

**Analysis of Waste Derived Compost Materials:**  
**An Investigation - Full Report**

PREPARED FOR ENVENTURE NORTHERN

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## **Abstract**

Steve Maslen and Associates were commissioned by Enventure Northern to investigate the matter of compost quality assurance in the UK following the discovery of interlaboratory analytical discrepancies. It is concluded that the industry would benefit from increased clarity regarding a definitive compost standard and universal analytical methods including standardised digest extractants and determination methods.

## **Summary**

As consultants to a major waste management company producing green waste compost, Steve Maslen & Associates (SMA) were commissioned to devise a routine compost sampling strategy and carry out analyses on samples of green waste compost collected. Analyses undertaken during 1996 gave rise to some unexpected results. When a further subsample of the same material was sent to a second laboratory to verify the results, differences were revealed which required investigation.

During a further round of sampling and analyses undertaken during 1997, two replicate samples of the same well mixed compost were submitted to two laboratories at the same time. Results revealed that while precision appeared generally to be good within each of the two laboratories - repeatability, differences in results between the two laboratories were inconsistent with each other - reproducibility. This prompted further research.

SMA were commissioned by Enventure Northern, to research the matter further at the end of 1997. This followed ENTRUST project approval. The objectives of the research were to:-

- Undertake a desk-based investigation regarding compost standards and compost quality assurance in the UK and other European countries, with particular

reference to standardised or accredited test methods.

- Identify any other Green Waste producers having experienced similar cases of analytical discrepancy
- Raise and discuss the issues with the Composting Association, the National Compost Development Association and other interested parties.
- Investigate how more standardised analytical techniques can be agreed for Green Waste composts.
- Re-assess the results of the 1997 SMA trials and comment with regard to reproducibility of results and standardisation of analytical techniques in the light of the above.
- Publish the results in an appropriate scientific/waste management journal.

Standardised, reliable, precise laboratory methods, together with a system of laboratory, producer and product accreditation are required for reliable characterisation and quality assurance of compost materials, if composted organic waste materials are to be marketed more successfully. It is in the interest of the industry as a whole to raise confidence in the market and to reduce potential liabilities with regard to the use of a waste-derived product by ensuring reliable and standardised techniques are established.

A short review of compost standards and quality assurance of compost products in the UK and other European countries has been carried out and recommendations made for the UK composting sector.

It is worth noting that although analytical techniques are being examined here, just as important is the sampling procedure used to collect the samples. These too are in need of industry-wide standardisation. Although this aspect is outside the scope of this study, it is recognised that the sampling process is the single biggest potential source of error in the total analytical procedure (Lustenhouwer et al. 1991).

It is concluded that the industry would benefit from increased clarity regarding a definitive compost standard and universal analytical methods including standardised digest extractants and determination methods.

### ***Background***

Wastewise, a client of SMA, began a composting program for municipal source separated organic wastes on two of their operational landfill sites. Composting is carried out on open air concrete pads on which the organic materials are stacked and windrowed following an initial shredding. As an integral part of this program, a strategy for the sampling and analysis of materials was devised by SMA. Composted materials are sampled from 25 points around the windrow and thoroughly homogenised before replicate samples are taken by spoon sampling and sent to a laboratory for analytical characterisation including macronutrients, heavy metals, pH and physical properties.

### ***Identifying the Problem***

During a round of analyses carried out for Wastewise during 1996 when a single sample was sent for analysis, certain unexpected results from the heavy metal analyses gave rise to suspicions of analytical imprecision. The determination for manganese in particular gave cause for concern, being in excess of The Composting Association Standard for Compost Produced by Aerobic Methods limit (now out of date and currently under review) and also the ICRCL limits for Soil Class A Uncontaminated Soil (ICRCL, 1987). Two replicate subsamples of the same material were sent to a different

laboratory and detected concentrations were considerably lower, revealing discrepancies between laboratories. It was proposed that during the next round of sampling, replicate samples would be submitted from the same sample to two different laboratories for comfort and verification.

During 1997 a further composted green waste sample was collected for analysis, once again comprised of 25 subsamples taken from the windrow. Two replicates of the sample were sent, each to two different laboratories (4 subsamples in total). Replicates were separated off by the spoon sampling technique.

This exercise proved to be very useful in identifying that the problems of imprecision appeared not to be caused by poor repeatability, both laboratories giving two sets of results generally in close agreement with each other, also validating the sampling precision. However, poor reproducibility between the two laboratories appeared to point towards a problem of non-standardised analytical procedures and techniques.

These analytical discrepancies were seen to have implications for quality assurance of waste derived compost products, and established the need for more thorough investigations.

### ***Research Objectives***

Several questions emerge from the reproducibility problems experienced. It is the aim of this research to make some initial observations and recommendations addressing the following points:

- Standardised, reliable, precise laboratory methods, together with a system of laboratory, producer and product accreditation are required for reliable characterisation and quality assurance of compost materials, if the composted organic waste materials are to be marketed successfully.

- Ultimately, the question must be asked as to whether the full range of concentrations that have been determined (discounting any imprecision) constitute a significant toxicity risk to the soil and wider environment.

Once standardised techniques have been agreed, more exhaustive rounds of sampling could be undertaken if necessary to re-assess reproducibility between laboratories.

### ***A Review of Compost Standards & Quality Assurance - Composting in the UK***

In the UK there are as yet no official standards for potentially toxic element (PTE) concentrations in compost treated soils or the composts themselves, the nearest relevant universal standards are those for sewage sludge amended soils, although the predecessor of the Composting Association, OWCA (the Organic Waste Composting Association) and the Soil Association, to name two, each have their own standards for composted materials. The Composting Association (CA) is currently undertaking an investigation of existing standards and a feasibility study into the development of a new standard.

A compost standard, if adopted would give a quality bench mark to be met by all producers of composts for soil improvers and soil substitutes. The Environmental Services Association (ESA) have drafted recommendations for the EC Composting

Directive working paper to be produced in 1999. It is the opinion of the ESA that a voluntary national quality scheme would be more effective and more appropriate (Composting News, Spring 1998). It is likely that any standards recommended by the CA will be based on those being developed by the Council of European Nations (CEN/TC/233). Once the CEN has arrived at a set of agreed standards, the BSI will implement any national standards that are to be adopted. These would be at least as stringent as the CEN standards. However, if an industry standard is embraced rather than a national standard, criteria may be less stringent.

### ***Composting in Europe***

It is evident that although there is a general consensus that separating and recycling organic wastes rather than landfilling is one of the key challenges facing the waste industry in the late twentieth century, composting as a widespread activity has been adopted at different rates from country to country. However, of the 12 European countries studied in the report by the Organic Composting and Reclamation Association (ORCA), some 9 had implemented or proposed standards.

The working group, CEN/TC/233, have long advised on the development of compost legislation in Europe and have just released their draft document on standards. In addition, the EC Composting Directive working paper is to be published in 1999, and is currently being compiled in Brussels.

Among the European countries, Austria stands out as having one of the most advanced

national composting programs, and a three tier compost class system has been developed as defined by the well established Ö-NORM S2200 and the Ö-NORM S2022 standards. The Ö-NORM S2200 standard classifies composts of classes 1 and 2, which are high class products (esp Class 1) produced from source separated VFG (Vegetable Fruit and Garden) wastes. In Austria VFG must be collected and treated separately nationwide under the Separate Collection of Organic Wastes law. Class 2 compost is defined by heavy metal limit values equal to the Austrian limits for soil - *bodengrenzwerte*. Class 1 is a higher quality product with limits at 70% of *bodengrenzwerte*. Importantly, accreditation to the standards is managed entirely by the Ö-NORM institute; both sampling and analysing are performed by governmental institutes or by government authorised organisations. The third class is comprised of materials which fail to comply with class 2 heavy metal limits - the standard is defined by Ö-NORM S2022 and its application is regulated by Ö-NORM S2024. In general in Austria, a market only exist for the Ö-NORM S2200 Class 1 compost.

Considerable work has also been done in Germany by the independent Association for the Control of Compost Quality (known as ACCQ or FCQAO), devising a standard accredited by the German quality assurance label RAL, in establishing a detailed standardised strategy for sampling and analytical techniques, but also in carrying out extensive interlaboratory testing. In 1997 the FCQAO gave assessment to approximately 240 composting plants, monitoring the capabilities of registered laboratories to carry out compost analyses, in addition to assessing the quality assurance of the compost products. Although the scheme is voluntary and unofficial, uptake has been considerable.

### ***Results: 1997 Trial***

Discussions with the two laboratories, referred to here as Lab A and Lab B to disguise their identities, have identified areas of non-standardised procedure and differences of opinion, regarding choice of analytical methods. These differences exist in respect to

the extractant mix chosen for the digest, as well as the determination method utilised. The 1997 analytical results and methods are included in Tables 2a and 2b.

The trial results have been compared with the UK maximum permissible PTE (Potentially Toxic Element) limits in sewage sludge amended soils (UK, 1989), the Compost Association Standard for Compost Produced by Aerobic Methods (currently being revised), and the FCQAO Limits for heavy Metal Concentrations in Quality Compost. These various standards are represented in Table 1.

In addition, the results are compared to typical soil background concentrations and the ICRCCL Limits for Uncontaminated Soil - Class A. While not wanting to confuse compost standards with soil concentration limits, it is important to compare the two, as in many cases, composts may be used as topsoil making materials, and must therefore compare favourably with accepted soil quality standards.

**TABLE 1 - Standards for Evaluating Compost Quality - Acceptable PTE Concentrations**

	SI, 1989, No 1263 (pH 6-7)	CA Standard (pH 5-9)	FCQAO Quality Compost	ICRCL, 1987 Class A
Total Lead	300	250	150	500
Total Nickel	75	100	50	20 (available)
Total Selenium	(3)*	5	-	1
Total Zinc	200	1000	400	250 (available)
Total Manganese	-	200	-	500
Total Cadmium	3	10	1.5	1
Total Molybdenum	(4)*	10	-	-
Total Arsenic	(50)*	15	-	30
Total Boron	-	5	-	2 (available)
Total Chromium	(400)*	1000	100	100
Total Copper	135	400	100	100 (available)
Total Mercury	1	2	1	1
Fluoride	(500)*	-	-	-

\* - Additional figures in brackets from DoE, 1989 - not determined by SI 1263

**TABLE 2a - Lab A PTE Analytical Results and Methods: 1997 Trial Replicates**

	W1 (mg/kg)	W2 (mg/kg)	Analytical Methods
Total Lead	43.9	43.2	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Nickel	15.6	13.9	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Selenium	0.52	0.53	BSI/ISO/MEWAM* - Atomic Fluorescence
Total Zinc	105	102	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Manganese	347	347	BSI/ISO/MEWAM* - AAS or ICP-AES

	W1 (mg/kg)	W2 (mg/kg)	Analytical Methods
Total Cadmium	0.33	0.31	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Molybdenum	1.55	1.55	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Arsenic	12.1	11.4	BSI/ISO/MEWAM* - Atomic Fluorescence
Total Boron	17.9	15.5	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Chromium	12.2	10.7	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Copper	23.2	23.3	BSI/ISO/MEWAM* - AAS or ICP-AES
Total Mercury	0.16	0.17	BSI/ISO/MEWAM* - Atomic Fluorescence
Fluoride	100	103	HClO4 - Ion selective electrode

BSI/ISO/MEWAM\* approved method = extraction using boiling mix of conc HNO<sub>3</sub> + HCl

**TABLE 2b - Lab B PTE Analytical Results and Methods: 1997 Trial Replicates**

	W3 (mg/kg)	W4 (mg/kg)	Analytical Methods
Total Lead	94.4	54.1	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Nickel	27.3	22.0	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Selenium	0.2	0.2	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Zinc	163.0	173.0	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Manganese	535	560	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Cadmium	0.6	0.6	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Molybdenum	3.3	6.2	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Arsenic	1.58	1.81	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Boron	57.6	54.2	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Chromium	43.7	39.3	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Copper	43.7	40.7	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Total Mercury	<0.1	<0.1	HNO <sub>3</sub> /HClO <sub>4</sub> - ICP-AES
Fluoride	2.4	2.4	H <sub>2</sub> O soluble - Colorimetry

Zinc (Zn), chromium (Cr), nickel (Ni), cadmium (Cd), copper (Cu), and arsenic (As), as

analysed by both Laboratory A and Laboratory B all fall below the various limit values. However this disguises the significant discrepancies between the sets of results from the two laboratories. For all these elements, Laboratory B analyses were consistently more sensitive than Laboratory A. The likely explanation for this is Laboratory B's use of a perchloric/nitric acid digest mix, which is more aggressive than the aqua regia used by Laboratory A.

The recorded Zn concentrations averaged around 100 mg/kg and 165 mg/kg for Laboratories A and B respectively, interlaboratory analyses are clearly not reproducible. The results are in excess of the Zn soil average concentration, although they are within the range of values naturally occurring in soils and the maximum permissible concentration of Zn in sludge amended soils; soil average 60 mg/kg, soil range 1.5 - 2000 mg/kg; and limited at 200 mg/kg in the UK, respectively. With such a magnitude of difference between Laboratory A and Laboratory B analytical results, a different sample with a slightly higher Zn content would sooner exceed the 200 mg/kg UK sludge amended soil limit for example, if analysed at Laboratory B rather than Laboratory A. An obvious consequence is that the material may be considered suitable for use if analysed at Laboratory A while considered too contaminated for use by Laboratory B's result.

Chromium in soils is naturally occurring at concentrations between 0.9 and 1500 mg/kg, although the mean value is around 84 mg/kg. The concentrations that have been reported in the 1997 trials are all under this value, however, the reproducibility between Laboratory A and Laboratory B for the Cr test is very poor. Laboratory A gave results in the region of 10 mg/kg, and Laboratory B around 40 mg/kg. This order of difference is highly significant a highly toxic element such as Cr. In this instance, however, all recorded values are well below the recognised limits for Cr. The FCQAO standard limits Cr at 100 mg/kg, as does the ICRCL, Soil Class A, while the provisional UK limit concentration for Cr in sewage sludge amended soil is 400 mg/kg.

Although the Ni concentrations as determined by Laboratories A and B are all below limit levels, there is a factor of between 1.5 and 2 difference between Laboratory A and Laboratory B results. The Ni results are in the region of some of the more stringent standards found in Europe, for example that of the Netherlands (20mg/kg). If a similar standard were to be adopted in the UK, the Laboratory B results would fail the compost, while the Laboratory A results are below this limit value and the compost could be passed as suitable for use as a soil additive.

Cadmium may be naturally occurring in soils in the order of fractions of mg/kg, and the UK sets a 3 mg/kg limit value on Cd in sewage sludge amended soils; the ICRCL Soil Class A limit is more stringent at 1 mg/kg. The results of the 1997 trials were below these limit values, but reproducibility was poor. Laboratory B's results were a factor of two greater than Laboratory A's 0.3 and 0.6 mg/kg respectively.

Copper in soils commonly ranges from 2.5 to 60 mg/kg, although the maximum permissible in sludge amended soils above pH 7.0 is 200 mg/kg (135 mg/kg between pH 6-7), and a typical figure in natural soils would be 26 mg/kg. The concentrations themselves in the 1997 results did not give cause for concern; however reproducibility was again poor between Laboratory A replicates at around 20 mg/kg and Laboratory B replicates, at around 40 mg/kg.

Reproducibility of the As determination between the two laboratories, was also found to be in question; there was a factor of ten disagreement between the two laboratories. However, the actual As levels recorded did not exceed the natural range in soils of 5 - 50 mg/kg.

The results for manganese (Mn) also demonstrated the reproducibility problems during

the analytical tests carried out in 1997. Although Mn is not regarded as a toxic element, the results illustrate the problem of poor reproducibility when two or more replicate results fall either side of a given standard. Mean test results in 1997 were 347 mg/kg and 548 mg/kg for Laboratory A and Laboratory B respectively. There is no limit specified for Mn in sewage sludge amended soils in the UK. Both results are below the mean concentration in soils 761 mg/kg (Davies & Jones, 1988), however, the result from Laboratory A is in excess of the ICRCL limit for Soil Class A Uncontaminated Soil - 500 mg/kg. Both results exceed The Composting Association Standard for Compost Produced by Aerobic Methods limit - 200 mg Mn/kg. The Mn results indicate reproducibility problems and this is complicated by a lack of agreement on a standard limit value.

The only significant repeatability problem was for the Laboratory B lead (Pb) analyses. The two samples W3 and W4 differed by a factor of two - 94.4 mg/kg and 54.1 mg/kg respectively. A probable explanation in this instance would be the presence of a small piece of lead in sample W3. Without further replicates it is not possible to ascertain whether this difference was due to a repeatability error.

Finally, fluoride (F) determinations, were not at all reproducible. This was also caused by methodological differences; Laboratory A performed a perchloric digest, whereas Laboratory B analysed for water soluble F only. The mean F concentration in naturally occurring soils is 270 mg/kg - the Laboratory A digest was well under this at 100 mg/kg.

## ***Discussion***

### ***- Assessment of Risks to Soils***

At the time of writing there is no established official standard for compost quality in the UK, therefore any assessment of compost quality been carried out with reference to mean and range background concentrations of determinants found in naturally occurring soils, alongside the various standards for composts, soils and other soil additive or soil

making materials such as sewage sludge.

Despite poor interlaboratory reproducibility of the 1997 analyses, all the results in this instance (with the exception some of the Mn determinations) fell within acceptable limits for the protection of soils. Having said that there is currently no universal standard for composts in the UK, therefore it is difficult to comment exactly on what “acceptable limits” are. Nor are there any officially accredited test methods or laboratories for the analysis of compost materials. Many of the results clearly illustrate that where interlaboratory reproducibility discrepancies and a range of different independent standards exist, compost material could either be passed or rejected for use as a soil additive, depending on the standard value chosen as an acceptable limit and the laboratory result that is selected.

In the absence of a definitive standard and accredited test methods, a comprehensive assessment of the risks to soils through the addition of compost materials is not possible. The industry would benefit from increased clarity regarding a definitive compost standard and universal analytical methods including standardised digest extractants and determination methods. Work is currently being undertaken by the Composting Association into the issue of composting standards.

### ***- Qualitative Assessment of Laboratory Performance***

From the combined experiences of SMA compost analysis trials and those carried out under the auspices of the German FCQAO certain heavy metal/potentially toxic element (PTE) analyses appear to be susceptible to variance and imprecision. Those from the German research are Cd, Hg and Cr. Typically, Cd and Hg are present in composts at very low concentrations (in the order of mg/kg) and consequently being determined at the limits of detectability. Determinations at or near the limits of detectability suffer a significant increase in variance and imprecision (FCQAO, 1996). The relatively much smaller scale trials carried out by SMA in 1997, revealed reproducibility problems

between laboratories which have been traced to differences in analytical methodologies.

Reproducibility discrepancies were found in the cases of Zn, Cu, Cr, Pb, Ni, Cd, As, F and Mn - as shown above.

A comparison of methods as shown in Tables 2a & 2b reveals that different extractants for example are used by the two laboratories; while Laboratory A uses boiling aqua regia, Laboratory B uses a mixture of boiling concentrated nitric and perchloric acids (a more aggressive mixture), which explains why most of Laboratory B's results are elevated relative to those of Laboratory A. The F results from the two laboratories are in fact different determinations, Laboratory A uses a perchloric digest, while Laboratory B has analysed for water soluble F. There are also differences in the choice of determination method. Laboratory A recognises the importance of using Flame AAS in all materials with a high concentration of iron, for example soils. Iron interferences using the ICP technique for soils, give rise to false results particularly for cadmium and arsenic. Interestingly, while in general, the Laboratory B methodology is more sensitive, the Laboratory A As result is a factor of 10 greater than the Laboratory B determination.

It is unclear whether this is due to a methodological difference or another source of imprecision. Compost materials could be analysed by either the AAS or ICP technique, but if elevated iron concentrations were detected, AAS would be used by Laboratory A; normally Laboratory B would use ICP, not AAS. In addition Laboratory A makes use of the higher sensitivity of the Flame Adsorption technique for determination of As and selenium (Se), which are typically near to the limits of detection of AAS and ICP.

The establishment of a compost quality standard (or standards) is important for the protection of soils and the environment. However, the workability of any given standard is drawn into question where analytical reproducibility discrepancies, such as have been demonstrated in this article, exist between two Laboratories offering, for all intents and purposes, the same service.

## ***Conclusions & Recommendations***

It has been shown above that despite poor reproducibility between laboratories, PTE determinations in both the FCQAO and SMA trials, are generally all within ranges found in naturally occurring soils and the limit values for sewage sludge amended soils. However, a serious question exists over the reproducibility of the results between UK laboratories as experienced in the 1997 trials and the results illustrate the quality assurance problems in the absence of definitive laboratory methods and quality standards. A short review of the composting frameworks in some of the other European countries (Austria and Germany have been focussed on) reveals well established standards, accepted and recognised in the market place, as well as much more fully developed systems of accredited test methods, laboratories, and producers of composts. Similar systems should be developed in the UK to ensure the quality assurance of waste-derived compost products and to reduce the associated liabilities of marketing a waste product for beneficial use.

As stated earlier, a two fold problem exists that would be partly solved by establishing a definitive standard for compost materials. Less widely appreciated, but highlighted by this study is the problem of poor inter-laboratory reproducibility caused by non-standardised analytical methodologies. It is concluded that a system of accredited analytical methods is required to enable a Compost Standard to be reliably, fairly and safely administered. Additionally, industry standardised sampling techniques are lacking and should be established.

While both laboratories in this study have legitimate reasons for exact choices of analytical method, the problem of non-standardised techniques is thrown into sharp focus by these results. Clearly, administering and awarding a Compost Standard to a particular producer relies on the reliable analysis of their product, which must require attention to be given to the standardisation of laboratory methods.

This study recommends the drafting of standardised test methods for compost materials. It would be pertinent to concentrate upon some of the PTE determinations that have caused concern regarding reproducibility - Zn, Cr, Ni, Cu and As are suggested. This could be followed up by multiple replicate analyses being carried out at several laboratories using these draft standard methods.

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