm achievable limits of detection – the smallest level which it is possible to determine with acceptable confidence of detection;
- provide a measurement of precision i.e. the closeness of agreement between measurements made on identical test portions. Precision will be more important at concentrations close to the PAS quality limits;
- identify laboratories for which there may be systematic error or bias (the closeness of agreement between the mean of a number of replicate determinations and the true value). Bias, once determined, can be corrected for, provided its source is understood. (Note: this approach will be dependent on having a known test value or sufficient participating laboratories to generate a statistically sound value);
- identify matrices for which interferences or test procedures need to be better controlled i.e. factors that change precision or bias (usually adversely if not addressed).

It is important to realise that whilst a PTS provides a comparison of the test result from one laboratory to another, the method used could, and often does, vary. Whilst the PASs list the required method for each determinand, there will be differences in how the method is adopted within each laboratory. Even where a basic method is adopted by the appointed laboratories, common variants will include sample preparation equipment, extraction methods (e.g. aqua regia digestion to determine PTEs may use different acid concentrations, extraction periods, reflux conditions and equipment), detection instrumentation and calibration procedures.

### 4.4 A PTS Framework

Considerable staff resource is required to develop, monitor and run a PTS. Care is needed in the collection of suitable samples and in preparation of 'identical and homogeneous' test samples. Homogeneity of test materials is essential if an unbiased comparison or rating is to be given – for comparative purposes the samples should be as near identical as makes no difference.

It is considered that a UK PAS specific PTS would be unaffordable for the current size of the business, especially where only a small number of distributions are required per annum.

PTS participation is the final step in the QC process and therefore the frequency of participation is typically found to be two to four distributions per annum as an on-going independent check of laboratory performance.

Economics are only part of the problem; with only a small number of participating laboratories there will be huge uncertainties in test result interpretation and performance evaluation, as the most commonly used z-score approach relies on a sufficient number of participating laboratories to reduce the impact of 'outlier' results or results that are not close to the mean.

## 5.0 Steps to improve the performance of PAS appointed laboratories

The report on the ring test results undertaken by the laboratories in 2012 provides initial evidence to suggest that the comparability of data produced by laboratories appointed under PAS schemes is poor for a selection of parameters (WRc, 2012).

A number of steps are proposed to improve the reliability and accuracy of PAS data. These are ranked in terms of the most immediate requirements for action and based on the information that has been supplied by the laboratories regarding existing laboratory practice.

### 5.1 Internal QA QC improvements

Having reviewed the information provided by the current appointed laboratories it is suggested that as an initial step (and in line with good laboratory practice), greater consistency in method validation and the use of simple quality control procedures could be used to improve the robustness of data being reported for PAS 100 and 110.

Good quality control is one of the most important ways of achieving good laboratory performance and ensuring it is maintained. Quality Management Systems (QMS) such as ISO17025 and Good Laboratory Practice can provide a valuable role in controlling the quality of the data generated by the laboratory. A QMS defines the processes being undertaken in the laboratories, the control and monitoring systems used and documentation management. QMS provide the framework under which QC can be effectively operated.

Actions include:

5.1.1 Making improvements to internal quality control procedures and monitoring of this data. A very valuable reference document is 'A Manual on Analytical Quality Control' (WRc 1989) which details all necessary actions and procedures needed to maintain control for data reporting.

5.1.2 All appointed laboratories should complete validation of test methods for the relevant PAS matrix using established performance testing procedures, for example the UKAS / DWi suggested approach of 11 replicates (WRc, 1989 & DWi 2012).

5.1.3 Laboratories should produce a bulk internal reference or control sample that should be included in all test batches. A common preservation method for compost is drying but other procedures such as freezing may be more appropriate for digestate e.g for the PAS110 RBP test. The laboratories should undertake a suitable investigation to identify a technique which provides the most consistent control.

5.1.4 Control charts should be set up to record the results of the internal control sample, to provide a continuing check on analytical performance. These charts can be used to identify random step changes or longer term data trends that may be due to equipment maintenance or failure. Regular monitoring of these charts will ensure prompt remedial action.

## 5.2 PAS laboratory organised PTS

Once routine QC measures are tightened there would be value in undertaking a ring test to assess between laboratory variability. This could involve a single distribution to make comparison with the original ring test exercise and look for improvements driven by tightening of routine QC procedures or, if participation in a PTS scheme in the immediate future is not required, the same sample could be repeatedly tested over time. A dried bulk control sample could be prepared by an appointed laboratory or third party independent organisation for distribution amongst the remaining current appointed laboratories to provide an internally run 'mini' PTS. If testing is repeated over time REAL or an appointed organisation could produce Youden control charts to identify systematic bias between laboratories assuming the laboratories are prepared to share their test data.

The cost for producing a bulk control sample is relatively minimal (1-2 days effort) and this approach controls costs incurred by the laboratories for testing. The evaluation of test data could be undertaken by REAL for ca. 2 days effort per distribution. This approach would keep costs to a minimum and avoid the costs of purchasing standard reference materials which are commonly provided in only 50-100g quantities. It is recommend that a single ring test is completed regardless of recommendations for participation in an externally run PTS.

## 5.3 External PTS

5.3.1 Once the laboratories have filled any gaps in their internal quality control procedures the appointed laboratories should be encouraged to join an existing scheme which has the benefit of a data set from a large number of participants; even if a few parameters are not covered. This is because the scoring is less affected by outlier data and costs for participation (if these are to be borne by the laboratories) are likely to be minimised. Participation in a PTS will provide the opportunity to assess how PAS laboratories perform against each other which monitoring of internal QC arrangements do not provide. International schemes operate between two and four distributions per annum. It is recommended that a minimum of two distributions should be participated in per annum. If the current number of samples being analysed by the PAS appointed laboratories increases, frequency of participation should be reviewed.

5.3.2 There are two PTS schemes that have particular synergy with PAS 100 and 110 and which cover the majority of the parameters required. The first is a French run BIPEA scheme (PTS 45 Organic fertilising materials / PTS 38 - Activated sludge & sediments) which, with the omission of pathogens and the digestate stability tests, covers the majority of parameters required by PAS 100 and 110. All current UK appointed laboratories already participate in the VETQAS scheme for microbiological parameters and WRc recommend that this continues. The second most likely match for the PAS 100 scheme is the US CAP programme which already distributes dried compost samples outside of the US and includes pathogen parameters. A similar scheme (MAP) provides more limited coverage of PAS 110 and solely for manure samples. Participation in the CAP programme alone would still provide value for laboratories under PAS110, especially for fibre testing. The value of such participation will only, however, be realised if performance data is reviewed and anomalies or bias acted upon.

No scheme has been identified that includes the volatile fatty acid and residual biomethane potential tests in the PAS 110.

5.3.3 A potential solution to accommodate the lack of any currently available PTS for VFAs and the RBP test for PAS110, would be to ask the appointed laboratories to undertake a ring-test. With the permission of current AD operators and receipt of a larger sample, laboratories could alternate preparation and distribution of the test sample or this could be undertaken by an independent third party. Again, results could be shared if agreeable to all participating laboratories. Whilst the small participating cohort would restrict the use of performance rating using a z-score approach, simple tools such as Youden graphs would allow a laboratory to see if their results are consistently higher, lower or similar to the other participating laboratories, which could be a prompt for improved method control or adjustment. REAL could ask for this data to be returned on (for example) an annual basis. This could be for a minimum of two samples per annum (see 3.1). Freezing aliquots of such a sample would allow for more regular testing.

# References

A Manual on Analytical Quality Control for the Water Industry R.V. Cheeseman and A.L. Wilson. Revised by M.J. Gardner, June 1989 (WRc Report NS 30)

Statistical Evaluation of Laboratory Precision and Performance for selected PAS100 Specification Parameters, N Agbasiere, S. Blake, E. Glennie and F. Vigo August 2012 (WRc Report UC9016.01)

DWi, Guidance on the Implementation of the Water Supply (Water Quality) Regulations 2000 (as amended) in England, version 1.1, March 2012, Appendix A, Section 3.6-3.9 http://dwi.defra.gov.uk/stakeholders/guidance-and-codes-of-practice/WS(WQ)-regs-england2010.pdf

The Test Method for the Examination of Composting and Compost (TMECC) US Composting Council

US EPA - Chapter 9 Sampling Procedures and Analytical Methods for sludge and compost. (EPA 1993) *www.epa.gov/nrmrl/pubs/625r92013/625R92013chap9.pdf*

# Appendix A: Adopted methods and quality control procedures within current PAS 100 / 110 appointed laboratories

The data summarised in this Appendix provides background on the range of procedures / approaches being used by currently appointed PAS 100 /110 laboratories for all parameters in the PAS schedules. The summarised data are presented using the same spreadsheet format sent to each facility to gather information. As of April 2014, one laboratory is seeking to be re-appointed, but their data is included for completeness.

A1 PATHOGENS PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-house | Yes | No | | No | - | No | | No | VETQAS | Compost | |
| 2 | Sub-contracted | Yes | Yes | UKAS* | Yes | - | Yes | Culture | | VETQAS | ABP | |
| 3 | Sub-contracted | Yes | Yes | UKAS* | Yes | - | | | | VETQAS | Compost | |
| 4 | In-house | Yes | Yes | BSI-ISO | Yes | Records | Yes | Oxoid | No | VETQAS | Compost | ISO 16140 |
| 5 | In-house | Yes | Yes | ISO 16140 | Yes | - | Yes | - | - | VETQAS | Compost | ISO 16140:2003 defines the general principle and the technical protocol for the validation of alternative methods in the field of microbiological analysis |

A2 PATHOGENS PAS110

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | Sub-contracted | Yes | Yes | UKAS* | Yes | - | - | - | - | - | - | |
| 5 | In-house | Yes | Yes | ISO 16140 | Yes | - | Yes | - | - | VETQAS | - | |

* UKAS – Dwi requirements inferred

## B1 POTENTIALLY TOXIC ELEMENTS PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes (Except Hg) | No | - | No | - | In-house | Soil | Yes | Aquacheck | Soil / Sludge | |
| 2 | In-House | Yes | Yes | Inter-lab trials * | Yes | Yes | Yes | Compost | Yes | Only inter-lab trials | Compost | Method validated against other labs on the PAS100 Scheme circa once a month in addition to internal validation |
| 3 | In-House | Yes | Yes | DWI 11*2 | Yes | Yes | No | - | - | Contest, Aquacheck WEPAL MARSEP | Soil/ Sludge/ Compost | |
| 4 | Sub-contracted | Yes | Yes | DWI 11*2 | Yes | Yes | No | - | - | Contest, AquacheckW EPAL MARSEP | Soil/ Sludge/ Compost | |
| 5 | In-House | Yes | Yes | 10 batches against CRM in ashed biomass | Yes | Yes | Yes | Ashed biomass | Yes | Aquacheck | Soil, acidified solution | |

* Note : response provided by lab; but not a recognised procedure for validation

## B2 POTENTIALLY TOXIC ELEMENTS PAS110

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | In-house | Yes (for separated fibre) | BS EN 15587-1: 2002 for Whole Digestate and Liquor | 11*2 MCERTS for sandy/loamy/clay soil | Yes | Yes | No | - | - | Contest, Aquacheck, WEPAL MARSEP | Soil, sludge, compost | |
| 5 | In-house | Yes | - | 10 batches against CRM in ashed biomass | Yes | Yes | Yes | Ashed Biomass | Yes | Aquacheck | Soil, acidifed solution | |

## C1 STABILITY (MICROBIAL RESPIRATION RATE) PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details * | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | | | | | | | |
| 2 | Sub-contracted | Yes | Yes | | Yes | No | No | | | Ring-test | | |
| 3 | Sub-contracted | Yes | Yes | | Yes | No | No | | | Ring-test | | |
| 4 | Sub-contracted | Yes | No | | No | | | | | | | |
| 5 | In-House | Yes | No* | | Yes | No | No | | | No | | Validation carried out over 15 years ago - no documentation |

## C2 STABILITY PAS110 (VFAS, RESIDUAL BIOGAS POTENTIAL TEST) PAS110

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details * | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | Sub-contracted | | | | | | | | | | | ** |
| 5 | In-house | Yes | No | | Yes | No | No | | | No | | |

**Key:** * No details provided

** Information was not provided

D1 WEED SEEDS AND PROPAGULES PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | - | Yes | Peat | No | No | | |
| 2 | In-House | Yes | Yes | Inter-laboratory trials with peat * | Yes | Yes | Yes | Peat | Yes | Inter lab trial | | |
| 3 | Sub-contracted | Yes | | | Yes | No | No | | | Ring-test | | |
| 4 | In-House | Yes | Yes | Test validity criterion * | Yes | No | No | | | Afor inter lab trial | Compost / Growing media | |
| 5 | In-House | Yes | No* | | Yes | No | No | | No | No | | Validation carried out over 15 years ago - no documentation |

* Note : response provided by lab; but not a recognised procedure for validation

E1 PHYSICAL CONTAMINANTS PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | No | No | | | No | | |
| 2 | In-House | Yes | Yes | Interlab trials * | Yes | Yes | Yes | Compost | Yes | Inter lab trials / in-house * | | |
| 3 | In-House | Yes | No | | No | | | | | | | |
| 4 | In-House | Yes | Yes | Afor MT PC&S 2012 * | No | | Yes | Sand | No | | | |
| 5 | In-House | Yes | No | | No | No | No | | No | No | | Validation carried out over 15 years ago - no documentation |

* Note : response provided by lab; but not a recognised procedure for validation or PTS

E2 PHYSICAL CONTAMINANTS PAS110

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | In-House | Yes | Yes | No details | | | | | | | | |
| 5 | In-House | Yes | Yes | No document ation | No | No | No | - | - | No | - | |

F1 PARTICLE SIZE DISTRIBUTION, DRY MATTER, LOSS ON IGNITION, TOTAL ORGANIC CARBON, C/N RATIO PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | | Yes (except DM,PSD) | Soil, compost | Yes (except DM,PSD) | Aquacheck (LOI,pH only) | Soil / Sludge | |
| 2 | In-House | Yes | Yes | Interlab trials | Yes (Only PSD) | Yes | PSD only | Compost | Yes | Inter lab trial | | |
| 3 | In-House | Yes (except DM) | LoI only | DWI / UKAS 11*2 | Yes (Only LOI) | Yes | | | | LoI (WEPAL ISE and MARSEP) | Soil / Manure / Compost / Organic fertilizers | |
| 4 | In-House | Yes | Yes | BSI/ISO or internal | No | | Yes | Sand | No | AfOR interlab trial & interlab study prEN 0023078 | Peat / Bark / Green Waste Compost / Perlite | |
| 5 | In-House | Yes | Yes (DM & LOI only) | 10 batches against CRM in food | No | No | Yes (except PSD) | Ashed biomass, food | Yes (except PSD) | QFCS | Food | |

G1 ELECTRICAL CONDUCTIVITY, PH, BULK DENSITY, TOTAL NITROGEN, TOTAL PHOSPHORUS (P), TOTAL POTASSIUM (K), CALCIUM, MAGNESIUM, SULPHUR, BORON, COPPER, IRON, MANGANESE, ZINC, SODIUM PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | - | No | - | Yes | Soil | Yes | Aquacheck / Other | Soil/ Sludge Food/ Feed | |
| 2 | In-House | Yes | Yes | Interlab trials | Yes | Yes | Yes | Liquid | Yes | Inter lab trials | Compost | |
| 3 | In-House | Yes | Yes (TN only) | DWI / UKAS 11*2 | Yes | Yes | No | | | Aquacheck, WEPAL MARSEP for Total N only | Soil/ Manure/ Compost/ Organic fertilizers | |
| 4 | In-House (pH/EC only remainder Sub-contracted | Yes | Yes | Externally validated BSI/ISO | Yes (Only EC) | Yes | Yes | Liquid | Yes | Afor interlab trial / Own interlab trial with PAS100 labs | Compost/ Growing media | |
| 5 | In-House | Yes | Yes (except TP, Ca, Mg, S, B) | 10 batches | Yes | No | Yes (except TP, Ca, Mg, S, B) | Ashed biomass, water, food | Yes | Aquacheck / QFCS | Water/ Soil | |

G2 ELECTRICAL CONDUCTIVITY, PH, BULK DENSITY, TOTAL NITROGEN, TOTAL PHOSPHORUS (P), TOTAL POTASSIUM (K), CALCIUM, MAGNESIUM, SULPHUR, BORON, COPPER, IRON, MANGANESE, ZINC, SODIUM PAS110

| **Lab No.** | **In-house or Sub-contracted** | **Uses Method Specified** | **Method Validated** | **Validation Details** | **Control standards used?** | **Control Charts maintained** | **Reference materials used** | **Matrix** | **Control Charts used** | **Participation in PTS** | **Matrix** | **Notes** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | In-House | Yes | | | | | | | | | | |
| 5 | In-House | | | | | | | | | | | |

H1 EXTRACTABLE CALCIUM CHLORIDE AND DTPA SOLUBLE: PHOSPHORUS, POTASSIUM, MAGNESIUM, SULPHUR, BORON, COPPER, IRON, MANGANESE, ZINC, SODIUM PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | | Yes | Compost | Yes | No | | |
| 2 | In-House | Yes | Yes | * | Yes | Yes | Yes | | Yes | Inter lab trials | Compost | * Multipoint calibration with standards and matrix spikes and blanks used in every run |
| 3 | In-House | Yes | Yes | In-house | Yes | Yes | No | | | No | | |
| 4 | Sub-contracted | Yes | No | | No | | Yes | Compost | Yes | No | | |
| 5 | In-House | Yes | No* | | | | | | | | | * Validation carried out over 15 years ago |

## I1 EXTRACTABLE WATER SOLUBLE: AMMONIA-N, PHOSPHORUS, POTASSIUM, CALCIUM, MAGNESIUM, SULPHUR, BORON, CHLORIDE, COPPER, IRON, MANGANESE, ZINC AND SODIUM PAS100

| Lab No. | In-house or Sub-contracted | Uses Method Specified | Method Validated | Validation Details | Control standards used? | Control Charts maintained | Reference materials used | Matrix | Control Charts used | Participation in PTS | Matrix | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | In-House | Yes | No | | No | | Yes | Compost | Yes | No | - | |
| 2 | In-House | Yes | Yes | * | Yes | Yes | Yes | - | Yes | Inter lab trials | Compost | * Multipoint calibration with standards and matrix spikes and blanks used in every run |
| 3 | In-House | Yes | Yes | In-house | Yes | Yes | No | - | - | No | | |
| 4 | Sub-contracted | Yes | No | - | No | - | Yes | Compost | Yes | No | | |
| 5 | In-House | Yes | Yes (except Ca, Mg, S, B) | 10 batches | Yes (except Cl) | No | Yes (except Ca, Mg, S, B) | Ashed biomass, water | Yes (except Ca, Mg, S, B) | Aquacheck | Water, soil | |

# Appendix B: Data summary of worldwide PTS of relevance to PAS 100 / 110

| No | Web address / Source | PT Scheme name | Contact | Accreditation | Matrix | PTEs | *E. Coli* | *Salmonella* | Physical contaminants | Plants response tests | Compost stability - Aerobic Digestion | Dry Matter, Total, water & pH, EC, Organic matter | Fertiliser nutrients (N, P, K, Na, Cl) | Digestate stability - anaerobic (RBP) | VFAs | Round / year | Cost* | Assessment method |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | http://www.lgcpt.com/productviewnarrow.aspx?SchemeID=100 | CONTEST (AQUACHECK) | | UKAS 0001 | Soil | X | | | | | | X | | | | 5 | | Z scores |
| 2 | http://ahvla.defra.gov.uk/ahvla-scientific/vetqas/PTpriceList.html | VETQAS (PT0039/PT0040 & PT0087/PT0088/ PT0084) | Vetqas – Quality Assurance Unit Vetqas@ahvla.gsi.gov.uk | UKAS 0004 ISO/IEC 17043 | Biological tissue | | X | X | | | | | | | | 4 | £631/513 & £276/467 | |
| 3 | http://www.isprambiente.it | APAT ICXXX | Maria Belli<br>Tel.: +39(0)6 50072952<br>Fax: +39(0)6 50072313<br>Email: maria.belli@isprambiente.it | ACCREDIA | Compost | X | | | | | | | | | | 3 | 0 | |
| 4 | http://www.bipea.org/node/98#sol | Scheme 45 | Carolina Mazzoni cmazzoni@bipea.org | COFRAC | Compost | X | | | X | | X | X | | | | 5 (2 compost) | £551 + £68 for association | Z scores |
| 5 | http://www.lhl.hessen.de/irj/LHL_Internet | LÜRV-A Bioabfall | Dr. Harald Schaaf<br>Tel.: +49-561-9888170<br>Fax: +49-561-9888300<br>Email: harald.schaaf@lhl.hessen.de | | Compost | X | | X | X | X | | X | X | | | 1 | £123-287 | Z scores |

| No | Web address / Source | PT Scheme name | Contact | Accreditation | Matrix | PTEs | *E. Coli* | *Salmonella* | Physical contaminants | Plants response tests | Compost stability - Aerobic Digestion | Dry Matter, Total, water & pH, EC, Organic matter | Fertiliser nutrients (N, P, K, Na, Cl) | Digestate stability - anaerobic (RBP) | VFAs | Round / year | Cost* | Assessment method |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6 | http://www.qrlservices.com/ipg/proficiency-testing,37142581 | LD001 | Ms. Sandra Suarez-Noguerol<br>Tel.: +34 981803884<br>Fax: +34 981803884<br>Email: info@qrservices.com | | Sludge | X | | | | | | | | | | 1 | £123 (excludes shipping) | |
| 7 | http://www.wepal.nl | MARSEP (MAnure and Refuse Sample Exchange Programme) | Drs. A. Eijgenraam<br>Tel.: +31 317 482349<br>Fax: +31 317 485666<br>Email: Info.Wepal@wur.nl | RvA on the basis of ILAC G13 | Compost | X | | | | | | | | | | 4 | £582 (excludes shipping) | z scores |
| 8 | http://www.hu.hamburg.de | Leistung test | Dr. Karla Ludwig-Baxter<br>Organisational unit: Institut für Hygiene und Umwelt<br>Tel.: +49-40-428453645<br>Fax: +49-40-428453847<br>Email: karla.ludwig-baxter@hu.hamburg.de | | Biological Tissue | | X | | | | | | | | | 2 | £241-803 (excludes shipping) | |
| 9 | http://www.ilvo.vlaanderen.be/Portals/68/documents/Mediatheek/Mededelingen/154_Vlarisub_ringtest_nov13.pdf | VLARISUB | Mr. Bart Vandecasteele<br>Tel.: +32 9 2722699<br>Fax: +32 9 2722701<br>Email: bart.vandecasteele@ilvo.vlaanderen.be | | Compost | X | | | | | | X | | | | 2 | | Z scores |
| 10 | https://www.vito.be/EN/HomepageAdmin/Home/Pages/Homepage.aspx | COALLA | Mr. Siegfried Hofman<br>Tel.: +32 14 335915<br>Fax: +32 14 321185<br>Email: siegfried.hofman@vito.be | VLAREL and VLM | Manure / Soil | | X | X | X | | | X | X | | | 2 | £722 (excludes shipping) | |

| No | Web address / Source | PT Scheme name | Contact | Accreditation | Matrix | PTEs | *E. Coli* | *Salmonella* | Physical contaminants | Plants response tests | Compost stability - Aerobic Digestion | Dry Matter, Total, water & pH, EC, Organic matter | Fertiliser nutrients (N, P, K, Na, Cl) | Digestate stability - anaerobic (RBP) | VFAs | Round / year | Cost* | Assessment method |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 11 | http://www.lgcpt.com/ | Animal Feeds Scheme - AFPS | Mr Abdul H Sheikh<br>Tel.: +44 (0)161 762 2500<br>Fax: +44 (0)161 762 2501<br>Email: abdul.sheikh@lgcpt.com | UKAS 0001 | Animal Feed | X | | X | | | | | | | | 4 | | |
| 12 | http://www.association-aglae.fr/?lang=en | AGLAE '9' | Mr. Philippe GUARINITel.: +33-320169140<br>Fax: +33-3-20169141<br>Email: contact@association-aglae.fr | SOFRAC | Soil | X | | | X | | | X | X | | | 2 | £317 (excludes shipping) | |
| 13 | http://www.association-aglae.fr/?lang=en | AGLAE '42' | Mr. Philippe GUARINITel.: +33-320169140<br>Fax: +33-3-20169141<br>Email: contact@association-aglae.fr | SOFRAC | Sludge | | X | X | | | | | | | | 1 | £361 (excludes shipping) | |
| 14 | http://www.lgcpt.com/ | Microbiology Scheme - QMS | Mr Abdul H Sheikh<br>Tel.: +44 (0)161 762 2500<br>Fax: +44 (0)161 762 2501<br>Email: abdul.sheikh@lgcpt.com | UKAS 0001 | Food, simulated on oatmeal matrix / Tea | | X | X | | | | | | | | 12 | | |
| 15 | http://eagri.cz/public/web/en/ukzuz/portal/ | MPZ UKZUZ - Soils | Dr. Jaroslava Srnkova<br>Tel.: +42(0) 543 548 220<br>Fax: +42(0) 543 210 444<br>Email: jaroslava.srnkova@ukzuz.cz | ISO/IEC 17043 | Soil | X | | | | | | | | | | 3 | £79 (excludes shipping) | |

| No | Web address / Source | PT Scheme name | Contact | Accreditation | Matrix | PTEs | *E. Coli* | *Salmonella* | Physical contaminants | Plants response tests | Compost stability - Aerobic Digestion | Dry Matter, Total, water & pH, EC, Organic matter | Fertiliser nutrients (N, P, K, Na, Cl) | Digestate stability - anaerobic (RBP) | VFAs | Round / year | Cost* | Assessment method |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 16 | https://www.collaborativetesting.com | CTS Agricultural Laboratory Proficiency (ALP) Program: Botanicals | Christopher Czyryca Tel.: +1+571-434-1925 x 103 Fax: +1-571-434-1937 Email: cczyryca@cts-interlab.com | | Agricultural Materials | X | | | | | | X | X | | | 3 | £520 (+£80 UK shipping) | |
| 17 | http://www.bipea.org | Scheme 38 | Carolina Mazzoni cmazzoni@bipea.org | Recognised by the Lloyd's Register Quality Assurance on the basis of ISO 9001 | Sludge | X | | | X | | X | X | X | | X | 5 (3 sludge) | £803 (+£68 for association), excludes shipping | z scores |
| 18 | http://compostingcouncil.org/compost-analysis-proficiency-program/ | CAP | Robert O. Miller, Ph.D. Colorado State University Fort Collins, CO 80550 Tel: 970-686-5702 Fax: 970-686-5709 Email: rmiller@lamar.colostate.edu | | Compost | X | X | X | X | X | X | X | X | | | 3 | | 95% CL of the median and evaluation of intra-laboratory precision established based on the consensus sample RSD |
| 19 | http://www.mda.state.mn.us/licensing/licensetypes/mapprogram.aspx | MAP - Manure Analysis Proficiency | Jerry Floren jerry.floren@state.mn.us Tel: 651-201-6642 Fax: 651-201-6112 | | Manure / Soil | X | | | | | | X | X | | | 2 | | |
| 20 | http://fapas.com/prog.cfm?currsch=leap&ny=1 | FAPAS/FEPAS/LEAP | FERA | UKAS 0009 | Soil | X | X | X | | | | | | | | 1 | | z scores |

* Approximate quote provided by PTS in US dollars or Euros and converted using following exchange rates 1 USD = 0.59 GBP, 1 EUR = 0.80 GBP (@25 June 2014)

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